APPENDIX A

AGGREGATE SWELLING IN A WETTING PATH: ESEM IMAGES

A.1 Equipment, sample preparation and wetting path

Environmental scanning electron (ESEM) photomicrographs (magnification ×2500) of high-density packings have been carried out on an Electroscan 2020 equipment at constant electron beam voltage of 20 kV in order to complement the information of aggregate swelling in a low-pressure wetting path. Boom clay powder at hygroscopic humidity (relative humidity of $h_r \approx 40\%$) has been compacted at a dry density of 2 Mg/m^3 and further destructured to obtain an adequate size range of high-density aggregations. These aggregations are carefully stuck on the sample holder with a special adhesive tape. No further charging of the non-conductive specimen is required. Chamber pressure is lowered to an absolute pressure of 8.0 torr (1 torr $\approx 1.333 \times 10^2$ Pa) at ambient temperature (point A in Fig. A.1). At this stage the charged vapour phase surrounding the specimen (H_2O^+) acts as the conducting film surface which ensures an adequate viewing mechanism for the electron bombardment of the sample surface. Chamber pressure is isothermally lowered to an absolute pressure of 4.9 torr for the beginning of the wetting path (point B). A thermoelectric Peltier cooling stage up to 10°C is imposed at constant pressure to the sample holder (point C) followed by an isothermal pressure increment up to 6.5 torr (point D), 7.5 torr (point E) and 9.5 torr (point F). This wetting path is performed at conditions under which vapour is the stable phase presenting the lowest chemical potential (points below the phase boundary corresponding to a flat liquid-gas interface) and over the triple point pressure (4.58 torr). However, hydrated samples are assumed to remain in a meta-stable state when the phase boundary is transgressed without the liquid changing phase (transgression path in Fig. A.1). Under these circumstances and the development of curved liquid-gas interfaces, it is possible to reach a state normally associated to a stable vapour phase without the vapour phase developing (Apfel, 1970 in Marinho and Chandler, 1995). However, this meta-stable condition can be spontaneously destroyed if vapour cavities are formed within the liquid itself or at its boundaries (Trevena, 1987 in Marinho and Chandler, 1995). This way, the chamber pressure : specimen temperature plot is assimilated in points below the phase boundary (or saturation vapour pressure boundary) to a vapour pressure : temperature plot. An apparent relative humidity h_{ra} is defined as the ratio of the chamber pressure u_c to the saturation vapour pressure u_{co} in the phase boundary at the same temperature. An apparent dew point is defined at the sample holder temperature to which unsaturated air must be cooled at constant pressure for condensation to occur. Apparent relative humidity values are indicated in Fig. A.1 for the different wetting stages, which give only an indication of the hydration evolution. Temperature and chamber pressure are equalised for approximately 10 min in order not to affect the charged vapour phase surrounding the specimen (H_2O^+) with its consequences on image resolution.

A.2 Volume change evolution

Measurement of volume changes in ESEM photomicrographs have been calculated using digital imaging techniques at the same magnification, where edges have been detected based on negative images and non-edge pixels outside the aggregate are suppressed. Unfortunately, digitised images have areas were relatively high contrast is encountered due to shadows, mainly in the bottom part of the aggregate. To overcome this problem the bottom part is manually traced on the screen. Fig. A.2 shows the representation of aggregate swelling for the different wetting steps. In the case of the aggregate at point B (h_{ra} = 28%), which is the initial configuration, the black pores represent 280331 pixels (294589

pixels if other transition colours are considered) with respect to the 1048576 pixels of the image, corresponding to an area between 26.7% and 28.1%. The high-density aggregate at point C (h_{ra} = 53%) presents 290487 black pixels (305502 pixels if other transition colours are considered) with respect to the same total pixels of the image, representing an area between 27.7% and 29.1%. The aggregate at point D (h_{ra} = 71%) presents 304847 black pixels (320069 pixels if other transition colours are considered) with respect to the same total pixels of the image, corresponding to an area between 29.1% and 30.5%. Finally, the aggregate at point E (h_{ra} = 81%) presents 315057 black pixels (330343 pixels if other transition colours are considered) with respect to the same total pixels of the image, corresponding to an area between 30.1% and 31.5%. Starting from the initial configuration a 2-D based volumetric strain evolution can be estimated, which is indicated in Fig. A.2, displaying a maximum value of around $\delta\varepsilon_{v\,2D} \approx (12.1 \div 12.7)\%$ at point E.

Equivalent wetting stages have been performed on bentonite aggregations, where similar volume change trends have been observed, giving some confidence to the experimental procedure and results obtained from this computer vision based technique. Irreversible features of aggregate volume change behaviour, as well as the detection of spurious test problems, are currently being analysed following a controlled drying path after main hydration.

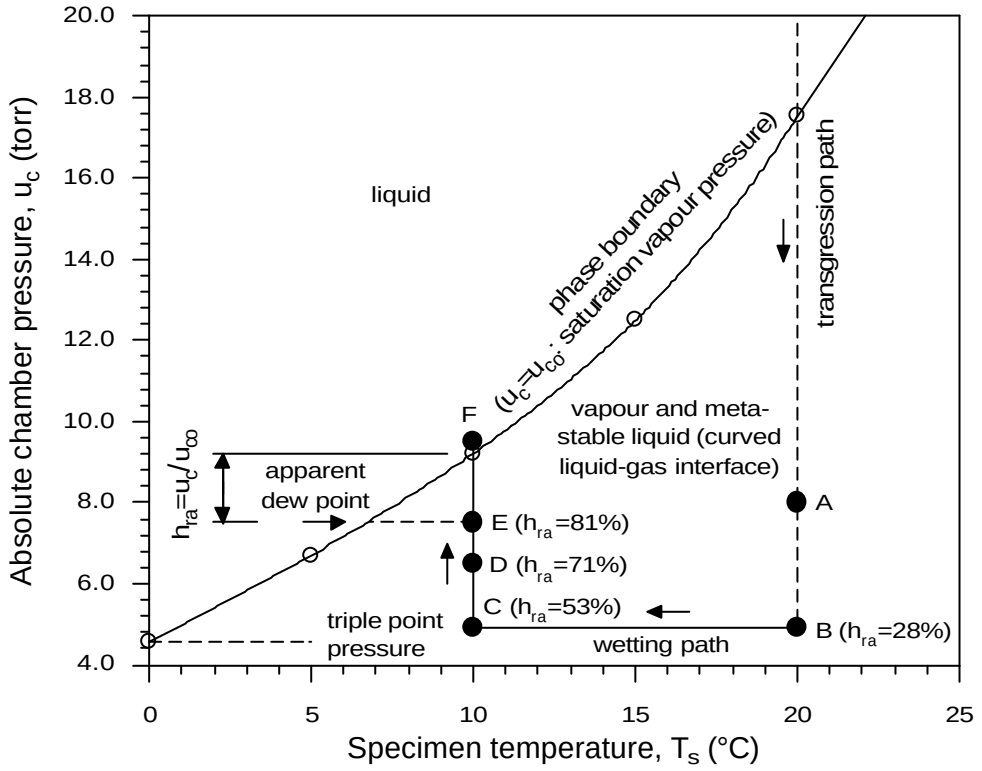
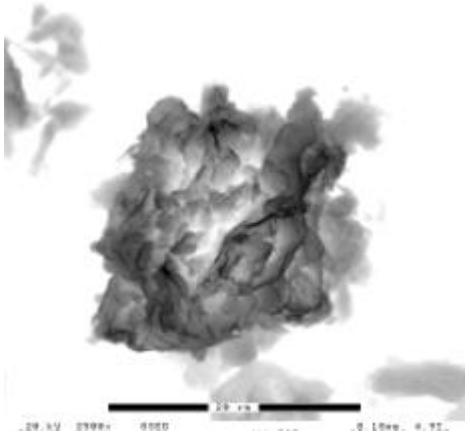
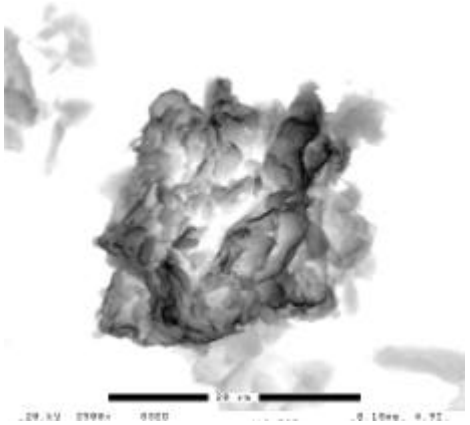


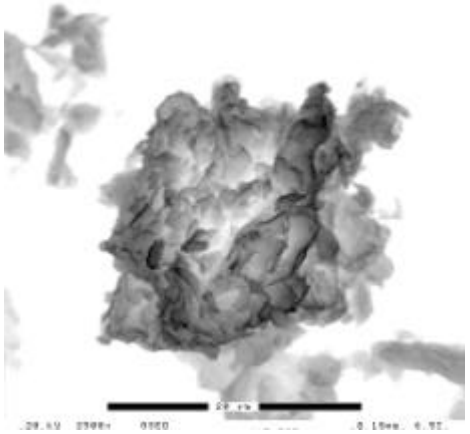
Figure A.1 Wetting path followed by the high-density aggregate in the ESEM.



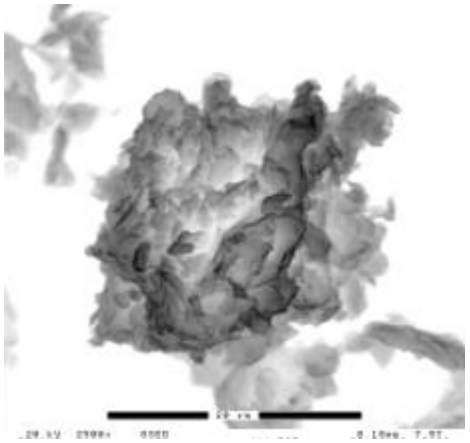
Point B
(h_{ra} = 28%);
initial configuration



Point C
(h_{ra} = 53%);
$\delta\varepsilon_{v\ 2D} \approx (3.5 \div 3.7)\%$



Point D
(h_{ra} = 71%);
$\delta\varepsilon_{v\ 2D} \approx (8.5 \div 9.0)\%$



Point E
(h_{ra} = 81%);
$\delta\varepsilon_{v\ 2D} \approx (12.1 \div 12.7)\%$

Figure A.2 Image analysis evolution of volume change behaviour (horizontal bar represents 20 μm).