Influence of household washing on the variation of the properties of intrinsic natural color organic cotton fabrics

Hesam Aliei

Institut d'Investigació Tèxtil i Cooperació Industrial

intexter

UNIVERSITAT POLITÈCNICA DE CATALUNYA

PhD program in TEXTILE AND PAPER ENGINEERING

Influence of household washing on the variation of the properties of intrinsic natural color organic cotton fabrics

Hesam Aliei

Supervisors:

Enric Carrera Gallissa **Diana Cayuela Marín**

Institut d'Investigació Tèxtil i Cooperació Industrial de Terrassa

Universitat Politècnica de Catalunya

Terrassa 2024

Abstract

Natural colored organic cotton fabrics gained considerable attention in recent years owing to due to their environmental friendliness and sustainability production. This imparts unique colors and patterns to the textiles. The colorimetric characteristics of these fabrics, including lightness and saturation, can be influenced by various factors being household washing one of them. To assess this effects, the colorimetric properties of knitted and woven fabrics were measured before and after 30 washing cycles with a skincare detergent with tap water at 40 ºC.

The most significant difference in colorimetric properties was observed after the initial wash, highlighting the reduction in both parameters, lightness and saturation, after the first wash. Also, there was a notable colorimetric difference between the second wash and the fifth washout from the fifth wash onward, the changes were minimal. Additionally, the FTIR-ATR analysis of the extracts in petroleum ether and subsequently in ethanol of the NaCOC fabrics, before and after home washing, in conjunction with a comparison with shrinkage, demonstrated that the latter process is accountable for the darkening of the sample.

Furthermore, the impact of various washing conditions on the fabric's color change was investigated. Specifically, three detergents (Fox Fiber® Colorganic®, Klar, and Pure Nature), two types of water (tap and distilled), and three different temperatures (20, 40, and 60°C) were considered as variables. In this study, the effect of washing variables on the color and integrity of fabrics using colorimetric measurements was evaluated. The findings demonstrated that water hardness was the most influential variable in terms of color changes in fabrics.

Overall, these findings highlight that household washing significantly affected the colorimetric properties of NaCOC fabrics, with the initial wash having the most substantial impact. The hardness of the water used in washing has been identified as a crucial factor in color changes, providing insights into the quality and color of natural-colored organic cotton textiles.

Keywords: natural color, organic cotton, household washing, color change, colorimetric.

Resum

Els texits de cotó orgànic naturalment colorejat (NaCOC) han guanyat una considerable atenció en els darrers anys degut al seu respecte pel medi ambient i la producció sostenible. Això atorga colors i patrons únics als tèxtils. Les característiques colorimètriques d'aquests teixits, incloent-hi la lluminositat i la saturació, poden ser influenciades per diversos factors, essent un d'ells el rentat domèstic. Per avaluar aquests efectes, es van mesurar les propietats colorimètriques de teixits de punt i de calada abans i després de 30 cicles de rentat amb un detergent per a la cura de la pell amb aigua de l'aixeta a 40 ºC.

La diferència més significativa en les propietats colorimètriques es va observar després del rentat inicial, destacant la reducció en ambdós paràmetres, lluminositat i saturació, després del primer rentat. A més, hi va haver una notable diferència colorimètrica entre el segon rentat i el cinquè rentat; a partir del cinquè rentat, els canvis van ser mínims. Addicionalment, l'anàlisi FTIR-ATR dels extractes en èter de petroli i posteriorment en etanol de les teles NaCOC, abans i després del rentat a casa, juntament amb una comparació amb l'encongiment, van demostrar que aquest darrer procés és responsable de l'enfosquiment de la mostra.

A més, es va investigar l'impacte de diverses condicions de rentat en el canvi de color del teixit. Específicament, es van considerar com a variables tres detergents (Fox Fiber® Colorganic®, Klar i Pure Nature), dos tipus d'aigua (de l'aixeta i destil·lada) i tres diferents temperatures (20, 40 i 60°C). En aquest estudi, es va avaluar l'efecte de les variables de rentat en el color i la integritat dels teixits utilitzant mesuraments colorimètrics. Les mesures van demostrar que la duresa de l'aigua va ser la variable més influent en termes de canvis de color en els teixits.

En general, aquestes troballes destaquen que el rentat domèstic afecta significativament les propietats colorimètriques de les teles NaCOC, essent el rentat inicial el que té el major impacte. S'ha identificat que la duresa de l'aigua utilitzada en el rentat és un factor crucial en els canvis de color, proporcionant informació sobre la qualitat i el color dels tèxtils de cotó orgànic de color natural.

Paraules clau: color natural, cotó orgànic, rentat domèstic, canvi de color, colorimetria.

Resumen

Los tejidos de algodón orgánico naturalmente coloreado (NaCOC) han ganado una considerable atención en los últimos años debido a su respeto por el medio ambiente y la producción sostenible. Esto otorga colores y patrones únicos a los textiles. Las características colorimétricas de estos tejidos, incluyendo la luminosidad y la saturación, pueden ser influenciadas por varios factores, siendo uno de ellos el lavado doméstico. Para evaluar estos efectos, se midieron las propiedades colorimétricas de tejidos de punto y calada antes y después de 30 ciclos de lavado con un detergente para el cuidado de la piel con agua del grifo a 40 ºC.

La diferencia más significativa en las propiedades colorimétricas se observó después del lavado inicial, destacando la reducción en ambos parámetros, luminosidad y saturación, después del primer lavado. Además, hubo una notable diferencia colorimétrica entre el segundo lavado y el quinto lavado; a partir del quinto lavado, los cambios fueron mínimos. Adicionalmente, el análisis FTIR-ATR de los extractos en éter de petróleo y posteriormente en etanol de los tejidos NaCOC, antes y después del lavado dom’estico, junto con una comparación con el encogimiento, demostraron que este último proceso es el responsable del oscurecimiento de la muestra.

Además, se investigó el impacto de varias condiciones de lavado en el cambio de color del tejido. Específicamente, se consideraron como variables tres detergentes (Fox Fiber® Colorganic®, Klar y Pure Nature), dos tipos de agua (del grifo y destilada) y tres diferentes temperaturas (20, 40 y 60°C). En este estudio, se evaluó el efecto de las variables de lavado en el color y la integridad de los tejidos utilizando mediciones colorimétricas. Los hallazgos demostraron que la dureza del agua fue la variable más influyente en términos de cambios de color en los tejidos.

En general, estos hallazgos destacan que el lavado doméstico afecta significativamente las propiedades colorimétricas de los tejidos NaCOC, siendo el lavado inicial el que tiene el mayor impacto. Se ha identificado que la dureza del agua utilizada en el lavado es un factor crucial en los cambios de color, proporcionando información sobre la calidad y el color de los textiles de algodón orgánico de color natural.

Palabras clave: color natural, algodón orgánico, lavado doméstico, cambio de color, colorimetría.

Preface

In contrast to synthetic dyes, which often fade after washing, naturally colored cotton fabrics display enhanced color retention and vibrancy post-wash. This distinctive behavior is theorized to be connected to the inherent qualities of cotton fibers, including the presence of waxes and the specific environmental conditions in which cotton is cultivated.

The naturally colored cotton present benefits such as its eco-friendly nature, reduced reliance on chemical treatments and potential applications across various sectors such as fashion and healthcare owing to its intrinsic attributes such as UV protection and low flammability. Additionally, its antibacterial properties make it suitable for sensitive applications. Eliminating the dyeing process reduces environmental pollution and production costs, aligning with the increasing demand for sustainable and environmentally friendly textile solutions.

Through this investigation, our objective has been to enhance the comprehension of how naturally colored cotton responds to washing, thereby boosting its usage and advocating for sustainable practices in the textile industry. This endeavor aims to offer valuable insights for consumers and manufacturers by encouraging the adoption of naturally colored cotton as a feasible substitute for conventionally dyed fabrics.

This study aims to support the adoption of naturally colored cotton as a viable and sustainable alternative to conventionally dyed fabrics, promoting a shift towards more environmentally friendly textile production practices. This study also aligns with the demand for sustainable and environmentally friendly textile solutions by providing valuable insights into the response of naturally colored cotton to washing. This encourages the use of naturally colored cotton and advocates for sustainable practices in the textile industry.

Objectives of the thesis

1) To find an explanation for the color change experienced by the articles of colored organic cotton, after domestic washing.

2) To study the physical properties of fabrics made from natural colored cotton and to compare the properties of fabrics made from these pieces of cotton and their color that will be changed after the domestic wash process and recommend information for consumer information for products made of naturally colored cotton.

3) Additionally, in this project, we are explored other properties of these fabrics, such as UV absorption, UPF, colorimetric values or extraction of waxes.

Table of Contents

List of Figures

List of Tables

Abbreviations

NaCOC	Natural Colored cotton
USSR	Union of Soviet Socialist Republics
BF cotton	Brown cotton fiber
BCI cotton	Better Cotton Initiative
COTS cotton	Conventional Open Top Spindle
OCS	Organic Content Standard
BCI	Better Cotton Initiative
OEKO-TEX®	global certification system for textile products
CMiA	Cotton made in Africa
RCS	Recycled Claim Standard
GRS	Global Recycled Standard
RDS	Responsible Down Standard
RWS	Responsible Wool Standard
GMO	Genetically Modified Organism
SOM	Soil Organic Matter
BC	Better Cotton
qRTPCR	quantitative Reverse Transcription Polymerase Chain Reaction
UV	Ultraviolet
ICP	Inductively Coupled Plasma
SEM	Scanning Electron Microscope
SDS	Safety Data Sheet
HVI	High Volume Instrument
AFIS	Advanced Fiber Information System
FMT	Fecal Microbiota Transplantation
DPPH	2,2-diphenyl-1-picrylhydrazyl
ABTS	2,2-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid
UTMs	Universal Testing Machine
ASTM	American Society for Testing and Materials
FAST	Fabric Assurance by Simple Testing
UPF	Ultraviolet Protection Factor
KES	Kawabata Evaluation System
DP	Degree of Polymerization
MW	Molecular Weight
FTC	Fair Trade Certification

RCS	Real Customer Service
GRS	Global Reference System
GMOs	Genetically Modified Organisms
WOB	World of Business
FMT	Fineness and Maturity Analyzer
IA	Image Analyze
FTIR	Fourier Transform Infrared Spectroscopy
CRE	Constant Rate of Extension
DSC	Differential Scanning Calorimetry
CIE	International Commission on Illumination

1. State of The Art

1.1 Fiber overview of conventional cotton cultivation

1.1.1 Botanical description

Cotton fiber is a single-celled outgrowth of the seeds of the cotton tree, a dicotyledonous plant of the malvaceous family of the genus Gossypium.

The genus Gossypium, which includes all cotton in the world, has 39 species, of which 33 are diploid (diploid cells are those that contain two similar copies of each chromosome, unlike haploid cells, which contain only one set). The vast majority of cells in eukaryotic organisms, including animals and plants, are diploid, a circumstance that gives them a great advantage in the face of natural selection because errors in the genetic material are more easily compensated for by the second copy and six tetraploids (a cell with four sets of homologous chromosomes).

There are four cultivated species.

- Two diploids are cultivated in India and Pakistan which are classified as below:
    - Gossypium arboretum
    - Gossypium herbaceous.

They represent only 4 % of the global cotton fiber production and have a fiber length of less than 21 mm.

- Two tetraploids are classified as below:
    - Gossypium barbadense. It is cultivated in Egypt, Peru, Sudan, USA and Central Asia. This represents 5 % of the global production. It is an extra-long fiber greater than 34.9 mm.
    - Gossypium hirsutism. It represents more than 90 % of the global fiber production. The main producers are China, Central Asia, the USA, India, Pakistan and Brazil. It is an intermediate-length fiber. In the US, varieties vary from a minimum of 1 inch (2.54 mm) to 1 $\frac{3}{16}$" (30.2 mm).

1.1.1.1 General characteristics of Gossypium hirsutum (Uppland type)

It is the most widespread species in the world and constitutes approximately 90 % of the global production of cotton fiber. It has a length that oscillates between 25 and 30 mm and a micronaire that oscillates between 3.5 and 5.0.

There are four main types in the United States.

- Type Acala. It is produced in the West (California, Arizona) and it is cotton of great quality and resistance.
- Delta type. It is produced in the Mississippi Valley, Tennessee, Arkansas, Mississippi and Louisiana. The Delta pine and Stoneville varieties are the most representative of this group.
- Plains type. It includes a heterogeneous group planted in the highlands of Texas and Oklahoma. These are mostly low-quality short cotton collected with "Strippers'.
- Eastern type. The Coker and MacNair varieties are planted in the states of Carolina and Georgia.

The cotton in Central Asia (former Soviet Union) are characterized by the use of precocious varieties that mostly come from the Tashkent Research Station (Uzbekistan).

Other countries such as Turkey, Greece, Spain, Syria, America (from Mexico to Argentina) and Africa use varieties from previous countries or select materials from American, Russian or African varieties in general.

1.1.1.2 General characteristics of Gossypium barbadense (Egyptian or Sealand)

Long staple fibers represent 3 % of the world's cotton production, with an approximate length of 37 mm and micronaire index of less than 4.

This species is cultivated because it is a very long, fine and resistant fiber of exceptional quality.

The main producers are Egypt, Sudan, the countries of Central Asia (former USSR), Peru and the USA. A relatively smaller production of this species has also been obtained in Israel, Morocco, Colombia, China and other countries.

Egyptian cotton is divided into two main groups:

- Long fiber (1 1/4" to 1 3/8"). Is produced in Upper Egypt and South of the Nile Delta.
- Extra-long fiber (greater than 1 3/8” or 34.4 mm). It is produced in the Central and Northern parts of the Nile Delta.

1.1.2 Geography of cultivation and production

Cotton is grown in various climates, including tropical, subtropical and temperate, but its development is very sensitive to extreme temperatures. The temperatures must not be lower than 10 °C or higher than 35 °C. It requires a long period without frost, much sun and moderate atmospheric precipitation, generally on the order of 600 to 1,200 mm.

In general, soils must be fairly heavy, although the level of nutrients does not need to be exceptional. In general, these conditions are met in the dry periods of the tropics and subtropical areas of the northern and southern hemispheres; however, today, a large part of cotton is grown in drier regions owing to irrigation.

The total growth period is 150–180 days. Depending on the temperature and variety, 50–85 days are needed for the formation of the first flower buds, 25–30 days for the first flowers to appear and 50–60 days for flowering.

Harvesting requires more than 160 days above 15 °C and the plant becomes inactive at this temperature. The geographical limitations of cotton cultivation are climatically determined by the length of the growing season, temperature and days of sunshine. Because the growing season varies from one area to another, varieties adapted to the number of days between sowing and harvesting are needed.

In the US, the growing season varies depending on area.

- High Plains Texas: 120-150 days
- Southeast: 130-170 days
- West: 180-210 days

In Spain, the growing season is somewhere longer than 200 days.

The d-ay (photoperiod) also influences flowering, although this does not occur in some varieties. Cotton is a plant that requires only a few days to grow. Optimal germination is achieved with temperatures of 18 ºC to 30 ºC, with a minimum of 14 ºC and a maximum of 40 ºC.

At the beginning of the vegetative growth stage, the daytime temperature should oscillate between 20 ºC and the night temperature should not fall below 12 °C, without exceeding 40 °C and 27 ºC, respectively. The optimal temperature for the development and maturity of the capsules is between 27 ºC and 32 ºC, but the yield is reduced when it exceeded 38 ºC.

Currently, cotton is cultivated on 35 million hectares (2.5 % of the cultivated surface of the world) in more than 40 countries reaching 43 ºN in Ukraine and 45 ºN in China (Manchuria). On the other hand, little cotton is grown (comparatively speaking) in the Southern Hemisphere, where it reaches 30 ºS in Australia and Northern Argentina. More than 50 % of the world's cotton is grown above 30 ºN and more than 60 % corresponds to irrigated cultivation (Figure 1).

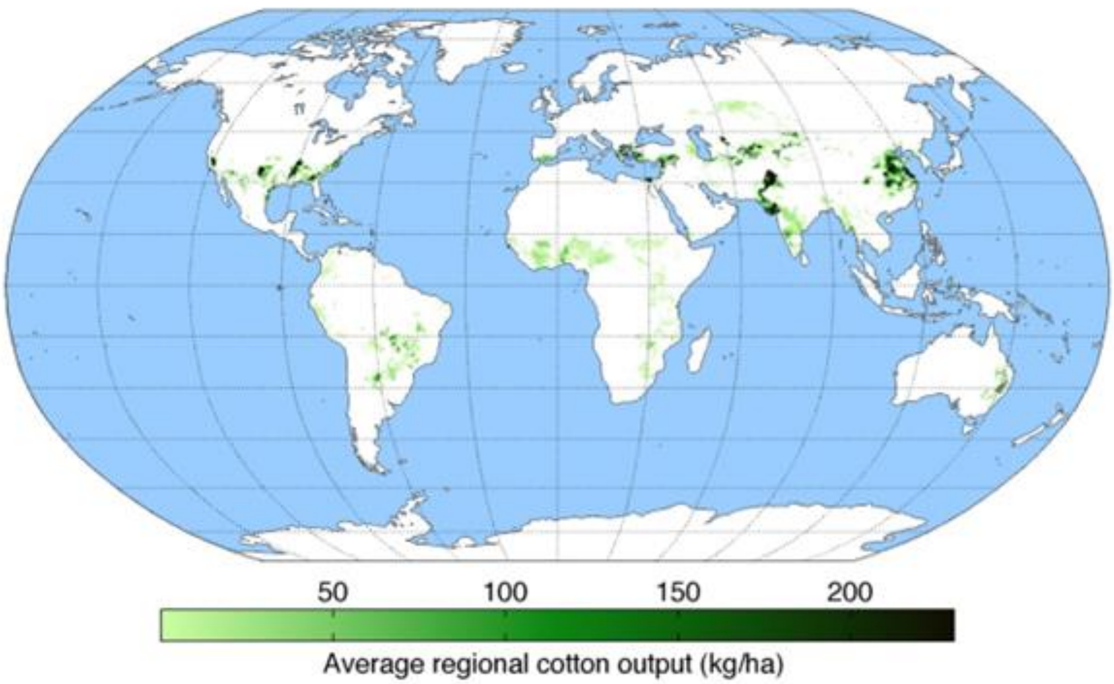


Figure 1. Distribution of cultivated area of cotton worldwide and indications of yield kg/ha [1].

US cotton grows below the isotherm, which provides 200 frost-free days, reaching 37 ºN in the San Joaquin Valley in California.

Because cotton is cultivated in both hemispheres, the sowing and harvesting periods vary significantly between countries,

Table 1, which allows the fiber to enter the world market practically throughout the year [2].

Table 1. Planting period, harvesting and approximate net weight of the bales of some cotton-producing countries.

Producing country	Sowing period	Collection period	Approximate net weight of the bales (kg)
Argentina	15 September - 30 October.	February – May	200
Brasil North	January – March	Julio – Agosto	190
Brasil South	October - November	March – June	190
Colombia	February - March July – August	July - August December – January	250 250
Costa Rica	July – 15 August	15 December – 20 January	220
China	April – June	September – October	-
Spain	April – May	September – November	235
EEUU	February – June	July – January	220
Greece	March-May	September – November	250
Guatemala	July – August	January – March	230
India	April – November	September – June	180
Israel	March – April	September – November	230
Mexico	January – February	June – July	220
Morroco	March – April	September – November	250
Mozambique	November - December	May – June	180
Nicaragua	15 June – August	November – January	230
Pakistan	April – June	September – January	180
Peru	November - January	June – July	260
Egipt	February – March	August – October	326
Sudan	June – September	October – May	195
Syria	April – May	15 August – December	205
Tanzania	November – March	May – September	185
Turkey	April – May	August – November	215
Uganda	April – June	November – April	185
URSS	April – May	September – November	210

The current world production of raw cotton is approximately 26 million tons, representing 22 % of the total global production of textile fibers (Figure 2). The main production countries are shown in Figure 3. China, India and the United States are the main producers of cotton in the world, representing almost 60 % of the global production of this fiber. Pakistan is another important country in the production of cotton, while Australia and Egypt produce the highest-quality cotton. The demand for cotton has increased steadily since the 1950s at an average annual rate of 2 %.

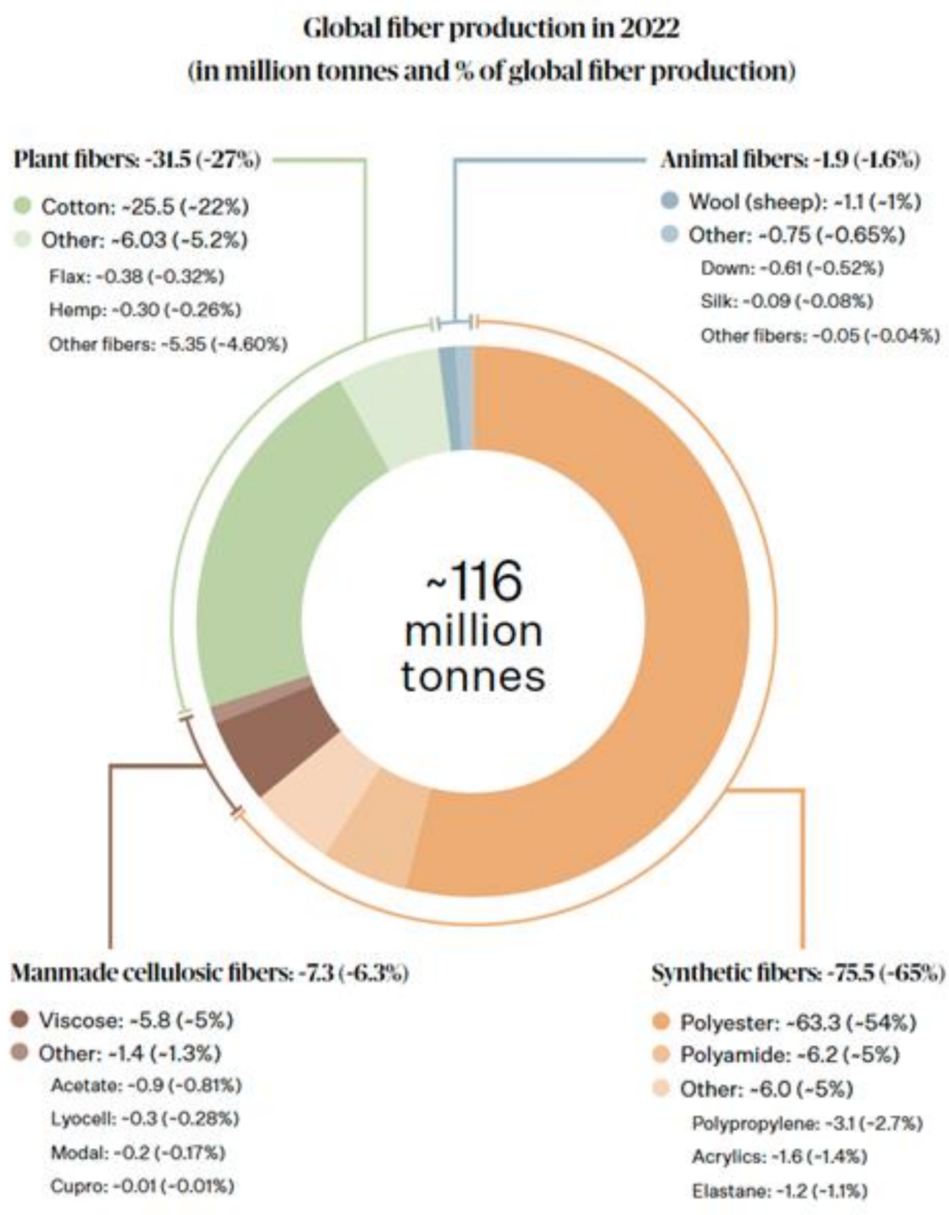


Figure 2. World production of textile fibers by 2022 [3].

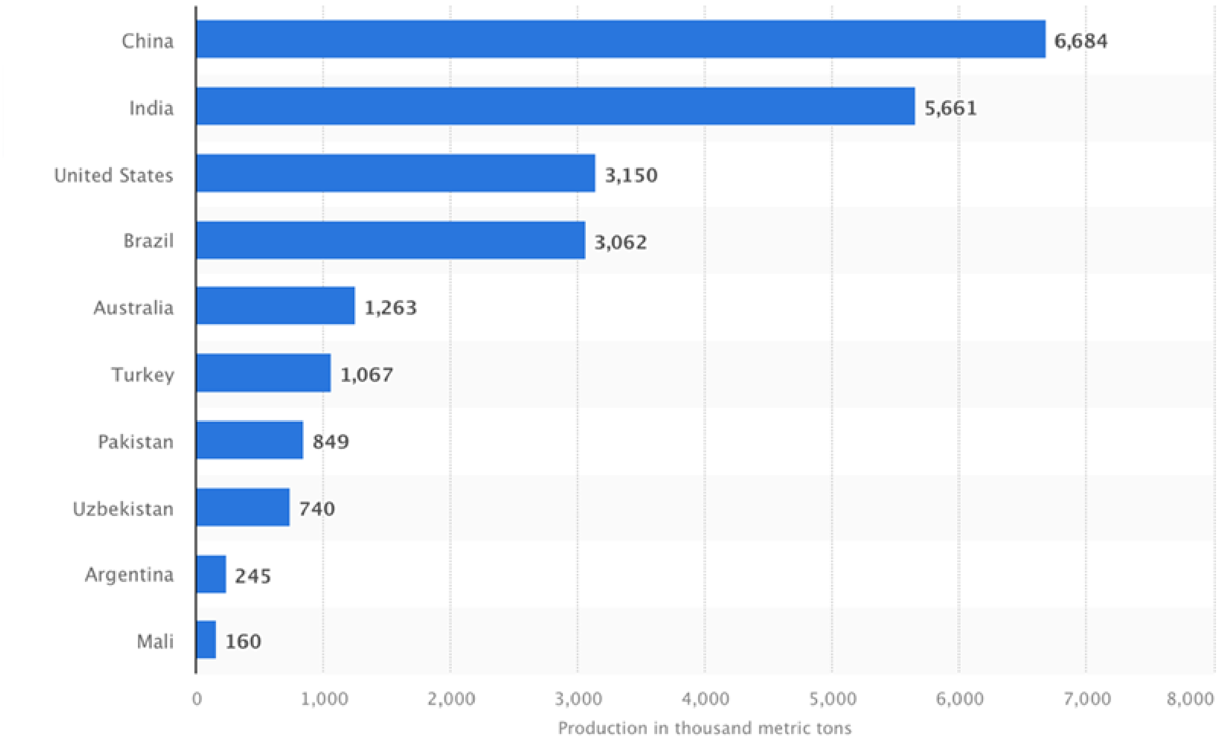


Figure 3. Main cotton-producing countries [3].

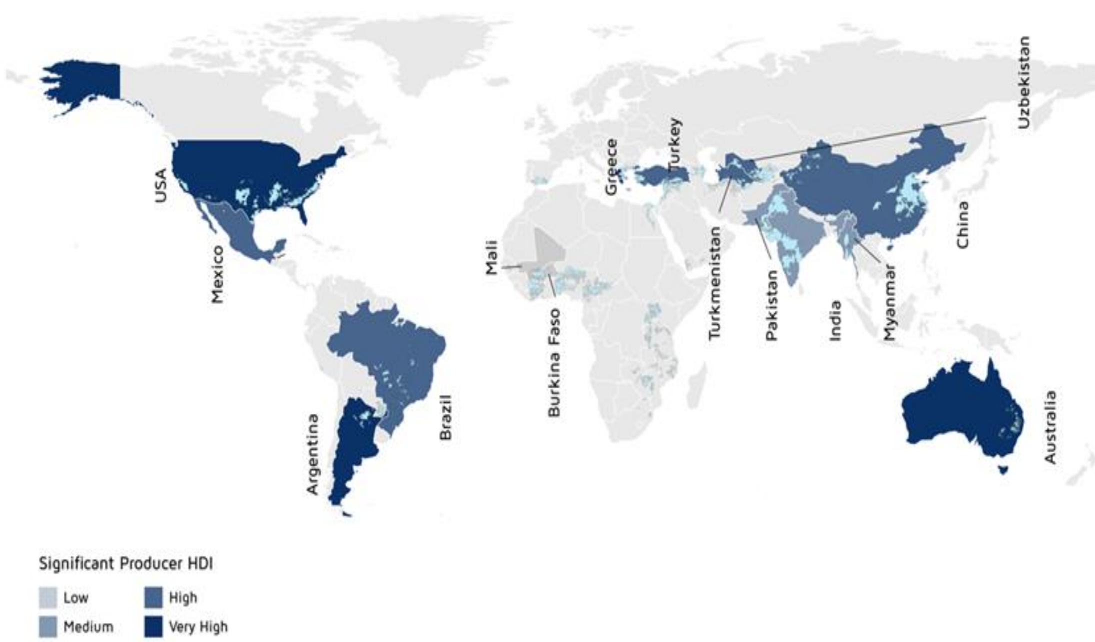


Figure 4. Map of the main cotton-producing countries [4].

1.1.3 Growth of the plant

The cotton plant is indeterminate, meaning that the main stem never ends in an inflorescence but instead produces a new node every 2 or 3 days.

The plant has two types of branches including:

- Vegetative branches or monopods

- Fruit-bearing branches or sympods

The growth point of the fruit-bearing branch ends in a flower and all subsequent developments occur from the axillary bud at the base of the leaf accompanying the flower.

Vegetative branches are usually located in the lower nodes of the stem and generally do not bear fruit. Growth is based on the first bud of the main stem node.

Flower buds are formed on the symptoms from the 5th to the 8th node, requiring maximum temperatures above 26 ºC.

Germination occurs when the soil temperature exceeded 18 ºC. The duration from emergence to the appearance of the first flower bud depends on the variety, being approximately 30 days for early varieties and approximately 50 days for later varieties. The duration from the appearance of the first bud to the first flower was approximately 22 days.

It takes approximately 24 days for the capsule to be perfectly formed and it takes an additional 24–40 days for the capsule to mature and open (Figure 5).

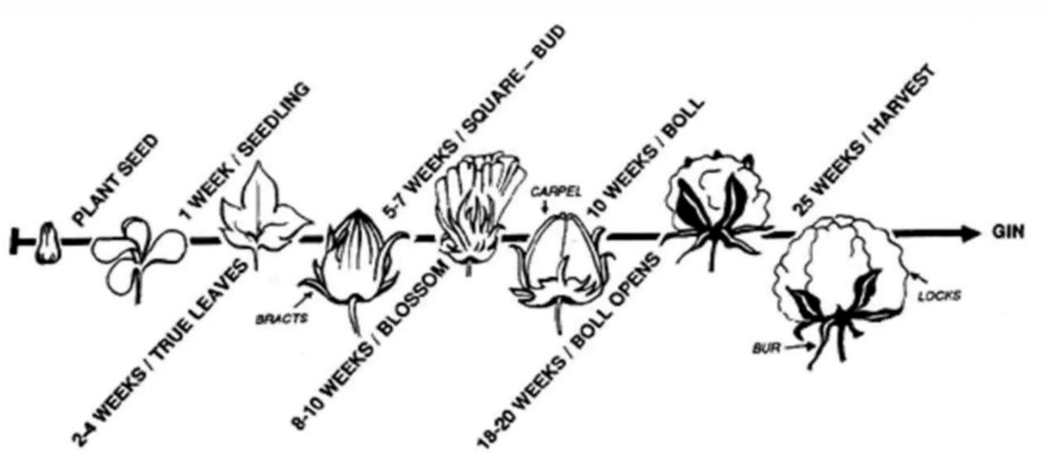


Figure 5. Cotton planting and harvest duration [4].

The outer part of the flower consists of the calyx (5 sepals), 3 bracts and 5 petals all joined at the base. White flowers bloom from the buds approximately five to seven weeks after planting.

The first flower blooms in the lower part of the plant, near the main stem. As the plant grows, several flowers open daily in the upper part of the plant and further out of the branches. The flowers opened mid-morning and withered the next day. Their color evolves from white to pink, blue and finally purple, as they wilt. Pollination occurs from 24 to 30 hours after pollination; the developed ovule transforms into a seed and the ovary takes on the shape of a capsule ,The maturation period of the capsule ranged from 45 to 60 days (Figure 6), reaching a size similar to that of a golf ball (Figure 7).

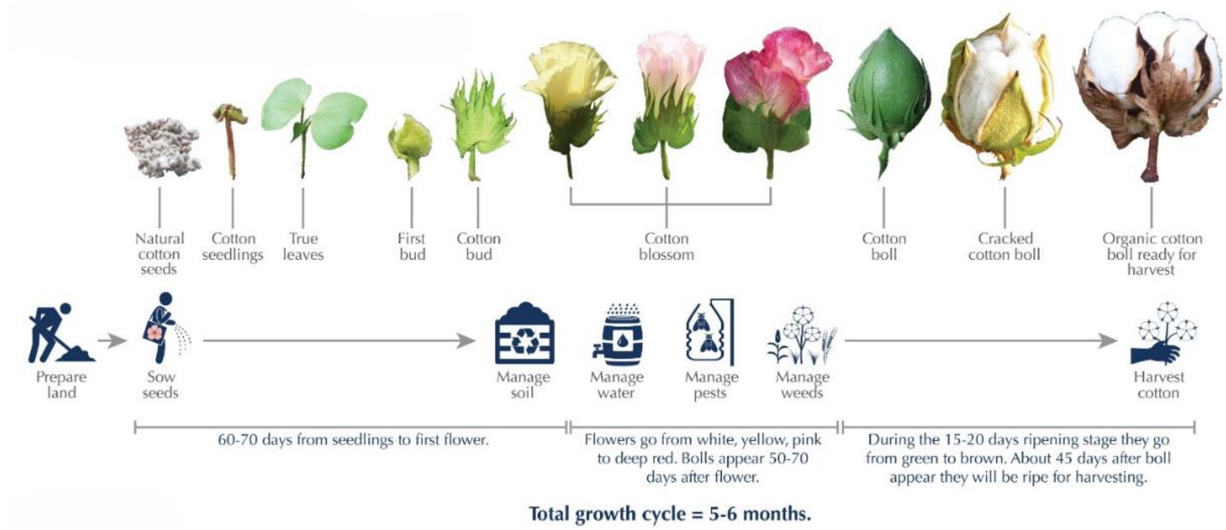


Figure 6. The growth evolution of cotton from the seed to the fiber capsule has undergone significant changes [5].

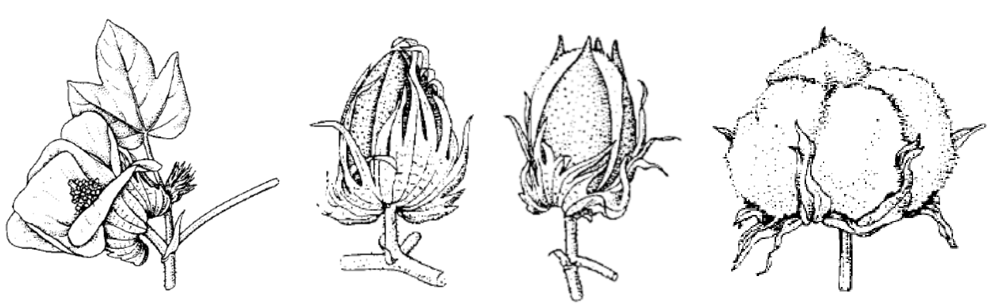


Figure 7. Cotton evolution. From left to right: flower, capsule at 90 % of its final size, the beginning of capsule opening and open capsule [6].

The capsule, which contains cotton fibers, begins to form when the flower withers (Figure 8).

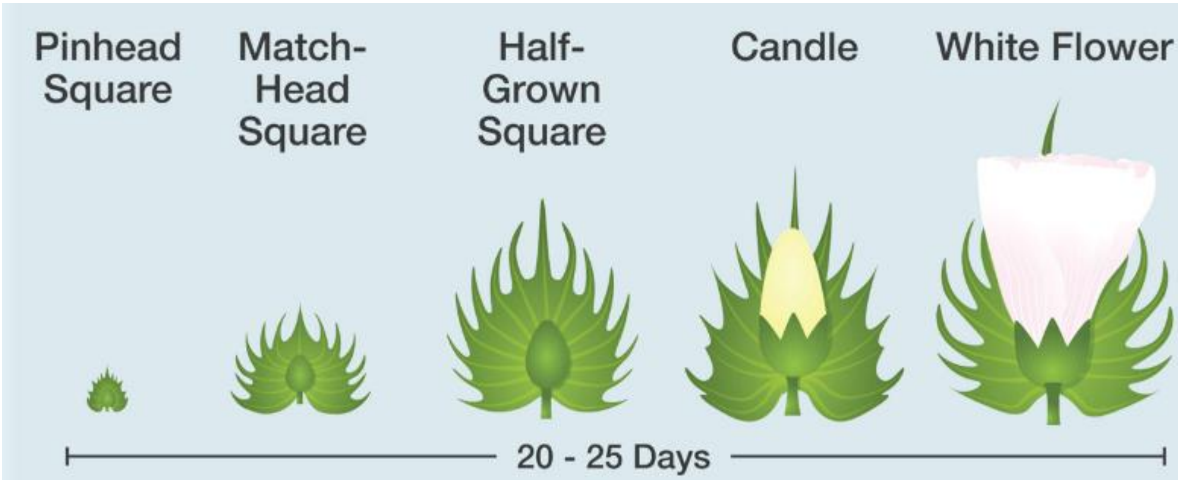


Figure 8. Growth of cotton plants from planting to harvesting [9].

Therefore, each capsule contained approximately 500,000 cotton fibers. Each plant can support up to 100 capsules. One pound (0.4536 kg) of cotton fiber contains 100 million or more individual fibers. Photographs of flowers, blossoms and cotton bolls are shown in Figure 9, Figure 10, Figure 11 and Figure 12, respectively.

Figure 9. Yellow cotton flowers [7].

Figure 10. Pink cotton flower [8]

Figure 11. Green cotton capsule. Right: Open and mature capsules [9].

Figure 12. Farmers holding a mature cotton capsule (left) and an unripe one [10].

The capsule breaks open in four or five straight lines from the tip. It then splits and opens, revealing four or five lobes (each containing 8 to 10 seeds). Up to 20,000 fibers can emerge from each seed (Figure 13).

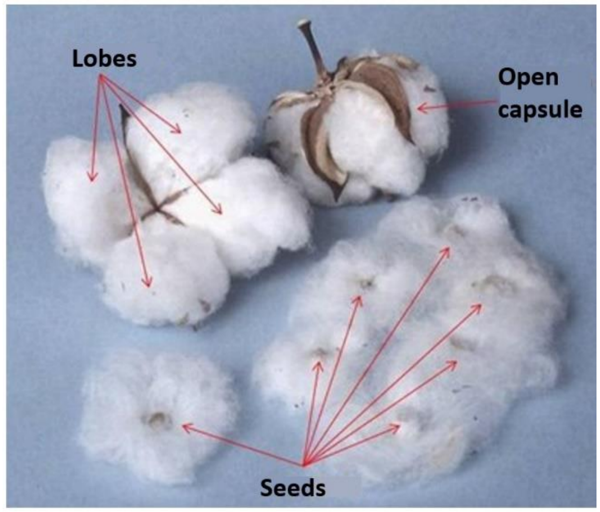


Figure 13. A cotton capsule consists of four or five lobes, each contains eight–ten seeds [11].

1.1.3.1 Structural development of the fiber

Two forms of fibers emerge from the cotton seed as simple cells of the seed's cortex epidermis, the lint and the fibers. The lint begins the day after flowering, once its development is complete, whereas the fiber starts 5 or 6 days after flowering and continues until the tenth or eleventh day.

The fibers developed from their beginning as a thin tubular structure, whose diameter is defined during the first five days and do not change afterward. The length of the fiber progressively increased, extending the process for approximately 13 – 20 days, depending on the variety of development conditions. Once the fiber has reached its maximum length, the walls of each fiber begin to fill by the aggregation of successive layers of cellulose inside the wall. In this process, the protoplasm is converted into cellulose.

The membranous wall or envelope of the fiber is called the primary wall and has a thickness of 0.1 to 0.2 microns. It constitutes the outer layer of the fiber after its complete development. The cross-section of the fiber is approximately circular during its length growth period and its diameter, except at the angles, is approximately constant in its middle portion with slight tapering towards the base and a more pronounced tapering towards the ends. This is approximately the same for all fibers emerging from the same seed.

As the fiber grows in length within the young capsule, it does not extend in a straight line, but rather in bands back and forth, forming acute angles, with the fibers tightly packed. The length of the fiber can be 1,000 to 3,000 times its diameter, remaining turgid or swollen until maturity (Figure 14).

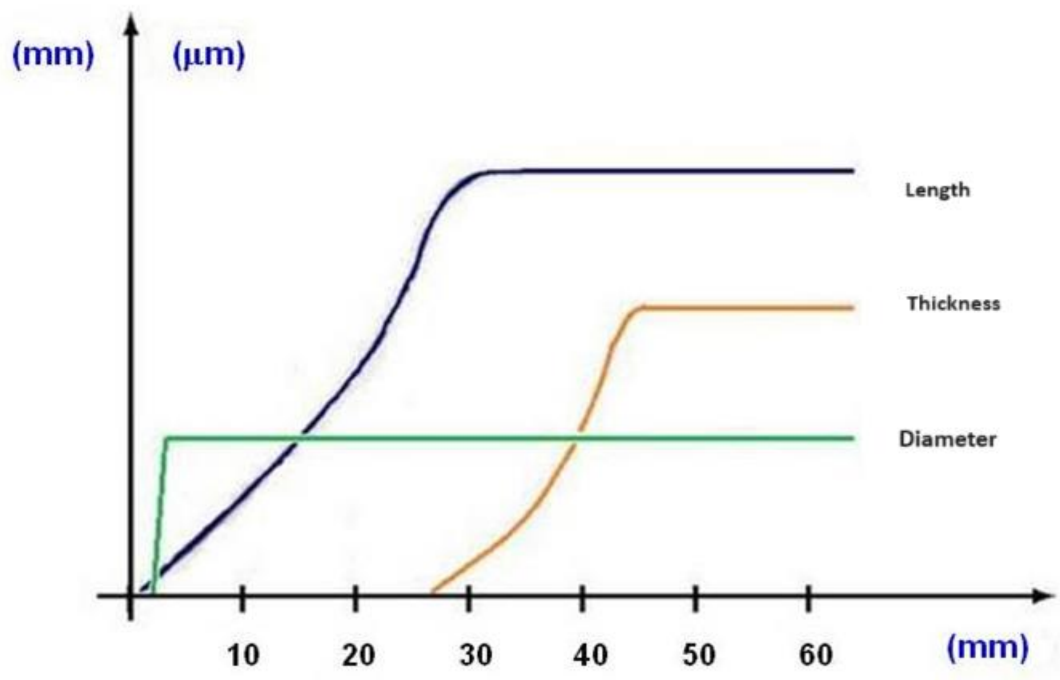


Figure 14. Stages of cotton fiber diameter, length and thickness [2].

As mentioned earlier, when the fiber reaches its maximum length, internal fiber growth begins through the addition of cellulose layers, a process that continues for 3 or 4 days before capsule opening. During this thickening process, the sugars produced by photosynthesis are converted into cellulose and deposited because of protoplasmic activity in successive layers on the inner surface of the primary wall. Formation of this wall occurs within 25 – 40 days. This structure does not fill the cells. It always leaves some space in the center of the fiber called the "lumen" Thickening of the wall depends on variety, species and environmental conditions.

When the capsule opens, there is a loss of water and a collapse of the fiber cells, which die, turning the lumen into a residue.

The cotton fibers appeared as flat twisted ribbons (50 to 100 turns per inch). The fiber was conical at one end and fibrillated at the other end attached to the cotton seed. The location where twisting or spinning occurs is known as "convolution." These twists are due to thickening of the wall, size of the lumen and perimeter of the fiber. When observed in its entirety, the cotton fiber is composed of the following parts (Figure 15):

- Cuticle: outer waxy layer of fiber-containing pectin and protein materials. It serves as a coating and protection and is resistant to water. This layer was removed during the scouring process before dying to allow the penetration of dyes and other chemical agents.
- Primary wall: original thin cell wall. They are formed by a network of fine cellulose fibrils.

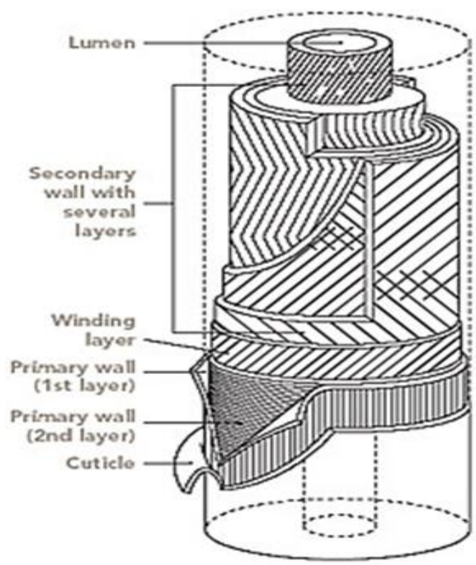


Figure 15. Cotton fiber layer structure [12].

The growth layer (also known as the S1 layer) is the first layer to thicken the secondary wall. It differs in structure from the primary wall and the rest of the secondary wall. It consists of fibrils aligned at angles of 40 º – 70 º with the fiber axis in an open mesh pattern.

The secondary wall (also known as the S2 layer) consists of concentric layers of cellulose, which constitute the main part of the cotton fiber. After the fiber reached its maximum diameter, new layers of cellulose were added to form a secondary wall. The fibrils were deposited at angles of 70 º – 80 º to the fiber axis, reversing the angle at points along the length of the fiber. The secondary wall accounts for 90 % of the fiber weight and its structure is responsible for most of its strength. Cellulose is more crystalline, and the fibril orientation is more parallel to each other than in the other layers.

The lumen (also known as S3 layer) separates the secondary wall from the lumen and is more resistant to certain reagents than the secondary layers of the wall. It constitutes a hollow channel that runs along the length of the fiber. It is filled with live protoplasm during the growth period. After the fiber matures and the capsule opens, the protoplasm dries up, leaving a central hollow or pore space in each fiber.

The Lumen is the central area containing the living and active parts of the cell. It is a channel in the center of the cell-free from cellulose deposits and before fiber drying (opening of the capsule), it contains the nucleus and protoplasm.

Throughout the cotton fiber structure and in each of its different layers, pores or capillary spaces exist between fibrils of varying sizes. Thus, cotton fiber can be considered as a microscopic sponge with a complex porous structure. This uniqueness of the internal capillary facilitated the absorption of liquids and vapors by the cotton fibers (Figure 16).

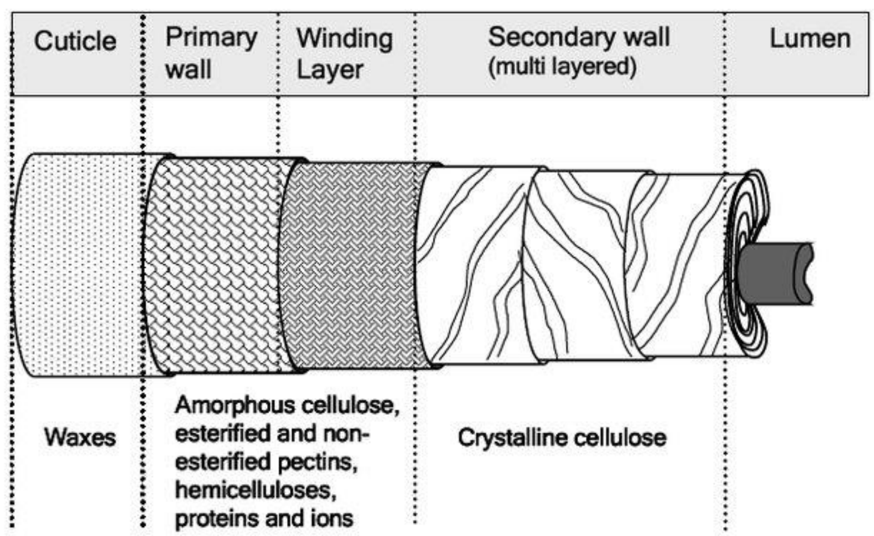


Figure 16. Composition of cotton fiber layers [12].

Not all fibers from the same seed develop in the same manner or reach the same length or internal thickness. Some had almost no deposits; therefore, they were immature.

Examination of the cross-section prepared from a mature fiber shows the alternation of bright and dark rings in the secondary thickening. Each pair of adjacent bright and dark rings represents cellulose deposited during the day of growth. Daytime deposits have a higher density than rings deposited after the sun sets. These daily deposits are similar to the annual growth rings observed in the cross-sections of trees.

1.1.3.2 Maturity

When the cotton fiber reaches its maximum length, it begins to grow inward, generating concentric layers of cellulose. A fiber is considered mature when all the protoplasm has been converted into cellulose, whereas an immature fiber, which has been collected during the period of cellulose sap segregation, has not yet fully converted all the protoplasm into cellulose. Dead cotton fibers are fibers without a protoplasm because they do not receive protoplasm at the time of formation. Therefore, maturity refers to the degree (quantity) of thickening of the cell wall concerning the fiber diameter.

Mature cotton is firmer and fluffier than immature cotton and brighter because of its nearly circular profile. Mature cotton absorbs more dye than immature cotton and has a lower tendency to form neps.

Mature fibers have a swollen or rounded kidney-shaped cross-sectional form, whereas immature fibers are less swollen and have a flatter shape. The dead fibers had the appearance of a thin flat ribbon, as shown in Figure 17.

Figure 17. Description of the appearance of mature (a), immature (b) and dead (c) fibers [2].

1.1.4 Chemical Structure

Cotton fibers are composed of long chains of crystalline cellulose that are linked by hydrogen bonds. The bonds are not exactly parallel to the fiber axis, but form a spiral around it, first in one direction and then in another (Figure 18).

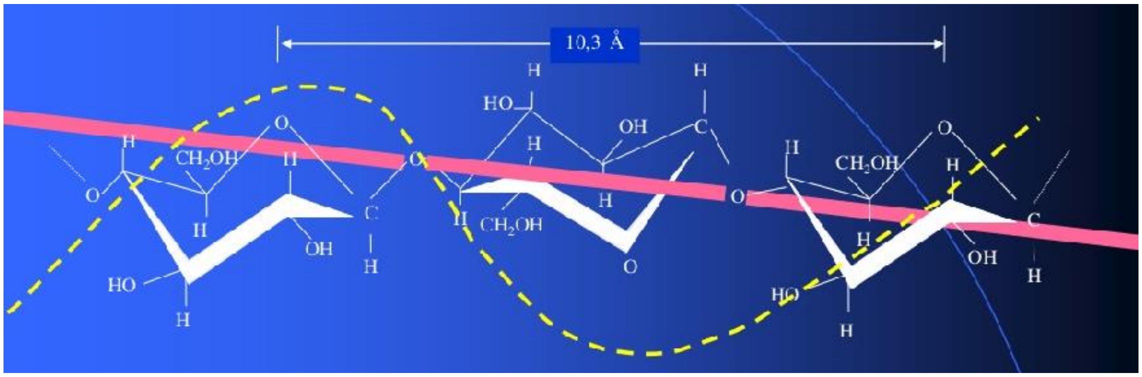


Figure 18. Chemical composition [13].

Cellulose is a lengthy polymer composed of multiple hydroglucose units that form a chain-like structure. Unlike glucose, a soluble and basic sugar, cellulose cannot dissolve in water because of its large size as a polymer. It falls under the carbohydrate category, with carbon accounting for 44.4 %, hydrogen 6.2 % and oxygen 49.4 % of its composition. The cellulose molecule depicted in Figure 19 is composed of cellobiose as its fundamental unit [14].

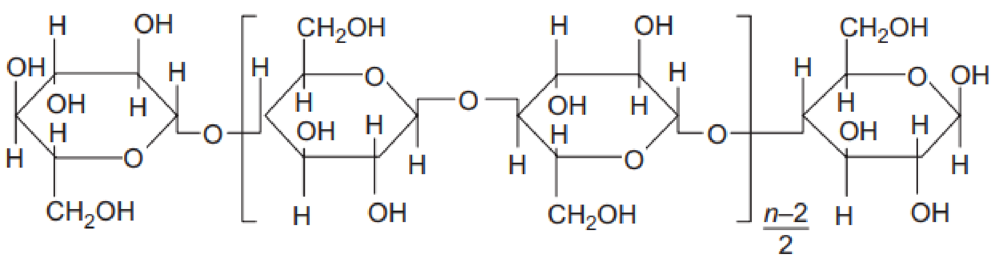


Figure 19. Diagram of the cellulose molecule.

The composition of cellulose is straightforward, as it consists of hydrocellulose units connected by β-1,4-glucosidic bonds to form linear polymeric chains. The length of these chains, known as the degree of polymerization (DP), indicates the number of hydrocellulose units linked together to create a chain molecule. In the case of cotton cellulose, the DP can reach up to 14,000, but it can be easily reduced to 1,000-2,000 through various purification treatments involving alkali. The crystalline regions of cellulose likely have a DP ranging from 200 to 300. Additionally, the molecular weight (MW) of cotton typically falls within the range of 50,000-1,500,000, depending on the source of the cellulose. The individual chains of cellulose adhere to each other along their lengths via hydrogen bonding and van der Waals forces. The physical properties of cotton fibers as a textile material, as well as their chemical behavior and reactivity, are determined by the arrangement of cellulose molecules in relation to each other and to the fiber axis.

The primary wall of a cotton fiber is approximately 1 μm thick and accounts for only approximately 1 % of the total thickness. The majority of non-cellulosic components of the fiber were found in or near the primary wall. These non-cellulosic impurities, including fats, waxes, proteins, pectin, natural colorants, minerals and water-soluble compounds, are predominantly present in the cellulose matrix of the primary wall and, to a lesser extent, in the secondary wall. These impurities significantly restrict the water absorbency and whiteness of cotton fibers. Pectin, in particular, is primarily located in the primary wall of the fiber.

Comprised predominantly of D-galacturonic acid residues connected through α(1→4)-linkages, which exhibit partial esterification of the carboxylic acid groups of certain galacturonic acid residues with methanol. A pectic molecule can be described as a block copolymer, alternating between esterified and non-esterified blocks. Within the primary cell wall, pectin forms covalent bonds with cellulose or hemicellulose in some plants, whereas in others, it forms strong hydrogen bonds with other components. Pectin acts as a potent biological adhesive and its mostly water-insoluble salts play a crucial role in binding waxes and proteins to create a protective barrier for the fiber.

1.1.5 Physical properties

High-quality cotton lint can be used to make high-quality yarn, fabric and, as a result, end products. High-quality cotton lint contains a variety of physical qualities, some of which are quantifiable, while others are not. High-quality lines also indicate the absence of hazardous chemicals. Cotton lint has the following crucial and quantifiable properties [15]:

- staple length
- uniformity of fiber length
- short fiber content (the percentage of short fiber)
- fiber strength
- fineness
- micronaire value
- maturity
- elongation

• Color grade

The properties of the cotton lint that are not measurable include the following.

• hand Feel

• luster

• silkiness

These are the primary attributes examined when cotton is categorized or graded, and they influence further processing and end-product quality. High-grade cotton also has low waste content. Cotton with a high trash content requires extreme processing to remove fibers, which may harm it and reduce the quality of the end products [16].

1.1.5.1 Length and fineness

The cotton fibers used in the textiles had an average length of 25–37 mm. The longer the fiber, the easier it is to process and the higher the quality of the output. Consequently, long-staple cotton is more valuable in textile processing and is utilized to produce high-quality yarn or end products. Because cotton is a natural product, the length of each cotton fiber may vary and the consistency of fiber length is critical because the higher the uniformity, the better the fiber length [17].

The ratio of the short fiber weight to the total tested fiber weight is known as the short fiber content and it is also an important aspect of processing. Short fibers are often defined as those that are less than a particular length. In high-volume instrument (HVI) testing, fibers less than 12.7 mm in length are termed short fibers and the higher the short fiber content, the poorer is the fiber processing ability and end-product quality. The fineness of fibers or yarns is commonly represented in terms of linear density (Tex or metric count). Tex is related to the weight of the fiber or yarn in grams of 10,000 meter length, whereas the metric count corresponds to the length of the fiber or yarn in meters [18].

1.1.5.2 Tensile behavior

The strength of a fiber or yarn is typically defined as the force required to break or rupture and is measured in units such as grams-force (gf), kilograms-force (kgf), newtons (N), centinewtons (cN), or millinewtons (mN). Conversely, tenacity is the resistance to breaking or rupturing per unit fineness of a fiber or yarn, calculated as the breaking force divided by the linear density of the unstrained material. For instance, single medium-staple cotton fibers exhibit a strength of 3.5 - 4.5 (gf), whereas long-staple cotton fibers range from to 4 - 6 (gf). A higher strength allows fibers to endure more processing without sustaining damage that would impair tenacity, durability and overall quality.

Yarn strength is influenced not only by the strength of individual fibers but also by fiber-to-fiber interactions determined by length, friction and the degree of twist. Breaking bundles of parallel fibers is an effective method to better predict yarn strength or tenacity because it simulates the combined effect of fiber strength and interactions. Cotton bundle tenacity ranged from 17 cN/tex for short coarse cotton to 43 cN/tex for long fine cotton. It is important to note that the strength or tenacity of cotton generally increases with moisture content and decreases with rising temperature [15].

Cotton elongation is defined as the percentage of elongation at the breaking point. Elongation at break, or simply elongation, is typically in the 6-9 % range for most cotton. Moisture has the most significant impact on the elongation. When the relative humidity is close to the saturation point, an elongation of approximately 5 % at low relative humidity increases to approximately 10 % [15].

1.1.5.3 Maturity and micronaire value

Cotton maturity is an important property; the higher its maturity, the stronger and thicker the fiber, or the higher its linear density, which is usually related to better dyeability, ease of processing and overall product quality. The micronaire is a maturity metric that assesses both maturity and fineness. The importance of the micronaire lies in its ability to predict obstacles that may arise during the process. Lower micronaire fibers break more easily during mechanical action and because they are generally more flexible, they entangle more easily to form neps [19].

The short fiber content has an impact on spinning performance, yarn and fabric quality, colored fabric appearance and happiness. Micronaire cotton that is too low (immature) or too high (over-matured) is typically avoided, with the optimal range for American Upland cotton being between 3.8 and 4.2 [15].

1.1.5.4 Elongation, stiffness resilience and rigidity

Cotton has minimal elasticity compared to other fibers, with an elongation of only 3-7 %. Cotton fibers, like other vegetable fibers, have little tenacity; therefore, goods made of pure cotton fold readily and do not recover well from wrinkling. Cotton fibers, which are finer than most other vegetable fibers, are soft and have low stiffness [15].

Cotton has little elasticity, in contrast to other fibers, with just 3-7 % elongation. Cotton fibers, like other vegetable fibers, have low tenacity; hence, items made of pure cotton fold easily and do not rebound after wrinkling. Cotton fibers are soft, flexible and finer than most other vegetable fibers [20].

1.1.6 Cotton collection

Cotton is harvested when bolls are fully open and mature. During manual harvesting, workers pick only mature bolls, which require several days of harvesting to ensure uniformity of fiber maturity. In mechanized harvesting, the harvester collects all bolls, regardless of their degree of maturity (Figure 20, Figure 21, Figure 22 and Figure 23).

Figure 20. Ripe cotton capsules [21].

Figure 21. Manual cotton picking [22].

Figure 22. Machine cotton picking (harvester) [23].

Figure 23. Appearance of the cotton harvester [24].

1.1.7 Cotton gin

After cotton is harvested in the field, it is necessary to separate the seeds from the cotton fiber. This process is known as ginning.

Initially, cotton bolls are placed in a hopper, where they are fed into a transportation system that takes them through a series of rollers and drums. These rollers and drums separate cotton fibers from seeds and other unwanted materials such as leaves and twigs. To facilitate this process, cotton is subjected to a pre-drying treatment (Figure 24, Figure 25 and Figure 26).

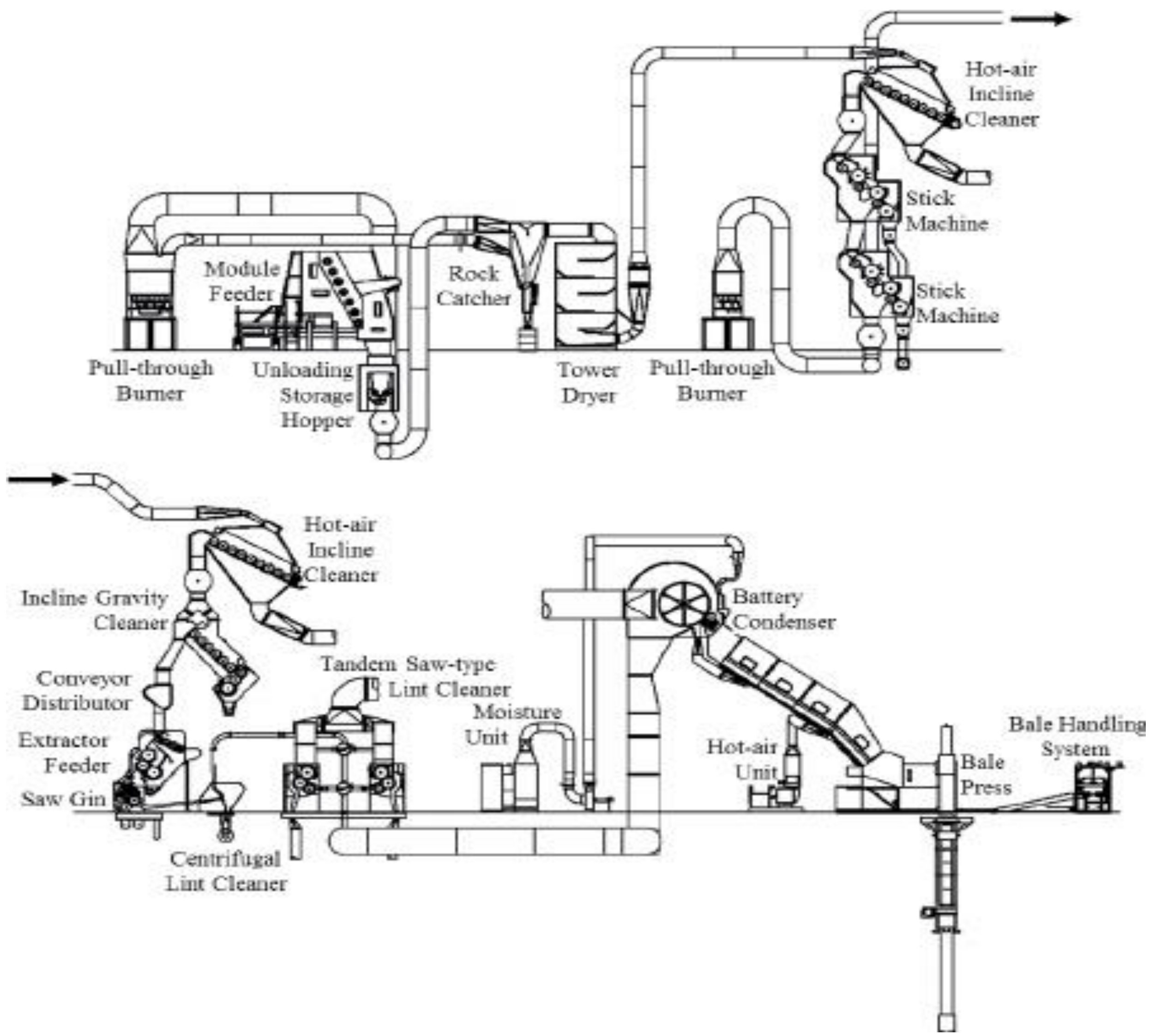


Figure 24. General outline of a cotton gin [2].

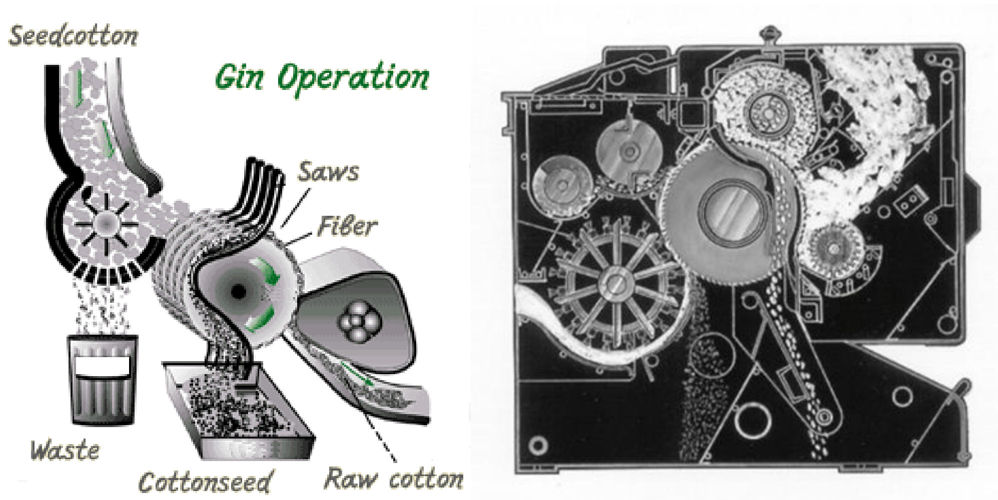


Figure 25. Basic outline of the central cotton gin [2].

Figure 26. External aspect of the ginning machine [2].

1.1.8 Environmental impact of conventional cotton cultivation

For more than a decade, transgenic insect-resistant cotton has been introduced into the environment for commercial production in China. They examined the microbial communities in two separate transgenic cotton fields and one non-transgenic cotton field for three years (2007-2009) to determine the environmental impact of the long-term growth of transgenic insect-resistant plants under believable field conditions. Cotton samples were collected at four distinct phases of development (seedling, budding, boll-forming and boll opening). The results revealed significant seasonal variation in the number of bacteria, fungi, acetobacter, denitrifying bacteria and ammonia-oxidizing bacteria, as well as in microorganism diversity indices, but no significant difference in the number of microbial populations or diversity indices attributable to long-term transgenic cotton cultivation [25].

Cotton production supports the livelihood of over 250 million people worldwide [26]. Despite occupying only approximately 2.5 % of the cultivable land, it is the most popular and profitable non-food crop globally. In developing countries, cotton cultivation accounts for approximately 7 % of the workforce. Cotton cultivation is economically important; however, it also has a high environmental impact.

Global warming

It is estimated that carbon emissions from cotton production amount to approximately 220 million metric tons per year. Conventional cotton uses a significant number of synthetic fertilizers to release nitrous oxide into the atmosphere. Nitrous oxide is a potent greenhouse gas that is 310 times more powerful than carbon dioxide. Other unsustainable practices, such as deforestation and intensive chemical use, also contribute to greenhouse gas emissions [27].

Water consumption

Cotton cultivation and manufacturing requires significant amounts of water. The dyeing process consumes approximately 5 billion liters of water annually worldwide. To produce just one cotton t-shirt, approximately 2.700 liters of water is required. Cotton plants rely heavily on water to thrive and yield profitable results. This accounts for approximately 69 % of the water footprint of textile fiber production. According to the World Economic Forum, water scarcity is listed as one of the top ten global challenges that the world may face in the next decade. A report by the Soil Association suggests that two-thirds of the world's population could potentially experience water scarcity by 2025 [28].

Soil degradation and erosion

Cotton plantations are often monocultures and as with these cultivation systems, the soil quality is degraded. The global area dedicated to cotton cultivation has remained relatively constant for the past 70 years. However, soil depletion has led to its expansion into new areas. This expansion directly causes deforestation and loss of wildlife habitats. The use of chemical products in cotton cultivation also damages the soil. This hinders the ability of the soil to filter water and sequester carbon, while also harming the natural fertility of the soil.

Pollution from cotton cultivation

Extensive and intensive cultivation of cotton requires the use of many harmful chemicals to control pests and increase production. Intensive use of synthetic pesticides and fertilizers has damaged the environment over time. These toxic chemicals also threaten human health, wildlife, water and the soil. Cotton accounts for 24 % and 11 % of global insecticides and pesticides, respectively. It also uses 4 % of the world's artificial phosphorus and nitrogen fertilizers [29].

Cotton is the third crop in the United States with the highest pesticide use. In 2017, producers used approximately 48 million pounds of cotton cultivation. Experts estimate that cotton consumes 8 million tons of synthetic fertilizers and 200,000 tons of pesticides annually worldwide. Of the top ten pesticides used in cotton cultivation in 2017, glyphosate, diuron and tribufos were considered human carcinogens. The remaining compounds are potential endocrine disruptors and toxic to bees. Farmers and people living near heavily contaminated cotton farms may suffer health damage caused by toxicity. In particular, glyphosate can cause genetic damage and congenital disability [30].

Sometimes, clothing and fabrics made from cotton cultivation, which require the intensive use of chemicals, contain traces of pesticides. This is a potential health hazard, particularly for people with sensitive skin.

Pollution from cotton textile production

Brightly colored or immaculately white cotton attracts consumers, but many people are unaware of its environmental costs. The fashion industry accounts for approximately 20 % of industrial water pollution. Production wastewater contains pesticides, toxic dyes, chlorine and other chemicals used to process cotton into fiber and clothing. These pollutants enter water systems, such as rivers, lakes, wetlands and underground aquifers, thereby contaminating them. Rainwater in Brazil's cotton-growing region contains 19 pesticides, of which 12 are directly linked to cotton cultivation [31].

Genetic modification

Scientists have genetically modified certain species of cotton to possess specific qualities that would increase productivity and profitability. Some qualities of genetically modified cotton include resistance to insects and tolerance to herbicides. Genetically modified cotton is used worldwide. Approximately 90 % of genetically modified cotton is grown in China, Argentina, Mexico, the United States, Paraguay, Australia, India, Pakistan and South Africa.

Genetically modified crops yield high yields. However, some types of genetically modified cotton are not eco-friendly. In 2017, ten farmers from the United States used the agricultural biotechnology company Monsanto. The farmers claimed that the company's dicamba-tolerant cotton and soybean crops resulted in the inevitable illegal spraying of the highly volatile and drift-prone herbicide dicamba, causing damage to crop and contamination.

The excessive use of inappropriate insecticides in cotton cultivation has led to at least 40 weed species developing resistance to glyphosate. This has also allowed some lepidopteran insects to develop resistance to the protective BT cotton genes. There is also the risk of genetically modified genes being transferred to wild crops [32].

Conclusion

Cotton is a natural plant-based fiber with exceptional textile properties. Nevertheless, extensive and intensive cultivation requires a significant number of synthetic fertilizers and agrochemicals (insecticides, herbicides, fungicides, defoliants and growth regulators), which have substantial environmental impacts on ecosystem health and human well-being.

Although 50 % of the world's cotton cultivation is rainfed, irrigated cotton consumes large amounts of water. When we add the water consumed in the dyeing and finishing processes, the water footprint of this fiber is extremely high. Moreover, water is used in these processes.

As an alternative to these environmental impacts, new cultivation systems have emerged that partially limit the use of certain agrochemicals, such as BCI Cotton (Better Cotton Initiative), which currently accounts for 22 % of global cotton cultivation. Although it is a crop with a lower environmental impact, it is not organic cotton, which requires a much more demanding agricultural process in terms of the environment and has strict control and certification systems. Currently, organic cotton represents only 1 % of global fiber production, is not treated or extracted improperly and has an even greater environmental impact.

1.2 Organic cotton

Cotton fiber is a historic, traditional, familiar and well-known natural fiber that is highly edible. Chemical fertilizers and insecticides are used extensively in conventional cotton farming. According to a preliminary estimate, the overall weight of fertilizer usage is 33 % of the annual raw cotton yield. In the 1980s, consumers and governments worldwide began to prioritize awareness and responsible conduct in many areas, including the fiber and textile industries. Some measures are taken in the textile and garment industries during the production, processing, consumption and waste processing stages, with organic cotton farming being globally adopted.

Organic cotton is grown using precise methods and controlled inputs, which significantly reduces the environmental impact. Organic fiber production aims to maintain natural resources and human health, as well as all animals, plantations and sustainable living situations. Organic agriculture replenishes and maintains soil fertility, reduces the use of toxic and persistent pesticides and fertilizers and promotes biological diversity. Organic fiber certification requires some difficult procedures, such as ceasing the use of pesticides and chemical fertilizers before three years of subsequent organic cultivation, using only permissible substances, physically enclosing cultivation fields, cultivating trap plants if necessary and using organic manure and organic soil enrichment. Organic cotton has emerged as a promising business opportunity with over 30 textile brands launching organic cotton textiles and clothing collections. The industry is projected to experience exponential growth in the future (Figure 27) [33].

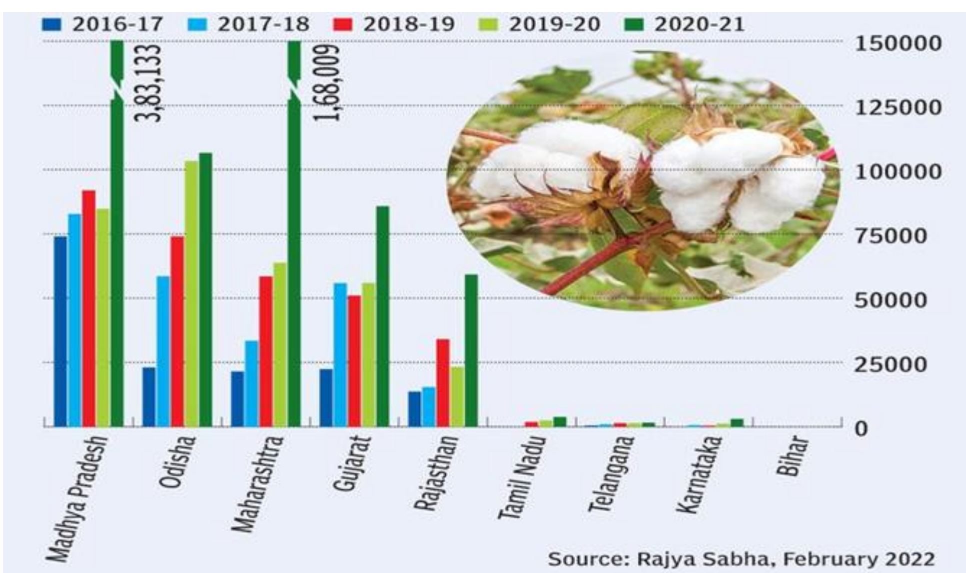


Figure 27. State-wise production of organic cotton during 2016-2021 [33].

1.2.1 Certifications

The certified organic cotton supply chain contrasts the traditional cotton supply chain. This analysis highlighted the most significant differences between the two supply chains. Cotton producers may benefit from switching to organic agriculture in various ways, including cheaper farm input costs, better soils, different sources of revenue and higher pricing. When the gross margins of both production systems are compared, organic Mali cotton may yield larger gross profits than traditional cotton cultivation [34].

Global Organic Textile Standard (**GOTS**): GOTS stands as a prominent standard in textile processing for organic fibers, notably cotton. It sets out environmental criteria across the entire organic textiles supply chain and mandates adherence to social criteria.

Organic Content Standard (**OCS**): The OCS verifies and monitors the presence of organically grown materials in the final product. It can be utilized for any non-food item containing 95-100 % organic material.

Better Cotton Initiative (**BCI**): BCI strives to enhance global cotton production for the benefit of producers, the environment and the industry's future. BCI certification emphasizes environmental, social and economic sustainability.

Fair Trade Certification (**FTC**): This certification guarantees fair compensation for cotton producers in addition to extra funds for community development. Fair Trade also upholds stringent environmental and labor standards.

STANDARD 100 by **OEKO-TEX®**: A worldwide standard for textile products across all processing stages, from fiber to finished garment. It confirms that the product has undergone testing for harmful substances, ensuring human health safety.

Cotton Made in Africa (**CmiA**): An initiative dedicated to improving the living conditions of African cotton farmers, advocating for eco-friendly cultivation methods and prohibiting genetically modified cotton.

Recycled Claim Standard **(RCS**) and Global Recycled Standard (**GRS**): These certifications pertain to products crafted from recycled materials including cotton. GRS incorporates social and environmental processing criteria, as well as chemical restrictions.

ISO 14001: Although not specific to cotton, ISO 14001 certification for Environmental Management Systems can be pertinent for cotton producers and manufacturers striving to reduce their environmental footprint.

Textile Exchange Standards: Encompassing various certifications like the Responsible Down Standard (**RDS**), Responsible Wool Standard (**RWS**) and others that uphold ethical and sustainable practices.

These certifications are essential for both businesses and consumers to recognize products that align with their beliefs in environmental conservation, social equality and product safety. They are crucial in advancing sustainable methods throughout the supply chain of the cotton industry.

1.2.2 World production and market evolution

Organic cotton production totaled 1.08 million tons. The largest organic cotton-producing countries are India, China, Kyrgyzstan, Turkey and the United States, with respective production (in tons) of 60.184, 14.187, 7.981, 7.577 and 4.524 in 2015-16. Organic cotton production is not connected to farming by simply substituting organic fertilizers and insecticides with synthetic fertilizers and pesticides. Organic cotton is defined as cotton that has been grown and certified by organic agricultural standards. Cotton is cultivated, harvested and processed without the use of chemical fertilizers, pesticides, herbicides, growth regulators, or defoliants. The specific agricultural procedures vary by location or even from farmer to farmer, but the use of genetically engineered seeds, agrochemical synthetic pesticides, fertilizers and growth regulators that are toxic and persistent is unavoidable [35].

1.2.3 Environmental impact of organic cotton

Organic agriculture is a farming strategy that improves soil fertility by optimizing the effective use of local resources while avoiding the use of agrochemicals, Genetically Modified Organisms (GMOs) and many synthetic substances used as food additives. Organic agriculture employs a variety of farming approaches based on ecological cycles, to lower the environmental effect of the food business, conserve long-term soil sustainability and minimize the use of non-renewable resources, which considerably decreases soil loss and increases soil organic matter (SOM) content under organic management. The biochemical and ecological properties of the soil appear to have improved. Furthermore, organically maintained soils have a far better water-holding capacity than conventionally managed soils, resulting in substantially higher yields during water shortages than conventional farming. Organic agriculture, owing to its better potential to store carbon in the soil, might provide a technique to enhance CO2 abatement if implemented on a broad scale. The influence on biodiversity is then highlighted; organic agricultural methods often have greater floral and faunal biodiversity than conventional systems; however, both may boost biodiversity when properly managed.

Importantly, this indicates that the environment surrounding cultivated land has the potential to improve biodiversity in agricultural settings. The study then discusses energy utilization in several agricultural settings, which offers greater energy efficiency (input/output) but lower yields and, hence, poorer productivity. Nonetheless, organic agriculture appears to perform better than conventional farming in general and it also provides significant environmental benefits, such as reducing the use of harmful chemicals and their spread in the environment and along the trophic chain as well as reducing water use. Based on the findings presented in this review, there is a clear need for more research and investment directed toward exploring the potential of organic farming to reduce the environmental impact of agricultural practices. However, the implications of decreased productivity on the socioeconomic system should also be considered and appropriate agricultural policies should be developed [36].

1.3 Natural Organic Colored Cotton (NaCOC)

1.3.1 Origin, varieties, producing countries and the evolution of production.

NaCOC was cultivated and used 4,500 years ago, as evidenced by excavations at Huaca Prieta in Peru. In this regard, De Carvalho et al, state that some researchers analyzed fabric fragments from this era and observed cotton fibers colored blue, purple, pink, brown, green, bronze and red [37].

In North America, the Acadians (Louisiana French) grew and spun naturally colored cotton for hand weaving. Brown cotton was then known as " *slave cotton* " in the South since it was used for slave clothing [38] and was thought to have been brought to Mississippi from Mexico [39].

In the mid-19th century, cotton production and exports were interrupted in the United States because of the Civil War. Records indicate that Peru exported brown cotton to England during this period. Between 1865 and 1937, this country produced and exported up to 12 different colors of cotton to English manufacturers, who mixed them with wool. Brown cotton was a cash crop in Louisiana until World War I [38-41].

NaCOC cotton trade has a long history. For example, Russia sold approximately 770 tons in 1945 [42-43].

However, the large-scale cultivation of NACOC cotton is not due in no small measure to developments by Californian entomologist Sally Fox, who first saw wild brown cotton seeds while working as a grower's assistant, developing cotton strains genetically resistant to pests [41]. Fox was enthusiastic about these colored cotton fibers; however, their low textile quality made spinning virtually impossible and he decided to improve their textile properties [44].

Through proper selection and cross-pollination, Fox obtained colored cotton hybrids with fibers long enough to be spun with current machinery in 1988. The success of growing a naturally colored spinnable cotton fiber led Sally Fox to create the company *Natural Cotton Colors, Inc.*, also obtaining a plant variety protection certificate for its cotton and the trademark FoxFibre ®, which markets the FoxFibre® green, Coyote brown, Buffalo brown and Palo Verde green varieties.

In 1984, the cotton grower Raymond Bird began experimenting in Reedley, California, with red, green and brown cotton to improve fiber quality [45].

The NaCOC cotton production has fluctuated widely. In 1993, 1,575 ha were planted in the US, reaching a maximum in the period 1993-1994 and 1995, when world production reached 53,600 bales, of which 30,940 were grown in the US. In 1996, however, many small producers withdrew from the market and planting in the US did not exceed 20 ha [46].

Currently, there is an interest in the use of NaCOC cotton in spinning mills in Europe and Japan. The main problem facing US producers is not the marketing of the lint, but the difficulty they face in finding a gin to process the lint. Regulations to protect white cotton from color contamination are stringent, such as the California Code of Regulations and the Arizona Department of Agriculture.

During World War II (1939-1959) and due to the lack of dye supply, NaCOC cotton of brown and green varieties was temporarily cultivated and consumed again, which ended at the end of the war [47].

Currently, more than 27 countries, including the United States, China, Brazil, Egypt, India and Peru, are researching NaCOC cotton. Various colors such as mocha, tan, gray, brown, black, mahogany, purple, orange, red, pink, blue, green, gray and cream are commercially available natural-colored cotton shades after genetic development. In 2001, there were more than 11,000 acres of NaCOC cotton in the US, primarily in Texas, California and Arizona. This number was later reduced to less than 5,000 acres due to increased competition from foreign producers, such as China and some South American countries [48-49].

After the first planting in the 1990s, China cultivated more than 46, 600 hectares of NaCOC cotton in 2005, an increase of more than 200 % from the 20,000 hectares cultivated in 2004.

Xinjiang province currently grows about 46,700 ha of NaCOC cotton. Xinjiang has developed seven varieties of colored cotton, including brown and green cotton. Xinjiang researchers Genetically Modified Colored Cotton Institute have been working with the Hereditary Science Institute of the Chinese to produce red, blue and black cotton by transferring an external color gene into naturally grown white cotton with sophisticated genetic engineering technology. World NaCOC cotton production reached 160,000 tons per year and cotton yarn sales increased more than 300 times compared to 2000 [50].

Colored cotton production in China currently accounts for 30 % of the world's total cotton production [51-52].

In the last 20 years, interest in NaCOC cotton has increased remarkably, owing to its environmental benefits and unique colorimetric properties. These studies have addressed various aspects of these fibers: the origin of natural color varieties of cotton, structure, composition, pigments, effects of scouring and washing of fibers, wax, textile properties of fibers, heavy metal content, behavior against UV radiation, fire behavior, bactericidal properties and environmental advantages.

Researchers believe that old World Asian diploid cotton originated before New World allotetraploid cotton. Color varieties were known in diploid cotton and were grown in Asia, especially in the Indian subcontinent, China and the Central Asian republics of the former Soviet Union for a long time [47].

It is generally accepted that cotton originated separately in the two hemispheres of the Earth, in the Old World and in the New World. Each hemisphere appeared to have two intersecting centers of origin. However, the cotton of the two separated hemispheres did not interbreed successfully (interbreeding did not produce fertile offspring). American cotton has 26 chromosomes, whereas Asian cotton has only 13 chromosomes [53].

Commercial cotton fibers are unicellular seed outgrowths of plants belonging to the genus *Gossypium* of the Malvaceae family, which also include hibiscus, okra and various mallows. Each type of cotton tends to produce a characteristic amount of fiber [54-55]. In 1987, Fox et al. identified 39 distinct cotton species [44], identification of *Gossypium* species *arboreum, G. herbaceum, G. raimondii, G. darwinii, G. tomentosum, G. barbadense and G. hirsutum.* According to Rollins, cultivated species include *G. arboreum, G. herbaceum, G. barbadense and G. hirsutum[56]*.

G. herbaceum is the original cotton from India. It is currently commercially grown in India, China, Iran, Iraq, Turkey and Russia. *G. arboreum* and *G. herbaceum* are commonly known as Asian cotton and include the shorter basic cultivated cotton. *G. arboreum* was the "cotton tree" of India and Africa. Thus, it is of minor commercial importance. *G. peruvianum* is generally considered indigenous to Brazil and other South American countries and is now considered part of the species *G. barbadense[56]*.

Most cotton species have variations in fiber color, primarily in browns ranging from white or extremely light brown to brown, reddish brown, grayish brown and extremely dark brown. Many species have been genetically examined and the gene loci identified where the alleles that produce color variations reside. The inheritance of fiber color in *G. barbadense* was studied by Balls in 1906 [54, 57].

Endrizzi et al. [57]. Identified the color variation in *G. raimondii* as "dirty white fiber" and the gene location as Dw. Harland discovered in 1935 that alleles at different loci determine brown fiber. The genes for the brown variations were found in Lc1 (incompletely dominant) in *G. hirsutum* and Lc2 (uniformly homozygous) in *G. barbadense, G. darwinii and G. tomentosum*. Some G. hirsutum lines also carried Lc2.

The dominant mutant gene was found in Lg in *G. hirsutum*, which produces green fiber and fiber that fades to a brownish color during capsule opening. Green fiber mutants have been identified as alleles at the Lg locus [46, 57-58].

Both Fox and Kohel identified the cotton species currently being studied in the United States for the marketability of their fiber color variations as *G. barbadense* and *G. hirsutum*. These two species are the two New World crop species commonly known in the United States as Pima cotton and upland cotton, respectively. Both have been identified as pre-Columbian species [54, 58].

Colored variants of the species *G. barbadense* (also called Long Staple, Sea Island, Egyptian, or Pima cotton) originated in the northern Andes and have longer staple lengths, coarser fibers and a wider range of colors than the color variants of *G. hirsutum*. Seven color variations in South America are currently used by Indians in Pem. All seven were browns of varying intensities, six were browns from the orange family and one was taupe brown with a lavender tinge [41].

The commercialized upland cotton species is *G. hirsutum* (also known as short-staple cotton), which originated in the Central American civilization among the Mayan Indians of Mexico. In Mexico, *G. hirsutum* has spread to the southwestern United States of America. Compared to *G. barbadense, G. hirsutum* is short-fibered and has limited color variations. *G. hirsutum* is cultivated throughout the world, providing 85 % to 90 % of the world's supply of mill-seed cotton [54-55].

All the original cotton grew primarily in tropical areas, where it remained a perennial plant. Modern Pemvian native-colored cotton, for example, produces cotton continuously for six to ten years, never dying.

Fox (1987a) stated that some cotton plants grew to 30 feet in height [54]. Approximately 200 years ago, according to Vreeland, cotton growers began cultivating the naturally perennial *G. hirsutum* annually in the temperate climates of America. West Texas has the highest elevation at which cotton has successfully grown [59].

1.3.2 Physical and chemical properties

Various studies on the morphology of green NaCOC cotton have suggested that the secondary walls of the fibers are formed by alternate layers of cellulose and a waxy organic substance called suberin [60].

Previously, Yatsu et al. (1983) concluded that green cotton fibers have a different morphology than white and brown cotton fibers do. That is, the secondary cell wall in green cotton fiber is composed of alternating layers of cellulose-suberin [61-62].

Quantitatively, the suberin content was determined by ethanol extraction. Owing to its polymeric nature, suberin is more difficult to remove from fibers than regular cotton wax. Thus, in the studies cited above, a 24-hour ethanol extraction was required for the complete removal of suberin. The ethanol extraction results show that green cotton has a significantly higher suberin content than the wax content of white or brown cotton.

Analysis of the fiber structures by X-ray diffraction showed that in all fibers the cellulose structure is normally found in cotton. However, in the green fibers, the crystallites were significantly smaller than those in the brown and white fibers. This may be because the alternating layers of suberin and cellulose in the secondary wall inhibited crystallite development. When the green fibers were fully extracted with ethanol, the size of the crystallites increased to the level found in the other fibers [63].

Using electron microscopy techniques, it has been shown that green cotton fibers (Gossypium *hirsutum L.*) cells had numerous thin concentric rings around the cell lumen. These rings have a fine lamellar structure, characteristic of suberin. Depolymerization analysis and gas chromatography-mass spectrometry demonstrated the presence of a suberin polymer in green cotton, the main aliphatic monomers being omega-hydroxydocosanoic acid (70 %) and docosanedoic acid (25 %). Chemical and ultrastructural analyses revealed that ordinary white cotton was encircled by a thin polymer cuticle comprising less than 0.5 % aliphatic components, which was significantly lower than that found in green cotton.

Blas-Sevillanoa et al. analyzed different types of Peruvian NACOC fibers and characterized them based on their chemical and physical properties using FTIR techniques, XPS, XRD and TGA. In their study, they concluded that the color intensity of NaCOC fibers seems to play a key role in the physicochemical properties of these materials. The darker-colored samples showed different chemical compositions (presence of nitrogen), lower crystallinities and shorter length values compared to the white and/or lighter cotton samples. Finally, some NaCOC fibers (mainly those exhibiting lighter colors) showed physicochemical properties similar to those of the white cotton fibers. The crystallinity also appeared to decrease with increasing color intensity. Finally, the thermal stability of the white cotton fibers was similar to that of NaCOC [64].

Various studies have shown that the brown pigments in cotton fibers are proanthocyanidins (PA). To clarify the details of the PA biosynthesis pathway in brown cotton fibers, Xiao et al. compared gene expression profiles in developing brown and white fibers using digital gene expression profiling and Real-Time Quantitative Reverse Transcription PCR (qRT-PCR). Compared to white cotton fibers, all steps from phenylalanine to PA monomers (flavan-3-ols) were significantly upregulated in brown fibers. Liquid chromatography-mass spectrometry analysis showed that most of the free flavan-3-ols in the brown fiber were in the 2,3-trans (gallocatechin and catechin) form and the major polymeric PA units were trihydroxylated in ring B. According to the monomeric composition, the transcription levels of flavonoid 39, 59-hydroxylase and leucoanthocyanidin reductase in cotton fiber were much higher than their competitor enzymes acting on the same substrates (dihydroflavonol 4-reductase and anthocyanidin synthase, respectively). Taken together, the data from this study revealed a fully activated PA biosynthesis pathway in brown cotton fiber and demonstrated that flavonoid 39,59-hydroxylase and leucoanthocyanidin reductase represent the main components of PA biosynthesis in cotton fiber [65].

Many species have been genetically examined and the gene loci identified where the alleles that produce color variations reside. The inheritance of fiber color in *G. barbadense*, for example, was studied by Balls as early as 1906 [54, 57, 66]

Endrizzi et al. identified the color variation in *G. raimondii* as "dirty white fiber" and the gene location as Dw. Harland discovered in 1935 that alleles at different loci determine brown fiber[57].

Both Fox and Kohel identified the cotton species currently being studied in the United States for the marketability of their fiber color variations, such as *G. barbadense* and *G. hirsutum*. These two species are the two New World crop species commonly known in the United States as Pima cotton and upland cotton, respectively. Both have been identified as pre-Columbian species [54, 58].

The natural color of cotton is due to the inherent genetic properties of the plant. The hue of colored cotton can vary with season and geographic location due to weather and soil variations.

There are two important aspects to consider in the study of cotton fiber coloration:

1- **Germplasm banks (Genetic resources for the improvement of crops):** In India, approximately 40 genotypes of upland cotton (G. *hirsutum)*, mostly various shades of brown and green, are available from the National Cotton Genetics Bank of Central Cotton Research Institute., Nagpur. These gene pools are indigenous and exotic collections from the US, the former USSR, Israel, Peru, Mexico, Egypt and others. Most colored fiber germplasm lines have been evaluated for their economic importance and fiber characteristics [47].

2- **Wild species:** Wild species are important sources of colored fiber; many of the wild species of the genus *Gossypium, including today's* tetraploid cotton, that is, *G. herbaceum* race *africanum* and *G. raimondii,* have colored fibers. Brown color in the different shades was the most common. The fiber colors of the *Gossypium species* are listed in Table 2.

Table 2. Fiber colors are found in the Gossypium species [46].

Continent	Species	Genome	Color of the fiber
America	G.aridum	D 4	Brown
	G.armourianum	D 2-1	Brownish
	G.darwin	TO	Brownish
	G. musteline	TO	Brownish
	G.gossypioides	D 6	Greyish
	G.harknessii	D 2-2	Greyish
	G. laxum	D 9	Tan
	G. lobate	D 7	Tan, White
	G. Tribolium	D 8	Tan
	G. lanceolate	TO	White
	G. tomentosum	(AD) 3	Red Brown
	G. hairy	(AD) 1	Brown, tan, white, green
	G. barbadense	(AD) 2	Creamish, white
Africa	G. anomalum	B 1	Brownish
	G. capitis-virdis	B 4	Brownish
	G. somalense	E 2	Brownish
	G.herbaceum	A 1	Greyish, white
	G.longicalyx	F 1	Greyish
	G.triphyllum	B 2	Tan, creamy
Afro-Asia	G.arboreum	A 2	Brown, tan, white
	G.stocksii	E 1	Brownish
Arabia	G. areysianum	E 3	Brownish grey
	G.incanum	E 4	Tan
Australia	G.australe	C 3	Brownish
	G.sturtianum	C 1	Brownish
	G.robinsonii	C 2	Greyish
	G.sturtianum var nandevarense	C 1-n	Greyish

In addition to fiber color, some germplasm lines and wild species are resistant to insect pests, droughts and salinity.

To date, the pigments in NaCOC cotton are not well known. Brown cotton fibers are generated in the tannin vacuoles of the lumen [67]. The brown color did not appear until the capsule opened when the fibers were exposed to oxygen and sunlight [68]. Thin-layer chromatographic analysis revealed that the pigment components included tannin precursors, catechins and tannin derivatives [69].

Tannin is a large polyphenolic compound that contains sufficient hydroxyls and other possible groups that can form strong complexes with proteins and other macromolecules. The tea plant (Camellia sinensis) is an example of a plant with naturally high tannin content [70].

The color of green cotton originates mainly from caffeic acid (Figure 28), which is a derivative of cinnamic acid (Figure 29) found in the suberin layer. Caffeic acid is deposited in alternate layers with suberin outside the fiber [66].

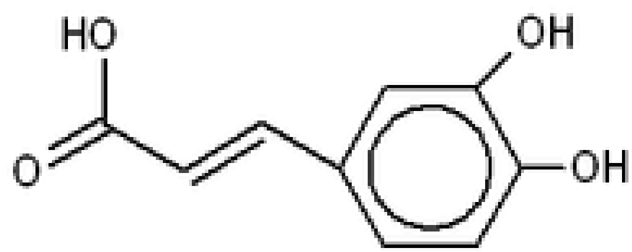


Figure 28. Caffeic acid [66].

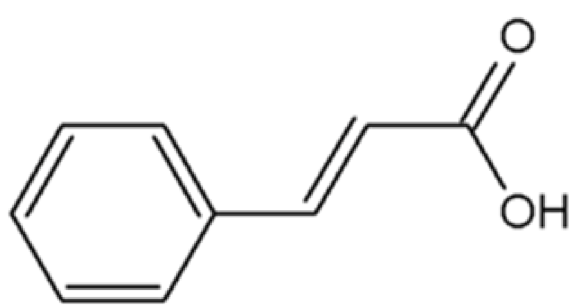


Figure 29. Cinnamic acid [66].

The amount of pigment was higher in the brown cotton than in the green cotton. As these coloring materials are bound to fiber molecules, they affect the formation of crystalline areas in the fiber, but the binding effects are different for brown and green cotton [71].

Fiber color is a genetically controlled trait. Pigment accumulation in the fiber lumen began before the capsule opened. In upland cotton (*G. hirsutum*), pigmentation begins to appear in the developing fiber 32 days after fertilization and takes nearly six days to develop color. In Asian cotton (*G. arboreum*), color pigments are observed 46-47 days after fertilization, taking 5-6 days to develop color. However, full expression of the fiber color occurs only when the capsule is opened and the fiber is exposed to sunlight. It takes approximately a week for the fiber to acquire a completely natural color. The intensity and time required for full-color development vary depending on the genetic background of the genotypes. Interestingly, although sunlight is essential for color development, continued exposure to sunlight leads to fading [47].

In the cultivated New World tetraploid species *G. hirsutum* and *G. barbadense*, the inheritance of fiber color has been more extensively investigated than that of Asian species. In several studies, each of the fiber colors (rust, dark brown, yellowish brown and green) was observed to be governed by a single incompletely dominant gene. The fiber color loci are allelomorphic and show admixture inheritance, indicating the involvement of plus modifiers [47].

The genetic background of fiber color has been extensively investigated in Asian and New World cotton species. In the Asian species (*G. arboreum* Y *G. herbaceum*), fiber color inheritance resembles a single major gene if segregates with variable color intensity are considered as a single group. However, the presence of modifiers obscures segregation, making it difficult to determine the exact number of genes responsible for fiber color. Later studies established the involvement of three loci, along with a variable complex of minor genes for fiber color. Following the rationalized system of gene nomenclature in cotton, these loci are symbolized as Lc1, Lc2 and Lc3. Subsequently, Lc4 and Lc5 were also reported. The characteristics of the individual loci and fiber color patterns governed by the respective genes are presented in Table 3.

Table 3. The characteristics of individual loci and fiber color patterns are governed by their respective genes [46].

Loci	genes	Characteristics
Diploid Cotton		
Lc1	lc1^k	The gene for khaki LC1 ^k is completely dominant, least affected by modifiers and shows little fading
Lc2	Lc2 ^ k	Determines khaki color which is very slightly lighter than Lc1 ^k and regarded as a duplicate of Lc1 ^k
	Lc2 ^M	Determines medium brown color
	Lc2 ^B	The gene for light brown lint, Lc2 ^B, shows low dominance and is highly susceptible to modifier effect and fading
	Lc2	Receive allele, with lint
Lc3	Lc3 ^B	Expression is similar to Lc2 ^B and regarded as duplicate Lc2
Lc4	Lc4 ^K	Khaki color lint
Lc5	Lc5 ^B	Light brown lint color, maybe a duplicate of Lc2 ^B
Tetraploid Cotton		
Lc1	Lc1 ^K	Governs khaki lint color in Guatemala upland cotton
Lc2	Lc2 ^K	Governs brown lint color in Egyptian enan's brown Lc2 ^K is independent but regarded as duplicate Lc1 ^K
Lc3		Dark brown lint color
Lc5		Determines light brown lint
Lc6		Determines brown lint
DW		Dirty white lint color
Lg		Green lint color

Zhao et al. have studied the presence of flavonoids and carotenoids in NaCOC cotton fibers. They quantified the flavonoid content in mature brown, green and white cotton fibers;

however, no carotenoids were detected. The pigment in the NaCOC cotton fibers was extracted with methanol at room temperature. The results of the pigment extraction color reactions showed that the methanol-extracted pigment consisted of flavones or flavonols with two adjacent hydroxide groups. The results of the UV spectral analysis further showed that the methanol-extracted pigment from the brown fiber belonged to the flavone with two adjacent hydroxide groups in the B ring and that the green fiber contained not only flavones but also flavonols with two adjacent hydroxide groups, both on rings A and B, indicating that they replaced a hydroxide group at three sites on ring A [72].

In the cultivated New World tetraploid species *G. hirsutum* and *G. barbadense*, the inheritance of fiber color has been more extensively investigated than that of Asian species. In several studies, each of the fiber colors (rust, dark brown, yellowish brown and green) was observed to be governed by a single incompletely dominant gene. The fiber color loci are allelomorphic and show admixture inheritance, indicating the involvement of plus modifiers [47].

The color of the NaCOC cotton fiber and its variation are influenced, to a large extent, by the effects of sunlight. Prolonged exposure of green fibers to sunlight during capsule opening causes rapid color fading to white or off-white. However, parts of the capsule that were not exposed to direct sunlight retained the original color of the fiber. Green has higher light fastness than brown, which also fades at a slower rate [47].

1.3.3 Studies on the characteristics, properties and influence of washing on the color variation of colored organic cotton fibers

There are several studies on the influence of scouring and washing on the color variation of NaCOC cotton, as well as the effects of exposure to sunlight.

NaCOC cotton fibers are quite different from conventional cotton fibers dyed with synthetic dyes. While in the latter, the color decreases with each wash, the color intensity of the NaCOC fibers increases with each wash.

According to Fox, brown colors reach their most intense colors between 10 and 20 washes and green in 30 washes. After maintaining the most intense intensities for some time, the colors gradually lightened between 100 and 120 washes to recover their original colors [58, 66].

In 1994, Williams, BLM studied the resistance to changes in color when exposed to detected stains and fabric care chemicals in cotton NaCOC Foxfiber [41].

Samples used in this study were 100 % white FoxFibre ®, 100 % brown (Coyote) FoxFibre ® and 100 % cotton green FoxFibre ® interlock knits. This study was divided into 2 phases. The samples for Phase I were untreated raw fabrics, whereas those for Phase II were washed raw fabrics.

Each piece of fabric was divided into six sections and numbered in permanent ink. Each of the six sections of a given piece of fabric received the same treatment, except for the number of care cycles; one section was removed after the first wash or dry cycle and another after 2, 5, 10 and 15 cycles. The sixth section underwent 20 cycles in Phase I and was not used in Phase II.

In Phase I, the samples were evaluated for the effects of dry heat, moist heat, wash detergent, water temperature, water hardness and number of wash cycles. Samples from Phase II were analyzed for the effects of stains, pretreatments and combinations of stains and pretreatments on the colors of washed and dry-cleaned coyote cotton, green and white. Pretreatments were applied immediately before the first wash or dry-cleaning cycles.

Dyes were applied to eight sets of samples (one set equaled one for each color). The eight sets of samples were divided into (1) wash or (2) dry clean: (a) within 16–30 hours, (b) after 7 days, (c) with pretreatment within 16–30 hours, or (d) with pretreatment after 7 days. Stains and/or pre-treatments were applied only once before the first care cycle, after which the samples were only washed or dry-cleaned.

The main conclusions of this study are as follows:

- The color changes were notably impacted by the number of wash cycles. Typically, color alteration reaches a stabilized state after approximately 15 cycles. Specifically, the Coyote color of cotton stabilized after 10 cycles, while the green color stabilized after 15 cycles. Notably, the green cotton experienced more pronounced color changes compared to the Coyote cotton. Additionally, it was observed that for both colors under examination, the most significant intensity of color change was observed after the initial wash cycle.

- A clear contrast was observed in how soft and hard water affected the color change of cotton. Hard water usage resulted in a higher average alteration score. The impact of water hardness was particularly noticeable in green cotton.

- The color variations in the wash are also affected by the temperature of the water. When the temperature decreased, the color variations decreased as well.

- A significant change in color reflectance was observed between fabrics washed with detergent and those that were not washed with detergent.

The number of cycles that the samples in Williams et al. (1994) study had was similar to the number of cycles used by other investigators [41, 73]. Raheel et al. subjected the samples to 0–400 cycles using a launderometer [74]. Instead, Breen et al. analyzed samples after five wash cycles [75].

Later (1999) Dickerson et al. studied naturally colored cotton and its resistance to changes in color and durability when refurbished with laundry aids. The purpose of this study was to evaluate the effects of various laundry aids, detergents with optical brighteners, phosphates and chlorine and non-chlorine bleaches on the color variation and durability of fabrics made from NaCOC cotton fibers. The fabrics evaluated were interlock jerseys knitted from single 20-count open-end spun. BC-1 (green), BC-2 (brown) and BC-3 (red) colored yarns, whereas the control fabric was conventional white Pima cotton. Five washing systems were used according to Table 4.

Table 4. Laundering Methods.

Method	Laundry Aid(s) Contents	*Amount used
1	AATCC 1993. Standard Reference Detergent. Without Brighteners (WOB). No Phosphates	66 grams
2	AATCC Standard Reference Detergent 124. With Brighteners. With Phosphates	90 grams
3	AATCC Standard Reference Detergent. (WOB) With Phosphates	90 grams
4	AATCC 1993 Standard Reference Detergent. WOB. No Phosphates. Non-Chlorine Bleach (Colorox II Brand)	66 grams (1/2 cup)
5	AATCC 1993 Standard Reference Detergent WOB. No Phosphates. Clorine Beach (Clorox Brand)	1 cup

*Amounts used as per test method or consumer label instructions

Most of the color change occurred for all fabrics by five washes for all methods except wash method 5 (detergent plus chlorine bleach), which had larger ΔE values at each interval for all methods. These values were significantly higher than those obtained for all other washing methods for all fabrics after 20 washes. NaCOC fabrics not only showed increases in ΔL (lightness) but also higher Δa and Δb values. This is in fundamental agreement with the test results reported by Williams [41]. Except for wash method 5, the similarities in the test results between fabrics and between methods were very low. Naturally colored white and brown Pima cotton fabrics showed the least amount of color change (insignificant to each other) with wash method 1. Neither method resulted in inconsistently lower ΔE values (less change) for cotton fabrics of natural color.

Natural-colored green cotton had the lowest ΔE with wash method 2 but was not significantly lower than that with wash methods 1 or 4. The ΔE for red NaCOC steadily decreased with each wash period, reaching the lowest ΔE after 20 washes of any fabric using method 4.

Subjective and visual assessments conducted by a consumer panel consistently assigned the lowest ratings (ranging from 1.0 to 1.3 on a scale of 1 to 5) to all fabrics subjected to wash method 5, indicating the highest degree of color alteration with this method. The consumer panel did not discern significant differences in color change among other methods for white or red fabrics. However, notable discrepancies in consumer ratings were observed for green and brown fabrics, with no consistent preference for washing methods between the two colors. Across all methods, fabrics were consistently ranked in the following order: Pima White exhibited the least change, followed by Brown, Red and Green, with the lowest rank indicating the most significant change. Interestingly, subjective consumer assessments exhibited a more uniform pattern across methods compared to the ΔE values. Consequently, consumer panel ratings for color change did not always align with the ΔE values. The consumer panel found that fabric color change was still acceptable for all fabrics with Methods 1, 2, 3 and 4, except for green cotton fabrics, which were primarily rated "slightly acceptable," and can be used with reservations. All fabrics were found to be only slightly acceptable or inferior (1.6 for green) using Method 5.

As previously mentioned, the inclusion of chlorine bleach notably influenced both the value and tone of all fabrics. Across all laundering methods, significant distinctions were observed in the NaCOC cotton fabrics. Fabric assessments consistently followed the same order (white, brown, red, green) for methods 2, 3 and 5. However, for methods 1 and 4, brown received a higher rating compared to white [45].

In 2009, Kang et al. conducted a study on the characterization of pigments in three naturally colored cotton varieties (buffalo brown, coyote brown and green), as well as their alterations in moisture recovery during finishing and maintenance processes. The application of enzymatic scouring with pectinase and cellulase did not produce significant color changes in naturally colored cotton. However, two alkali scouring methods (using CaCO3 and NaOH) resulted in decreased lightness and chroma (a* and b*) of NaCOC cotton. Various analytical techniques including wax extraction, light microscopy, scanning electron microscopy (SEM), X-ray surface analysis and inductively coupled plasma (ICP) analysis were employed to elucidate the mechanism behind the observed color changes. The findings revealed that following alkali scouring, the cross-sections of the fibers expanded in green cotton, leading to the migration of pigments to the outer surface and a substantial reduction in potassium content within the fibers. Moreover, the color intensity of the laundered cotton increased notably after enzyme treatment. Subsequent exposure to UV radiation resulted in a decrease in lightness for cotton treated with a single enzyme after five hours, followed by an increase, while cotton treated with multiple enzymes exhibited a continuous rise in lightness during UV exposure. Remarkably, green-colored cotton lost its original color and acquired a red hue upon exposure to UV rays [76].

Han et al. conducted a study to investigate color changes in naturally colored organic cotton (NaCOC) fibers following laundering and to assess the human sensory perception of these fibers. They aimed to explore the relationship between color coordinates and sensory perception. Three NaCOC fiber colors (ivory, coyote brown and green) underwent four treatments (boiling water, enzyme, sodium carbonate and sodium hydroxide). Color coordinates (*L*, a*, b**) were measured in *CIELAB* using a spectrophotometer (SP62, X-Rite) and color differences (*ΔL*, Δa*, Δb** and *ΔE*) were calculated. Sensory perception of NaCOCs was evaluated by 27 female participants using a questionnaire consisting of nine pairs of bipolar visual sensory adjectives via SDS. Results indicated that *L** and *b** values decreased, while a* values increased after treatment. *ΔE* was highest when treated with alkaline solutions compared to other treatments. Human sensory perception, including brightness, clarity, lightness and freshness, generally decreased, while vividness and strength increased. Notably, significant color factors predicting brightness and lightness were *L** and *ΔL*, respectively, while factors predicting vividness and sensory strength were *ΔL* [77].

1.3.4 Textile properties of fibers

The initial investigations into the textile quality of colored cotton fiber and pilot spinning trials trace back to the 1940s and 1960s [78].

Colored cotton was discovered to be weaker and shorter compared to white cotton. Despite achieving agricultural yields ranging from 50 % to 70 % of those obtained with white cotton, colored cotton was priced at five times the cost of white cotton [79-81].

Several breeding projects have been initiated in Brazil, Greece, Israel, Peru, Turkey and the Soviet Union to enhance the processability of colored cotton [82].

Dutt *et al.* conducted a study comparing three varieties of NaCOC cotton - white, brown and green, in terms of fiber quality and performance. The research indicated that colored staple cotton exhibited lower quality compared to white staple cotton. The study also analyzed the impact of cellulose, mineral elements, nitrogen (N), phosphorus (P), potassium (K) and pH levels on fiber quality at various stages of development. It was observed that cellulose content played a significant role in determining fiber quality, with white staple cotton showing higher quality due to its elevated cellulose content in comparison to NaCOC cotton. Changes in pH levels in white and colored cotton could potentially influence fiber elongation rates. Additionally, potassium levels were found to have a positive correlation with fiber quality characteristics among the mineral elements studied. The study also noted similar patterns of pigment development in brown and green cotton, with green fiber cotton taking longer to achieve a deeper color compared to brown fiber cotton [83].

The traditional physical characteristics of NaCOC fibers have been evaluated using HVI equipment, the AFIS instrument, the Shirley Fineness and Maturity Analyzer (FMT) equipment and an Image Analyzer (IA). Both HVI and AFIS were used to measure the tensile, length and fineness properties of the natural-colored cotton fibers. The fiber circularity and perimeter were measured using IA and the fiber fineness and maturity ratio were measured using FMT. The results showed that the white cotton fibers were longer and stronger than the colored cotton fibers, although there were differences between the different types of brown fibers.

Compared to the brown fibers, the green fibers showed lower micronaire, lower surface friction, less round cross-section, higher wax content and lower tenacity and specific work of rupture, although they are acceptable for commercialization. White cotton is superior to colored cotton in terms of the typically measured fiber properties. Carried out an FTIR (Fourier Transform Infrared Spectroscopy) study on colored cotton fiber, comparing it with white cotton fiber. This study revealed that white cotton has more intermolecular hydrogen bonds than colored cotton. This explains the greater mechanical resistance of white cotton. However, green-colored cotton has higher intermolecular hydrogen bonding and lower mechanical strength than brown-colored cotton, owing to its higher wax content. White cotton is superior to colored cotton in terms of the typically measured fiber properties [84-86].

1.3.5 Waxes

In higher plants such as cotton, the protective layer for the cell wall is composed of the biopolymer suberin. Usually, suberin-insoluble polymers are dissolved in organic solvents. Early examinations of white cotton wax indicated that it comprises around 25 percent free fatty acids, a higher proportion of esters and approximately 40 percent higher alcohols, specifically long-chain primary alcohols [87].

Based on a hypothetical model, these polymers contained aliphatic and aromatic domains. Another detailed study on cotton wax showed that the main compositions are 1-triacontanol, montanol, beta-sitosterol and some high molecular weight esters [66].

Table 5 shows the wax content of brown and green cotton compared with that of white cotton. After 6 hours and 24 hours of ethanol extraction, the white and brown cotton samples showed little difference. The wax percentage was very high for green cotton even after 24 hours of ethanol extraction.

Table 5. Percentage of wax in the cotton fiber samples.

Samples	**Percentage of wax (by weight)**	
	After 6 hours of ethanol extraction	After 24 hours of ethanol extraction
white cotton	0.76	1.24
brown cotton	0.60	0.82
green cotton	4.66	5.28

Ryser isolated and characterized the caffeoyl fatty acid glycerol ester of wax associated with suberin from green cotton fiber. All constituents of caffeoyl ester are related to the antioxidant and radical-scavenging properties of caffeic acid [87].

Some studies have been conducted on the wax content of green-colored cotton fibers. The major aliphatic monomers of the green cotton suberin have been observed to be 70 percent ω-hydroxydocosanoic acids and 30 % docosanedioic acids. Green cotton wax is composed of long-chain (>C16) 1-alkanols and alkanoic acids and exhibits phenolic and polymeric characteristics [61, 88-89]. Caffeic acid and glycerol were also found to be components of the suberin layers in green cotton fibers [90].

It was also observed that caffeic acid is a better antioxidant and has higher radical scavenging activity than cinnamic, p-coumaric and ferulic acids, which are known to scavenge free radicals [91].

White cotton and NaCOC brown cotton have very similar wax content. On the other hand, green cotton had a much higher wax content. The higher wax content of green cotton can markedly reduce the moisture absorption capacity of the fiber [17]. To prove this, Gu et al treated green cotton with warm water and NaOH solution to reduce the hydrophobic content and increase moisture absorption. After caustic solution treatment, the green-colored cotton showed increased moisture absorption and a reduced crystalline region [51].

Pan et al. studied the effects of wax content on colored organic cotton. The results of this investigation showed that the darker the brown fiber, the higher the fiber wax content. However, the behavior of green cotton was the opposite. Among forty-eight varieties of colored cotton, the average wax content was 2.81 %. The wax content of green cotton was the highest at five–eight times that of white cotton. The next up was cotton-brown, with twice the wax content of white and that of white cotton was the lowest. Thus, it is obvious that the low stability of the green fiber, especially the light-green ones, may be related to the high wax content of the fiber. This study also showed that wax content had a positive effect on fiber elongation and a significantly negative correlation with cellulose content and other fiber properties. However, the cellulose content was significantly positively correlated with lint percentage, boll weight, fiber length, fiber strength, fiber fineness and fiber uniformity. These results imply that the quality of the fiber was mainly determined by the accumulation of cellulose and that the wax content also affected the properties of the colored cotton fiber. Therefore, it is important to select low-wax-colored fiber lines to improve the color stability of the fiber and colored cotton textiles [92].

1.3.6 Heavy metal content

Parmar and Chakraborty et al. stated that the total heavy metal content in NaCOC cotton fibers is higher than that in white cotton fibers. shows that the heavy metal compositions of the three types of cotton fibers are different. These cotton fiber samples were obtained directly from a single cotton farm in India Table 6 [93].

Table 6. Heavy metal analysis of cotton grown in India [93].

	white cotton	White cotton	Brown cotton	Brown cotton	Green cotton	Green cotton
Heavy metals	mg/kg of fibers	No. of mol/kg fibers x10^3	mg/kg of fibers	No. of mol/kg fiber x 10^3	mg/kg of fiber	No. of mol/kg fiber x 10^3
Cr	1.34	2.56	2.34	4.50	2.65	5.07
Co	3.95	6.70	2.70	4.58	4.68	7.92
Cu	4.19	6.59	4.25	6.68	3.05	4.80
Pb	10.10	4.86	7.90	3.80	7.44	3.60
Ni	10.67	18.17	12.07	20.55	11.18	18.17
Zn	3.78	5.78	7.83	11.96	4.42	6.76
Total		44.66		52.07		47.15

Other studies have reported the heavy metal content of cotton samples from farms in China [94]. The results in Table 7 showed very different heavy metal components and rates in the three cotton compared with those grown in China.

Table 7. Heavy metals content in cotton from China

Chemical components	White cotton (µg/kg)	Brown cotton (µg/kg)	Green cotton (µg/kg)
Mn	1863.8	1364.3	1439.1
W	393.0	1180.1	1275.2
Zn	-	1052.5	399.1
Rb	2422.7	2015.5	1259.0
Sr	1623.6	2221.9	1239.8
Na	2396.0	1864.5	1871.0
Th	162.7	48.2	203.6
Pb	1202.1	596.2	101.3
Fe	203.8	747.8	249.4
Al	883.4	1221.5	860.8
Mg	309.5	567.3	429.7

1.3.7 Behavior against UV radiation

Hustvedt et al. studied the behavior of green, tan and brown NaCOC cotton when exposed to ultraviolet radiation (UPF) and the effect of light exposure and washing.

NaCOC cotton was exposed to xenon light and subjected to accelerated bleaching. Ultraviolet transmission values were measured and the UPF values were calculated after light exposure and bleaching. NaCOC cotton exhibited significantly higher UPF values than conventional cotton (bleached or unbleached).

Although the NaCOC cotton exhibited some fading following exposure to xenon light and repeated laundering, the UPF values for all three shades remained high enough to provide adequate sun protection even after the equivalent of five household washes [68].

1.3.8 Behavior in fire

Van Zandt (1994) investigated the fire resistance of NaCOC cotton, concluding that FoxFibre® green and Coyote brown exhibited favorable fire resistance properties, making them suitable for use in fabrics intended for upholstering motor vehicles and airplanes. The NaCOC cotton fabrics tested demonstrated higher limiting oxygen index values compared to white Pima cotton, with red cotton displaying the highest oxygen index value among all fabrics tested [44].

In 2001, Parmar and Chakraborty grew camel brown and olive-green cotton and studied their physical properties. The ash content of these cotton samples was higher than that of white J-34 cotton because of the presence of more heavy metals. The oxygen limit of NaCOC cotton is also higher than that of white cotton, which makes its flammability low. DSC was used to determine its thermal behavior, clearly showing that the decomposition of white cotton into hemicellulose and cellulose and the volatilization of the degradation product completed at approximately 370 ºC, while for brown and green cotton, devolatilization was completed at approximately 390 ºC [95].

1.3.9 Bactericidal properties

Recently, the antibacterial activity of NaCOC compared to conventional white cotton and brown cotton was found to exhibit excellent antibacterial activity with a reduction rate of 89.1 % and 96.7 %, respectively, when in contact with two species of bacteria, Staphylococcus aureus and Klebsiella pneumoniae, while the antibacterial effect of green cotton was weak. To investigate the antibacterial mechanism of NaCOCs, pigments were extracted from the fibers for further determination of their antibacterial activity using the disk diffusion method and the chemical nature of the extracted pigments was analyzed. The results were as follows: the brown cotton fiber pigment showed significant inhibition against the two challenge bacteria, while the resistance capacity of the green cotton fiber pigment was negligible, indicating that the antibacterial efficacy of NaCOC fibers is closely related to the chemical nature of the pigment. The results indicated that the brown cotton pigment contained condensed tannins and the green cotton fiber pigment contained flavonoids. The effect of high-temperature treatment on the antibacterial activity of the pigments extracted from brown cotton was also examined. It was shown that the general conditions of high-temperature textile treatments decreased the antibacterial activity, but the antibacterial activity still performed satisfactorily [96].

Subsequently Ma, M, et al. Once again studied brown cotton fibers, concluding that the antioxidant capacity of this color is significantly greater than that of white cotton. A brown variety with a darker color has a much higher capacity than 1,1-diphenyl-2-picrylhydrazyl (DPPH) and 2,2′-azinobis(3-ethylbenzothiazoline-6-sulfonic) acid (ABTS) They eliminate free radicals. The activities are 6.9 and 6.1 times higher, respectively than those of white cotton. The antioxidant properties of natural, brown-colored cotton were long-lasting after scrubbing and washing. The phenolic pigment composition of the fibers was found to contribute to their antioxidant properties. The results presented have improved our understanding of the polyfunctional properties of natural brown cotton fibers and their pigmentary components [97].

1.3.10 Environmental and benefits

Approximately 25 % of the world's insecticide use and over 10 % of the world's pesticide use are attributed to cotton crops. In 2003, an estimated 55 million pounds of pesticides were applied to 12.8 million acres of cotton.

Gülümser et al, compared all the steps and costs of the dyeing process of a brown NaCOC cotton knit fabric with a white one. The calculations were performed by considering all fabric treatment steps in the production process. NaCOC cotton was evaluated during the laundering and softening steps. Dyeing of the white cotton fabric was carried out with the same NaCOC cotton color in the laboratory and the bleaching, dyeing, washing and softening processes were evaluated. Calculations and comparisons were made taking into account the costs of energy, water, labor, electricity, chemicals, auxiliary agents and dyes. It was concluded that the process followed for NaCOC cotton is 2.9 times less expensive than the dyeing process for white cotton [98].

1.3.11 Dermatitis

Clothing made with NaCOC cotton have been proven effective in preventing skin diseases such as atopic dermatitis [99].

Quite a few cases of textile dermatitis are known in workers in the textile industry, workers in clothing stores and consumers who wear clothing dyed with dispersed dyes [38, 100-101].

2. Methodology

The main purpose of this study was to examine the resistance of naturally colored cotton (Coyote) to color changes when subjected to household laundry. Four samples were selected for the study. Table 8 details the various stages of the thesis that will be elaborated upon in this chapter. Figure 30 presents images corresponding to each stage of the process.

Table 8. General steps of the work carried out.

<table>
<tr><th>Tasks</th><th colspan="3">Activities</th></tr>
<tr><td>1</td><td>Characterization properties of raw material.</td><td colspan="2">Physical and mechanical tests on raw material</td></tr>
<tr><td>2</td><td>Produce and characterize yarn from naturally colored cotton</td><td colspan="2">Physical test on yarn</td></tr>
<tr><td rowspan="8">3</td><td rowspan="4">Produce, characterize and wash knitted fabrics.</td><td colspan="2">Physical test on knitted fabrics.</td></tr>
<tr><td rowspan="3">Washing process on knitted fabrics:</td><td>Dimensional</td></tr>
<tr><td>Colorimetric test, UPF test</td></tr>
<tr><td>study of the number of washings on color changes.</td></tr>
<tr><td rowspan="4">Produce, characterize and wash woven fabrics.</td><td colspan="2">Physical test on woven fabrics.</td></tr>
<tr><td rowspan="3">Washing process on woven fabrics:</td><td>Dimensional</td></tr>
<tr><td>Colorimetric test, UPF test</td></tr>
<tr><td>Study number of wash, type of water, type of detergent and water temperature on color changes</td></tr>
</table>

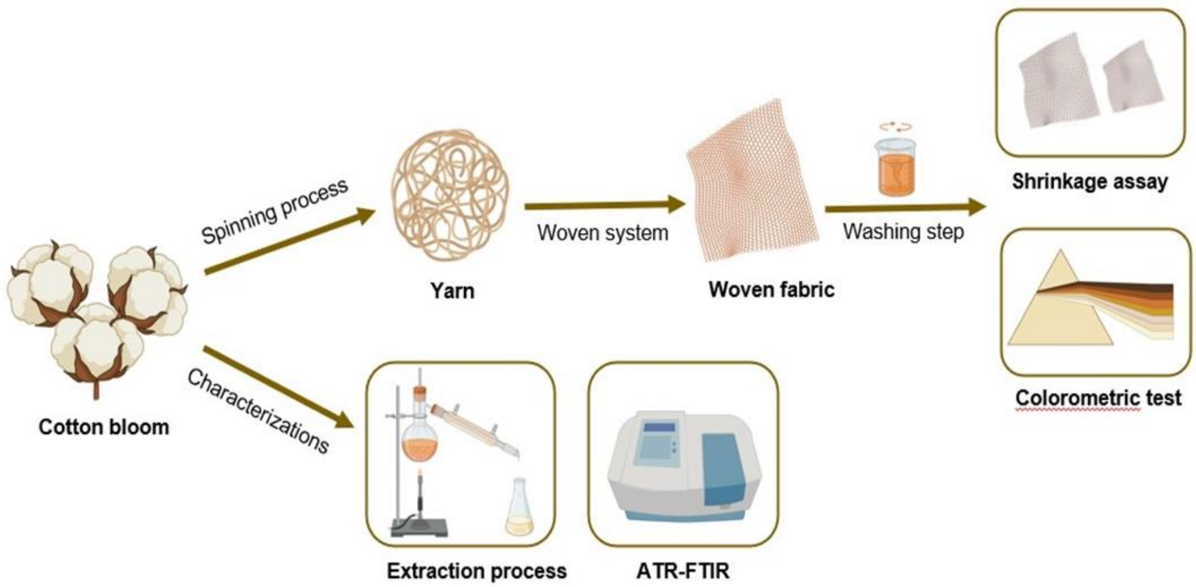


Figure 30. Graphical abstract of the work carried out.

2.1 Material

Cotton

This study employed naturally colored organic cotton provided by the Organic Cotton Colors Company (hereafter referred to as OCC) cultivated in Brazil [102]. This study included four varieties of naturally colored cotton cultivated in Brazil: Topaz, Light Ruby, Dark Ruby and White Raw. Each sample was grown under identical climate conditions. This is crucial because climatic conditions and cultivation practices are significant determinants of cotton quality.

2.2 Production and treatments

2.2.1 Production of yarn (spinning)

The raw material (2.1) was mixed with OCC in equal proportions to obtain the sliver used to prepare the yarn via continuous ring spinning.

The spinning process followed has been that of combed cotton, that is, opening, cleaning, flat carding (carding machine), combing, the first step of the draw frame (drawing frame), the second step of the draw frame (drawing frame) and Roving frame. The spinning company produced all of this "Hilaturas Gonfaus," located in the town of Barcelona of Puigreig. The company gave us roving coils and spinning using a ring spin machine in the spinning workshop of the Higher School of Industrial, Aerospace and Audiovisual Engineering of Terrassa of the Polytechnic University of Catalonia. As indicated below, the yarns were spun using a PINTER S.A ring-spinning machine. The spinning conditions are presented in Table 8.

Table 8. Spinning conditions.

Type of spinning machine	PINTER mode spa
Sliver count	600 N tex
Draft	20.33 (g/m)
Linear spinning speed	16.65 m/min
Revolutions per minute of the spindle	7500 rpm
Hoop diameter	42 mm
Type of traveller	BRÄCKER 2DR

2.2.2 Production of knitted fabric

To evaluate the possible color changes of the fabrics with domestic washing, a knitted fabric was obtained to serve as a substrate for analyzing these behaviors. Knitted fabric was initially chosen because it is much easier, faster, and cheaper to produce than woven fabric, with the hope that it would yield significant results.

Textile samples were produced using a lab-knitting machine and naturally colored cotton yarn. A yarn with a count of 29.5 tex was chosen for its ideal combination of fineness and strength, which is suitable for creating high-quality knitted fabrics.

The natural tons of cotton yarn not only enrich the visual appeal of textiles but also support sustainability efforts by reducing reliance on synthetic dyes. This decision reflects our dedication to eco-friendly manufacturing.

For this project, the knitting machine was configured with a gauge of 18 needles per inch to accommodate the thickness of the 29.5 tex yarn, ensuring tight and consistent stitches. Operating at a controlled speed of 0.8 meters per minute, the machine guarantees precision in the knit pattern while minimizing the yarn stress and potential breakage.

We programmed the machine to produce a plain knit structure, highlighting the natural texture and color variations of cotton. This setup allowed the inherent qualities of the yarn to shine through in the final fabric.

Careful adjustments in tension settings are meticulously fine-tuned to prevent any distortion in the fabric and to maintain uniformity and dimensional stability throughout the knitting process. Following knitting, the fabric underwent a series of finishing treatments, such as steaming and light pressing, to enhance its softness and dryness. The configurations of the knitted fabrics are listed in Table 9.

Table 9. Configuration of the knitting machine.

Knitting machine	CELEX (1.6.24)
Diameter of machine	14 cm
Number of needles	214
Gage	12
Speed	Flexible
Fabric width	14.5 cm

2.2.3 Production of the woven fabric

As will be seen in the results section, the behaviours of the knitted fabric - due to its high yarn mobility–were not suitable for analysing the washing behaviors. We proceeded to obtain a fabric in which the warp, as shown later, is made up of conventional cotton yarn and the weft with NaCOC cotton yarn obtained in our weaving workshop. In this study, woven fabrics were prepared and their properties were examined before and after washing. The specifications of the fabric produced using the weaving system are listed in Table 10.

Table 10. Weaving parameters.

Weave machine system	Air-jet Dornier loom
Jacquard machine	Staübli
Loom width	150 cm
Ligament	Five satin
Frame insertion speed	4000 rpm
Fabric density	Warp: 38.4 yarn/cm 2/30 Nm Weft: 19 pics/cm 2/34Nm
Fabric width	146 cm

Conventional cotton yarn was used for warp and colored cotton yarn was produced for the weft. Five satin patterns were used for weaving, as these patterns allowed better visualization of the surfaces with colored cotton yarns during the measurement of elements and treatments before and after the washing process (Figure 31).

Figure 31. Weaving device (top) and woven fabric (bottom).

2.2.4 Domestic washing

Samples of the naturally colored fabrics were washed in the INTEXTER laboratory. The type of washing machine was FAGOR, model INNOVATION, with a dry capacity of 8 kg and 16 automatic program washings (Figure 32).

Figure 32. FARGO washing machine.

The conditions for washing in this study depend on the development of the work and several parameters can be changed as follows:

1- Type of water used for washing

2- Washing temperature

3- Type of washing detergent

1. **Type of water**
    - Three different types of water were used in different studies in this thesis: conventional plumbing water in the city of Terrasa (in the study of washing on circular fabrics, we are aware that the degree of hardness of water is very high at 400 ppm).
    - Water was added to a mixture of 50/50 tap water/distilled water.
    - Distilled water.
2. **Washing temperature**

To wash and compare the effects, three different types of temperatures were considered.

- 20 ºC or cold water
- 40 ºC
- 60 °C

3. Type of washing detergent

The three types of detergents are shown in Table 11.

Table 11. Types of detergents used.

Reference	Name of detergent	Country of product
Det A	Fox Fibre® Colorganic®	Spain
Det B	KLAR ECO SENSITIVE	Germany
Det C	PURE NATURE	Germany

The **FOX FIBER** detergent brand appeared to prioritize the performance of washing clothes. The presence of "Fox Fiber" implies that it may contain specific additives or technologies aimed at preserving the quality and color of the fabric. This detergent is formulated to effectively remove stains and maintain fabric integrity, making it suitable for various clothing types.

KLAR ECO SENSITIVE is an eco-friendly and hypoallergenic detergent ideal for individuals with sensitive skin. Products under this label are typically free from fragrances, dyes and preservatives, thus reducing the risk of skin irritation and allergic reactions. Consumers seeking environmentally sustainable cleaning options that are gentle to both the environment and their skin would prefer this detergent.

Pure Nature brands emphasize the use of natural ingredients. Detergents under this name are likely to be made of biodegradable components and possibly plant-based cleaners. They are often marketed to consumers who prioritize minimizing ecological impacts and prefer products without harsh chemicals. Pure Nature detergents are suitable for all types of laundry, especially for those with health concerns or environmental awareness.

The color properties of the fabrics were assessed before and after washing. Numbered fabric pieces were detached and weighed for each wash, with white cotton used for internal weight balance adjustments. Despite the immovable washing machine, the washing procedures were repeated as often as possible. The fabrics were shielded from sunlight after washing to prevent color alterations. Cotton from the same region and harvest season in Brazil was used to ensure uniformity in yarn and textile production. The INTEXTER laboratory employed conventional washing machines for cleaning, preceded by a novel natural wax extraction technique from cotton. Home-based washing methods were chosen to create fabrics appropriate for the apparel industry. Washing frequency varied based on fabric type and detergent, with a fully automated washing machine program (Nos. 1, 4 and 5) used for the textiles. Details of the washing process are presented inTable 12.

Table 12. The washing process conditions is detailed.

Washing machine	FAGOR, F2810
Washing temperature (ºC)	20, 40 or 60
Washing time (min)	Depends on the temperature: 50, 57 and 80 min, respectively
Spin speed (rpm)	1000
Load (kg)	8
Soap dose (ml)	15 ml for Det A 30 ml for Det B 30 ml for Det C
Water hardness (ppm)	0, 200, or 400, depending on wash conditions

Each sample was washed under varying conditions and subsequently, the fabric was dried using a ROMEER model 92 drying machine, employing different automatic dryer programs as illustrated in the study (Figure 33). The drying conditions were approximately 100-120 min using the chosen program.

Figure 33. ROMEER dryer machine.

2.3 Characterization

2.3.1 Physical properties of raw material

The physical properties of cotton were determined using high-volume instrument (HVI) equipment at Spain's National Cotton Center located in Barcelona, as presented in Table 13.

Table 13. Description of HVI parameters.

Parameters	Signification
SCI	The Spinning Consistency Index is a calculation for predicting the spinnability of the fibers. a multiple regression equation can provide valuable information to anticipate the strength and spinning potential. the regression equation uses most of the individual HVI measurement results to calculate the SCI. This index can be used to simplify the category system used in the cotton warehouse. In general, the higher the index. the higher the yam strength and the better the overall fiber spinnability.
MIC	Micronaire A fiber sample of constant weight is measured by passing air through the fibers and measuring the pressure drop. The micronair scale has been established empirically with a standard set of cotton and is not linear. Other factors such as fineness and maturity influence micronaire results
MAT	The maturity index is a relative value that is calculated using a sophisticated algorithm including other HVI measurements. such as microlayer. strength and elongation. This indicates the degree of cell wall thickness in the cotton sample. The HVI Maturity Index correlates very well with the AFIS Maturity Ratio and the reference method of microscopy (cross-sectional analysis).
UHML	The by-weight measurement of the Upper Half Mean Length is calculated from the fibro gram. A fiber beard of randomly clumped fibers is scanned optically across its length and the fibro gram is derived from it. The Upper Half Mean Length corresponds to the classer's staple length as well as to the AFIS Upper Quartile Length by weight. Please note that a long-range is assigned in inches for each length staple or code. The ranges calculated in millimeters do not line up exactly due to the conversion calculation. However, inches or 32nds are used for staple length determination in the international cotton trade and are therefore binding. UHML is the mean length by the number of fibers in the largest half by the weight of fibers in a cotton sample, usually measured from the fibro gram

Table 14. Description of HVI parameters.

Parameters	Signification
UI %	The Uniformity Index expresses the ratio of the Mean Length to the Upper Half Mean Length. It is an indication of the distribution of fiber length within the fibrogram
SF %	The Short Fiber Index is a value that is calculated using a sophisticated algorithm. The fibro gram is mathematically converted into a length distribution curve. The SFI is an indication of the number of fibers in percent that are less than 0.5 inches (12.7 mm) in length. It correlates very well with the AFIS Short Fiber Content by weight (SFC.).
STR	The bundle strength is the breaking strength of the cotton fibers in grams per tex. The fineness is calculated from the microwave value. The fiberboard is broken at a continuous deformation rate (CRE= Constant Rate of Extension) and with a 1/8-inch distance between the clamps.
ELG	Elongation is a measurement of the elastic behavior of the fibers in the bundle. The fibers are clamped in the bundle with a 1/8-inch distance between the clamps. The first pair of clamps is stationary and the back pair of clamps is pulled away at a constant rate. The distance the fibers extend before they break is recorded and expressed as percent elongation. For example, if you were to measure 50 % elongation, the fibers would have extended 1/16th of an inch before breaking. Below is a table describing ranges of actual elongation values in cotton fibers
RD	This value expresses the Whiteness of the light that is reflected by the cotton fibers. It corresponds to the reflectance (Rd) represented in the Nickerson/Hunter color chart. It is used in conjunction with the yellowness (+b) to determine the color grade of the cotton.
+B	This value expresses the yellowness of the light that is reflected by the cotton fibers. The yellowness (+b) of the sample is determined by using a yellow filter. It corresponds to the +b value represented in the Nickerson/Hunter color chart. The yellowness is used in conjunction with the reflectance (Rd)

2.3.2 Physical properties of yarn

The regulations and equipment for the characterization of the yarn obtained are listed in Table 15.

Table 15. Characterization of yarn (instrument and standards)

Parameter	Device	Standard
Count	Aspe and balance	UNE-EN ISO 2060:1996
Twist	Twistmatic MESDAN Torsiometer	UNE-EN ISO2061
Strength	Textechno Statimat ME Dynamometer	UNE-EN ISO 2062:2010
Tenacity	Textechno Statimat ME Dynamometer	UNE-EN ISO 2062
Elongation	Textechno Statimat ME Dinamometer	UNE-EN ISO 2062
Unevenness	Keisokki Regularimeter	UNE 40-225-74 Part 1
Hairiness	Zweigle G 5675 Hairinessmeter	Own method MO 500-39

The methodology of the tests for characterizing these yarns is described below.

Count

The yarn count determines the thickness or thinness of the yarn by comparing its length with its weight. Yarn count plays a crucial role in selecting the appropriate yarn thickness for different types of fabrics, ultimately affecting the texture and weight of the fabric.

The yarn count, indicating the thickness or fineness of the yarn, was measured using systems such as Metric Count (Nm), Denier and English Cotton Count (Ne). The count was determined using the international standard UNE-EN ISO 2060:1996, which specifies the methods for determining yarn linear density [103].

Twist

Twist measures the number of twists per meter of yarn (twists per meter or T/m), indicating the level of twisting in the yarn. A high torque enhances the strength of the yarn and reduces its stretchability, whereas a low torque makes the yarn softer and more flexible.

To obtain accurate results, the twist was measured using an automatic device. For this purpose, a TWISTMATIC-CFT 500-38 MESDANLAB, manufactured by INTEXTER LAB under the UNE-EN ISO2061 standards, was used (Figure 34), [104].

Figure 34. Twist the automatic counter device.

Strength

The primary evaluation of yarn strength involves conducting tensile strength tests using devices such as a Constant Rate of Extension (CRE) Tester and Universal Testing Machines (UTMs). The assessment is based on key standards, such as ASTM D2256, which provides guidelines for determining the breaking force and elongation of textile yarns using the single-strand method. Furthermore, ISO 2062 specifies methods for measuring the breaking force and elongation at the break of different yarn types. These standards play a crucial role in ensuring that yarns meet the quality and performance criteria necessary for their intended applications.

Yarn strength tests were performed using an automatic tensile test with a TEXTECHNO machine under standard laboratory conditions. This test was carried out on five bobbins (UNE-EN ISO 2062) of naturally colored cotton, as in the other tests (Figure 35) [105].

Figure 35. TEXTECHNO machine.

Tenacity

Tenacity measures the tensile strength supported by each unit of yarn thickness and is obtained by dividing the maximum breaking strength by the yarn count, given in cN/tex. Yarn tenacity, a measure of the maximum stress a yarn can endure before breaking, is determined using tensile testing machines and is expressed in units such as Newtons per tex. The yarn is stretched until it reaches its breaking point and the tenacity is calculated by dividing the breaking force by the linear density. Esteemed standards, such as UNE-EN ISO 2062:2010, are utilized to establish procedures for evaluating the tensile properties and breaking forces of yarns. This evaluation plays a vital role in guaranteeing the strength and longevity of yarns for various textile applications [106].

Elongation at Break

This test measures the extent to which a yarn can stretch before it breaks and is expressed as a percentage. The ductility of the yarn was evaluated by measuring its elongation at break, indicating the percentage of stretch it can undergo before breaking. To measure this, the yarn was subjected to tension in a testing machine until it ruptured and the lengths before and at the point of break were recorded. The increase in length was then calculated to determine the elongation at break. These measurements follow established standards, such as UNE-EN ISO 2062:2010, which outline procedures for assessing tensile properties, including elongation. This property is crucial for selecting materials suitable for applications requiring flexibility and energy absorption [106].

Evenness Test

The mass regularity of the yarn obtained has been analyzed using the Keisokki regularimeter (Figure 36). The unevenness test, a fundamental test for comparing and studying fiber properties, was conducted at the INTEXTER laboratory under standard laboratory conditions on both wicks and yarns [107].

Figure 36. Inverness device.

Variations in the thickness or weight of the yarns and fabrics were measured using the unevenness test, which plays a crucial role in evaluating the quality of textiles. During the test process, samples were passed through a measurement device, where data were recorded and analyzed to determine unevenness, typically expressed as a coefficient of variation. The standards governing the use of capacitive testing instruments for assessing yarn and fabric unevenness include UNE 40-225-74 Part 1 standards. This test is indispensable for optimizing production and ensuring the quality of the final textile products [108].

Hairiness

Hairiness was tested for yarn in the INTEXTER laboratory using the ZWEIGLE G565 testing device, which examined approximately 400 m of yarn according to the standard UTF500-039 (internal method), as shown in Figure 37.

This equipment determines the number of fibers that protrude from the yarn structure based on its length. For example, the results obtained in category N1 indicate the number of fibers that protrude 1 mm from the yarn, N2 means the number of fibers that protrude 2 mm from the yarn and so on. The S3 value indicates the sum of values obtained between N3 and N12.

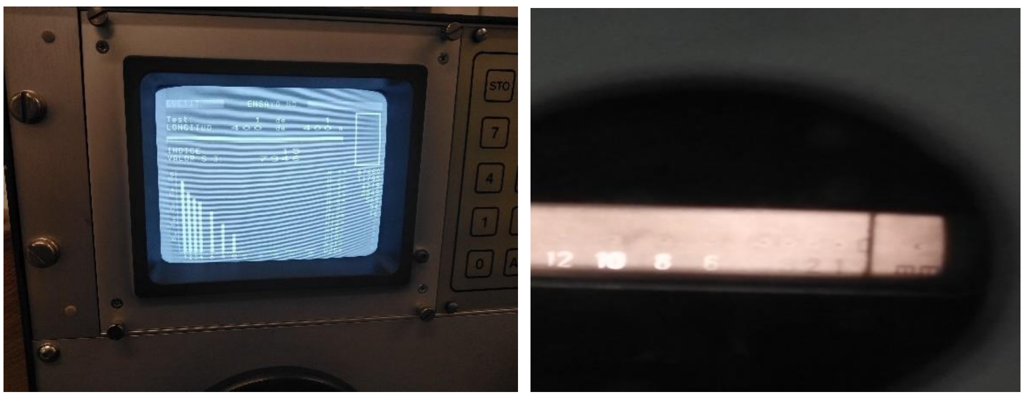


Figure 37. Hairiness automatic test device.

2.3.3 Physical properties of fabrics

For the characterization of the fabrics obtained, the following tests have been conducted:

a) Dimensional stability

b) Weight per square meter

c) FAST Test. This is a set of tests to determine the tolerability of wool and blended fabrics. It is based on analyzing the physical-mechanical behaviors of tissues when they are subjected to small tensions. Although it is a device designed to exclusively analyze wool fabrics and blends, in this thesis, it has been used to evaluate the physical-mechanical properties of the NaCOC shed fabrics obtained because it is a rapid test and allows obtaining information of interest.

d) Tensile strength and elongation in the axial direction by the strip method,

e) Drape test

f) Colorimetry

g) UPF The ultraviolet protection factor

The test results are presented below.

Dimensional stability

Specificity for weaving

To measure the dimensionality and weight of the woven fabrics, 30 cm × 30 cm samples were prepared to facilitate the work and prevent the edges of the fabric from being twisted and the warp and weft from being separated after the cutting operation in the INTEXTER workshop. The samples were embroidered with an INTERLOCK round stitch using a sewing machine.

To measure dimensional stability, eight points on the surface of the fabric were considered, 5 cm from the edges, as shown in Figure 38.

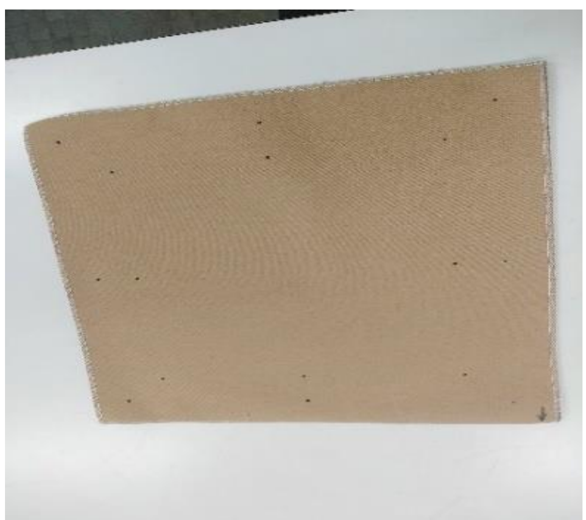


Figure 38. Dimensional stability of fabrics.

To measure the dimensional stability, three measurements were obtained in the vertical direction. The warp direction and three measurements in the horizontal and weft directions were compared before and after washing.

Specificity for knitting

The dimensional stability of the knitted fabrics was determined by counting the number of rows and columns (representing vertical and horizontal rows). The number of rows in different parts of the fabric was selected and repeated four times for each piece (Figure 39).

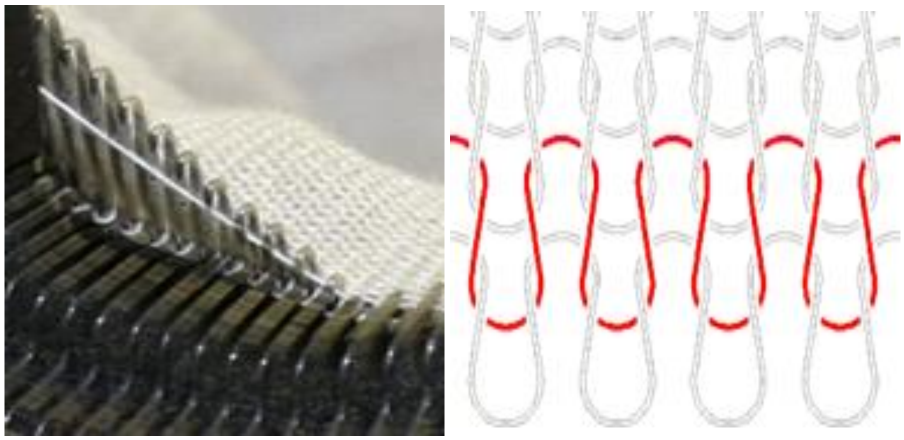


Figure 39. Knitting process and schematic of the loops.

It should be borne in mind that this test will be performed for both unwashed and washed fabrics and will ultimately be compared.

There were eight LOOPS 8 in the horizontal direction and nine in the vertical direction on a scale of 1 cm × 1 cm (Figure 40).

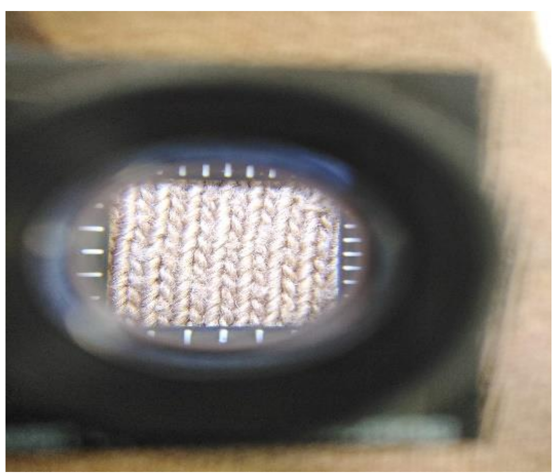


Figure 40. Loop counting.

This changed after washing the number of loops horizontally to eight and nine loops vertically to 1 cm × 1 cm.

Weight per square meter

Certainly, any fabric undergoes weight change after washing. To determine the extent of these changes, we calculated the weight of the samples at the previous point (10 cm × 10 cm) and obtained the weight per square meter of the fabric (Figure 41). The procedure for determining the weight of the fabric in grams per square meter (g/m²) is outlined in ISO 3801:1977. A specific area of fabric was cut with a circular cutter and weighed on a precise scale and the weight per square meter was obtained by dividing the weight of the sample by its area. To ensure accuracy, measurements were taken multiple times and the average result was calculated for consistency [109].

Figure 41. Specimen knitted fabric (10 x 10).

To increase the accuracy and meet laboratory standards, this procedure was repeated five times for each selected piece of fabric. It should be noted that these values must be obtained for fabrics both before and after washing. Finally, the results were compared.

Certainly, any fabric undergoes weight change after washing. To determine the extent of these changes, the weight at 10 cm × 10 cm was calculated to obtain the weight per square meter of fabric, as shown (Figure 42).

It is necessary to note that these values must be obtained for the fabrics both before and after the washing process and the results were compared.

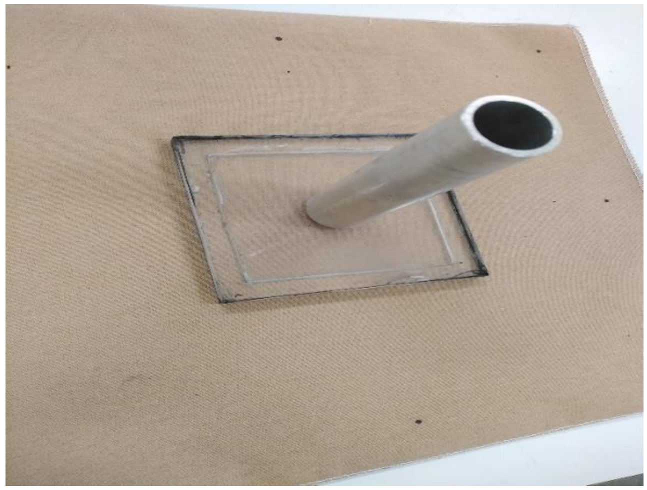


Figure 42. Woven sample (10 x 10).

FAST TEST (Fabric Assurance by Simple Testing)

The FAST test was applied only to the woven fabric. For a long time, researchers have examined the connection between a fabric's dimensional, physical and mechanical features and its aesthetic qualities, such as how it feels to touch, how it performs when used to make clothing and how it looks after washing and wearing. Unfortunately, the industry did not frequently measure fabric quality under suitable conditions until 1980. In recent years, several innovations have rekindled the interest in objectively measuring technology.

Highly sensitive devices have been developed to test the physical, mechanical and surface characteristics of textiles under suitable circumstances. New methods have been created to analyze the data obtained from different instruments in a way that is acceptable to the industry, in addition to the availability of suitable instrumentation.

Finally, there is a need for technology to predict how a fabric will behave in the production of clothing, owing to the ongoing trend toward lightweight fabrics, which poses more challenges to garment makers than fabrics of more traditional weights, as well as the mounting commercial pressures brought on by the increased use of automation and quick response programs.

Fabric Assurance by Simple Testing (FAST) is a set of instruments and test methods developed by the CSIRO Division of Wool Technology (Australia) to measure the properties of fabrics that affect their tailoring performance and the appearance of the garment in wear. The system consists of three simple instruments and a test method and is designed to be used by fabric manufacturers, finishers and garment makers, *FAST-1 Compression Meter*

The FAST-1 system comprises a compression meter that gauge's fabric thickness under two specific stresses. The measurement principle is as follows: By adding weights to the measuring cup, the pressure at which the thickness is measured may be adjusted (Figure 43).

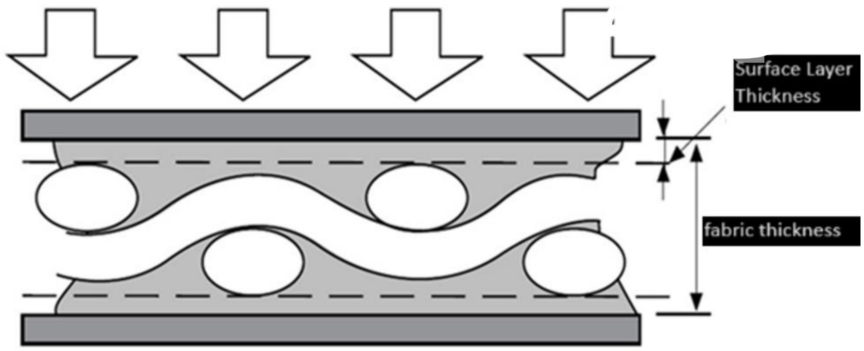


Figure 43. Measuring principle of the FAST-1 compression meter.

FAST-2 Bending Meter

The bending length of the cloth was measured using a FAST-2 bending meter. The bending rigidity of the fabric was determined using a measurement method. The instrument uses the British Standard method (BS:3356(1961)) of the cantilever bending theory. Nevertheless, unlike other test equipment, the edge of the cloth was detected by a photocell in FAST-2 instead of by the eye. FAST-2 is more accurate than the competing instruments because of the removal of this source of operator error, in addition to making the instrument easier to operate. An instrument display was used to read the bending length values immediately. This is an illustration of the measurement concept shown in (Figure 44) [110].

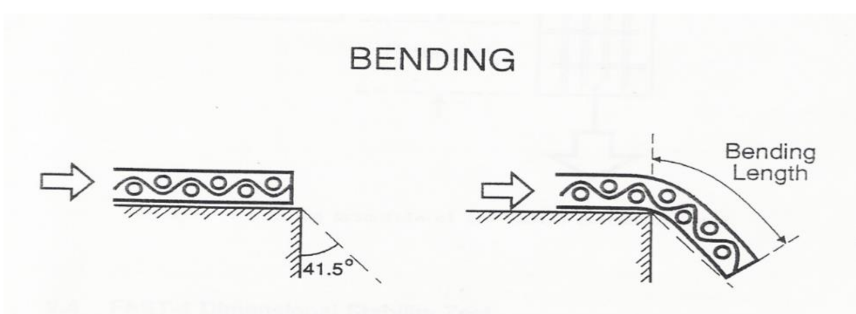


Figure 44. Measuring principle of the FAST-2 bending meter [97].

FAST-3 extension meter

The FAST-3 extension meter uses a straightforward lever principle to evaluate the extensibility of a fabric under three distinct loads, replicating the deformation that the fabric is expected to experience during garment manufacturing. In theory, any angle to the warp (or weft) yarn can be used to measure the fabric's extensibility. Only the warp, weft and bias directions were typically extensible (Figure 45).

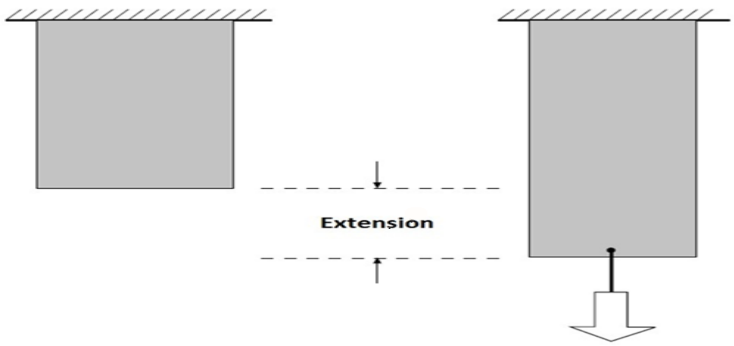


Figure 45. Measuring principle of the FAST-3 extension meter [97].

FAST-4-Dimensional Stability Test

The final component of the FAST is a test method that measures the dimensional stability of a fabric. This method involves measuring fabric dimensions before and after the wet relaxation process. The test is a modification of the conventional wet-dry test, which was first formalized by Shaw. The FAST-4 test can be completed in less than two hours and does not require a conditioned atmosphere.

FAST requires a specific sample size for both the instrumental and dimensional stability tests. In practice, half a meter of fabric at full width is adequate for conducting a full range of tests.

Mechanical behavior

The strip method described in EN ISO 13934-1:1999 provides guidelines for measuring the maximum force (tensile strength) and elongation at the maximum force of textile fabrics. This method involves cutting fabric strips, which are usually 50 mm wide and securing each end in a tensile testing machine. The strips were then subjected to tension until they broke, allowing for determination of the force at which the fabric strip fractured (maximum force) and the elongation of the strip at the point of rupture [111].

The fabric strip technique involves aligning fabric samples with dynamometer jaws to secure yarns and to evaluate the risk of breakage and impact on the fabric structure. This method is not recommended for fabrics with less than 20 yarns per width because it compares the strength of individual yarns to the overall fabric strength. Five warp and weft samples were selected, positioned 3 m from the fabric edge, free of any defects and placed away from the selvage to prevent yarn-sharing. Before conducting the tests, the samples were conditioned to ensure standardized moisture and temperature levels (Figure 46).

Figure 46. Fabric piece [97].

Once the warp and weft directions were determined, the test tubes were labeled on the fabric using permanent ink before being cut. Arrows were used to show the warp direction on each tube, which was then positioned in alignment with either the warp or weft yarns depending on their orientation (Figure 47, Figure 48, Figure 49).

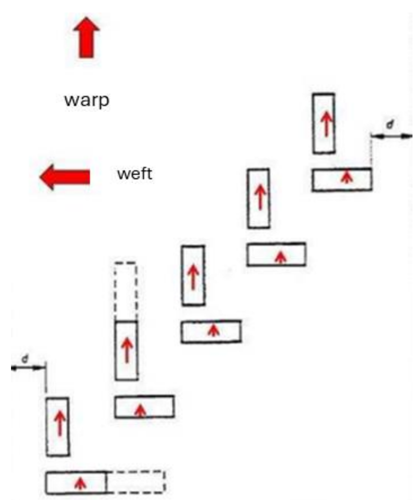


Figure 47. Distribution of warp and weft test pieces in the fabric.

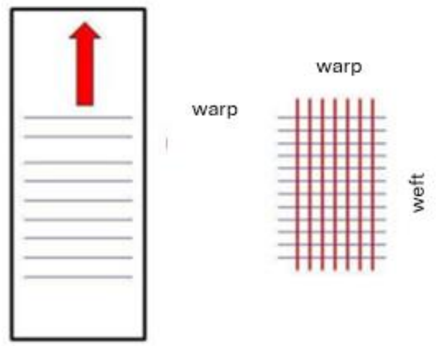


Figure 48. The arrow drawn on the test piece marks the direction of the warp [97].

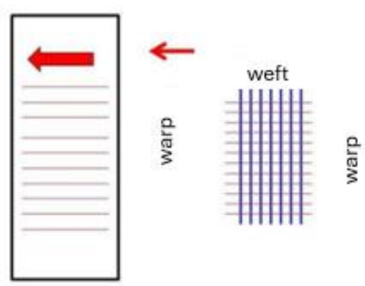


Figure 49. The arrow drawn on each test tube marks the direction of the warp. plot essay [97].

Each test piece will have a width of 50 mm (excluding fringes) and a length that will allow 200 mm of fabric to be tested, plus any additional length needed to fit between the dynamometer's jaws. This is decreased to 100 mm in textiles with an elongation-to-break of more than 75 % of the nominal gauge length (Figure 50 and Figure 51).

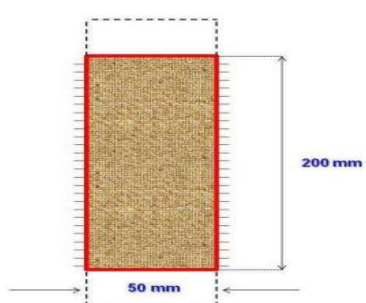


Figure 50.Test tube for strip method test.

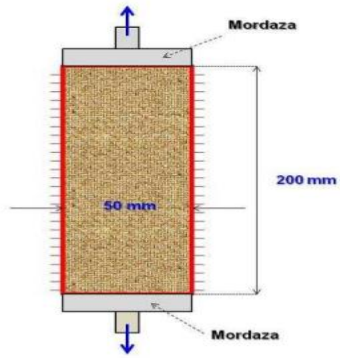


Figure 51. Strip method test. The width of the jaw is equal to that of the specimen [97].

To ensure the integrity of the fabric, the specimens were placed in the jaws of the dynamometer with a pretension equivalent to 0.25 % of the anticipated breaking force. The testing was carried out using a dynamometer programmed to break the fabric within a time frame of 30 ± 5 s, with the jaws lowered at a speed of 200 mm/min. The load (F) represents the tensile force applied to the specimen, which was measured in centinewtons (cN) or grams (gf). The tensile strength or breaking load (R) is the maximum load that a specimen can withstand before fracturing. The elongation (∆φ) measures the deformation of the specimen under axial force and is typically expressed as a percentage. The load-elongation curve demonstrates the deformation of the specimen as the force increases and is the increase in the length of the specimen owing to the application of the breaking load (Figure 52).

Figure 52. Strip method test. Sources: Left Instron. Right: Vidal protection [97].

The load-elongation curve depicts the axial deformation of the specimen as the applied force increases, the tangent line drawn from the origin to the curve represents Young's modulus or the initial modulus, which indicates how the fabric deforms under a small force. This parameter is essential for evaluating the textile characteristics. Tests such as the Shirley Stiffness Test or the Kawabata Evaluation System (KES) are commonly employed to quantify the initial modulus, typically measured in units such as millinewtons per centimeter (MN/cm) or grams of force per centimeter (gf/cm). Fabrics with a high initial modulus usually have a stiff and firm feel, whereas those with a low initial modulus are more delicate. A higher modulus results in fabrics with increased flexibility reduced sagging and fewer wrinkles (Figure 53).

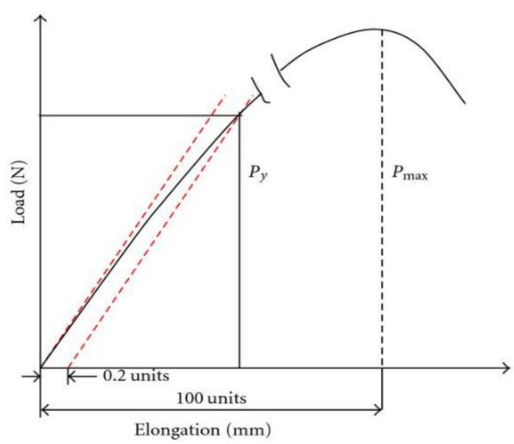


Figure 53. Load-elongation curve [97].

The energy absorbed by the material until it breaks is quantified in joules as the work of rupture (placed beneath the face elongation curve). In (Figure 54, Figure 55 and Figure 56), observed the chart load-elongation and the behavior of the fabric under tension.

Figure 54. Tensile strength test [97].

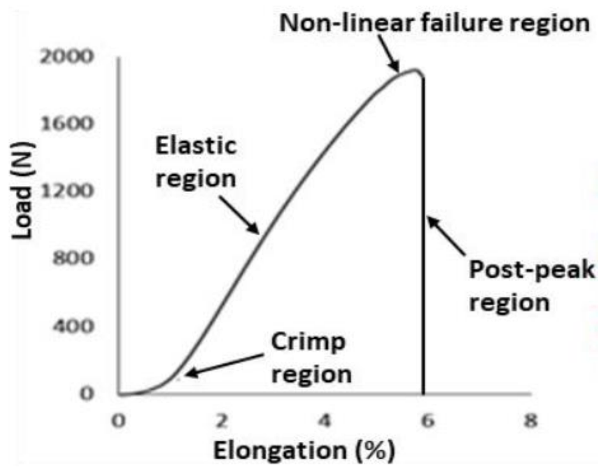


Figure 55. Breaking process of fabric specimens in tensile tests [111].

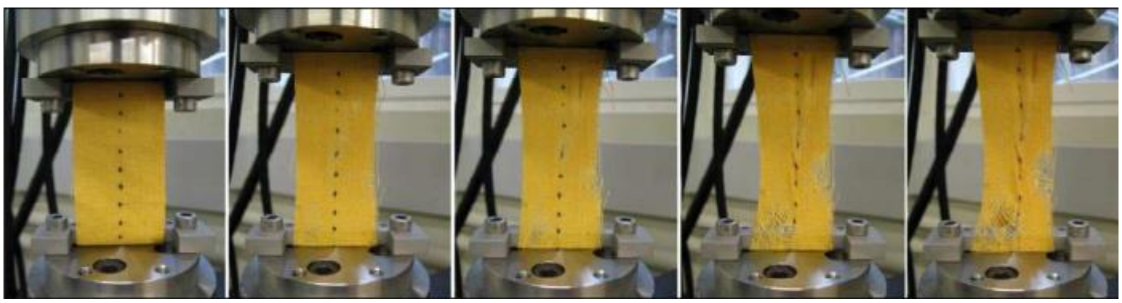


Figure 56. Zone load of elongation curve of fabric specimen [97].

The factors influencing test precision include specimen number, length, jaw feed speed and jaw quality. The fabric characterization compares the results across the specimen sizes. Fabric breaking near the jaws must be rejected. Under cyclic loading, the textile substrates exhibited viscoelastic behavior, with residual elongation observed after the application of the maximum stress (Figure 57).

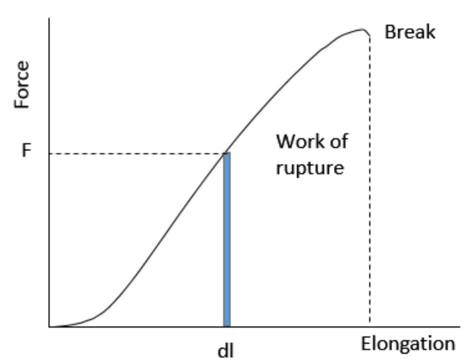


Figure 57. Work of rupture in the force-extension curve [111].

Drape test

A drape test has been conducted to measure the static flow of the fabrics. Its main purpose is to evaluate the drapability, which refers to how a fabric hangs and stretches under its own weight. Consumer perception of fabric drapes has been subjective and can be influenced by the tactile and visual sensations experienced when observing or touching the fabric. Although visually assessed, fabric drape has been subjectively evaluated based on sociocultural and historical trends that have varied across different times and locations.

In this test, a circular fabric sample was positioned horizontally on a metal disk with a small diameter. The fabric does not hang freely owing to gravity and a light bulb situated beneath the center of the sample projects light downwards onto a concave mirror. The mirror then reflects the light upward, creating a shadow of the fabric's drape on a circular paper placed above the fabric (Figure 58, Figure 59, Figure 60,Figure 61).

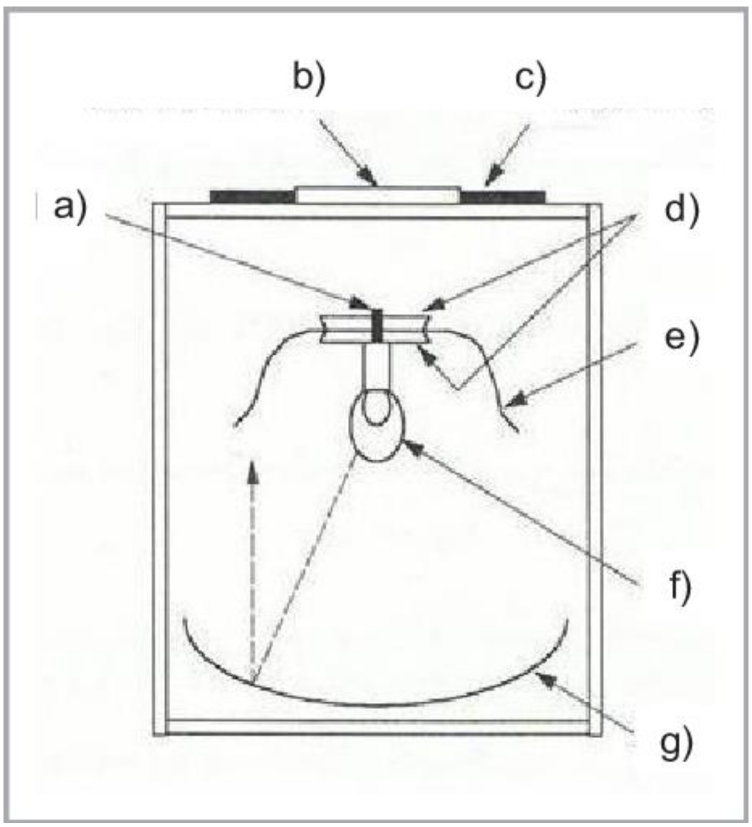


Figure 58. Typical Cusick drape meter: a) specimen fixing needle, b) central disc lid, c) paper ring, d) specimen support discs, e) fabric specimen, f) spotlight and g) parabolic mirror [97].

Figure 59. Top view of the current version of the Cusick diameter marketed by the SDL Atlas. Fountain [97].

The drap rate (DR) is defined as the proportion of the annular ring area covered by the spray projection of the sample, Equation 1.

$$DR=\frac{Area\ of\ the\ shadow\ cast\ by\ the\ sample - Area\ of\ the\ support\ disc\ Area\ of\ the\ specimen}{Area\ of\ the\ specimen - area\ pf\ the\ support\ disk} \qquad \text{Eq. 1}$$

Briefly, a circular piece of paper with a radius R was placed in the middle of the device. In this study, the perimeter of the cast shadow of the fallen fabric was sketched. Following that, the paper is sliced along the previously specified line to get area "A" (Figure 62), W1 was obtained by weighing this area of the paper. The pieces of paper from sections "A" and "C" are then weighed together to give the weight W2. The drape ratio (DR) is the quotient of the two weights, as is expected in Equation 2.

$$DR=\frac{W1}{W2} \qquad \text{Eq. 2}$$

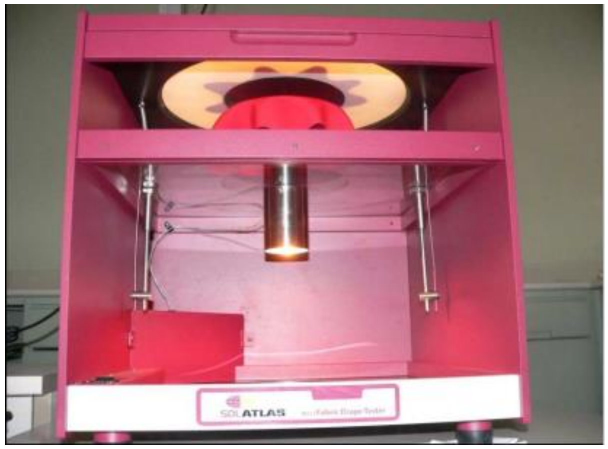


Figure 60. Bottom view of the current version of Cusick diameter [97].

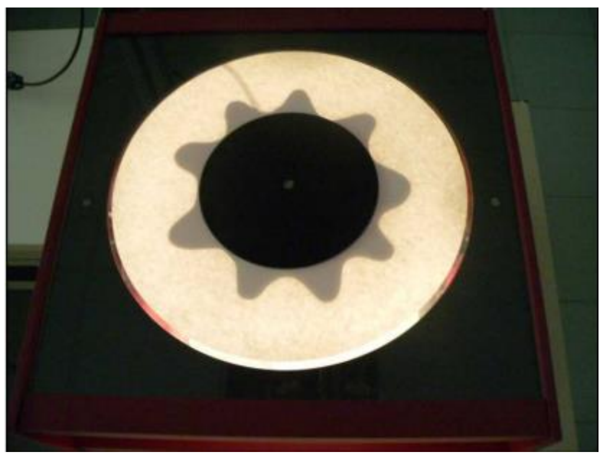


Figure 61. The profile of the cast shadow of the fabric falls on a Cusick drosometer [97].

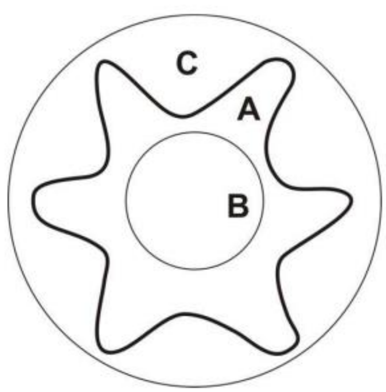


Figure 62. The profile of the cast shadow of the fabric was measured using a Cusick drosometer. Zone "C" corresponds to the area of the shadow cast by fabric [97].

Experiments were conducted on the front and back of each test tube and the mean of two readings from the four test tubes have been employed. The Drape Ratio (DR %), presented as a percentage, offers an impartial evaluation of fabric distortion, although not completely. A low DR % indicates that the fabric is susceptible to distortion, whereas a high DR % indicates that it is easily deformed (Figure 63).

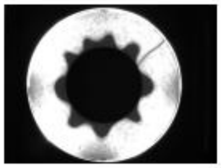
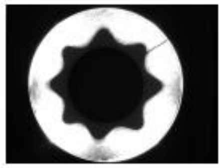
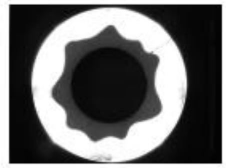
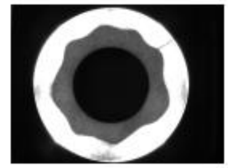
				
DR%	25,072	45,013	55,90	73,334

Figure 63. Aspects of the falling profile of four fabrics with different DR % [97].

However, this indicator is insufficient to explain the complicated three-dimensional phenomena of falling textiles because two textiles with the same DR % might exhibit drastically diverse fall morphologies, as shown in Figure 64. It is essential not to lose sight of the fact that this approach involves analyzing a three-dimensional reality (the falling item) and reducing it to a two-dimensional reality (the shadow of the falling object projected onto a plane).

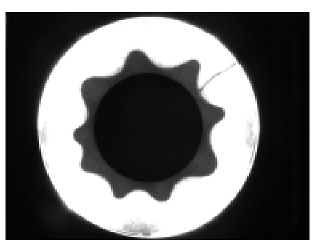
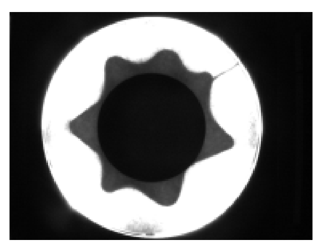
Ref	A	B
Imagen		
DR%	39,518	39,698
FN	9	7

Figure 64. Two fabrics with the same DR % value may exhibit different drape profiles [97].

Cusick found that falling fabric leads to multi-directional deformation, which is influenced by both shear and flexural stiffness. They devised an equation connecting the Drape Ratio (DR %) to the flexural stiffness (measured by cantilever length) and shear stiffness.

Both BS-5058-1973 [112] and ISO 9073-9 [113] employ an 18-inch backing disk cm and circular specimens of 24, 30, or 36 cm, depending on the declining index range.

Normative applicable: UNE 40-383-79 [114], Determination of the cayenne of fabrics- ISO 9073-9 [113] Textiles. Nonwoven Testing Procedures. Part 9: Durability determination, including drape coefficient.

BS-5058-1973 [112] and techniques for Drape Fabric Evaluation DIN-54306 [115], testing textiles for drape fabric determination JIS-R-3418 [116]. All these standards and test related to the drape.

2.3.4 Colorimetry

Colorimetric analysis is important in the textile industry because it guarantees the uniformity and accuracy of colors in fabrics. This entails the implementation of precise standards and procedures to quantitatively assess and define colors.

Any device (such as a colorimeter or spectrophotometer) used to measure the relative amount of radiation reflected from a specimen in the visible region of the spectrum (comprising the wavelengths from 360 nm to 780 nm) and including as a minimum the region from (400 nm to 700 nm).

The International Commission on Illumination (CIE) defines several color space models, including *CIELAB** and CIE*LCh**, which are widely used for fabric color measurements. These models provide a consistent and reliable method for numerically describing colors in a reproducible manner.

Traditional color spaces like the three-dimensional CIE tristimulus values (X,Y,Z), the (x,y,z) space, and the two-dimensional CIE (x,y) chromaticity diagram are not visually uniform. Equal distances in these spaces do not represent equally perceptible differences between color stimuli. To address this, the CIE introduced and recommended the *CIELAB* and CIELUV color spaces in 1976. These spaces use non-linear functions of X, Y, and Z coordinates to create a more perceptually uniform representation. Consequently, numerical values representing the magnitude of color differences can be described using simple Euclidean distances or more sophisticated formulas that better correlate with perceived differences.

The central vertical axis in the *CIELAB* color space represents lightness (L^*), ranging from 0 (black) to 100 (white). The color axes (a^* and b^*) reflect the opposing nature of colors: a^*

represents the red-green axis, and b* represents the blue-yellow axis. In this model, a color cannot be both red and green or both blue and yellow simultaneously, as these colors oppose each other.

Overall, the *CIELAB* and *CIELUV* color spaces have become well-accepted standards for color measurements due to their improved uniformity and the ability to describe color differences accurately.

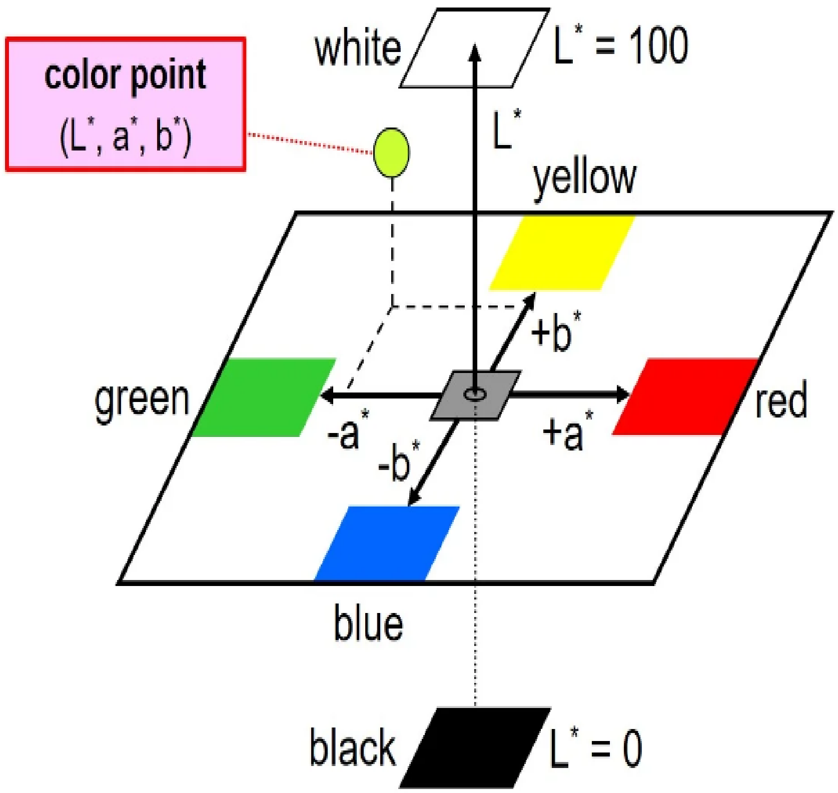


Figure 65. The 3-dimensional *CIELAB* color space.

*L** (lightness): ranging from 0 (black) to 100 (white).

*a** (green-red axis): with negative values indicating green and positive values indicating red.

*b** (blue-yellow axis): with negative values indicating blue and positive values indicating yellow.

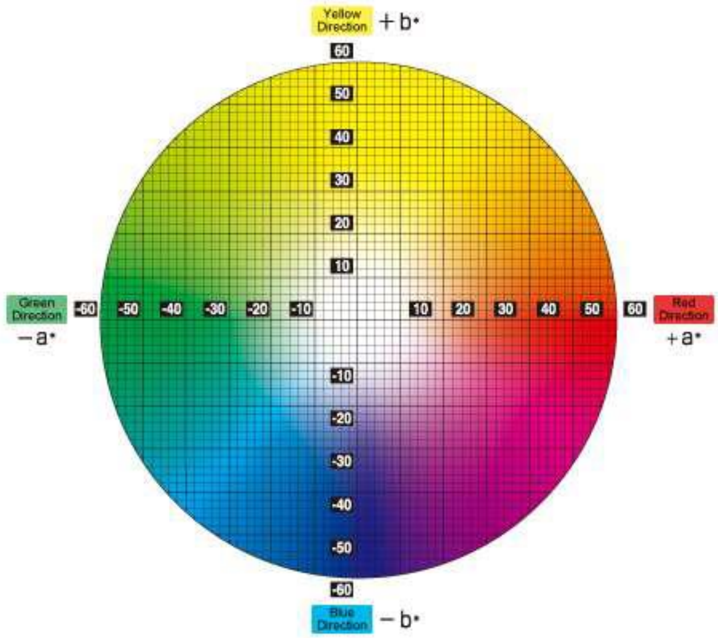


Figure 66. *L*a*b** Color Specification System Chromaticity Diagram.

Changes in *L** affect lightness (lighter or darker), changes in *a** shift the color towards red or green and changes in *b** shift the color towards yellow or blue, allowing precise control over color representation.

Standard Illuminants and Observers: Standards often specify the conditions under which fabrics should be evaluated. This includes the type of light used (standard illuminants) and the observer conditions (typically a 10-degree observer). These standardized conditions ensure that color measurements are consistent and can be reliably compared. By employing these standard approaches and practices in colorimetry, the textile industry can maintain the desired consistency, quality and accuracy of colors in fabrics [117].

The *CIELab* values will be calculated based on the measured reflectance spectra using Spectro magic (v:3.61) software for D65 illumination and a 10º observer. SPECTROPHOTOMETER CM-3600D (KONICA MINOLTA) and standard AATC, ISO 105-J03 [118].

Where *L**, *a** and *b** are the lightness, green–red and blue-yellow coordinates, respectively. One of the main goals of this project is to check the color properties and color changes of fabrics made from natural cotton fabrics. The machine was placed inside, and the test was performed. This test was repeated to measure the amount of color change after washing.

The Colorometry i7 device operates using advanced color measurement technology to accurately assess the color of a sample. It employs standardized light sources, such as D65 (daylight) or A (incandescent), to illuminate the sample. The device uses different illumination and viewing geometries to capture accurate color data.

In the Diffuse/0 (sphere) geometry, the sample is placed against a port that opens into a diffusely illuminated integrating sphere, coated with a highly reflective material for even light distribution. The device views the sample at an angle between 0º and 10º from the perpendicular, capturing all light reflected from the sample for precise color measurement. In the 0/Diffuse (sphere) geometry, the illumination path is reversed. The sample is illuminated at an angle between 0º and 10º and the reflected light is measured by a sensor inside the integrating sphere, ensuring accurate measurement of reflected light.

The device also uses 45/0 (or 0/45) geometry, where the sample is illuminated at a 45º angle and viewed at a 0º angle (perpendicular to the sample surface) or vice versa. This geometry ensures that the measurement is consistent with how the human eye perceives color.

To begin the measurement process, the device is first calibrated using a white standard reference for accuracy. The sample is then placed in the measurement area, ensuring proper alignment. Using a spectrophotometer, the device measures the light reflected from the sample, capturing data across the visible spectrum, typically from 400 nm to 700 nm. The captured data is processed to calculate color values, usually using the CIE (Commission Internationale de l'Éclairage) color space, including L_{ab^*} or L_{Ch^*} values, representing lightness, chroma and hue.

The color values are displayed on the device's screen or exported to a computer for further analysis, allowing for comparison to standard color references to ensure color accuracy and consistency. The Colorometry i7 device is widely used in various applications, such as the textile industry for ensuring color consistency and quality control in fabrics, in printing for accurate color matching, in manufacturing for quality control of colored products like plastics, paints and coatings and in research and development for developing new colors and materials with specific color properties.

By using standardized lighting conditions and precise geometric configurations, the Colorometry i7 device ensures highly accurate and reproducible color measurements, essential for quality control and color matching across various industries.

White calibrated standard, with which to standardize the instrument. The colorimetric values for this calibration standard are stored in the instrument or the software and require only that a specific standard be used to standardize the instrument. The correct white standard is usually identified with a serial number.

Black standard is required for some instruments. It may be of zero reflectance (a light trap), or it may be calibrated.

Preparation specimen, the perfect sample for color measurement should possess the qualities of being rigid, non-textured, inert, opaque and uniformly colored. However, since such flawless specimens are not found in textiles, it is essential to employ specific techniques to reduce the influence of specimen characteristics on color measurement. Important factors to consider include a) The presence of fluorescence from dyes or fluorescent whitening agents (FWAs) can distort results, particularly when instruments are unable to calibrate UV content. This issue is commonly observed in white or lightly colored materials treated with FWAs. b) Moisture content can have an impact on color and appearance, with the required conditioning time varying depending on the fiber, fabric structure, dyes and environmental factors. Materials such as cotton and viscose are notably susceptible to moisture.

Non-flexible fabric samples might extend into the viewing window of the device, resulting in inaccuracies in measurements. Fabrics that are not opaque permit light to penetrate, causing inaccurate outcomes. The sensitivity to light (photochromism) and heat (thermochromism) can cause measurements that cannot be replicated. The accuracy of color measurements is influenced by factors such as the size and surface texture of the sample, including pile, gloss and twill. Inconsistent results can also occur due to variations in color within the sample. Some examples are fiber, yarn, knits, lightweight materials, carpet, corduroy, denim and leather.

Tristimulus values are essential in colorimetric calculations, derived from spectral data. The specific values (X, Y and Z) obtained from spectral data are influenced by various factors such as the wavelength range, measurement interval and the illuminant/observer functions chosen by the user. For consistent results across users, it is recommended to calculate tristimulus values following ASTM E-308-96 standards. Typically, computer programs are used for these calculations and users should confirm with the instrument/software provider regarding the calculation method.

Calculation method of CIE 1976 *L**, *a**, *b** *C** and *h*$_{ab}$

*L**, *a**, *b**, C^*_{ab}, h_{ab} values are calculate from the *X*, *Y* and *Z* tristimulus values as shown in Equation(3-6).

$$L^* = 116\,(Y/Y_n)^{1/3} - 16 \quad \text{if } (Y/Y_n) > 0.008856$$
$$L^* = 903.3\,(Y/Y_n) \quad \text{if } (Y/Y_n) < 0.008\,856 \qquad \text{Eq. 3}$$

$$a^* = 500\left[f\left(\frac{x}{x_n}\right) - f\left(\frac{y}{y_n}\right)\right] \qquad \text{Eq. 4}$$

$$b^* = 200\left[f\left(\frac{y}{y_n}\right) - f\left(\frac{z}{z_n}\right)\right] \qquad \text{Eq. 5}$$

$$C^* = \sqrt{a^{*2} + b^{*2}} \qquad \text{Eq. 6}$$

where:

$f(x/x_n) = (x/x_n)^{1/3}$ if $(x/x_n) > 0{,}008\,856$

or

$f(x/x_n) = 7.787\,(x/x_n) + \frac{16}{116}$ if $(x/x_n) =< 0.008856$

$f(Y/Y_n) = (Y/Y_n)^{1/3}$ if $(Y/Y_n) > 0.008856$

or

$f(Y/Y_n) = 7{,}787\,(Y/Y_n) + \frac{16}{116}$ if $(Y/Y_n) < 0.008856$

$f(Z/Z_n) = (Z/Z_n)^{1/3}$ if $(Z/Z_n) > 0.008856$

or

$f(Z/Z_n) = 7.787\,(Z/Z_n) + \frac{16}{116}$ if $(Z/Z_n) < 0.008856$

$h_{ab^*} = arctan\left(\frac{b^*}{a^*}\right)$ expressed on a 0 º to 360 º scale with the *a** positive semi-axis being 0 º and the *b** positive semi-axis at 90 º.

$if\ a^* > 0\ \ and\ b^* > 0,\ \ 0 < h_{ab} < 90^\circ$

$if\ a^* < 0\ \ and\ b^* > 0,\ \ 90^\circ < h_{ab} < 180^\circ$

$if\ a^* < 0\ \ and\ b^* < 0,\ \ 180^\circ < h_{ab} < 270^\circ$

$$if\ a^* > 0\ \ and\ b^* < 0, \qquad 270^\circ < h_{ab} < 360^\circ$$

for these equations, X_n, Y_n and Z_n are the tristimulus values of the illuminant. For daylight, the preferred illuminant/observer combination is D65/10.

The color differences *(ΔE_{ab}*)* of the naturally colored cotton lines, compared with a reference (0 wash) fabrics, is calculated according to Equation 7.

$$\Delta E_{ab}{}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad \text{Eq. 7}$$

2.3.5 Ultraviolet Protection Factor (UPF)

Ultraviolet Protection Factor (UPF) is determined by assessing the level of UV radiation that can pass through the material and come into contact with the skin. This evaluation is conducted using a spectrophotometer or a comparable tool capable of gauging the strength of UV radiation that traverses the material. The UPF value is established by comparing the amount of UV radiation exposure with and without the protective material. For example, a UPF rating of 25 indicates that only 1/25th of the UV rays from the sun can permeate the material [119].

The sun-blocking properties of textiles are enhanced when a dye pigment that dilutes the ultraviolet absorber finish absorbs ultraviolet radiation and blocks its transmission from fabric to skin. For (UPF) test, using a UV TRANSMITTANCE ANALYZER (UV 1000) device under the AS/NZS 4399:1996 standard [120] and the ultraviolet protection factor (UPF) was calculated using the mean percentage transmission in the UVA region (320-400 nm) and mean percentage transmission in the UVB region (280-320 nm) according to Equation 8 and Figure 67.

Figure 67. UPF device test.

Ultraviolet transmittance spectra average transmittances UVR, UVA and UVB When direct light falls on a textile, part of the radiation is reflected, the material absorbs another part and the remainder passes through it and is transmitted diffusely. The proportion of the incoming light that is transmitted is different for every wavelength. The diffuse transmittance spectrum is the representation of the proportion of ultraviolet radiation transmitted against the wavelength (from 290 to 400 nm). The transmittance spectra were determined using the Ultraviolet Transmittance Analyses UV1000F of the Lab sphere, designed especially for this task. According to the Standard AS/NZS 4399:1996, the UVR transmittance through the fabric is defined as the arithmetic mean of the transmittances in the ultraviolet range wavelengths, from 290 to 400 nm (Equation 8).

$$UVRAV = \frac{\mathrm{T290} + \mathrm{T295} + \cdots + \mathrm{T400}}{23} \qquad \text{Eq. 8}$$

where T_λ is the spectral transmittance at wavelength λ. Because there is a great difference in the effect that the UVA and UVB radiation have on the human skin, it is interesting to have a parameter that quantifies the amount of both UVA and UVB radiation that passes through the fabric. To this aim, the average UVA and UVB transmittance is defined as the arithmetic mean of the transmittance in the wavelengths of the UVA and UVB, respectively (Equation 9 and equation 10).

- T290, T295,T400 are the temperature measurements at wavelengths 290 nm, 295 nm, ..., and 400 nm.
- The number 23 represents the total number of temperature measurements taken at these wavelengths.

This formula indicates that the UVRAV is the mean temperature over 23 measurements from the specified range of wavelengths.

$$UVAAV = \frac{\mathrm{T315} + \mathrm{T320} + \cdots + \mathrm{T400}}{18} \qquad \text{Eq. 9}$$

$$UVBAV = \frac{\mathrm{T290} + \mathrm{T295} + \cdots + \mathrm{T315}}{6} \qquad \text{Eq. 10}$$

Especially important that the UVB transmission is as low as possible, as the radiation in this wavelength interval is much more damaging for the human skin, Table.

Table 16. UPF Classification System.

UPF Range	UVR Protection Category	Effective UVR Transmission (%)
15–24	Good protection	6.7–4.2
25–39	Very good protection	4.1–2.6
40–50, 50+	Excellent protection	≤2.5

Ultraviolet protection factor The UPF of the fabrics has been determined by the in vitro method, according to the indications of the standard AS/NZS 4399:1996, [120]. The UPF of each specimen is calculated as follows (Equation 11):

$$UPF = \frac{\int_{\lambda=280}^{\lambda=400} S_\lambda \cdot E_\lambda \cdot d_\lambda}{\int_{\lambda=280}^{\lambda=400} S_\lambda \cdot E_\lambda \cdot T_\lambda \cdot d_\lambda} \qquad \text{Eq. 11}$$

where: S_λ is the solar spectral irradiance,

E_λ is the CIE reference erythema dose spectrum,

d_λ is the bandwidth, in nm,

T_λ is the diffuse transmittance spectrum,

λ is the nanometer wavelength.

SPF is based on the time it takes for UV-exposed skin to redden; if you burn after 20 minutes, an SPF 15 sunscreen may protect your skin 15 times longer (if used correctly). UPF testing measures the amount of UV radiation that can penetrate fabric and reach your skin. UPF tests measure both UVB and UVA rays.

3 Results

In this section, provided an overview and then classify the results presented in this section. The results of this chapter are presented in the following sections.

1- Raw materials characterization
2- Yarn preparation and characterization
3- Study on knitted fabrics
4- Study in woven fabrics
5- Effect of washing on woven fabrics
6- Influence of household washing on the colorimetric properties of intrinsically natural organic cotton fabrics
7- Evaluating the Impact of Washing Conditions on the Colorfastness of Naturally Colored Cotton Fabrics: A Focus on Detergents, Water Types and Temperature

3.1 Raw materials characterization

Raw materials have been characterized as described in section 2.3, Results are included in Table 17 ten samples of Topaz and Raw materials have been and 5 samples of Light ruby and Dark ruby.

Table 17. Physical test on raw cotton.

Sample	SCI	Mic	Mat	UHML (mm)	UI (%)	SF (%)	Str (g/tex)	Elg (%)
Topaz	84	4.23	0.84	26.67	79.5	9.5	25.0	8.6
Light ruby	-	4.26	0.84	20.50	78.8	11.9	19.6	9.9
Dark ruby	-	4.15	0.84	21.01	79.2	12.0	21.7	9.0
Raw	99	3.76	0.84	25.73	78.0	10.3	26.3	7.0

The results of the test of the four cotton with the HVI equipment show that it is an acceptable quality with very good micronaire values, an acceptable fiber length and a relatively low short fiber content that can subsequently be improved by combing, an operation this last that has been carried out in the spinning company where the fiber has been processed. The resistance results are also correct for obtaining medium-quality yarns.

3.2 Yarn preparation and characterization

Yarns were characterized according to the methodology described in Section 2.3.1. In Table 18 presented the yarns characterizations and after can see the details of each test in separate parts.

Table 18. Yarn characterizations

Parameter	Average	SD	CV%	Q (95%)	min	max
Count (tex)	29.5	-	0.08	-	-	-
Twist (t/m)	706.6		9.0			
Breaking Strength (cN)	406.74	30	7.28	11.85	344.52	466.47
Tenacity (cN/tex)	13.79	1.006	7.29	0.4	11.678	15.806
Elongation at break (%)	6.41	0.33	5.202	0.134	5.56	7.068
Breaking Work (cN·cm)	700	78	11.2	31.3	549	859
Mass Unevess (CV%)	13.932			-	-	-
DR% (1.37m 5.00%)	24.0	7.9	32.7	34.3	13.9	37.1
Thin point (50%) (1.000 m)	17.3	13.1	75.8	30.8	2.0	31.0
Thick points (50%) (1.000 m)	29	19	35	51	0	53
Neps (+200%) (1.000 m)	6.8	7.9	32.7	11.8	0	13.0
Hairiness N1	65823	6932	-	-	-	-
Hairiness N2	12377	1396	-	-	-	-
Hairiness N3	5201	627	-	-	-	-
Hairiness N4	2912	391	-	-	-	-
Hairiness N6	727	136	-	-	-	-
Hairiness N8	130.6	33.3	-	-	-	-
Hairiness N10	16.6	3.4	-	-	-	-
Hairiness N12	2.5	-	-	-	-	-
Hairiness Index	5	-	-	-	-	-
Hairiness S3	44949	-	-	-	-	-

The values of resistance, elongation and tenacity can be considered as usual for being woven on an air loom like the one used in this thesis and the values of irregularity of mass and hairiness as of an acceptable average quality.

3.3 Study on knitted fabrics

The yarns produced were transformed into a knitted fabric following 2.2.2. Fabrics were characterized with the following tests:

1. Weight per square meter test (section 2.3.3).

2. Color measurement (section 2.3.4)

3. UV radiation absorption test (section 2.3.5).

These By conducting tests were conducted to assess not only the fabric properties but also the confidence in the quality and improved performance of the final product. Compliance with industry standards and requirements is of great importance to ensure that the final product delivers an optimal performance.

analyzed to investigate the effects of domestic washing on fabrics made from naturally colored cotton. Samples were prepared using the knitting method and washed with detergent, as suggested by the FOX FIBER company (Det A). The samples were washed at varying intervals, ranging from 1 to 30 times, to observe qualitative alterations, such as weight per square meter, UPF and color variations(colorimetric) before and after washing. These data are valuable for determining the resilience and excellence of fabrics under typical usage circumstances.

To determine the weight per square meter of fabric, it was necessary to obtain a 10 cm × 10 cm sample and subject it to standard atmospheric conditions for a duration of 24 hours. The sample was then weighed using a precise scale and the resulting weight in grams was multiplied by 100 to obtain the weight per square meter (g/m²). This approach guarantees precise evaluation of fabric quality and material expenses while also accounting for variations in fabric types, such as knitted or woven fabrics. It is advisable to measure multiple samples to ensure consistency in results. The procedure for determining the weight of the fabric in grams per square meter (g/m²) is outlined in ISO 3801:1977. A specific area of fabric was cut with a circular cutter and weighed on a precise scale and the weight per square meter was obtained by dividing the weight of the sample by its area. To ensure accuracy, measurements were taken multiple times and the average result was calculated for consistency, Table 19, [109].

Table 19. Weight per meter square of fabrics before and after washing and weight changes.

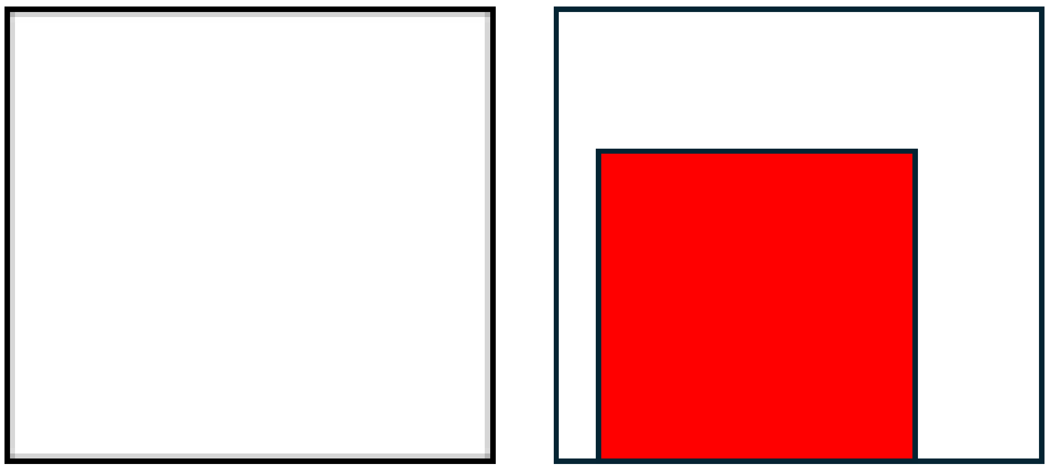
	Number of washes										
	0	1	2	3	4	5	10	15	20	25	30
Before wash	195.2	188.4	199.0	208.5	196.4	189.7	187.4	202.8	202.8	185.3	195.3
After wash	195.2	215.5	215.5	209.8	218.4	213.6	202.4	209.0	207.9	212.1	205.2
Weight changes (%)	0	14.4	8.3	0.64	11.2	12.8	8.0	3.0	2.5	14.5	5.0

Shrinkage in fabrics following laundering is widely recognized. Samples with a knitting structure are used in experiments, which predispose them to edge curling and thus complicate the measurements of dimensional changes across both length and width. This challenge is intensified by the tiny dimensions of the samples (10 cm x 10 cm), making the assessment of shrinkage percentages through loop counting ineffective. To navigate these challenges, a new methodological approach is proposed. Every sample's dimension and weight are thoroughly recorded both before and after washing. Given a uniform shrinkage rate across all axes, the post-wash dimensions of the fabric are anticipated to be considerably smaller than the pre-wash dimensions. Through the application of the updated measurements and weight, the shrinkage percentage is accurately calculated as outlined in the formulas below. These processes and their underlying methodologies are depicted schematically in the designated (Figure 68).

Calculation of shrinkage area that used the equations 12 and 13.

$$shirinkage\ area\ (\%) = \frac{w1 * 100}{w2} \qquad \text{Eq. 12}$$

$$percentage\ of\ shirinkage = \frac{x - 100}{100} \qquad \text{Eq. 13}$$



(a) (b)

Figure 68. Schematic of shrinkage, (a) The initial sample and (b) final sample are in red.

Table 20. Shrinkage area and thickness of knitted fabrics in function of the number of washed.

	Number of washes										
	0	1	2	3	4	5	10	15	20	25	30
Shrinkage area (%)	0	-12.6	-7.7	-0.6	-10.1	-11.2	-7.4	-3.0	-2.4	-12.7	-4.8
Thickness (mm)	1.1	1.1	1.05	1.10	1.08	1.10	0.98	1.07	1.08	1.00	1.02

Due to the particular structure of knitted fabrics that have high yarn mobility and low stability, the results obtained in the study of the influence of domestic washing on dimensional stability have presented extremely variable results that cannot be used for a rigorous analysis of how fabric shrinkage can affect color changes. For this reason, it is rejected to continue analysing this phenomenon with knitted fabrics and it is decided to analyse it from woven fabrics, Table 20.

Colorimetry of knitted fabrics

Colorimetric testing is utilized to assess the color characteristics of textiles to guarantee colorfastness, uniformity of shade and consistency, particularly following washing procedures. The objective is to uphold fabric colors of superior quality and dependability that adhere to both industry regulations and customer requirements. This process encompasses techniques such as spectrophotometry and visual evaluations, which assist in ensuring quality control and adherence to standards. In the following, we presented the colorimetric analyses on knitted fabrics after 30 washes, Table 21.

Table 21. Colorimetric analysis of knitted fabrics.

Number of washes	*L**	*a**	*b**	*C**	*hº*
0	56.49	9.74	20.02	22.26	64.06
1	56.06	9.81	20.01	22.29	63.88
2	55.71	9.60	19.44	21.68	63.71
3	56.37	9.67	19.84	22.07	64.01
4	55.80	9.69	19.69	21.94	63.80
5	56.59	9.45	19.37	21.55	64.00
10	56.15	9.64	19.93	22.14	64.18
15	55.96	9.47	19.30	21.50	63.86
20	55.73	9.69	19.65	21.91	63.74
25	56.06	9.58	19.53	21.75	63.86
30	56.39	9.65	19.73	21.96	63.95

Various parameters were investigated for each sample during the analysis of the table. The brightness or darkness of each sample was determined by examining the light (*L**) values, with higher values indicating brighter samples and lower values indicating darker ones. The color components (*a** and *b**) were also examined, representing the red, green and yellow-blue components respectively. Positive a* values indicated the presence of red tones, while negative values indicated green tones. Similarly, positive *b** values indicated yellow tones, whereas negative values suggested blue tones. These values were analyzed to uncover the color characteristics of each sample.

In addition, the colorfulness or saturation of a sample was assessed through the evaluation of chroma (*C**), with higher values indicating more vibrant colors and lower values indicating less saturated or more desaturated colors. The hue angle (*hº*) was also examined to identify the dominant hue of a sample, measured in degrees to represent its position on the color wheel. Analyzing the hue angle helped in identifying the primary colors present in each sample.

Furthermore, differences *(ΔL^*, Δa^*, Δb^*, ΔC^*, Δh^** and ΔE^*_{ab}) between each sample and the standard sample were evaluated. Positive values indicated an increase in a specific attribute compared to the standard, while negative values indicated a decrease. Analyzing these differences provided insights into how each sample deviated from the standard in terms of color.

By scrutinizing these parameters for each sample, a comprehensive understanding of the color characteristics, variations and deviations from the standard could be obtained. This analysis proved valuable for comparing and evaluating the color properties of different samples, especially under the D65/10° lighting conditions.

Eleven samples similar to those utilized in analyzing knitted fabrics were produced. The details regarding the items are available in Section 2.4.2. Evaluations of the physical properties were carried out on the woven fabrics.

UPF test knitting fabrics

As explained about the UPF parameters in last parts in summary before and after washing treatment in Table 22 and Table 23.

These findings suggest that the UPF treatment is highly effective in enhancing UV protection, with significant improvements observed after several washes and sustained effectiveness even after 25 washes. The higher UPF values and lower UV transmittance percentages confirm the efficacy of the treatment over time,

UPF 15-24: Good protection

UPF 25-39: Very good protection

UPF 40-50+: Excellent protection

Table 22 and Table 23, analysis reveals that the UV protective performance of the fabric diminishes significantly within the first few washes, as evidenced by the initial drop in UPF values and the corresponding increase in UV transmission percentages. However, after around 5 washes, the fabric begins to regain some of its UV blocking ability, stabilizing in its performance from 10 washes onwards. This suggests that while the fabric's initial integrity is compromised by washing, it reaches a point of stabilization where further washes do not significantly degrade its UV protective properties.

Table 22. Change of the UPF with the number of washes (before wash).

Number of washes										
	0	1	2	3	4	5	10	15	20	25
UPF Melb	47.38	41.52	38.40	32.29	26	30.08	35.38	35.87	36.69	35.48
UPD Albur	47.21	41.29	38.24	32.15	25.90	29.99	35.26	35.74	36.60	35.36
UPF Albur (EN13757:2001)	47.21	41.29	38.24	32.15	25.90	29.99	35.26	35.74	36.60	35.36
UVA 315-400 (%)	2.53	2.82	3.03	3.62	4.44	4.07	3.28	3.21	3.29	3.28
UVB 290-315 (%)	2.13	2.37	2.56	3.07	3.84	3.58	2.76	2.75	2.82	2.83
UVR 290-400 (%)	2.44	2.71	2.93	3.50	4.30	3.96	3.17	3.10	3.19	3.18

Table 23. Change of the UPF with the number of washes (after wash).

Number of washes										
	0	1	2	3	4	5	10	15	20	25
UPF Melb	63.88	174.81	33.29	110.55	70.65	60.08	26.40	36.69	35.48	33.66
UPD Albur	63.65	174.52	32.15	110.38	70.50	60.03	26.35	36.60	35.36	33.60
UPF Albur (EN13757:2001)	63.65	174.52	32.15	110.38	70.50	60.03	26.35	36.60	35.36	33.60
UVA 315-400 (%)	1.84	0.62	3.62	1.00	1.56	1.77	4.11	3.29	3.28	3.19
UVB 290-315 (%)	1.61	0.57	3.07	0.90	1.41	1.66	3.48	2.82	2.83	2.97
UVR 290-400 (%)	1.79	0.61	3.50	0.98	1.51	1.74	4.05	3.19	3.18	3.14

The data shows initial significant fluctuations in UPF values across different washing stages, with unusually high UPF values at 1 wash. Following these fluctuations, UPF values stabilize at lower levels after 5 washes. Conversely, UV transmission percentages initially decrease significantly at 1 wash before increasing and stabilizing after 5 washes. This indicates that the fabric undergoes an initial alteration in its UV protective properties before settling into a more consistent performance range. Despite these initial fluctuations, the fabric maintains reasonable UV protection with continued washing.

Color change and UPF that can be done to the fact that working with knitted fabrics is difficult due to their high elasticity, dimensional instability and looped surface texture that can affect test consistency when measuring color and UPF.

As woven fabrics are more stable, uniform and easier to handle and also provide more reliable results in textile testing, woven fabrics of the same material are preferred for such tests in the following processes.

3.4 Study in woven fabrics

Bearing in mind the conclusions of the results of the knitted fabrics, woven fabrics were produced following the methodology in section 2.2.3.

The results show that the domestic washing process of the studied shed fabric causes a shrinkage of the fabric both in the warp direction and in the weft as shown in Figure 69.

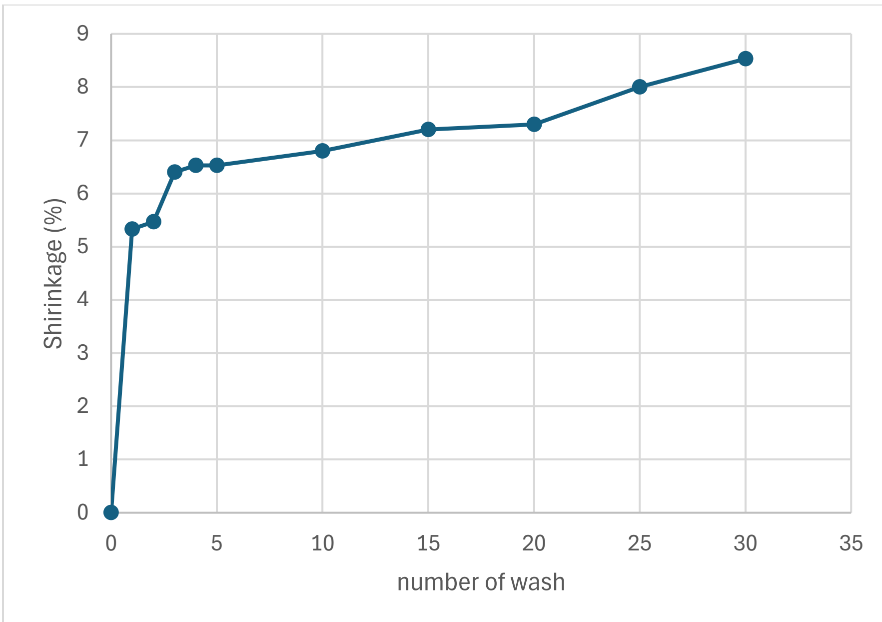


Figure 69. Evolution of shrinkage in function of the number of washes.

Changes in shrinkage cause, as expected, an increase in the weight per square meter of the fabrics, as shown in the figure below (Figure 70).

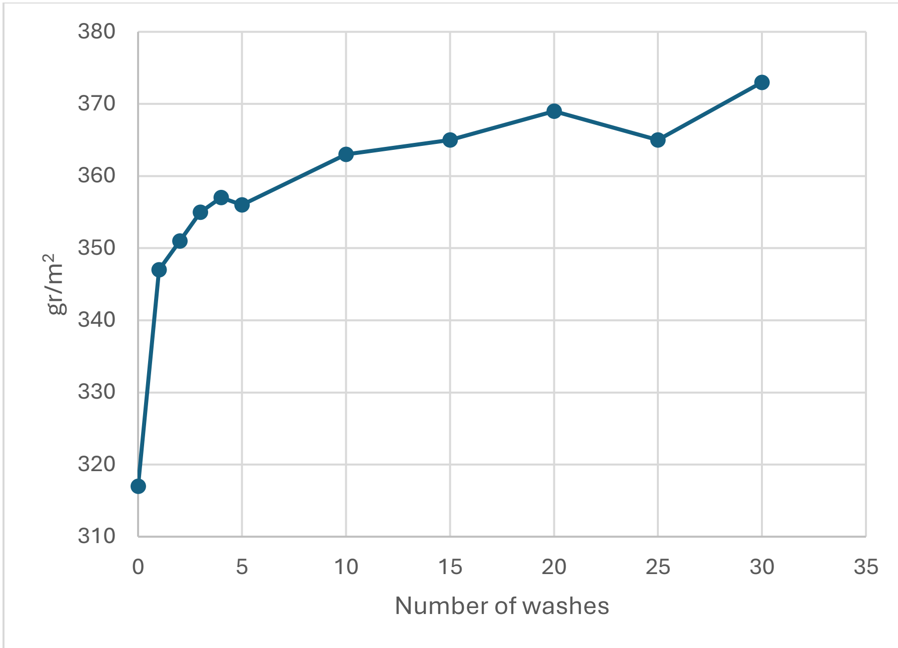


Figure 70. Evolution of weight per square meter in function of the number of washes.

On the other hand, shrinkage also causes and also as expected, a slight increase in the thickness of the fabric (T100): Thickness value in millimetres at a pressure of 100 grams, (measured in the FAST test), as shown in Figure 71.

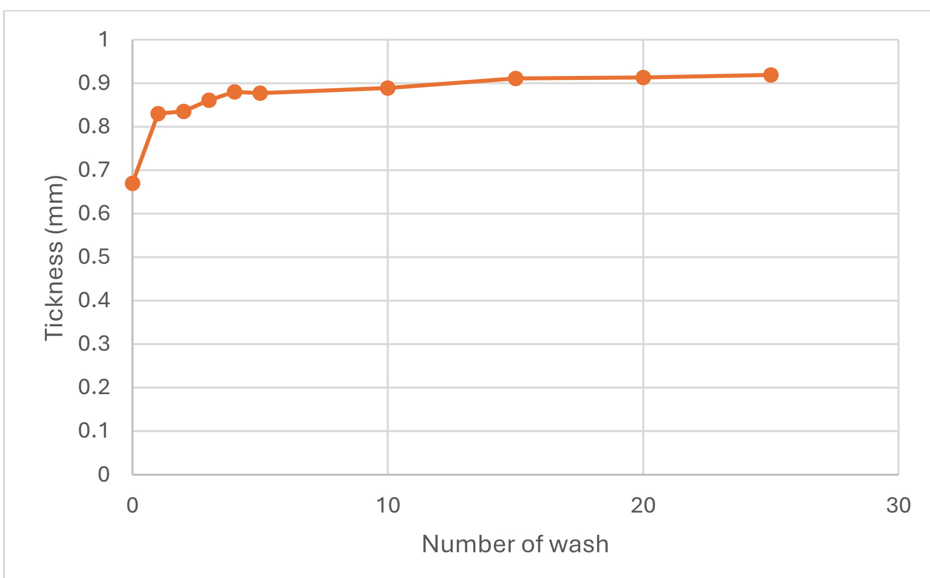


Figure 71. Evolution of T100 in function of the number of washes.

The relationship between fabric shrinkage and fabric thickness is shown in the linear correlation between both parameters as shown in the following in Figure 72.

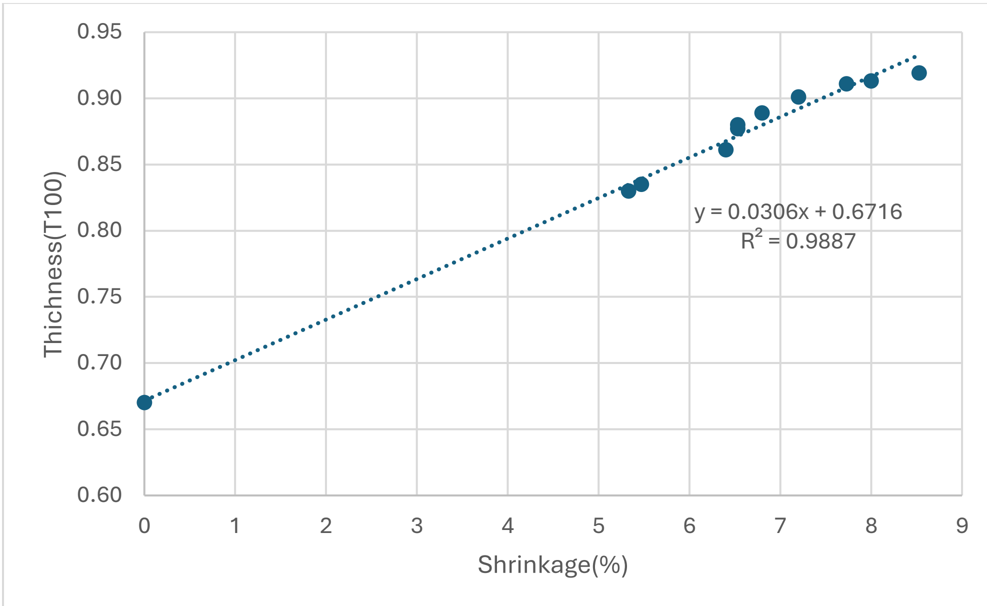


Figure 72. Coloration between shrinkage and thickness of the fabric.

The high R-squared value of 0.9887 demonstrates that the linear model is an excellent fit for the data, explaining 98.87 % of the variance in the shrinkage percentage based on T100 values. The predictive equation (y=0.0306x+0.6716) can be used to estimate the shrinkage percentage for any given T100 value.

The shrinkage derived from domestic washing causes both an increase in the density of the fabric, its weight per square meter and an increase in thickness, which entails an increase in the mass of fibers per unit of surface that can cause (as will be seen later), in turn, an increase in color intensity due to a change in the physical structure of the fabric.

The rest of the physical tests carried out on the fabric as described in the methodology section (FAST, tensile strength, drape, etc.) have not led to significant relevant changes that could affect the color changes detected throughout domestic washing and which deserve a comment. For this reason, they are not included in this section. Just comment, as an example, that domestic washing, as was also foreseeable, causes a loss of tensile strength due to the deterioration of the yarns and also a slight increase in bending rigidity as shown in the (Figure 73, Figure 74 and Figure 75).

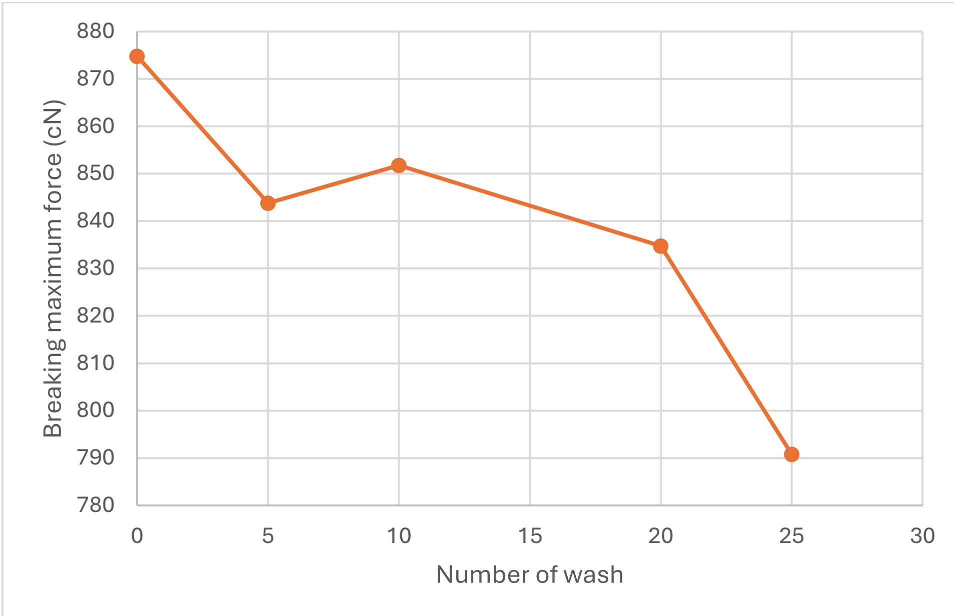


Figure 73. Breaking maximum force (cN)

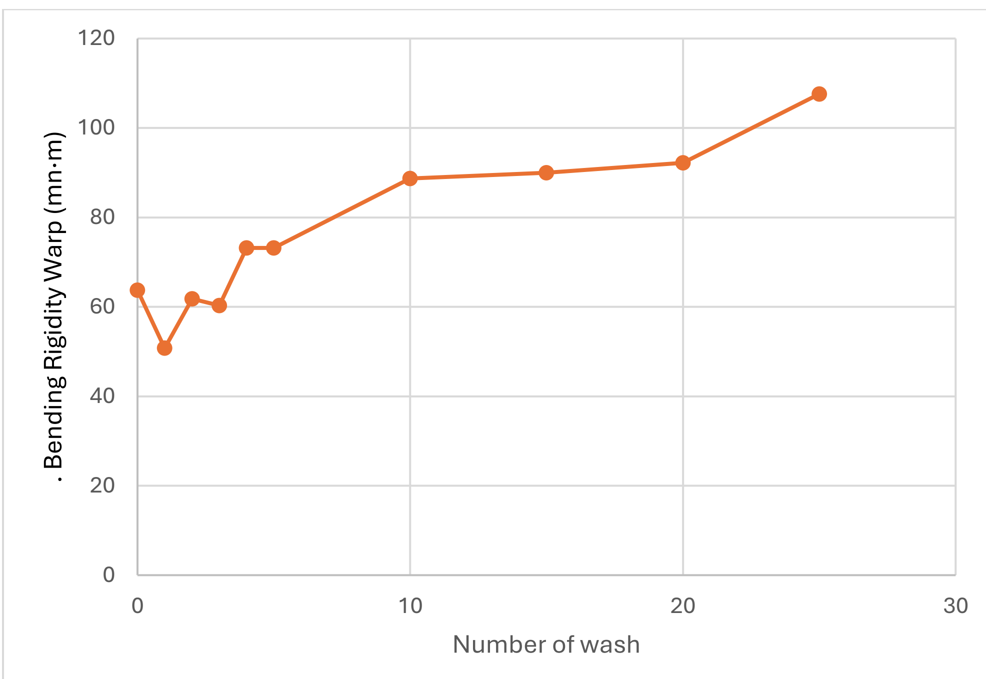


Figure 74. Bending rigidity of warp direction

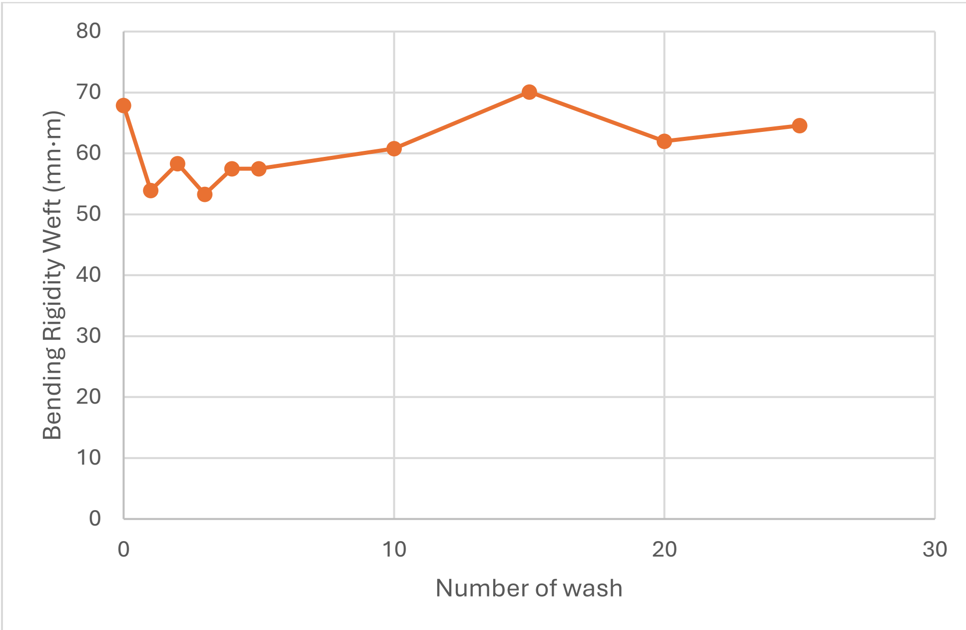


Figure 75. Bending rigidity of weft direction.

3. 5 Effect of washing on woven fabrics

This part of the thesis is divided in two parts. In the first part of the study, the effect of the number of washes on the color differences was analyzed. This research examined the impact of household washing on the colorimetric characteristics of natural colored organic cotton (NaCOC) fabrics, assessed before and after up to 30 wash cycles. Significant changes in lightness and saturation were observed, especially after the first wash, with minimal variation after the fifth wash. FTIR-ATR analysis indicated that the washing process contributed to the darkening of the samples. These results were published in the Journal of Natural Fiber (Aliei, H., Cayuela, D., Capdevila, F. J., & Carrera-Gallissà, E. (2024). Influence of Household Washing on the Colorimetric Properties of Intrinsically Natural Organic Cotton Fabrics. Journal of Natural Fibers, 21(1). https://doi.org/10.1080/15440478.2024.2351160).

Considering the results of the first study, were the most important washes in terms of color changes were between the first five washes, in a second paper (to be published, as second part of the study, the influence of the temperature, type of detergent and the temperature were studied. This part of the work carried out investigates how washing conditions affect the color of naturally colored organic cotton (NaCOC) fabrics, focusing on the impact of different detergents, water types and temperatures. The study uses colorimetric measures to evaluate the effects on color and fabric integrity. Findings indicate that water hardness is the most significant factor influencing color changes.

3.5.1 Influence of household washing on the colorimetric properties of intrinsically natural organic cotton fabrics

3.5.1.1. Abstract

This research aimed to examine how household washing influences the colorimetric characteristics of natural colored organic cotton (NaCOC) fabrics. The colorimetric properties of (NaCOC) fabrics were assessed both before and after undergoing washing for up to 30 cycles. The findings indicated that washing significantly influenced the colorimetric properties of naturally dyed cotton fabrics, as evidenced by changes in lightness and saturation. The most significant difference in color between the two samples was observed after the initial wash, highlighting the reduction in both parameters, lightness and saturation, after this first washing. Between the second and fifth washes, there are notable differences in these parameters. However, from the fifth wash, the variation in color difference was minimal. Additionally, the FTIR-ATR analysis of

the extracts in petroleum ether and subsequently in ethanol of the NaCOC fabrics, before and after home washing, in conjunction with a comparison with shrinkage, demonstrated that the latter process was accountable for the darkening of the sample.

Keywords: Natural colored cotton; colorimetric; color changes; household washing

3.5.1.2 Introduction

Cotton, the most vital natural fiber, is utilized by nearly every individual worldwide daily. Supporting the livelihoods of approximately 250 million people, it stands as one of the foremost fiber crops cultivated in over 80 countries. While being a renewable natural resource, responsible management of cotton is imperative. Natural-colored cotton, characterized by its pigmented fibers in shades of green, brown and red, is available commercially in brown and green hues [45]. Previous research on natural-colored organic cotton [45, 68, 121-124] can be categorized into three distinct groups: cellulose synthesis and genetic diversity [45, 121], pigment migration, color variations and property changes [68, 123-124] and performance after repeated laundering [121].

Evidence from archaeological findings suggests that naturally colored cotton has been used for at least 2500 years. However, the use of this type of cotton gradually declined due to its lower fiber quality and lower yields compared to white cotton, resulting in reduced profitability [125]. In recent times, interest has been resurgent in naturally pigmented cotton, driven by growing ecological concerns. This renewed interest has also led to an increase in the market share of naturally colored organic cotton (NaCOC). The textile and fashion industries are particularly drawn to NaCOC because it eliminates the need for dyeing, thereby reducing environmental pollution [121-123].

The dyeing process results in an excessive amount of effluent, which is not only expensive but also requires immediate action to minimize its harmful effects. In order to prevent this action, recent studies have been conducted proposing more sustainable processes for dyeing cotton with tannin derivatives [8-10]. Another way to prevent the action of dye is to avoid it. Therefore, the increasing environmental concerns have prompted different nations, including Israel, the United States and Brazil, to enhance their endeavors in creating strains with vibrant colors [97, 126]. These new strains are characterized by improved fiber properties and a wide range of natural colors that exhibit desirable fastness, aligning with modern spinning and finishing techniques [85]. Spinning and finishing processes are essential in transforming raw cotton into marketable goods. Cellulose, which constitutes approximately 90-95 % of cotton fibers, is the primary component, while non-cellulosic elements like waxes and salts make up the remaining percentage. Waxes play a crucial role in facilitating spinning; however, they need to be eliminated through appropriate chemical treatments [127]. Typically, wet alkali treatments (scouring) involving various caustic solutions are employed to remove non-cellulosic constituents and the addition of wetting agents enhances the absorption properties of cotton fibers [128].

Naturally pigmented cotton and conventional white cotton can be compared in various aspects. The dissimilarities between them do not solely arise from their color variations. Their chemical compositions, structures and certain other characteristics exhibit remarkable similarities. Nevertheless, there exist fundamental disparities between the two types of cotton [97].

The definitive identification of the specific constituents of the pigments present in colored cotton remains an ongoing challenge. Numerous research studies have been conducted to determine the composition and properties of the natural pigments found in colored cotton. It has been discovered that the brown coloration observed in colored cotton fibers is attributed to the presence of tannin vacuoles within the lumen of the fiber cells. Conversely, the green coloration in green-colored cotton primarily originates from caffeic acid, a derivative of cinnamic acid, which is predominantly found in the suberin layer. Importantly, the quantity of pigments is more prominent in brown cotton compared to green cotton. This difference in pigment types serves as one of the justifications for selecting brown cotton fabrics in the current study [129], which aims to investigate the impact of household washing on the color variation of cotton fabric.

Fabrics produced with NaCOC cotton offer several benefits, one of which is their absence of agrochemicals and chemicals used in the dyeing process. This makes them highly suitable for individuals with multiple chemical sensitivity and skin hypersensitivity. In this study, we have specifically examined the impact of domestic washing with gentle soap on the colorimetric properties of the fabric. It is well known that the color of this type of cotton intensifies with washing and our analysis has focused on this intriguing characteristic.

3.5.1.3 Experimental part

Material

This study uses NaCOC, grown in Brazil, supplied by the Spanish company “Organic Cotton Colors (OCC)” [130]. For the realization of the yarn used in this study, the following cotton varieties of this company have been used: Topaz, Light ruby, Dark ruby and Raw (white) cotton. Characteristics of the corresponding varieties are included in Table 24.

Table 24. Characteristics of the NaCOC fibers used.

Sample	SCI	MIC	MAT	UHML (mm)	UL (%)	SF (%)	STR (g/tex)	ELG (%)
Topaz	84	4.23	0.84	26.67	79.5	9.5	25.0	8.6
Light ruby	-	4.26	0.84	20.50	78.8	11.9	19.6	9.9
Dark ruby	-	4.15	0.84	21.01	79.2	12.0	21.7	9.0
Raw	99	3.76	0.84	25.73	78.0	10.3	26.3	7.0

Were:

- SCI: Spinning Consistency Index. It is a parameter for predicting the spinnability of the fibers. In general, the higher the index, the higher the yarn strength and the better the overall fiber spinnability.
- MIC: Micronaire Index. The pressure drop is measured by passing air through the fibers.
- MAT: Maturity index. This indicates the degree of cell wall thickness in the cotton sample. The HVI correlates very well with the AFIS Maturity Ratio and the microscopy reference method (cross-sectional analysis).
- UHML: upper half mean length. It is the mean length by the number of fibers and corresponds to the classer's staple length and the AFIS Upper Quartile Length by weight.
- UL%: Uniformity Index. It expresses the ratio of the Mean Length to the Upper Half Mean Length. It is an indication of the distribution of fiber length within the fibrogram.
- SF%: Short Fiber Index. Indicates the number of fibers in percent less than 12.7 mm in length. It correlates very well with the AFIS Short Fiber Content by weight (SFC).
- STR: Bundle strength. Is the breaking strength of the cotton fibers in grams per tex.

- ELG: Elongation. It is a measurement of an increase in the length of the cotton test piece, expressed in %, as a result of the application of a load.

With these fibers, the Spanish company OCC made a mixture in equal parts of the four cotton described in Table 1 and proceeded to carry out a combed spinning process (opening, cleaning, carding, drawing, combing, drawing and finally roving machine). Ring spinning was carried out on a PINTER machine model Merlin Spa 1803, under the conditions indicated in Table 25, which includes the characteristics of the yarn obtained.

Spinning conditions and characteristics of the yarn obtained

Table 25. Spinning conditions and characteristics of the yarn obtained.

Type of ring spinning machine	PINTER model Merlin spa1803
Roving count (ktex)	0.600
Yarn count (tex)	29.5
Twist (t/m)	705
Linear spinning speed	16.65 m/min
Revolutions per minute of the spindle	7,500 rpm
Ring diameter	42 mm
Type of traveller	Bräcker 2DR

In the present study, the fabric employed is made up of a conventional cotton warp and a NaCOC weft. These components were woven using an air-jet Dornier loom and a Staübli Jacquard machine. The fabric is constructed in a five-satin pattern, with a density of 38.4 yarn/cm (2/30 Nm) for the warp and 19 pics/cm (2/34 Nm) for the weft. The areal density is 314 g/m^2. Its width measures 146 cm. No other treatment has been applied to the fabric.

Washing

The Fox Fibre® Colorganic® detergent has been used in this work. This laundry detergent is specifically designed for washing delicate fabrics. It is formulated with a blend of cleaning agents, surfactants and grease solvents to effectively remove dirt and stains without damaging the fibers.

The soap used in this detergent is carefully selected to control foam and maintain a regular pH level. Additionally, natural organic acids and additives are included to ensure the integrity of the fibers is maintained throughout the washing process. This detergent has no environmental impact on aquatic life and it is fast and completely biodegradable.

The cotton fabrics were subjected to washing in a conventional household washing machine (FAGOR, F2810) under typical Spanish household conditions, which included a temperature of 40 ºC, a duration of 1 hour, 15 ml of Fox Fibre® Colorganic® detergent and a water hardness of 400 mg $CaCO_3$/L. The pH of the detergent at the concentration used in household washing is 9. Fabric specimens measuring 170 x 150 cm were utilized and underwent 1 to 30 washes under the aforementioned conditions. After each wash, the samples were dried in a Frommer model dry 92 tumble dryer and then conditioned in a standard climate of 20 ºC and 60 % RH for 24 hours.

Characterization

The CIE*L*a*b** coordinates and UPF were utilized to measure the optical parameters, which were then correlated with the number of washes and the shrinkage of the samples. UPF was also related to the weight per surface unit and thickness.

The color of the samples was assessed using a GRETAGMACBETH COLOR I7 equipment, following the ISO 105-J03 standard [131], with D65 illuminant and a ten-degree observer. To determine the color differences between each washed fabric and the original, the parameter ΔE_{ab}^* was employed (Eq. 13):

$$\Delta E_{ab}{}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad \text{(Eq. 13)}$$

were L^*, a^* *and* b^* are the lightness, green–red coordinate and blue-yellow coordinate, respectively, in the CIE*L*a*b** space.

The Ultraviolet Protection Factor (UPF) of the fabrics was determined by the in vitro method using an Ultraviolet Transmittance Analyzer UV1000F (Labsphere) and according to Standard AS/NZ 4399: 1996 [130].

The UPF of each specimen is calculated as follows:

$$UPF = \frac{\int_{280}^{400} S_\lambda E_\lambda \Delta\lambda}{\int_{280}^{400} S_\lambda E_\lambda T_\lambda \Delta\lambda} \quad \text{(Eq. 14)}$$

where:

E_λ CIE relative erythemal spectral effectiveness,

S_λ solar spectral irradiance,

T_λ spectral transmittance of the fabric,

Δ_λ wavelength step in nm,

λ wavelength in nm.

The weight per surface unit, g/m^2, of the fabrics was determined according to ASTM D3776 [132] and thickness was assessed through the ISO 5084 standard [21].

The shrinkage of the fabrics after laundering was determined according to ISO 3759:2011 [132]. The extraction of waxes and pigments was conducted using a soxhlet apparatus. A duplicate of 10 g of the sample was subjected to petroleum ether in the soxhlet for a duration of 4 hours. The resulting petroleum ether was then evaporated to obtain a yellowish and transparent oily residue. The extracted cotton was dried at room temperature and subjected to another 4 hours of treatment in the soxhlet apparatus, this time with ethanol 95 %. This treatment facilitated the extraction of pigments that are soluble in polar solvents [133].

Following the evaporation of the petroleum ether and ethanol, the residues collected were dried and analyzed using Fourier Transform Infrared Analysis-Attenuated Total Reflection (FTIR-ATR). A Nicolet 6700 FTIR spectrometer (Thermo Scientific, Waltham, MA, USA) with a SmartOrbit Diamond ATR accessory was used to determine the spectra of the extracts. Thirty-two scans were completed in the 400-4000 cm^{-1} range, at a spectral resolution of 2 cm^{-1}.

3.5.1.4 Results

Figure 76 illustrates the appearance of the samples obtained after each washing cycle. The upper part of the figure displays the scanned images of the actual samples, while the lower part represents the simulated RGB colors derived from the CIE*L*a*b** results [134].

Picture of fabric samples in function of the number of washes. (up: scanner of real samples; down: simulated color based on *L*a*b** results).

Figures 78, 79 and 80 contain the results of *L**, *a* and b**, respectively, obtained from the colorimetric determinations of each sample. *L** corresponds to the brightness of the sample, *a** corresponds to the *a** axis (green, red) and *b** corresponds to the *b** axis (yellow, blue) in the CIE*L*a*b** space.

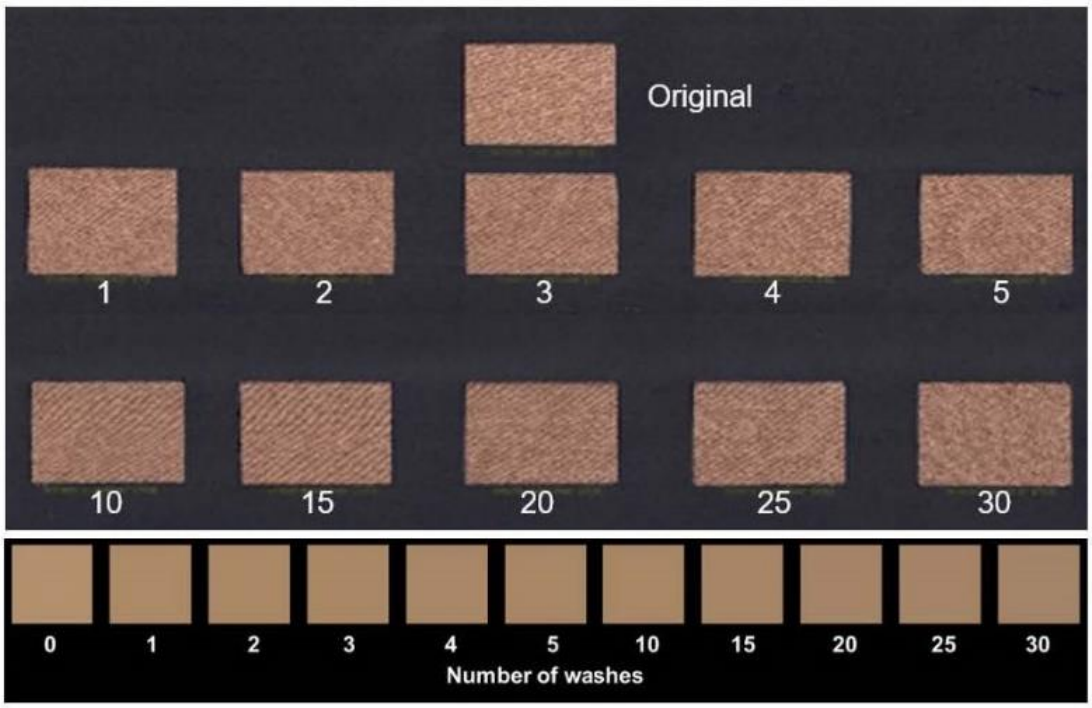


Figure 76. Picture of fabric samples in function of the number of washes. (up: scanner of real samples; down: simulated color based on L*a*b* results).

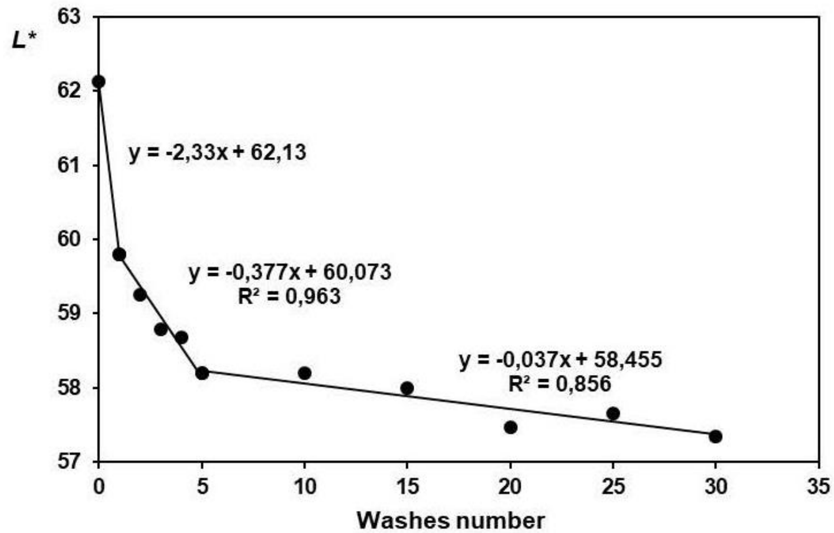


Figure 77. Evolution of *L** in function of the number of washes.

Contrary to what typically occurs in industrially dyed cotton, the color of the samples becomes darker with each wash, as evidenced by the decrease in the lightness (*L**) of the samples with the number of washes (Figure 77).

The initial washing produces the greatest loss of brightness, with the slope of lightness loss of 2.33 *L** units/wash. Lightness decreases further during subsequent washes, where the lightness loss rate is 0.38 *L** units/wash and then again between five and thirty washes, with a lightness loss rate of 0.04 *L** units/wash.

The change in a* value in relation to the number of washes is depicted in Figure 78. Initially, there is a decrease in a* value which indicates a loss of red color. However, after five washes, this decrease becomes minimal or non-existent. Therefore, some amount of red color is lost during the first few washes.

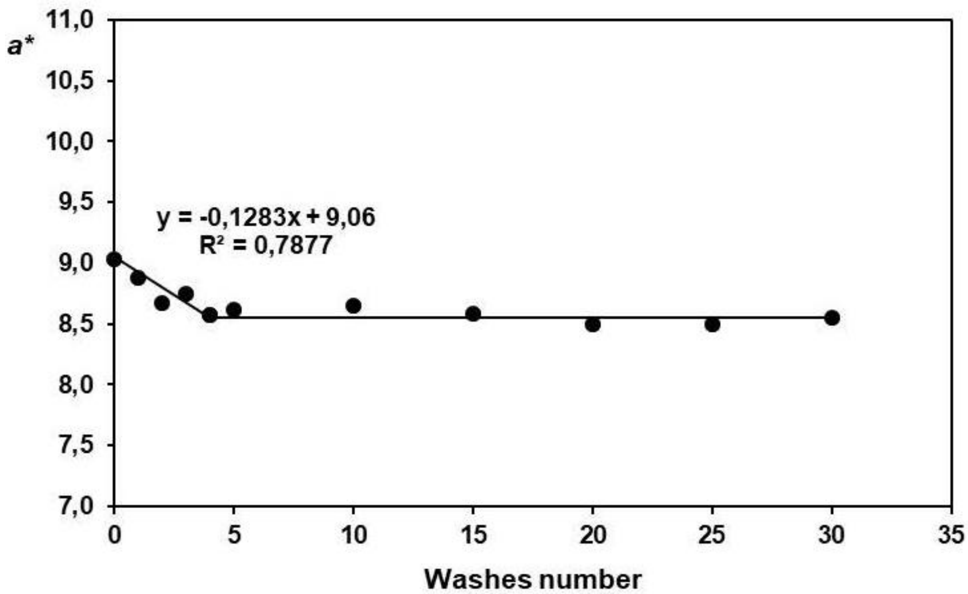


Figure 78. Evolution on *a** in function of the number of washes.

Evolution on *a** in function of the number of washes.

The relationship between the number of washes and the evolution of the *b** parameter is depicted in Figure 79. The *b** parameter ranges from yellow (+*b**) to blue (-*b**). A positive value of *b** indicates the presence of yellow color in this parameter, which linearly diminishes with each home washing.

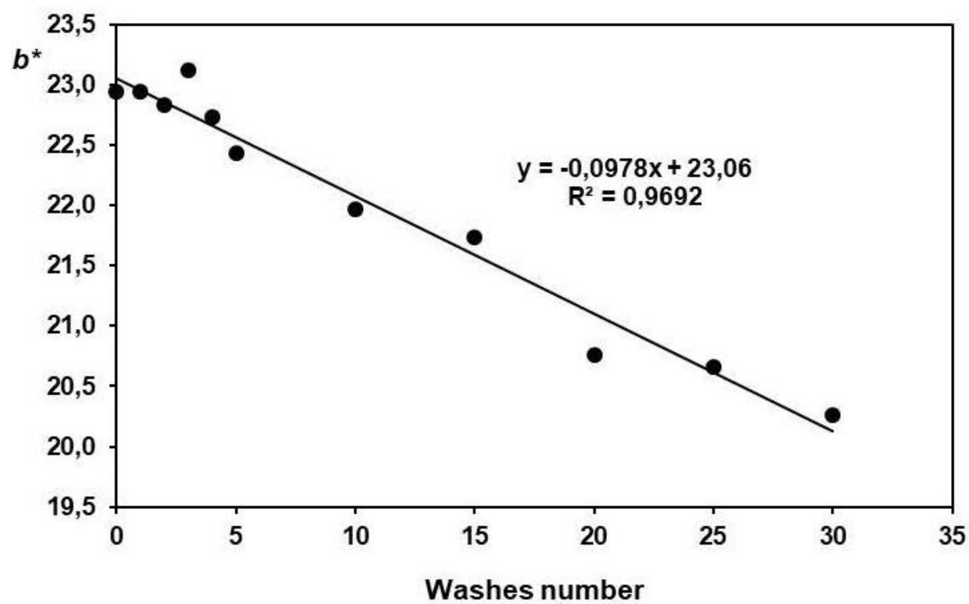


Figure 79. Evolution of *b** in function of the number of washes.

Consequently, the substrates experience a loss of the red and yellow colors, essential components of the brown color. To gain a more comprehensive understanding of the significance behind the decrease in both a^* and b^*, we will delve into the definition of chroma or saturation (C*) within the CIE$L^*C^*h^*$ color space. This color space, similar to CIE$L^*a^*b^*$ but utilizing cylindrical coordinates instead of rectangular coordinates, is preferred by certain professionals in the industry due to its strong correlation with human color perception.

$$C^* = \sqrt{a^2 + b^2} \qquad \text{(Eq. 15)}$$

After completing the relevant calculations and plotting *C** as a function of the number of washes (Figure 80), it becomes clear that there is a linear decrease in saturation with each subsequent wash. However, visually the intensity of the remaining colors increases, leading to a decrease in luminosity.

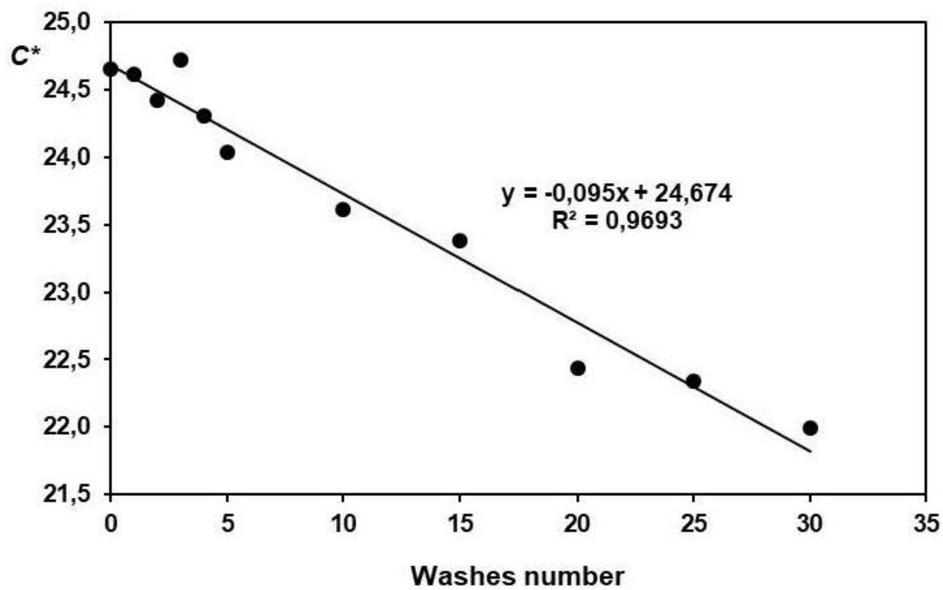


Figure 80. Evolution of *c** in function the number of washes.

Finally, ΔE^*_{ab} corresponds to *CIEL*a*b** color differences, all of them between washed and unwashed fabric (reference). The change in color difference, ΔE^*_{ab}, as the number of washes increases, is depicted by three distinct lines with different slopes, as shown in Figure 81. The first line corresponds to the color change caused by the initial wash. This represents the most significant difference in color between the two samples (ΔE^*_{ab} = 2.34) and can be attributed to the removal of dirt and waxes, as well as the pronounced effect on pigments under the washing conditions. In the second range ($y = 0.3878x + 2.0517$; $R^2 = 0.966$), which spans from one to five washes, there is a notable decrease in the rate of color change (from 2.34 units per wash in the first range to 0.39 units per wash in the second range). This indicates that there is some activity or influence on the substrate that leads to a significant change in color difference, although not as substantial as in the first wash. Finally, from five to thirty washes, although there is still a color change, the variation in color differences is minimal, with a velocity of color change of 0.06 units per wash ($y = 0.0581x + 3.7$; $R^2 = 0.896$).

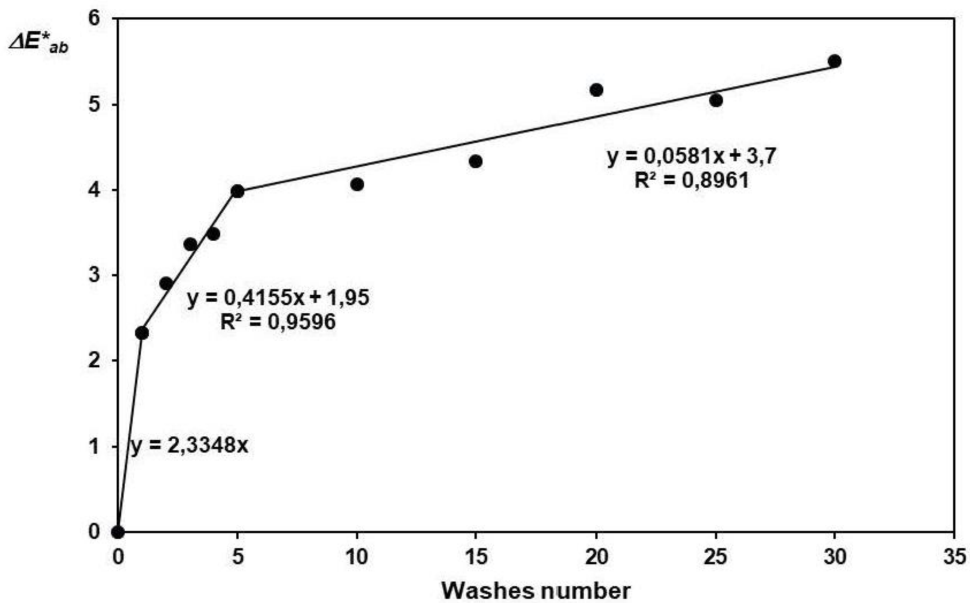


Figure 81. Color difference between ΔE^*_{ab} in the function of the number of washes.

Upon comparing the color differences between a sample and its preceding one, it is evident that the first wash causes a substantial color change (ΔE^*_{ab} = 2.33), aligning with the previously mentioned notable color loss. Nevertheless, the average color variation among the subsequent samples (compared to their respective previous samples) remains relatively stable ((ΔE^*_{ab} 0.53 units/wash)) and is challenging to perceive with the human eye. However, these differences become significant when the cumulative impact of minor color variations after each wash is considered. When we merge the initial samples, following one wash, five washes and thirty washes, into a unified figure (Figure 82), it becomes evident that the visual disparities are already noticeable.

Figure 82. Comparison between the color of original sample and washed 1,5 and 30 times.

To further comprehend the underlying causes of this color change, which results in reduced luminosity and diminished red and yellow hues, several parameters will be examined.

Figure 83. Wastewater of the four first washes.

As depicted by Figure 83, a significant amount of color is lost during the initial washes. However, the color of the fabric shows a marked increase during this stage, in contrast to the higher color intensity observed in the washing water. This paradoxical finding may be explained by two competing phenomena that could be occurring concurrently. The first of these mechanisms includes the removal of waxes, dirt and largely polar solvent-soluble pigments during the first wash. The second phenomenon involves the shrinkage of the cotton fabric during washing, which induces an increase in coloration.

In order to gain a better understanding of what happens to pigments during the initial wash, samples of Dark ruby cotton, which is known for being the darkest cotton, were subjected to the same washing conditions as previously described in order to assess the removal of washes and pigments in the first wash. Following conditioning, the original and washed cotton samples were extracted using petroleum ether and subsequently with 95 % ethanol, as outlined in the characterization section.

Upon evaporation of the petroleum ether, a transparent oily residue, representing the cotton waxes, was obtained and characterized using ATR-FTIR (Figure 84). It can be inferred from the obtained spectra that they correspond to waxes and its analysis revealed no significant differences between the original and washed samples. This indicates that the washing process does not have a significant impact on the composition of the waxes.

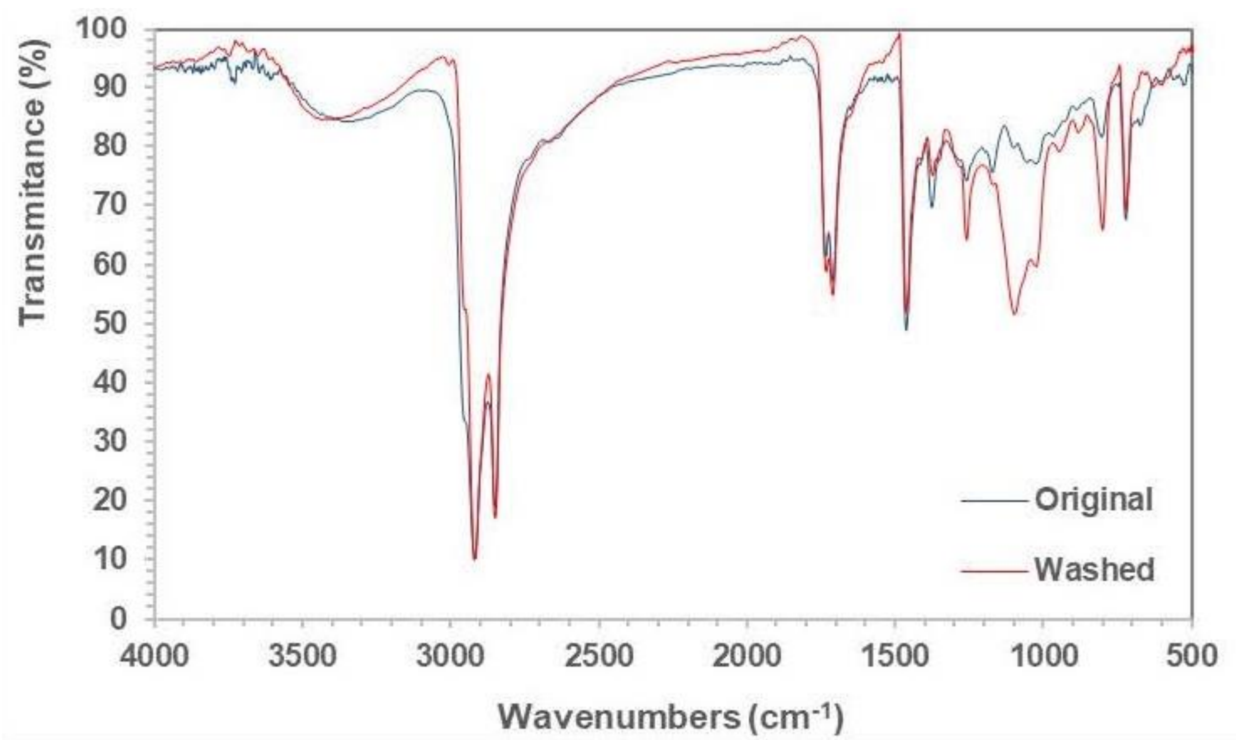


Figure 84. ATR spectra of petroleum ether extraction for original cotton (blue) and washed Ruby cotton (red).

Following the extraction with petroleum ether, the extraction with 95 % ethanol is carried out on the same yarn. After this last extraction, the extracts were subjected to evaporation and concentration. The resulting residue was then analyzed using ATR-FTIR, as depicted in Figure 85.

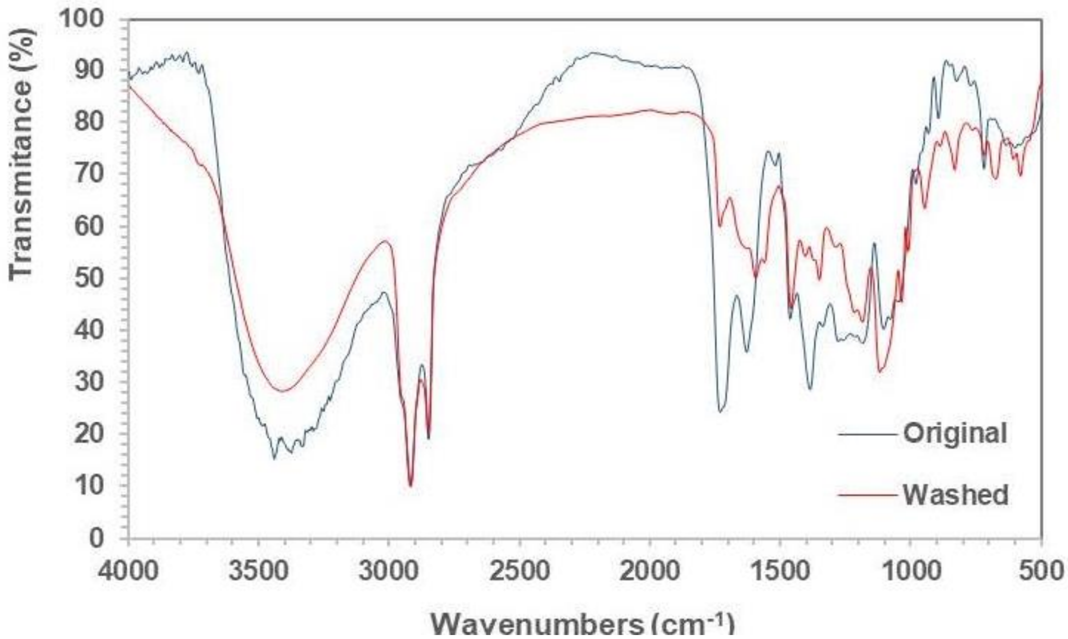


Figure 85. ATR spectra of the ethanol extraction for original cotton (blue) and washed Ruby cotton (red)

ATR spectra of the ethanol extraction for original cotton (blue) and washed Ruby cotton (red)

The two spectra acquired are notably dissimilar and while this method's ability to identify them is limited, it is evident that the ethanol-extracted substance (polar solvent-soluble pigments) varies between the two specimens examined. Thus, it has been established that the washing process results in the loss of pigments that are detectable in the washing water. Nevertheless, the extract's evaporation and measurement indicate that it is less than 1 %.

After providing a rationale for the loss of pigments in the fabric, the cause behind the darker appearance of the fabrics, despite the minor loss of pigments, will be determined. It is widely acknowledged that cotton undergoes shrinkage during the washing process. Therefore, this study aims to explore the relationship between fabric shrinkage and its impact on color. The changes in dimensional stability, specifically in the warp (COT, conventional cotton) and weft (NaCOC) directions are visually depicted in Figure 86. Notably, there is a significant shrinkage of 8.3 % and 5.3 % in the warp and weft directions, respectively, after the initial wash. Consequently, the substantial increase in darkness observed in the fabric samples after the first wash can be attributed to this considerable initial shrinkage.

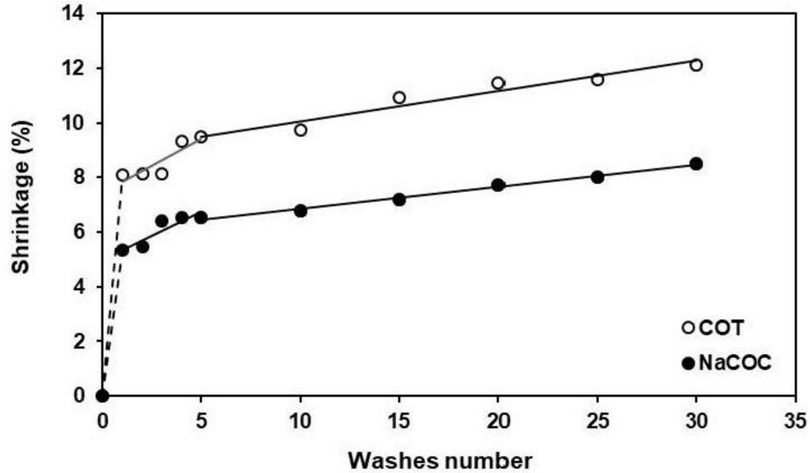


Figure 86. Shrinkage in the warp (COT) and weft (NaCOC) directions is a function of the number of washes.

After the initial significant reduction in the size of the sample, two distinct responses occur. The first one takes place between one and five washes, during which the fabric experiences a 0.35 % increase in shrinkage in both directions. Although this increase is less significant than the initial decrease, it is still greater than the final response. In the final response, the shrinkage increases linearly with the number of washes at a rate of 0.12 % (R^2 = 0.943) in the warp direction and 0.08 % (R^2 = 0.991) in the weft direction.

Figure 87 examines the relationship between the luminosity of the fabric, L* and the shrinkage. It reveals a strong linear correlation between the decrease in luminosity and the increase in shrinkage.

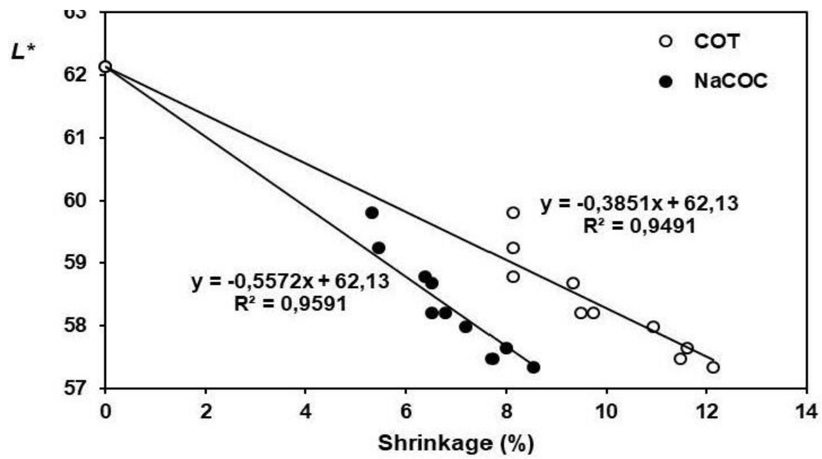


Figure 87. Variation of L* with shrinkage.

However, it should be noted that there is no correlation between the decrease in red (a*) and yellow (b*) values and the shrinkage of the fabric. This implies that the observed color change in the fabrics is a result of the fabric's brightness being altered due to shrinkage during the washing process.

In Table 26, the changes in UV-A, UV-B and UPF (Ultraviolet Protection Factor) are presented in relation to the number of washes. It is clear that there has been a notable rise in UPF during the first five washes. The UPF value has increased from 281 to 412 within two washes and, subsequently, it stabilizes at a relatively consistent fluctuating level between 450 and 540. This finding confirms that the most significant impact and alterations occur during the first five washes.

Table 26. Evolution of UVA, UVB, UPF, areal density and thickness of the samples in function of the number of washes.

Washes	0	1	2	3	4	5	10	15	20	25	30
UVA (%)	0.31	0.21	0.20	0.17	0.17	0.19	0.18	0.16	0.17	0.16	0.17
UVB (%)	0.37	0.27	0.26	0.23	0.23	0.23	0.23	0.23	0.21	0.21	0.24
UPF	281	402	412	473	474	499	453	479	541	530	445
Weight per m^2 (gr/m^2)	314	345	352	355	357	357	363	366	369	365	373
Thickness (µm)	7.23	8.93	8.90	8.87	9.23	9.10	9.37	9.37	9.67	9.27	9.70

It is well known that the relationship between the UPF (Ultraviolet Protection Factor) of fabric and its color can vary depending on several factors, including the type of fiber, the thickness of the fabric, the fabric design and the dyeing method. Thus, in the substrates studied, only two parameters can affect: the thickness of the fabric and the color (related to the type of dyeing in general terms).

Regarding the thickness of the fabric, in general, the denser the fabric, the higher its ability to block UV rays. Thicker fabrics may have a higher UPF regardless of color [135]. Algaba established that the weight per surface unit and thickness of the fabrics are structural parameters that are strongly correlated with UPF. An increase in any of these parameters leads to an increase in UPF. To examine this phenomenon, the values of areal density and fabric thickness have also been included in Table 3. It can be observed that both parameters increase with the number of washes, which is a response to the shrinkage of the fabrics that occurs during washing. Hence, it is evident that the UV protection of the fabrics examined is significantly impacted by shrinkage.

On the other hand, the color of the fabric can affect its ability to block UV rays to some extent. Algaba [119] concluded that the diffuse transmission of ultraviolet radiation through fabrics decreases as the color intensity of the fabrics increases. As transmission decreases, the UPF increases with the intensity of the dye. Dark colors tend to absorb more UV radiation than light colors, which can result in a slightly lower UPF for dark fabrics. However, this difference is usually relatively small and may be insignificant compared to other factors such as fiber type and fabric thickness. In this study, the decrease in brightness should result in a decrease in UPF. However, as this does not happen, it once again confirms that the shrinkage that occurs, especially in the first wash, is the driver of the darkening phenomenon that takes place during washes and in the increase of UPF protection.

3.5.1.5 Conclusions

The impact of home washing on the colorimetric properties of naturally colored organic cotton (NaCOC) fabrics has been investigated, revealing that washing can have a substantial effect on the visual characteristics of these textiles, particularly in terms of lightness and saturation. The initial wash is found to be the most influential in terms of color modification, although subsequent washes up to the fifth also exhibit a noticeable impact. During the first wash, two simultaneous phenomena occur. Firstly, the pigments present in NaCOC are extracted and transferred to the wash water, resulting in a reduction in the saturation components, a* and b*, of the CIE*L*a*b** coordinates. Secondly, the fabric undergoes shrinkage, leading to a darkening of the color. Considering the overall behavior, it can be inferred that the darkening of the fabric due to shrinkage is more significant than the loss of pigments, making it the dominant phenomenon in household washing. Furthermore, this shrinkage also contributes to a significant increase in the fabric's ultraviolet protection factor (UPF).

Acknowledgments: The authors would like to express their gratitude to Mr. Francisco José Barahona, Mr. Oliver García, Mrs. Montserrat Guerrero, Mr. Ferran Parés and Mrs. Zurine Ramírez for their assistance in the experimental work.

3.5.2 Evaluating the Impact of Washing Conditions on the Colorfastness of Naturally Colored Cotton Fabrics: A Focus on Detergents, Water Types and Temperature

3.5.2.1. Abstract

Intrinsically colored cotton is crucial for sustainability as it eliminates the need for chemical dyes, reducing water pollution and carbon footprint. It also preserves biodiversity by using fewer pesticides and supports eco-friendly, ethical product creation.

This research aims to examine the factors that influence the color change that occurs in naturally colored organic cotton (NaCOC) fabrics when washed under normal household conditions, focused in special detergents designed for people with skin hypersensitivity.

The study studies the impact of various washing conditions on the colour changes of the fabrics. Specifically, three specific detergents, two types of water (tap and distilled), and three different temperatures (20, 40, and 60°C) are taken into consideration as variables. By using colorimetric measures and correlating the results with the significant variables of the experimental design, the study evaluates how washing practices affect both the colour and the overall integrity of the fabric. The findings demonstrate that the water hardness is the most influential variable when it comes to the color changes in the fabrics. Additionally, higher washing temperatures exacerbate color changes, particularly in hard water conditions. These results provide valuable insights for maintaining the color integrity of NaCOC fabrics during washing.

Keywords: Natural-coloured organic cotton, household washing, color changes

3.5.2.2 INTRODUCTION

Cotton accounts for 22% of global textile fiber production and is the leading natural fiber in consumption due to its exceptional textile qualities and comfort [136]. In contemporary times, the growing concern for ecological issues has increased the fascination with naturally colored organic cotton (NaCOC). According to archaeological findings, this fiber has been used for at least 4,500 years [37, 97, 128, 137-138]. However, its applications in the industrial field remain relatively limited.

The extensive cultivation of NaCOC on a larger scale can be largely attributed to the advancements made by Sally Fox, a Californian entomologist. While working as an assistant to a producer [41], Fox first discovered wild brown cotton seeds and developed genetically resistant strains [40]. Although Fox was enthusiastic about colored cotton fibers, their poor textile quality posed a significant challenge for the spinning process. As a result, Fox focused on improving the textile properties of the fibers. Using careful selection and cross-pollination techniques, Fox developed colored cotton hybrids in 1988 that had fibers long enough to be spun using contemporary machinery. The success of cultivating naturally colored, spinnable cotton fibers eventually led Sally Fox to establish Natural Cotton Colors Inc. Additionally, she obtained a plant variety protection certificate for the cotton and registered it as FoxFiber.

The main appeal of NaCOC lies in its organic agricultural production, which eliminates the need for dyeing, thus avoiding the use of chemicals and water, resulting in a marked reduction of its environmental impact. The organic production of NaCOC contributes to agricultural sustainability by minimizing the use of pesticides and chemical fertilizers, promoting healthier practices for the environment and farmers. Furthermore, the absence of pesticides in the cultivation of the plant, along with the absence of chemicals associated with dyeing and finishing processes, makes garments derived from these fibers especially suitable for babies and people with hypersensitive skin and multiple chemical sensitivity (MCS). Determining the precise prevalence of MCS is challenging, as it varies significantly between different studies. Prevalence rates tend to be lower when patients undergo medical evaluation and can range from less than 1% to 33% in various studies conducted in Europe [139].

Grigorev et al. conducted a study analyzing the differences in Metabolites of White and Brown Naturally Colored Cotton in relation to their implications for Biofunctional and Aseptic Textiles. The findings on the properties of bioactive metabolites suggested therapeutic, delicate aseptic, repellent, UV protective, and metal toxicity reducing effects of the studied fibers, as well as their resistance to biodestruction by pecto- and cellulolytic bacteria and mold fungi. This would make biofunctional textiles more comfortable and hygienic [140].

To date, several studies have been conducted on the color changes experienced by fabrics made from NaCOC, focusing especially on the effects of different pre-treatments for industrial dyeing with chemical dyes (scouring, bleaching, etc.), as well as, though less extensively, on the effects of domestic washing. These studies have revealed that washing can have a substantial effect on the visual characteristics of these textiles, particularly in terms of lightness and saturation [40-41, 45, 141-145].

In a previous paper [146], authors studied how household washing influences the colorimetric characteristics of natural colored organic cotton (NaCOC) fabrics after undergoing washing for up to 30 cycles. Washing significantly changed lightness and saturation of samples, being the most significant difference in color between the two samples after the initial wash. Between the second and fifth washes, there are notable differences in these parameters. However, from the fifth wash, the variation in color difference was minimal. Additionally, the FTIR-ATR analysis of the extracts in petroleum ether and subsequently in ethanol of the NaCOC fabrics, before and after home washing, in conjunction with a comparison with shrinkage, demonstrated that the latter process was accountable for the darkening of the sample.

The present study examines the color changes in NaCOC garments during the first five home washes, excluding industrial bleaching impacts, focusing exclusively on the changes that fabrics undergo during the usage phase of these garments by a broad group of users who have high skin sensitivity and therefore are required to use only specific commercial detergents for this condition. This study builds on previous [146] focusing on how different detergents, water types, and temperatures affect the colorfastness of NaCOC fabrics under household washing conditions, aiming to enhance their durability and consumer satisfaction . By evaluating how these specific detergents affect the characteristics of NaCOC, valuable information can be obtained on the durability and maintenance of these textiles. This, in turn, promotes more conscious and sustainable consumption, as garments that maintain their visual and functional properties for longer reduce the need for frequent replacement, thereby decreasing the environmental impact associated with textile production. Ultimately, this study contributes to a broader understanding of how sustainable textiles can be effectively integrated into the daily lives of consumers, fostering more ecological and responsible practices in the fashion industry.

3.5.2.3 MATERIALS AND METHODS

Material

This study employed naturally colored organic cotton (NACOC) from Brazil, supplied by the Spanish company Organic Cotton Colours (OCC) [147]. The yarn used in this study have been produced from a selection of OCC cotton varieties, including Topaz, Light Ruby, Dark Ruby and Raw (white). The characteristics of these unique varieties are listed inTable 27.

Table 27. Characteristics of the NaCOC fibers used.

Sample	SCI	MIC	MAT	UHML (mm)	UL (%)	SF (%)	STR (g/tex)	ELG (%)
Topaz	84	4.23	0.84	26.67	79.5	9.5	25.0	8.6
Light ruby	-	4.26	0.84	20.50	78.8	11.9	19.6	9.9
Dark ruby	-	4.15	0.84	21.01	79.2	12.0	21.7	9.0
Raw	99	3.76	0.84	25.73	78.0	10.3	26.3	7.0

were:

SCI: Spinning Consistency Index. It is a parameter for predicting the spinnability of fibers. In general, the higher the index, the higher the yam strength and the better the overall fiber spinnability.

MIC: Micronaire Index. The pressure drop was measured by passing air through the fibers.

MAT: Maturity index. This indicates the degree of cell wall thickness in the cotton samples. The HVI correlates very well with the AFIS Maturity Ratio and microscopy reference method (cross-sectional analysis).

UHML: upper half mean length. It is the mean length by the number of fibers and corresponds to the class’s staple length and the AFIS Upper Quartile Length by weight.

UL%: Uniformity Index. It expresses the ratio of the mean length to the upper half mean length. This indicates the distribution of the fiber length within the fibrogram.

SF%: Short Fibre Index. Indicates the percentage of fibers less than 12.7 mm in length. This correlates well with the AFIS short-fibre content by weight (SFC).

STR: Bundle strength. is the breaking strength of the cotton fibers in grams per tex.

ELG: Elongation. This is a measurement of the increase in the length of the cotton test piece, expressed in %, as a result of the application of a load.

With these Fibers, the OCC company made a mixture in equal proportions with the four cotton samples described in Table 1 and proceeded to carry out a combed spinning process (opening, cleaning, carding, drawing, combing, drawing and finally, roving machine). Ring spinning was carried out on a PINTER machine model Merlin Spa 1803, under the conditions indicated in Table 28, which includes the characteristics of the yarn obtained.

Table 28. Spinning conditions and characteristics of the yarn obtained.

Type of ring spinning machine	PINTER model Merlin spa1803
Roving count (ktex)	0.600
Yarn count (tex)	29.5
Twist (t/m)	705
Linear spinning speed	16.65 m/min
Revolutions per minute of the spindle	7,500 rpm
Ring diameter	42 mm
Type of traveller	Bräcker 2DR

The woven fabric used in this study was constructed using a conventional cotton warp (raw white) and a NaCOC weft produced on an air-jet Dornier loom combined with a Staübli Jacquard machine. The fabric exhibited a five-satin weave pattern so that the NaCOC weft threads emerge better on the surface of the fabric, the hems are distributed in a more regular way and in this way the color of the fabric is dominated by the high quantity of NaCOC weft yarns.The density is 38.4 warp yarns per centimeter (2/30 Nm), 19 weft picks per centimeter (2/34 Nm), and total fabric width of 146 cm. The areal density is 314 g/m^2. No other treatment has been applied to the fabric.

Domestic wash

This study evaluated the cleaning effectiveness of three commercial detergents: Fox Fibre® Colorganic® (Det A, [148]), Klar (Det B, [149]) and Pure Nature (Det C, [150]). All of them are designed for washing delicate textiles and are compounded to effectively remove dirt and stains while safeguarding textile fibers. These detergents have a minimal environmental impact, Table 29.

Table 29. Detergent infirmation

Reference	Composition
Det A	Citrus Grandis Fruit Extract Ammonium Lauryl Sulfate Cetyl Alcohol EO Soyamide DEA Sodium Oleate Other ingredients: hydrolyzed cotton protein, sodium benzoate, potassium sorbate, citric acid, curcuma zedoaria, CL 16035
Det B	15 % - 30 % soap (soap and vegetable soap*) <5% non-ionic surfactants (carbohydrate surfactants). Other ingredients: water, ethanol, sodium citrate, lactic acid, citric acid. 100% of the total ingredients are of natural origin. * From controlled organic farming.
Det C	15 % - 30 % anionic surfactants 5-15% non-ionic surfactants 5-15% plant alcohol Other ingredients: water, potassium soap of rapeseed, oil (organic), sodium lauryl sulfate, alcohol denatured, lauryl glucoside, octyl sulfate

The household wash was applied to fabric samples measuring 30 × 30 cm. The tests were carried out following the concentration instructions provided by the producers for the three different detergents: 15 ml of Det A, 30 ml of Det B and 30 ml of Det C. The substrates were washed five times (X1) using either distilled water or tap water (X2) at 20, 40 and 60°C (X3). The hardness of the tap water has been 400 mg $CaCO_3$/L. The duration of the washing cycle varied with the temperature: 50 min at 20 °C, 60 min at 40 °C and 80 min at 60 °C.

After washing, the fabric specimens were dried using a Frommer Dry 92 tumble dryer. Subsequently, they were conditioned in a standardized environment maintained at 20 °C and 60% relative humidity for 24 hours.

characterization

Color changes were determined by colorimetry. Colorimetric measurements were performed using a GRETAGMACBETH COLOR I7 instrument conforming to the ISO 105-J03 standard [151] with measurements taken under a D65 illuminate and a 10-degree observer angle.

The degree of color change between each laundered fabric sample and its original state was determined using the color difference parameter, ∆E* (Eq. 16).

$$\Delta \mathrm{E} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad \text{(Eq. 16)}$$

where *L**, *a** and *b** are the lightness, green–red coordinate and blue-yellow coordinates, respectively, in the *CIEL*a*b** space.

The shrinkage of the fabrics after laundering was determined according to ISO 3759:201 [152].

The color difference response was analyzed by modelling with quantitative and qualitative variables as indicated in Table 30.

Table 30. Experimental plan variables and levels.

<table>
<tr><td rowspan="10">Quantitative variables</td><td rowspan="5">X1</td><td rowspan="5">Washes number
(5 levels)</td><td>1</td><td>-2</td></tr>
<tr><td>2</td><td>-1</td></tr>
<tr><td>3</td><td>0</td></tr>
<tr><td>4</td><td>1</td></tr>
<tr><td>5</td><td>2</td></tr>
<tr><td rowspan="2">X2</td><td rowspan="2">Type of water
(2 levels)</td><td>Distilled
(0 ppm hardness)</td><td>-1</td></tr>
<tr><td>Tap
(400 ppm hardness)</td><td>1</td></tr>
<tr><td rowspan="3">X3</td><td rowspan="3">Temperature
(3 levels)</td><td>20</td><td>-1</td></tr>
<tr><td>40</td><td>0</td></tr>
<tr><td>60</td><td>1</td></tr>
<tr><td rowspan="3">Qualitative variables</td><td rowspan="3" colspan="2">Type of detergent</td><td colspan="2">Det A</td></tr>
<tr><td colspan="2">Det B</td></tr>
<tr><td colspan="2">Det C</td></tr>
</table>

Categorical variables were used to introduce the detergent variable into the model, taking on the values specified in Table 31, for each level (Det. A, Det B, Det C).

When all categorical variables take a value of zero, the model will be estimated for detergent A. This detergent will be the reference and the model will indicate if there are significant differences in the other types of detergent. If in the final model, there are terms involving Q2 and/or Q3, it will indicate that there are statistically significant differences for the different types of particles.

By substituting the values of Q2 and Q3, separate models can be obtained for the different types of detergents.

Table 31. Categorical variables for each level.

	Level		
Categorical variable	**Det A**	**Det B**	**Det C**
Q2	0	0	1
Q3	0	1	0

3.5.2.4 RESULTS

Figure 88 summarizes the reflectance results for the substrates washed with all specified detergents, using both distilled and tap water at 60 °C.

Noticeable reflectance changes were primarily observed during the first washing cycle. Tables 32, 33 and 34 include the *CIEL*a*b** coordinate values derived from the reflectance measurements for washing with detergents A, B and C, respectively.

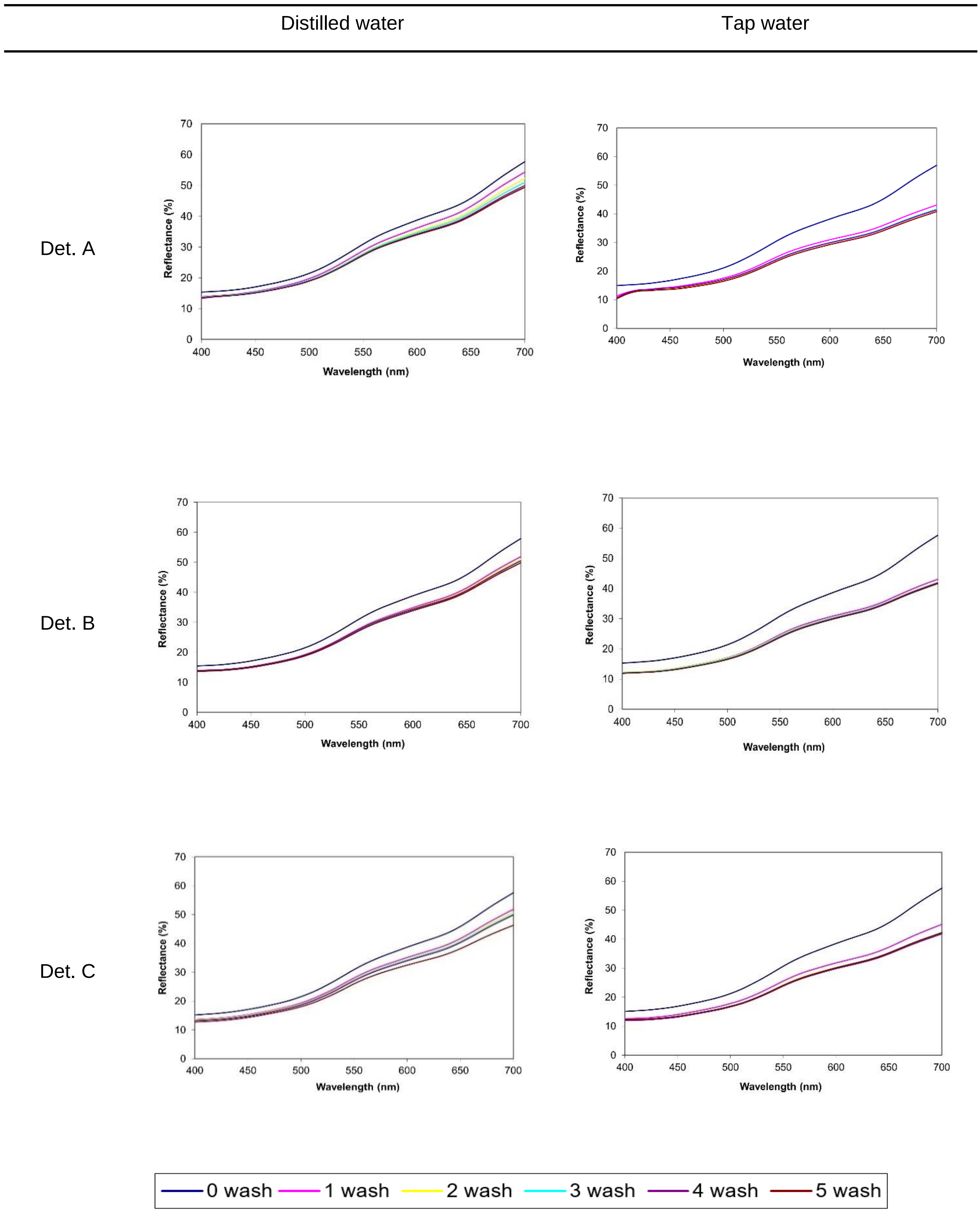


Figure 88. Reflectance curves of the substrates washed with detergents, distilled water and tap water at 60 °C.

Table 32. *CIEL*a*b** coordinate values before and after washing with detergent A under different conditions.

Type of water	T (°C)	Wash number	L*	a*	b*	C*ab	hab	∆E*ab
Distilled	20	0	62.15	9.23	23.14	24.92	68.25	
		1	59.46	9.35	23.33	25.14	68.16	2.70
		2	59.28	9.23	22.77	24.57	67.94	2.89
		3	59.27	9.11	22.71	24.47	68.15	2.92
		4	59.11	9.02	22.42	24.17	68.08	3.14
		5	58.80	9.04	22.60	24.34	68.20	3.40
	40	0	62.43	9.22	23.04	24.82	68.20	
		1	59.94	9.16	22.88	24.64	68.17	2.49
		2	59.94	9.08	22.62	24.38	68.12	2.52
		3	59.67	9.22	23.09	24.86	68.23	2.75
		4	59.46	9.25	22.81	24.62	67.93	2.98
		5	59.40	9.21	22.83	24.62	68.04	3.04
	60	0	62.31	9.29	23.20	24.99	68.18	
		1	60.44	9.30	23.28	25.06	68.23	1.87
		2	59.68	9.17	22.70	24.48	68.01	2.68
		3	59.49	8.90	22.16	23.88	68.11	3.03
		4	59.18	8.78	22.06	23.74	68.29	3.37
		5	59.08	8.69	21.82	23.49	68.29	3.56
Tap	20	0	62.31	9.21	23.01	24.78	68.19	
		1	58.75	8.81	22.12	23.80	68.29	3.69
		2	57.81	8.55	21.43	23.07	68.26	4.82
		3	57.96	8.44	21.03	22.66	68.12	4.84
		4	57.33	8.46	20.86	22.51	67.93	5.48
		5	56.97	8.53	20.81	22.49	67.73	5.82
	40	0	62.17	9.01	22.90	24.61	68.52	
		1	58.52	8.73	22.00	23.67	68.37	3.77
		2	57.65	8.44	21.07	22.70	68.18	4.91
		3	57.68	8.37	20.75	22.38	68.03	5.02
		4	57.12	8.37	20.28	21.94	67.58	5.72
		5	57.15	8.23	19.76	21.41	67.40	5.98
	60	0	61.93	9.22	23.24	25.00	68.36	
		1	56.82	8.34	20.22	21.88	67.58	6.00
		2	55.99	8.37	19.85	21.54	67.15	6.89
		3	56.05	8.35	19.51	21.22	66.83	7.02
		4	55.97	8.34	19.35	21.07	66.69	7.18
		5	55.39	8.48	19.51	21.27	66.52	7.57

Table 33. *CIEL*a*b** coordinate values before and after washing with detergent B under different conditions.

Type of water	T (°C)	Wash number	*L**	*a**	*b**	C^*_{ab}	h_{ab}	ΔE^*_{ab}
Distilled	20	0	62.24	9.30	23.17	24.97	68.14	
		1	59.66	9.26	22.97	24.77	68.05	2.58
		2	59.18	8.99	22.81	24.52	68.48	3.09
		3	58.86	9.08	23.02	24.75	68.47	3.39
		4	59.14	8.96	22.85	24.54	68.60	3.13
		5	58.99	9.13	23.10	24.84	68.43	3.25
	40	0	62.29	9.28	23.28	25.06	68.26	
		1	59.87	8.97	22.75	24.45	68.48	2.49
		2	59.73	8.99	23.09	24.78	68.73	2.58
		3	59.14	8.93	23.20	24.86	68.94	3.17
		4	58.91	8.89	22.84	24.51	68.74	3.43
		5	58.86	8.97	23.04	24.72	68.73	3.45
	60	0	62.39	9.23	23.07	24.85	68.19	
		1	59.56	9.07	22.57	24.32	68.10	2.87
		2	59.39	8.90	22.41	24.11	68.33	3.08
		3	59.17	8.85	22.40	24.09	68.44	3.31
		4	59.33	8.72	22.16	23.82	68.52	3.23
		5	58.91	8.82	22.08	23.77	68.21	3.63
Tap Tap	20	0	62.20	9.28	23.18	24.97	68.19	
		1	59.00	8.80	22.41	24.08	68.55	3.33
		2	58.00	8.73	22.45	24.08	68.74	4.31
		3	57.99	8.49	22.03	23.61	68.94	4.43
		4	57.58	8.46	22.03	23.60	68.99	4.83
		5	57.67	8.42	22.08	23.63	69.13	4.74
	40	0	62.11	9.31	23.06	24.87	68.01	
		1	58.27	8.51	21.88	23.48	68.75	4.09
		2	57.27	8.30	21.69	23.22	69.06	5.13
		3	56.89	8.40	21.73	23.30	68.86	5.47
		4	56.81	8.26	21.56	23.09	69.03	5.61
		5	56.53	8.34	21.62	23.17	68.91	5.84
	60	0	62.22	9.33	23.14	24.95	68.03	
		1	56.55	8.33	21.59	23.14	68.89	5.96
		2	56.29	8.22	21.13	22.67	68.75	6.36
		3	56.12	8.33	21.20	22.78	68.55	6.48
		4	55.81	8.42	21.22	22.83	68.35	6.75
		5	55.71	8.46	21.15	22.78	68.19	6.85

Table 34. *CIEL*a*b** coordinate values before and after washing with detergent C under different conditions.

Type of water	T (°C)	Wash number	L^*	a^*	b^*	C^*_{ab}	h_{ab}	ΔE^*_{ab}
Distilled	20	0	62.28	9.16	23.03	24.79	68.32	
		1	59.51	9.60	23.87	25.73	68.10	2.93
		2	59.30	9.44	23.37	25.21	68.01	3.01
		3	59.01	9.32	23.19	24.99	68.10	3.28
		4	59.44	9.16	22.81	24.58	68.12	2.85
		5	58.75	9.34	23.38	25.17	68.22	3.55
	40	0	62.33	9.22	22.94	24.73	68.10	
		1	60.10	9.25	23.15	24.93	68.22	2.24
		2	59.59	9.16	22.80	24.57	68.10	2.74
		3	59.23	9.10	22.71	24.46	68.17	3.11
		4	59.19	9.14	22.70	24.48	68.07	3.14
		5	59.15	9.17	22.76	24.54	68.06	3.19
	60	0	62.32	9.24	23.08	24.86	68.18	
		1	59.75	9.03	22.61	24.35	68.23	2.62
		2	59.27	9.03	22.31	24.07	67.96	3.15
		3	59.09	9.07	22.39	24.15	67.96	3.31
		4	58.87	9.03	22.35	24.11	67.99	3.53
		5	57.84	8.62	21.76	23.40	68.38	4.72
Tap	20	0	62.36	9.16	22.97	24.73	68.26	
		1	58.92	8.69	22.32	23.95	68.73	3.54
		2	58.20	8.46	22.12	23.69	69.06	4.31
		3	57.76	8.59	22.68	24.26	69.25	4.65
		4	57.26	8.44	22.30	23.84	69.28	5.20
		5	57.00	8.51	22.26	23.83	69.09	5.45
	40	0	62.38	9.23	23.01	24.79	68.14	
		1	58.06	8.45	21.95	23.52	68.95	4.52
		2	57.13	8.43	21.69	23.27	68.75	5.47
		3	57.05	8.37	21.63	23.20	68.84	5.57
		4	56.81	8.36	21.73	23.28	68.96	5.78
		5	56.95	8.42	21.85	23.41	68.93	5.62
	60	0	62.12	9.36	23.25	25.06	68.06	
		1	57.41	8.31	21.66	23.20	69.01	5.07
		2	56.13	8.31	21.14	22.71	68.55	6.43
		3	55.89	8.29	21.08	22.66	68.54	6.68
		4	55.78	8.33	20.98	22.58	68.35	6.81
		5	56.03	8.40	20.97	22.59	68.17	6.57

From the results, there were alterations in fabric color when the number of washes was increased under different washing conditions.

The lightness, L*, decreases importantly after the first wash, indicating noticeable darkening. The change in lightness, *ΔL**, in this first wash for the samples, is included in Figure 89.

After this first wash, although there exists a decrease of *ΔL**, the average difference between the second wash and the fifth is 0.65. There are no existing significant differences among the different conditions.

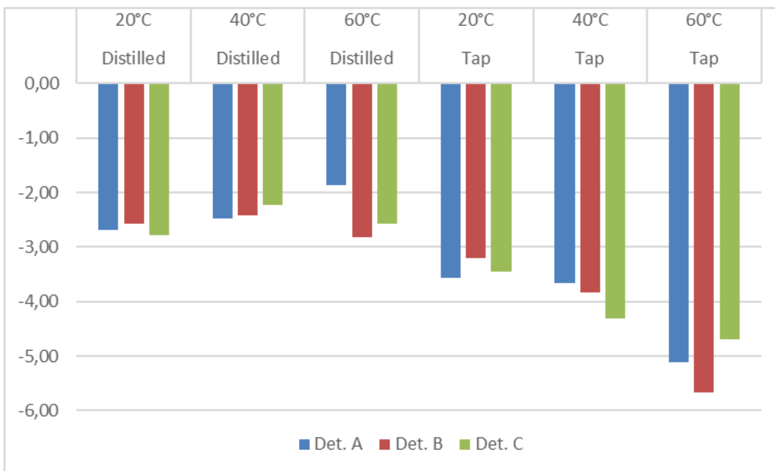


Figure 89. Δ*L** of all the samples after the first wash.

In order to identify accurately the variable or variables that have the greatest impact on the color change in the first domestic wash, the equation 17, model has been employed. In it, the X1 variable is not included, being the studied answer the *ΔL** of the first wash. The beta coefficients have been estimated from this initial model using the linear model, stepwise backward regression, with a significance level (α) of 0.05.

$$\Delta L^* = \beta_0 + \beta_1 \cdot X2 + \beta_2 \cdot X3 + \beta_3 \cdot X2 \cdot X3 + \beta_4 \cdot Q2 + \beta_5 \cdot Q2 \cdot X2 + \beta_6 \cdot Q2 \cdot X3 + \beta_7 \cdot Q2 \cdot X2 \cdot X3 + \beta_8 \cdot Q3 + \beta_9 \cdot Q3 \cdot X2 + \beta_{10} \cdot Q3 \cdot X3 + \beta_{11} \cdot Q3 \cdot X2 \cdot X3 \qquad \text{Eq. 17}$$

The resulting model of *ΔL** of the first wash is as follows:

$$\Delta L^* = -3.329 - 0.837 \cdot X2 - 0.223 \cdot X3 - 0.504 \cdot X2 \cdot X3 - 0.456 \cdot Q3 \cdot X3 \qquad R^2 = 0.945 \qquad \text{Eq. 18}$$

In the model, it has been observed that both variables X2 (type of water) and X3 (temperature) have a significant impact on the luminosity response *ΔL** of the first wash, along with their interaction. The type of water interacts with the temperature and the interaction of detergent B with temperature is also significant. The coefficient of determination for the fit is 94.45%, indicating a strong fit of the estimated model to the experimental data.

Upon substituting the values of the categorical variable Q3, the following models are derived:

$$\Delta L^* = -3.329 - 0.837 \cdot X2 - 0.223 \cdot X3 - 0.504 \cdot X2 \cdot X3 - 0.456 \cdot Q3 \cdot X3 \qquad \text{Eq. 19}$$

Det. A and Det C $$\Delta L^* = -3.329 - 0.837 \cdot X2 - 0.223 \cdot X3 - 0.504 \cdot X2 \cdot X3 \qquad \text{Eq. 20}$$

Det. B $$\Delta L^* = -3.329 - 0.837 \cdot X2 - 0.679 \cdot X3 - 0.504 \cdot X2 \cdot X3 \qquad \text{Eq. 21}$$

The results obtained indicate that the primary factor affecting the decrease in luminosity after the initial wash of NaCOC fabrics is water hardness. The higher the water hardness, the more pronounced the darkening effect. Additionally, although to a lesser extent, temperature also plays a significant role, particularly in the case of detergent B. When using distilled water, the darkening between the washed fabrics and the original one is reduced. However, as the water hardness increases, this difference becomes more significant, especially with higher temperatures.

In order to properly analyse chroma and hue, a prior understanding of the pigments responsible for color is necessary. The brown color in naturally coloured cotton Fibers is primarily due to the presence of tannins pigments [96, 153] and by Peng et al. [154].

Tannins can vary in their solubility in water depending on their chemical structure and molecular weight. Generally, smaller tannin molecules are more soluble in water, while larger tannin molecules tend to be less soluble. The solubility of tannins in water also depends on factors such as pH, temperature and the presence of other solutes.

Then, in the case of naturally colored cotton, some of these tannins present in the Fibers may be soluble and contribute to the color during processing, others may be tightly bound to the cotton fibers and less likely to leach out during washing or other treatments.

The Chroma of the samples (Tables 30, 31 and 32) is described through the *C** value and it, at the same time is related to *a** (green-red axe) and *b** (blue-yellow axe) in the CIE*L*a*b** colors space:

$$C^* = \sqrt{a^2 + b^2} \qquad \text{Eq. 22}$$

The hue (*h*) is also related with *a** and *b** with the following expression:

$$h = arctg\left(\frac{b}{a}\right) \qquad \text{Eq. 23}$$

Figure 90 represents Δa^*, Δb^*, ΔC^*_{ab} and Δh_{ab} for all the detergents, under the different washing conditions. All the differences have been calculated in relation to the unwashed original fabric. Based on the results, in the first wash, there is a decrease in the value of the green-red axis, *a**, which is more pronounced when washed with hard water than with distilled water. Additionally, the figures show a tendency for this decrease to be greater as the temperature of the domestic wash increases. On the other hand, there are no significant differences when increasing the number of washes. In other words, there is a significant change in this value with the first wash, but it does not significantly change further with subsequent washes.

The yellow-blue axis, *b** also experiences a decrease in its value in the first wash, which is more pronounced when washed with hard water than with distilled water. Furthermore, it can be observed in the figures that there is a tendency for this decrease to be greater as the temperature of the domestic wash increases. On the other hand, it seems that in some cases, especially when washing with detergent A, there is a certain decrease in this value as the number of washes increases. In other words, generally, there is a significant change in this axis with the first wash, but it no longer changes or changes very little with subsequent washes.

The decrease in both, *a** (in the positive semi-axis, corresponding to yellow) and *b** (in the positive semi-axis, corresponding to red), indicates a decrease in the colors red and yellow (components of brown).

The decrease in chroma values (ΔC^*_{ab}) follows a similar order and trend as those of *b**, given that the decrease in *a** is much smaller than that of *b**, resulting in the chroma trend being similar to that of *b**.

A decrease in chroma in the fabric after washing refers to a reduction in the intensity or purity of its color. When fabric undergoes washing, especially if it involves harsh detergents, agitation, or exposure to sunlight, the color molecules can break down or fade. This can result in a decrease in chroma, making the color appear duller or less vibrant. Factors such as the type of fabric, dye used, washing method and water temperature can all affect the degree of chroma loss.

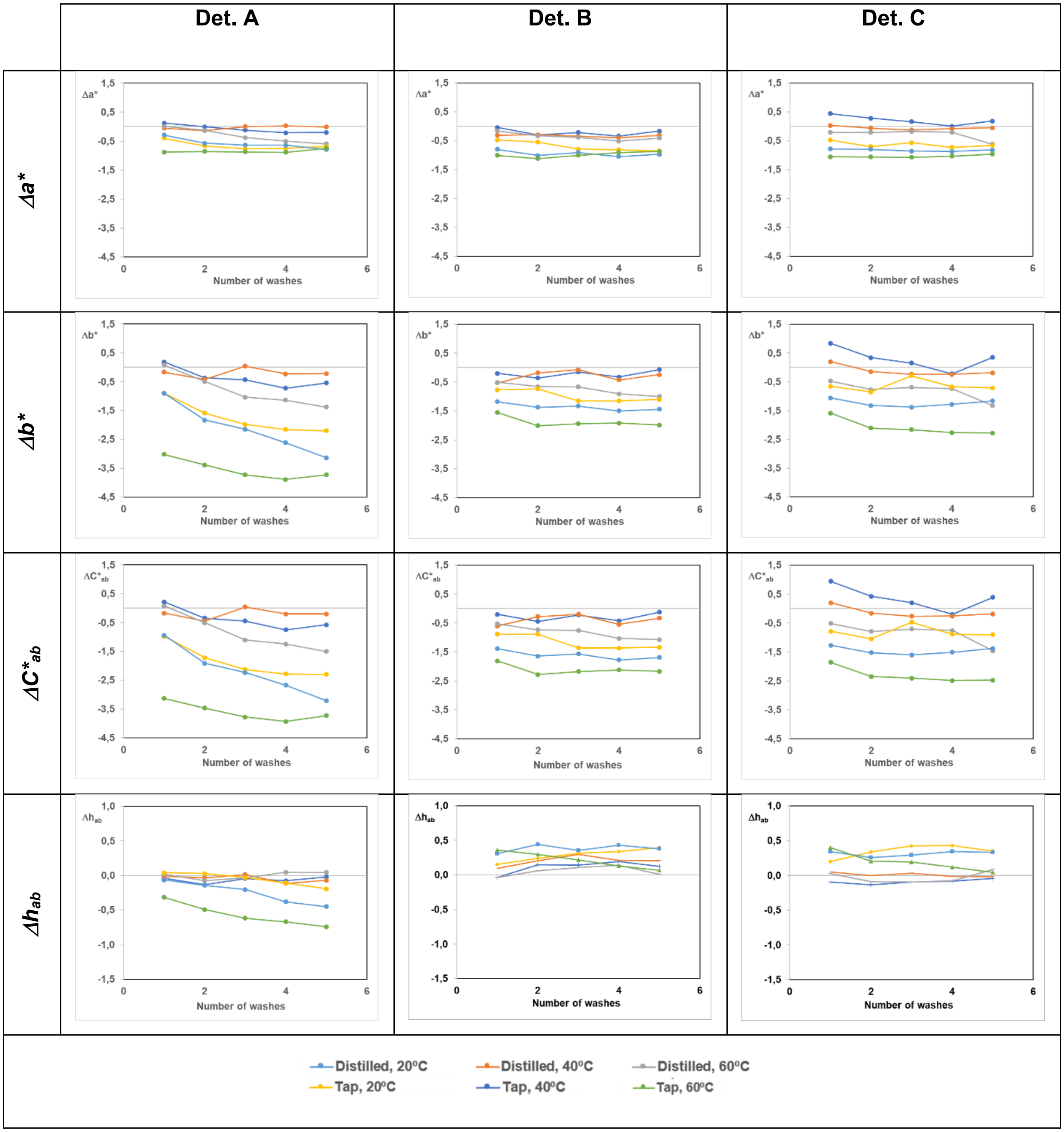


Figure 90. Δa^*, Δb^*, ΔC^*_{ab} and Δh_{ab}, with respect to the unwashed substrate, for all the detergents in all the washing conditions.

In this study, the pigments are the natural ones from cotton. Due to the small amount of lost chroma during the repetitive washes, it could be attributed more to the loss of soluble pigments during washing, as observed in the color of the waters after the first four washes (Figure 91), rather than any other type of phenomenon.

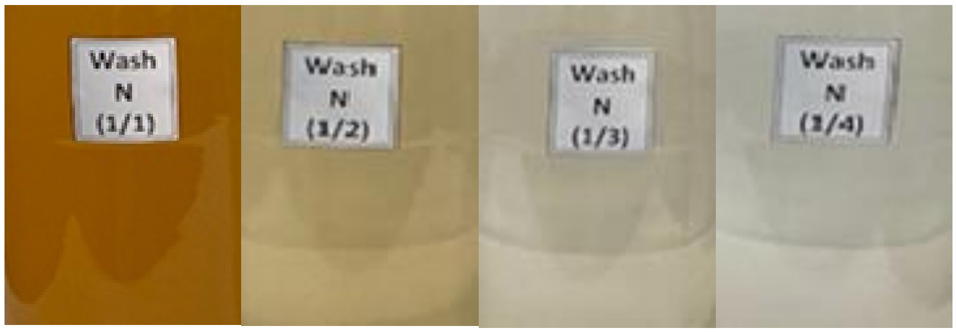


Figure 91. Wastewater of the 4 first washes.

The tannins concentration and/or type of tannin are related to de hue of the fabric. The hue, pure color of an object, remains virtually unchanged or slightly decreases when washing at higher temperatures in tap water with detergent A. For detergents B and C, it either remains unchanged or slightly increases in the first wash and then remains constant. Although the changes are very small and may not be observable to the naked eye, an increase in hue signifies that the dominant color or the specific shade of color becomes more intense or apparent. On the other hand, a decrease in hue signifies that the dominant color or the specific shade of color becomes less intense or apparent.

As for the color difference, ΔE^*_{ab}, it ranges from a minimum of 2.5 to a maximum of 7.6. These values are well above a maximum accepted error value of 2 units.

ΔE^*_{ab} values increased with temperature, particularly with tap water, demonstrating a substantial color change with each wash. This emphasizes the impact of tap water at elevated temperatures on hastening color alteration. To analyze the color change more thoroughly after multiple washes, Figure 92 illustrates the color difference for all samples under various conditions. No significant differences are observed when washing with distilled water, the detergent used, or the temperature. However, the most notable differences are found when washing with hard water.

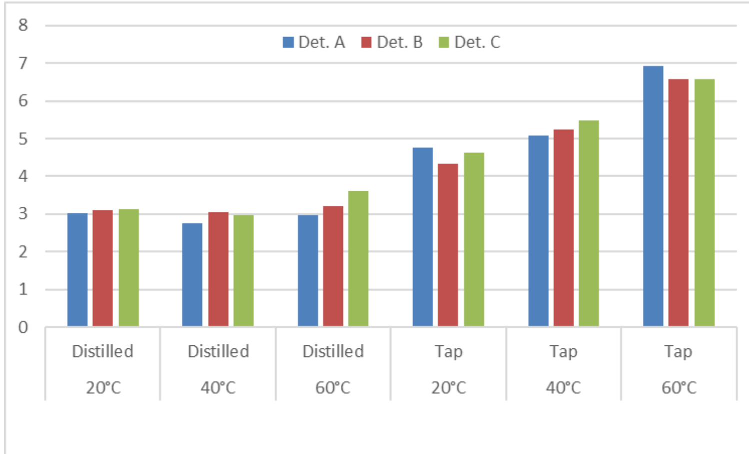


Figure 92. Comparison of *ΔE*ab* values after five washes between washed and original samples with different detergents, water hardness and washing temperature.

In order to identify accurately the variable or variables that have the greatest impact on the color change of substrates obtained after a domestic wash, the equation 18, model has been employed. Interactions between the number of washes and temperature or type of water are not considered. The beta coefficients have been estimated from this initial model using the linear model, stepwise backward regression, with a significance level (α) of 0.05.

$$\Delta E^*_{ab} = \beta_0 + \beta_1 \cdot X1 + \beta_2 \cdot X2 + \beta_3 \cdot X3 + \beta_4 \cdot X2 \cdot X3 + \beta_5 \cdot Q2 + \beta_6 \cdot Q2 \cdot X1 + \beta_7 \cdot Q2 \cdot X2 + \beta_8 \cdot Q2 \cdot X3 + \beta_9 \cdot Q2 \cdot X2 \cdot X3 + \beta_{10} \cdot Q3 + \beta_{11} \cdot Q3 \cdot X1 + \beta_{12} \cdot Q3 \cdot X2 + \beta_{13} \cdot Q3 \cdot X3 + \beta_{14} \cdot Q3 \cdot X2 \cdot X3 \quad \text{Eq. 18}$$

The resulting model of ΔE^*_{ab} is as follows:

$$\Delta E^*ab = 4{,}266 + 0{,}304 \cdot X1 + 1.379 \cdot X2 + 0.518 \cdot X3 + 0.456 \cdot X2 \cdot X3 - 0.236 \cdot Q2 \cdot X2 - 0.262 \cdot Q3 \cdot X2 \quad \text{Eq. 19}$$

$$R^2 = 0.949$$

In the model, it is observed that the variables X1 (number of washes), X2 (type of water) and X3 (temperature) are significant in the response ΔE^*_{ab}. The type of water interacts with the temperature. In the equation, there are significant differences in the qualitative variables because there are interactions of terms in Q with water hardness that are different from zero. The coefficient of determination of the fit is 94.87%, a very high value that indicates a good fit of the estimated model to the experimental data.

It is observed that the highest coefficient obtained is for X2 (type of water) and that this variable interacts with all the process variables, except for the number of washes that have been previously disregarded (due to previous individual analyses with each detergent).

By substituting the values of the categorical variables for each type of detergent, the following models are obtained:

Table 35. Model for each type of detergent.

Det. A	ΔE*ab =	$4.266 + 0.304 \cdot X1 + 1.379 \cdot X2 + 0.518 \cdot X3 + 0.456 \cdot X2 \cdot X3$
Det. B	ΔE*ab =	$4.266 + 0.304 \cdot X1 + 1.117 \cdot X2 + 0.518 \cdot X3 + 0.456 \cdot X2 \cdot X3$
Det. C	ΔE*ab =	$4.266 + 0.304 \cdot X1 + 1.143 \cdot X2 + 0.518 \cdot X3 + 0.456 \cdot X2 \cdot X3$

The obtained results show that water hardness is the variable that most influences the color change of NaCOC fabrics, so the higher the water hardness, the greater the color change. Additionally, this variable depends on the type of detergent, although the differences between detergents are not very significant (coefficients between 1.12 and 1.38). Thus, under the conditions of this study, the decrease in hardness of the washing solution, due to the presence of builders in the detergent, results in less darkening of fabrics. Builders work synergistically with surfactants to enhance the cleaning efficiency of detergents by softening water, preventing soil redeposition and improving the stability of the detergent solution. Therefore, even a small variation in detergent concentration can affect their action on the color of NaCOC substrates due to the presence of builders in the detergents.

Next, the washing temperature variable follows as an important role, which has a significant interaction with water hardness. The lower the water hardness (distilled water), the smaller the color difference between the washed fabrics and the original, and this difference increases significantly with temperature as water hardness increases.

The number of washes have less importance (coef = 0.304), so the color difference depends little on this value.

In the previous study, where 1 detergent, 1 temperature, and 1 type of water were examined, it was found that shrinkage in the first wash was the characteristic that most influenced the color change. The analysis of shrinkage in this study has shown that it is independent of the type of water and detergent and changes very little with the number of washes. In fact, the average shrinkage in the first wash of all substrates (considering all temperatures, detergents, and types of water) is 6.8 % ± 0.7 % and the average shrinkage considering all washes is 7.6 % ± 0.7 %. According to these results, the studied variables do not affect the shrinkage of the fabric, and therefore, the differences between the substrates are attributed to the significant variables.

Finally, it is very important to indicate that only the weft yarn is NaCOC cotton. In the case that both the weft and warp threads were NaCOC, the color difference would be much more significant.

3.5.2.5 CONCLUSIONS

A comprehensive examination of the influence of laundry variables, including the number of washes, type of water, and temperature, on the efficacy, intensity, and color variation (ΔE^*) of cleaned textiles employing special detergents designed for people with skin hypersensitivity A, B, and C uncovers crucial insights into the maintenance of fabric quality for naturally colored cotton. As concluded in a previous study, shrinkage is the variable that most influences the initial color change. In addition, in this study, it was found that, a part of shrinkage and among all the factors considered, the type of water is the most influential element affecting the overall alteration of fabric color, with the most significant change occurring after the initial wash. Specifically, distilled water consistently led to more prominent alterations in both the intensity and ΔE^*, signifying its substantial role in modifying the color properties of the fabric. Water hardness significantly affects the color change of NaCOC fabrics, suggesting that the use of water with lower hardness may be beneficial to preserve fabric quality. Using hard water requires larger amounts of detergent and adjuvants to compensate for the hardness, which can result in a greater environmental impact.

Temperature also plays a critical role, as higher temperatures increase color change, thereby highlighting the sensitivity of naturally colored cotton to washing temperature. As washing at high temperature influences the color change of the fabrics, washing at lower temperatures is advisable to prevent color changes and it is, generally, more energy efficient, reducing electricity consumption and greenhouse gas emissions associated with water heating. Although the number of washes contributes to a gradual decline in color efficacy and intensity, its impact is relatively less pronounced than that of water type and temperature. Notably, the interaction between the water type and temperature was particularly significant, suggesting that a specific combination of these factors can have a synergistic effect on color modification.

4. CONCLUSIONS

The comprehensive analysis of the impact of home washing on naturally colored organic cotton (NaCOC) fabrics has revealed several critical insights that underscore the importance of understanding textile behavior under household laundering conditions. The study has shown that washing NaCOC fabrics results in significant alterations to their colorimetric properties and ultraviolet protection factor (UPF), with the initial wash exerting the most profound effect.

One of the primary findings is the dual phenomenon occurring during the initial wash. Firstly, there is a noticeable extraction of pigments from the fabric, which are transferred to the wash water. This pigment loss results in a reduction of the saturation components, a^* and b^*, as measured by the *CIELAB* color space. Despite this, the shrinkage of the fabric during washing plays a more substantial role in altering the fabric's visual characteristics. The shrinkage leads to a darkening effect, which is more significant than the lightening caused by pigment loss. This darkening due to shrinkage is the dominant factor influencing the color change in NaCOC fabrics during home washing.

Moreover, the study indicates that this shrinkage also contributes to a notable increase in the fabric's UPF. This enhancement in UV protection is a beneficial side effect of the washing process, adding a functional advantage to the aesthetic changes in the fabric. Subsequent washes continue to affect the fabric's color and UPF, though their impact diminishes compared to the initial wash. The cumulative effect of repeated washing includes ongoing pigment loss and progressive shrinkage, which together influence the fabric's appearance and protective properties.

Further examination of various laundry variables, including the type of water and washing temperature, has provided additional insights into maintaining fabric quality. Among these variables, the type of water emerged as the most influential factor in color variation, with distilled water causing the most significant changes in color intensity and ΔE^*. Higher washing temperatures were also found to exacerbate color loss, underscoring the sensitivity of NaCOC fabrics to heat. Although the number of washes contributes to a gradual decline in color efficacy and intensity, its impact is relatively less pronounced than that of water type and temperature. Notably, the interaction between water type and temperature suggests that specific combinations of these factors can have a synergistic effect on color modification.

5. References

1. Data Seed cotton yields FAO. https://ourworldindata.org/grapher/cotton-yield (accessed 16.07.2024).
2. Carrera-Gallissà, E., . Algodón. Apuntes de Materias Textiles. ESEIAAT (UPC). Terrassa, 2024.
3. Materials Market Report 2023. https://textileexchange.org/knowledge-center/reports/materials-market-report-2023/ (accessed 06.07.2023).
4. The Journey of Cotton: Harvesting. https://barnhardtcotton.net/blog/journey-of-cotton-harvesting/ Posted on January 31, 2018 (accessed 16.07.2024).
5. Porter, J. Cotton Plant Growth Stages and Life Cycle Complete Details. https://gardenwisdomhub.com/cotton-plant-growth-stages-and-life-cycle/ (accessed 06.07.2024).
6. Clemencia Gómez de Er, I. M. A. H. I., MIliam Morales V,Arnulfo Díaz D, Malezas Problema en el Cultivo del Algodón (Gossypium hirsutum L,), en los Valles Cálido del Alto Magdalena y Río Sinú. Julio 2003, http://hdl.handle.net/20.500.12324/1174 (accessed 06.07.2024).
7. Photo, R. W. A. S. Gossypium darwinii, Galapagos Cotton Flower at Urbina Bay, Isabela Island, Galapagos Islands, Ecuador. https://www.alamy.com/stock-photo-gossypium-darwinii-galapagos-cotton-flower-at-urbina-bay-isabela-island-85873122.html?imageid=4C4639BE-11AB-4DB8-8A18-BFC6D6AECC51&p=177974&pn=1&searchId=37ee7e11501513a3ea7351a67fee551f&searchtype=0 (accessed 06.07.2024).
8. Close up view of a Cotton flower and cotton balls in a cotton fields ready for harvesting. https://cdn.pixabay.com/photo/2016/08/24/20/07/pink-cotton-flower-1617904_960_720.jpg (accessed 06.17.2024).
9. Cotton Growing: Proper Conditions And Smart Cultivation. https://eos.com/blog/cotton-growing/ (accessed 06.07.2024).
10. Después de 30 años: Táchira vuelve a producir algodón para el país. http://www.tachira.gob.ve/2017/03/10/despues-de-30-anos-tachira-vuelve-a-producir-algodon-para-el-pais/ (accessed 06.17.2024).
11. otton capsul and seeds http://www.asiantextilestudies.com/ATS/cot-33.jpg,
12. Dochia, M. S., C.; Kozlowski, R. M.; and Roskwitalski, Z., , Handbook of Natural Fibres. In *Handbook of Natural Fibres*, Cambridge.: 2012; Vol. 1, pp 11-23.
13. Cotton's chemical composition. https://es.slideshare.net/Nicolas1964/teido-de-algodn-con-colorantes-reactivos .
14. Heinze, T., Cellulose: structure and properties. *Cellulose chemistry and properties: fibers, nanocelluloses and advanced materials* 2016, 1-52, https://doi.org/10.1007/12_2015_319.
15. Yu, C., Natural textile fibres: vegetable fibres. In *Textiles and fashion*, Elsevier: 2015; pp 29-56, https://doi.org/10.1016/B978-1-84569-931-4.00002-7.
16. Mather, R. R.; Rana, S.; Wardman, R. H., *The chemistry of textile fibres*. Royal Society of Chemistry: 2023, https://doi.org/10.1039/9781782626534.
17. Mather, R. R.; Wardman, R. H., *The chemistry of textile fibres*. Royal Society of Chemistry: 2015.
18. Thomason, J.; Vlug, M., Influence of fibre length and concentration on the properties of glass fibre-reinforced polypropylene: 1. Tensile and flexural modulus. *Composites Part A: Applied science and manufacturing* 1996**,** *27* (6), 477-484, https://doi.org/10.1016/1359-835X(95)00065-A.

19. Betrabet, S.; Pillay, K.; Iyengar, R., Structural Properties of Cotton Fibers: Part II: Birefringence and Structural Reversals in Relation to Mechanical Properties. *Textile research journal* 1963**,** *33* (9), 720-727, https://doi.org/10.1016/1359-835X(95)00065-A.
20. Yan, Y., Developments in fibers for technical nonwovens. In *Advances in technical nonwovens*, Elsevier: 2016; pp 19-96.
21. Richardson, D. a. S. Asian Textile Studies. https://www.asiantextilestudies.com/cotton.html (accessed 06.07.2024).
22. Manual cotton picking. https://www.solidaridadnetwork.org/sites/solidaridadnetwork.org/files/commodity/_header.jpg.
23. Machine cotton picking (harvester). https://chdairiesdiary.files.wordpress.com/2015/04/cotton-is-my-life-3.jpg.
24. Appearance of a cotton harvester.: http://agriculturers.com/wp-content/uploads/2015/03/John_Deere_cotton_harvester_kv02.jpg.
25. Li, X.; Liu, B.; Cui, J.; Liu, D.; Ding, S.; Gilna, B.; Luo, J.; Fang, Z.; Cao, W.; Han, Z., No evidence of persistent effects of continuously planted transgenic insect-resistant cotton on soil microorganisms. *Plant and soil* 2011**,** *339* (1), 247-257.
26. Cotton. Sustainable Agriculture. World Wildlife Foundation. https://www.worldwildlife.org/industries/cotton.
27. *Cotton and the Environment*; Organic Trade Association July,2018, https://ota.com/sites/default/files/indexed_files/Cotton%20and%20the%20Environment%20Final%20July%202018.pdf.
28. The Apparel Industry's Environmental Impact in 6 Graphics. https://www.wri.org/insights/apparel-industrys-environmental-impact-6-graphics.
29. Shepherd, H. *Thirsty for Fashion? How organic cotton delivers in a water-stressed world*; Technical report, Soil Association: 2020, https://library.unccd.int/Details/books/1352#:~:text=How%20organic%20cotton%20delivers%20in%20a%20water%2Dstressed%20world,-TitleThirsty%20for&text=One%20kilogram%20of%20cotton%20takes,difference%20that%20saves%20precious%20water.
30. Parvez, S.; Gerona, R.; Proctor, C.; Friesen, M.; Ashby, J.; Reiter, J.; Lui, Z.; Winchester, P., Glyphosate exposure in pregnancy and shortened gestational length: a prospective Indiana birth cohort study. *Environmental Health* 2018**,** *17*, 1-12, http://10.1186/s12940-018-0367-0.
31. Environmental Justice Foundation & Pesticide Action Network. https://ejfoundation.org/resources/downloads/the_deadly_chemicals_in_cotton.pdf.
32. Rocha-Munive, M. G.; Soberón, M.; Castañeda, S.; Niaves, E.; Scheinvar, E.; Eguiarte, L. E.; Mota-Sánchez, D.; Rosales-Robles, E.; Nava-Camberos, U.; Martínez-Carrillo, J. L., Evaluation of the impact of genetically modified cotton after 20 years of cultivation in Mexico. *Frontiers in bioengineering and biotechnology* 2018**,** *6*, 82, https://doi.org/10.3389/fbioe.2018.00082.
33. Devrent, N.; Palamutcu, S., Mini review on organic cotton. *J Text Eng Fashion Technol* 2017**,** *3* (2), http://10.15406/jteft.2017.03.00093.
34. Lakhal, S. Y.; Sidibé, H.; H'Mida, S., Comparing conventional and certified organic cotton supply chains: the case of Mali. *International journal of agricultural resources, governance and ecology* 2008**,** *7* (3), 243-255, https://doi.org/10.1504/IJARGE.2008.018328.
35. Mageshwaran, V.; Satankar, V.; Shukla, S.; Kairon, M., Current status of organic cotton production. *Indian Farming* 2019**,** *69* (02), 09-14, http://10.1017/S1742170512000318.
36. Debuschewitz, E.; Sanders, J., Environmental impacts of organic agriculture and the controversial scientific debates. *Organic Agriculture* 2022**,** *12* (1), 1-15, https://doi.org/10.1007/s13165-021-00381-z.

37. Carvalho, L. P. d.; Farias, F. J. C.; Lima, M. M. d. A.; Rodrigues, J. I. d. S., Inheritance of different fiber colors in cotton (Gossypium barbadense L.). *Crop Breeding and Applied Biotechnology* 2014**,** *14*, 256-260, https://doi.org/10.1590/1984-70332014v14n4n40.
38. Oei, H.; Kartanegara, W.; Oei, R., Textile—Dye allergic contact dermatitis. *The Journal of Allergy and Clinical Immunology* 2003**,** *2* (111), S155, http://10.1016/S0091-6749(03)80504-3.
39. Patil, K. A.; Patil, R. S., The Inheritance Pattern of Brown Lint in Color-Cotton. In *Int. J. Curr. Microbiol. App. Sci* [Online] 2021; pp. 946-950.
40. Apodaca, J. K., *Naturally colored cotton: a new niche in the Texas natural fibers market*. Bureau of Business Research, Graduate School of Business, University of …: 1990, https://books.google.es/books/about/Naturally_Colored_Cotton.html?id=kr54HAAACAAJ&redir_esc=y.
41. Williams, B. L. FoxFibre naturally colored cotton, green and brown (coyote): resistance to changes in color when exposed to selected stains and fabric care chemicals. 1994, https://ttu-ir.tdl.org/items/a8fc7265-ac2f-43ae-a52e-d1bae9a1ae72.
42. Funk, P. A.; Gamble, G. R., Fiber properties of saw and roller ginned naturally colored cottons. 2009.
43. Vreeland Jr, J., Naturally colored and organically grown cottons: anthropological and historical perspectives. *Proceedings (USA)* 1993.
44. VanZandt, M. J. Development of fabrics from Foxfibre naturally colored cottons (coyote and buffalo) and evaluation of flame resistant characteristics. 1994, http://hdl.handle.net/2346/61056.
45. Dickerson, D. K.; Lane, E. F.; Rodriguez, D. F., Naturally colored cotton: resistance to changes in color and durability when refurbished with selected laundry aids. *California Agricultural Technology Institute, California State University, Fresno* 1999, 1-42, https://doi.org/10.1007/978-981-10-8782-0_4.
46. Katz, D.; Boone, N.; Vreeland Jr, J. M. In *Organically grown and naturally colored cotton: a global overview*, Beltwide Cotton Conferences (USA), 1997.
47. Singh, P.; Singh, V.; Waghmare, V., Naturally coloured cotton. 2001, http://krishi.icar.gov.in/PDF/ICAR_Data_Use_Licence.pdf.
48. Lee, J., A new spin on naturally colored cottons. *Agricultural Research* 1996**,** *44* (4), 20.
49. M. Vreeland, J., The revival of colored cotton. *Scientific American* 1999**,** *280* (4), 112-118.
50. Zhao, N.; Wang, W.; Grover, C. E.; Jiang, K.; Pan, Z.; Guo, B.; Zhu, J.; Su, Y.; Wang, M.; Nie, H., Genomic and GWAS analyses demonstrate phylogenomic relationships of Gossypium barbadense in China and selection for fibre length, lint percentage and Fusarium wilt resistance. *Plant biotechnology journal* 2022**,** *20* (4), 691-710, https://doi.org/10.1111/pbi.13747.
51. Gu, H., Research on the improvement of the moisture absorbency of naturally self-coloured cotton. *Journal of the Textile Institute* 2005**,** *96* (4), 247-250.
52. Soydan, A. S.; Yavaş, A.; Avinç, O. O.; Günaydın, G. K.; Kıvılcım, M. N.; Demirtaş, M.; Palamutcu, S., The effects of hydrogen peroxide and sodium hypochlorite oxidizing treatments on the color properties of naturally colored green cotton. *European Journal of Engineering and Natural Sciences* 2019**,** *3* (2), 1-10, http://10.1111/j.1365-313X.2004.02138.x.
53. Ware, J. O., Plant breeding and the cotton industry. 1936.
54. Fox, S., Naturally colored cotton. *Spin off* 1987**,** *46*, 29-31.
55. Rollins, M. L., The Cotton Fiber, in" The American Cotton Handbook," Vol. 1, DS Hamby. 1965.
56. Hamby, D. S., *The American Cotton Hadbook*. Interscience Publishers: 1965.
57. Endrizzi, J.; Turcotte, E.; Kohel, R., Qualitative genetics, cytology, and cytogenetics. *Cotton* 1984**,** *24*, 81-129, https://doi.org/10.2134/agronmonogr24.c4.

58. Kohel, R., Genetic analysis of fiber color variants in cotton 1. *Crop Science* 1985**,** *25* (5), 793-797, https://doi.org/10.2135/cropsci1985.0011183X002500050017x.
59. Vreeland Jr, J., Recuperando el algodón nativo: una tecnología nativa para la agricultura del desierto peruano. Ayancucho: 1987.
60. Richards, A. F.; Rowe, T.; Stankovic Elesini, U., Structure of naturally coloured cottons. *Journal of the Textile Institute* 1999**,** *90* (4), 493-499, https://doi.org/10.1080/00405000.1999.10750048.
61. Schmutz, A.; Jenny, T.; Amrhein, N.; Ryser, U., Caffeic acid and glycerol are constituents of the suberin layers in green cotton fibres. *Planta* 1993**,** *189* (3), 453-460,
62. Yatsu, L.; Espelie, K.; Kolattukudy, P., Ultrastructural and Chemical Evidence of the Suberisation of the Cell Wall of Green Cotton. *Plant Physiology* 1983**,** *73*, 521-524, https://doi.org/10.1104/pp.73.2.521.
63. Yatsu, L.; Espelie, K. E.; Kolattukudy, P., Ultrastructural and chemical evidence that the cell wall of green cotton fiber is suberized. *Plant Physiology* 1983**,** *73* (2), 521-524.
64. Blas-Sevillano, R.; Veramendi, T.; La Torre, B.; Velezmoro-Sánchez, C.; Oliva, A.; Mena-Martínez, M.; Herrera-Kao, W.; Uribe-Calderon, J.; Cervantes-Uc, J., Physicochemical characterization of several types of naturally colored cotton fibers from Peru. *Carbohydrate polymers* 2018**,** *197*, 246-252, https://doi.org/10.1016/j.carbpol.2018.06.006.
65. Xiao, Y.-H.; Yan, Q.; Ding, H.; Luo, M.; Hou, L.; Zhang, M.; Yao, D.; Liu, H.-S.; Li, X.; Zhao, J., Transcriptome and biochemical analyses revealed a detailed proanthocyanidin biosynthesis pathway in brown cotton fiber. *PLoS One* 2014**,** *9* (1), e86344, https://doi.org/10.1371/journal.pone.0086344.
66. Endrizzi, J.; Turcotte, E.; Kohel, R., Genetics, cytology, and evolution of Gossypium. *Advances in genetics* 1985**,** *23*, 271-375, https://doi.org/10.1016/S0065-2660(08)60515-5.
67. Halloin, J. M., Localization and changes in catechin and tannins during development and ripening of cottonseed. *New Phytologist* 1982**,** *90* (4), 651-657, https://doi.org/10.1111/j.1469-8137.1982.tb03274.x.
68. Crews, P. C.; Hustvedt, G., The ultraviolet protection factor of naturally-pigmented cotton. 2005, https://digitalcommons.unl.edu/textiles_facpub.
69. Wakelyn, P.; Chaudhry, M., Organic cotton: production practices and post-harvest considerations. In *Sustainable textiles*, Elsevier: 2009; pp 231-301, https://doi.org/10.1533/9781845696948.2.231.
70. Chung, K.-T.; Wong, T. Y.; Wei, C.-I.; Huang, Y.-W.; Lin, Y., Tannins and human health: a review. *Critical reviews in food science and nutrition* 1998**,** *38* (6), 421-464, https://doi.org/10.1080/10408699891274273.
71. Ying, C.; Xinyuan, S., Effect of coloring materials on microstructure of naturally colored cotton. *Colourage* 2006**,** *53* (4), 55-58.
72. Xiangqian, Z.; Xuede, W., Composition analysis of pigment in colored cotton fiber. *Zuo wu xue bao* 2005**,** *31* (4), 456-462.
73. Heap, S. A.; Greenwood, P. F.; Leah, R. D.; Eaton, J. T.; Stevens, J. C.; Keher, P., Prediction of finished relaxed dimensions of cotton knits—the starfish project: Part II: Shrinkage and the reference state. *Textile Research Journal* 1985**,** *55* (4), 211-222, https://doi.org/10.1177/004051758505500403.
74. RAHEEL, M.; DEVER LIEN, M., The Use of Scanning Electron Microscopy for Studying Abrasion Phenomena in Laundered Fabric. *Textile Chemist & Colorist* 1985**,** *17* (5),
75. Breen, N.; Durnam, D.; Obendorf, S., Residual oily soil distribution on polyester/cotton fabric after, laundering with selected detergents at various wash temperatures. *Textile research journal* 1984**,** *54* (3), 198-204, https://doi.org/10.1177/004051758405400310.
76. Kang, S. Y.; Epps, H. H., Effect of scouring and enzyme treatment on moisture regain percentage of naturally colored cottons. *The Journal of the Textile Institute* 2009**,** *100* (7), 598-606, https://doi.org/10.1080/00405000802125113.

77. Han, A.; Chae, Y.; Lee, M.; Cho, G., Effect of color changes of naturally colored organic cotton fibers on human sensory perception. *Fibers and Polymers* 2011**,** *12*, 939-945, https://doi.org/10.1007/s12221-011-0939-x.
78. Ware, J.; Benedict, L., Colored cottons and their economic value. *Journal of Heredity* 1962**,** *53* (2), 57-65, , https://doi.org/10.1093/oxfordjournals.jhered.a107122.
79. Conrad, C. M., HERITABLE RELATION OF WAX CONTENT AND GREEN. *Journal of Agricultural Research* 1943**,** *66*, 307.
80. Price, J. B.; Cui, X.; Calamari, T. A.; Mcdaniel, R. G., Assessing the quality of four naturally colored cottons. *Textile research journal* 2001**,** *71* (11), 993-999, https://doi.org/10.1177/004051750107101110.
81. Conrad, C. M., *HERITABLE RELATION OF WAX CONTENT AND GREEN PIGMENTATION OF LINT IN UPLAND COTTON ¹*. 1943; Vol. 66, p 307.
82. Matusiak, M.; Kechagia, U.; Tsaliki, E.; Frydrych, I., Properties of the naturally colored cotton and its application in the ecological textiles. 2007, https://doi.org/10.1046/j.1439-0523.2003.00938.x.
83. Dutt, Y.; Wang, X.; Zhu, Y.; Li, Y., Breeding for high yield and fibre quality in coloured cotton. *Plant Breeding* 2004**,** *123* (2), 145-151, https://doi.org/10.1002/(SICI)1097-4628(20000531)76:9%3C1466::AID-APP10%3E3.0.CO;2-Q.
84. Chen, H. L.; Yokochi, A., X-ray diffractometric study of microcrystallite size of naturally colored cottons. *Journal of Applied Polymer Science* 2000**,** *76* (9), 1466-1471, https://doi.org/10.1002/(SICI)1097-4628(20000531)76:9%3C1466::AID-APP10%3E3.0.CO;2-Q.
85. Meredith, R., Properties of textile materials. I—tensile strength, breaking extension and stress-strain relations of fibres. *Journal of the Textile Institute Proceedings* 1952**,** *43* (9), P755-P764, https://doi.org/10.1080/19447015208664103.
86. Kang, S. Y. Investigation of color change and moisture regain of naturally colored cotton. University of Georgia, 2007.
87. Schmutz, A.; Jenny, T.; Ryser, U., A caffeoyl-fatty acid-glycerol ester from wax associated with green cotton fibre suberin. *Phytochemistry* 1994**,** *36* (6), 1343-1346, https://doi.org/10.1016/S0031-9422(00)89721-6.
88. Ryser, U.; Holloway, P., Ultrastructure and chemistry of soluble and polymeric lipids in cell walls from seed coats and fibres of Gossypium species. *Planta* 1985**,** *163* (2), 151-163,
89. Ryser, U.; Holloway, P., Ultrastructure and chemistry of soluble and polymeric lipids in cell walls from seed coats and fibres of Gossypium species. *Planta* 1985**,** *163*, 151-163, https://doi.org/10.1007/BF00393501.
90. Schmutz, A.; Jenny, T.; Amrhein, N.; Ryser, U., Caffeic acid and glycerol are constituents of the suberin layers in green cotton fibres. *Planta* 1993**,** *189*, 453-460, http://10.1007/BF00194445.
91. Graf, E., Antioxidant potential of ferulic acid. *Free radical biology and medicine* 1992**,** *13* (4), 435-448, https://doi.org/10.1016/0891-5849(92)90184-I.
92. Pan Zhaoe, P. Z.; Sun DongLei, S. D.; Sun JunLing, S. J.; Zhou ZhongLi, Z. Z.; Jia YinHua, J. Y.; Pang BaoYin, P. B.; Ma ZhiYing, M. Z.; Du XiongMing, D. X., Effects of fiber wax and cellulose content on colored cotton fiber quality. 2010, https://doi.org/10.1007/s10681-010-0124-0.
93. Günaydin, G. K.; Avinc, O.; Palamutcu, S.; Yavas, A.; Soydan, A. S., Naturally colored organic cotton and naturally colored cotton fiber production. *Organic Cotton: Is it a Sustainable Solution?* 2019, 81-99, https://doi.org/10.1007/978-981-10-8782-0_4.
94. Angelova, V.; Ivanova, R.; Delibaltova, V.; Ivanov, K., Bio-accumulation and distribution of heavy metals in fibre crops (flax, cotton and hemp). *Industrial crops and products* 2004**,** *19* (3), 197-205, https://doi.org/10.1016/j.indcrop.2003.10.001.

95. Parmar, M.; Chakraborty, M., Thermal and burning behavior of naturally colored cotton. *Textile Research Journal* 2001**,** *71* (12), 1099-1102, https://doi.org/10.1177/004051750107101211.
96. Ma, M.; Li, R.; Du, Y.; Tang, Z.; Zhou, W., Analysis of antibacterial properties of naturally colored cottons. *Textile Research Journal* 2013**,** *83* (5), 462-470, https://doi.org/10.1177/0040517512447585.
97. Ma, M.; Luo, S.; Hu, Z.; Tang, Z.; Zhou, W., Antioxidant properties of naturally brown-colored cotton fibers. *Textile Research Journal* 2016**,** *86* (3), 256-263, https://doi.org/10.1177/0040517515588270.
98. GÜLÜMSER, T., Comparison between naturally colored cotton fabric and white cotton fabric in manner of processes in the dyehouse. *Textile and Apparel* 2016**,** *26* (3), 287-294,
99. Chang, Y.; Park, J.; Hong, W.; You, H.; Han, A.; Chae, Y.; Cho, G. Analysis of Visual Sensibility Evaluation of Naturally Colored Organic Cotton (NaCOC).
100. Lazarov, A.; Trattner, A.; David, M.; Ingbe, A., Textile dermatitis in Israel: a retrospective study. *American Journal of Contact Dermatitis* 2000**,** *11* (1), 26-29, https://doi.org/10.1016/S1046-199X(00)90029-0.
101. Ferreira, J. A.; Costa, J. C. T.; Castro, E. D.; Vaz, M., Assessment of atopy in textile industry workers: Comparison of two methods. *The Journal of Allergy and Clinical Immunology* 2002**,** *1* (109), S325.http://10.1016/S0091-6749(02)82141-8.
102. organiccolourcotton https://www.organiccottoncolours.com/.
103. Woven fabrics - Construction - Methods of analysis. *Part 5: Determination of linear density of yarn removed from fabric* 2020, *Volume* (Issue), https://www.iso.org/standard/13845.html.
104. Determination of twist in yarns - Direct counting method. 2015, *Volume* (Issue), https://www.une.org/encuentra-tu-norma/busca-tu-norma/norma/?c=N0055676.
105. Yarns from packages - Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester. 2010, *Volume* (Issue), https://www.une.org/encuentra-tu-norma/busca-tu-norma/norma?c=N0045183.
106. UNE-EN ISO 2062:2010. *Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester* 2010-04-14, *Volume* (Issue), https://www.une.org/encuentra-tu-norma/busca-tu-norma/norma?c=N0045183.
107. UNE 40225-1:1974 IN. *Determinación de la irregularidad de masa lineal de corto periodo de las cintas, mechas e hilos de lana mediante el regularímetro Zellweger-Uster.* 2000, *Volume* (Issue).
108. UNE 40225-1:1974 IN. *WOOL. DETERMINATION OF SHORT TERM IRREGULARITY OF LINEAR DENSITY OF SILVERS, ROVING AND YARNS, BY MEANS OF AN ELECTRONIC EVENESS TESTER.* 1974-05-15 *Volume* (Issue), https://www.une.org/encuentra-tu-norma/busca-tu-norma/norma?c=N0004475.
109. Determination of mass per unit length and mass per unit area. *woven fabric* 1977-09, *Volume* (Issue), https://www.iso.org/standard/9335.html.
110. BS 3356. *Method for determination of bending length and flexural rigidity of fabrics* 31/08/1990, *Volume* (Issue), https://landingpage.bsigroup.com/LandingPage/Undated?UPI=000000000000220852.
111. UNE-EN ISO 13934-1:1999. *TENSILE PROPERTIES OF FABRICS. PART 1: DETERMINATION OF MAXIMUM FORCE AND ELONGATION AT MAXIMUM FORCE USING THE STRIP METHOD* 1999, *Volume* (Issue), https://www.une.org/encuentra-tu-norma/busca-tu-norma/norma?c=N0014213.
112. BS 5058:1973. *Method for the assessment of drape of fabrics* 1973, *Volume* (Issue), https://www.en-standard.eu/bs-5058-1973-method-for-the-assessment-of-drape-of-fabrics/.

113. ISO 9073-9:2008. *Test methods for nonwovens,Part 9: Determination of drapability including drape coefficient* 2008, *Volume* (Issue), https://www.iso.org/standard/41375.html.
114. Normalización, I. N. d. R. y., *Determinación del cayente de las telas no tejidas: Norma española. UNE 40-383-79*. Instituto Nacional de Racionalización y Normalización, IRANOR: 1979.
115. DIN 54306:1979-02. *Testing of textiles; determination of the drape of textile fabrics* 1979, *Volume* (Issue), https://www.beuth.de/en/standard/din-54306/778208.
116. JIS R 3418:1991. *Textile glass fabric curtains for casement and drapery* 1991, *Volume* (Issue), https://www.orochem.org/53003.html.
117. Chae, Y.; Cho, G., Colorimetric properties and color sensibility of naturally colored organic cotton fabrics. *Fibers and Polymers* 2012**,** *13*, 1154-1158.
118. UNE-EN ISO 105-J03:2010. *Tests for colour fastness - Part J03: Calculation of colour differences* 2010, *Volume* (Issue), https://www.une.org/encuentra-tu-norma/busca-tu-norma/norma?c=N0044887.
119. Algaba, I. M.; Pepió, M.; Riva, A., Correlation between the ultraviolet protection factor and the weight and thickness of undyed cellulosic woven fabrics. *Fibres & Textiles in Eastern Europe* 2008**,** *16* (1), 85-89.
120. AS/NZS 4399:1996. *Sun protective clothing Evaluation and classification* 1996, *Volume* (Issue), https://www.saiglobal.com/PDFTemp/Previews/OSH/As/as4000/4300/4399.PDF.
121. Hua, S.; Wang, X.; Yuan, S.; Shao, M.; Zhao, X.; Zhu, S.; Jiang, L., Characterization of pigmentation and cellulose synthesis in colored cotton fibers. *Crop Science* 2007**,** *47* (4), 1540-1546, https://doi.org/10.2135/cropsci2006.12.0835.
122. Khan, A. I.; Awan, F. S.; Sadia, B.; Rana, R. M.; Khan, I. A., Genetic diversity studies among coloured cotton genotypes by using RAPD markers. *Pak. J. Bot* 2010**,** *42* (1), 71-77.
123. Frydrych, I. K., Textile Material Engineering: Ecotextiles from the Naturally Colored Cotton. 2007.
124. Kang, S. Y.; Epps, H. H., Effect of Scouring on the Color of Naturally-colored Cotton and the Mechanism of Color Change. *AATCC review* 2008**,** *8* (7).
125. Cardoso, G. D.; Alves, P. L.; Severino, L. S.; Vale, L. S., Critical periods of weed control in naturally green colored cotton BRS Verde. *Industrial Crops and Products* 2011**,** *34* (1), 1198-1202, https://doi.org/10.1016/j.indcrop.2011.04.014.
126. Demir, A.; Özdoğan, E.; Özdil, N.; Gürel, A., Ecological materials and methods in the textile industry: Atmospheric-plasma treatments of naturally colored cotton. *Journal of Applied Polymer Science* 2011**,** *119* (3), 1410-1416, https://doi.org/10.1002/app.32575.
127. Chae, Y.; Lee, M.; Cho, G., Mechanical properties and tactile sensation of naturally colored organic cotton fabrics. *Fibers and Polymers* 2011**,** *12*, 1042-1047, http://10.1007/s12221-011-1042-z.
128. de Morais Teixeira, E.; Corrêa, A. C.; Manzoli, A.; de Lima Leite, F.; de Oliveira, C. R.; Mattoso, L. H. C., Cellulose nanofibers from white and naturally colored cotton fibers. *Cellulose* 2010**,** *17* (3), 595-606, https://doi.org/10.1007/s10570-010-9403-0.
129. Matusiak, M.; Kechagia, U.; Tsaliki, E.; Frydrych, I. In *Properties of the naturally colored cotton and its application in the ecological textiles*, Proc. Fourth World Cotton Research Conference, Lubbock, TX, 2007; pp 10-14.
130. Gies, P.; Slevin, T.; Harrison, S.; Plowman, P.; Dain, S.; Moller, L.; Mawley, F.; Swift, N., Australian/New Zealand Standard, AS/NZS 4399: 2017: sun protective clothing–evaluation and classification. Standards Australia: 2017.
131. Ahmed, S., A study on the physical properties of 100% cellulosic woven fabrics. *J Textile Eng Fashion Technol* 2021**,** *7* (4), 127-132, http://10.15406/jteft.2021.07.00279.
132. Gordon, S.; Hsieh, Y.-I., *Cotton: Science and technology*. Woodhead Publishing: 2006.
133. da Silva, D. J.; da Silva Barbosa, R. F.; de Souza, A. G.; Ferreira, R. R.; Camani, P. H.; Nantes-Cardoso, I. L.; Rosa, D. S., Bactericidal activity of cotton fabrics functionalized by

ZnO and Cu via microwave. *Cellulose* 2021, *28*, 8153-8175, https://doi.org/10.1007/s10570-021-03990-9.
134. Geminiani, L.; Campione, F. P.; Corti, C.; Luraschi, M.; Motella, S.; Recchia, S.; Rampazzi, L., Differentiating between Natural and Modified Cellulosic Fibres Using ATR-FTIR Spectroscopy. *Heritage* 2022, *5* (4), 4114-4139, https://doi.org/10.3390/heritage5040213.
135. Rotzler, S.; Schneider-Ramelow, M., Washability of e-textiles: failure modes and influences on washing reliability. *Textiles* 2021, *1* (1), 37-54, https://doi.org/10.3390/textiles1010004.
136. Exchange, T., Organic cotton market report. *Available in: http://textileexchange. org/sites/default/files/te_pdfs/2011_Organic_Cotton_Market_Report_websize. pdf [Accessed 15th July 2014]* 2011.
137. Yuan, S.; Malik, W.; Bibi, N.; Wen, G.; Ni, M.; Wang, X., Modulation of morphological and biochemical traits using heterosis breeding in coloured cotton. *The Journal of Agricultural Science* 2013, *151* (1), 57-71, https://doi.org/10.1017/S0021859612000172.
138. Murthy, M., Never say dye: the story of coloured cotton. *Resonance* 2001, *6* (12), 29-35, https://doi.org/10.1007/BF02913764.
139. Zucco, G. M.; Doty, R. L., Multiple chemical sensitivity. *Brain sciences* 2021, *12* (1), 46, https://doi.org/10.3390/brainsci12010046.
140. Grigorev, S. V.; Illarionova, K. V.; Konarev, A. V.; Shelenga, T. V., Differences in metabolites of white and naturally colored cotton: implications for biofunctional and aseptic textiles. *Journal of Natural Fibers* 2022, *19* (13), 7060-7072, https://doi.org/10.1080/15440478.2021.1941490.
141. Zoeller, B. L.; Doyle, M. H., *List of Publications of the United States Department of Agriculture from January 1941 to December 1945, Inclusive*. US Department of Agriculture: 1946, https://www.amazon.com/-/es/1946-U-S-Department-Agriculture/dp/0371460735.
142. Bryant, M. M., Names and Terms Used in the Fashion World. *American Speech* 1970, *45* (3/4), 168-194, https://doi.org/10.2307/454832.
143. Kantor, N.; Moore, M. A., EFFECTS OF LAUNDERINGS TREATMENT ON NATURALLY COLORED COTTON KNIT FABRICS. *Undergraduate Research Journal for the Human Sciences* 2004, *3*, https://publications.kon.org/urc/laundering_with_addition.html.
144. Tsaliki, E.; Kechagia, U.; Eleftheriadis, I.; Xanthopoulos, F., Effect of wet treatments on color fastness of naturally colored cotton. *Journal of Natural Fibers* 2016, *13* (4), 502-505, https://doi.org/10.1080/15440478.2015.1069234.
145. Chen-Yu, J. H.; Wong, S.-W., Effects of repeated treatments of perspiration, weathering, and laundering on the ultraviolet protection of a naturally colored lightweight cotton fabric. *Clothing and Textiles Research Journal* 2017, *35* (2), 128-140, https://doi.org/10.1177/0887302X16687074.
146. Aliei, H.; Cayuela, D.; Capdevila, F. J.; Carrera-Gallissà, E., Influence of Household Washing on the Colorimetric Properties of Intrinsically Natural Organic Cotton Fabrics. *Journal of Natural Fibers* 2024, *21* (1), 2351160, https://doi.org/10.1080/15440478.2024.2351160.
147. Organic Cotton Colours. https://www.organiccottoncolours.com/ (accessed March 2024).
148. Lava Ropa (Fox Fiber Detergent). https://www.vreseis.com/shop (accessed March 2024).
149. Klar Detergent. https://www.almawin.de/en/our-products (accessed March 2024).
150. Pure Nature detergent. https://www.purenature24.com/liquid-detergent-2-liter-bottle (accessed march 2024).
151. ISO 105-J03:2009, Textiles: Tests for colour fastness. *Part J03: Calculation of colour differences* 2020, *Volume* (Issue), 10, https://www.iso.org/standard/51385.html#:~:text=Abstract,two%20specimens%20do%20not%20match.

152. ISO 3759:2007, Textiles Preparation, marking and measuring of fabric specimens and garments in tests for determination of dimensional change. 2011, *Volume* (Issue), 6, https://www.iso.org/standard/57309.html.
153. Ma, M.; Hussain, M.; Memon, H.; Zhou, W., Structure of pigment compositions and radical scavenging activity of naturally green-colored cotton fiber. *Cellulose* 2016**,** *23*, 955-963, https://doi.org/10.1007/s10570-015-0830-9.
154. Peng, Z.; Gao, Q.; Luo, C.; Gong, W.; Tang, S.; Zhang, X.; Song, W.; Wang, Z.; Liu, H.; Du, X., Flavonoid biosynthetic and starch and sucrose metabolic pathways are involved in the pigmentation of naturally brown-colored cotton fibers. *Industrial Crops and Products* 2020**,** *158*, 113045, https://doi.org/10.1016/j.indcrop.2020.113045.