NANOMORPHOLOGY OF ISOSTATIC PRESSED KAOLINS UNDER DRY AND WET CONDITIONS

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INTRODUCTION

Kaolin morphology has particular importance in paper filling and coating. Besides particle size distribution and shape, aspect ratio and aggregate structure have a dominant importance on the rheological behaviour of kaolin slurries, and in other properties such as opacity, gloss, printability and brightness (Zbik & Smart, 1998). On the other hand, Galán et al. (2006) have shown that isostatic pressure applied under dry/wet conditions produced variations on structural order degree of kaolinites. The results obtained in those experiments can be industrially used to improve or modify certain kaolin technological properties (viscosity, plasticity, brightness, BET, etc.) since they depend on kaolinite structural order. Thus, an industrial-pressure process under dry or wet conditions could optimize some of these industrial properties, inducing variations in kaolinite structural order.

The objectives of this investigation were to evaluate the changes produced by isostatic pressure on nanomorphology of kaolins and to determine the effect of the presence or absence of water during these experiments. These results were correlated with the variations on kaolinite structural order determined by Galán et al. (2006).

MATERIALS AND METHODOLOGY

Three samples containing kaolinites of different structural order were studied. Two of them, Alvaraes (Portugal) and Montecastelo (Spain), were formed by granite weathering, whereas the third, St. Austell (UK), is a hydrothermal alteration of granite. They were industrial (washed) kaolin samples used in ceramics, paper filler or coating, plastics and paints.

Isostatic pressure (compaction) was obtained by introducing a gum bag containing the sample into a high-pressure container filled with water, in this form the effect of pressure on the sample is similar in all directions, simulating isostatic pressure, but pressure was not measured in the sample. The time to reach the final pressure (4000 kg/cm^2) was 1 min and it was maintained for 10 min. In order to determine the influence of the presence of water, a water-kaolin (1:1) suspension was also used in the experiment.

Nanomorphology was evaluated by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM). The SEM study was carried out using Jeol JSM400 microscope with a field emission gun operating at 20 kV acceleration voltage. TEM study was performed with a Philips CM10 microscope operating at 100 kV. A Nanoscope III AFM (Digital Instruments) was used with a J-type scanner (150 μm scan range), working in «tapping mode». Phosphorus (n) doped Si tip-mounted cantilevers of nominal vibration frequency of about 300 kHz and spring constant k of 40 N/m were used.

RESULTS AND DISCUSSION

Montecastelo and Alvaraes kaolins present abundant stacks or «books», straight or curved (vermicular) formed by crystals of perfect pseudohexagonal morphology. Also twin of edges and angle very well defined were observed. Small aggregates of rounded crystals coexist with hexagonal kaolinite (Figure 1a and 1g). This morphology is similar to that described by Keller (1978) for weathering kaolins, with particle and books randomly oriented. On the other hand, in St. Austell kaolin the plates occur as single sheaves or thin packets, with crystals oriented face to face, rather than as expansive books. The present stacks are very compacted. In general kaolinite crystals of large size (> 1 μm) show pseudohexagonal morphology and well defined edges, whereas the edges of crystals of small size are rounded (Figure 1d). Some crystals of halloysite were also detected by TEM. This morphology is similar to that described by Keller (1978) for hydrothermal kaolins.

Confined pressure produces changes on the nanomorphology, but the behaviour is different if the compaction is performed in absence or in presence of

Sample	Roughness (nm)	I.dp	HI	AGFI
Montecastelo	230	29	1.10	1.19
Montecastelo 4000 kg cm^{-2} (dry)	160	---	0.59	0.62
Montecastelo 4000 kg cm^{-2} (wet)	50	43	1.49	1.45
St. Austell	200	24	0.89	0.97
St. Austell 4000 kg cm^{-2} (dry)	240	----	0.58	0.81
St. Austell 4000 kg cm^{-2} (wet)	140	38	1.03	1.30
Alvaraes	170	14	0.79	0.96
Alvaraes 4000 kg cm^{-2} (dry)	940	----	0.60	0.60
Alvaraes 4000 kg cm^{-2} (wet)	131	13	0.92	0.96

Table 1: Kaolin nanomorphology data compared to kaolinite structural order (ldp, HI and AGFI).

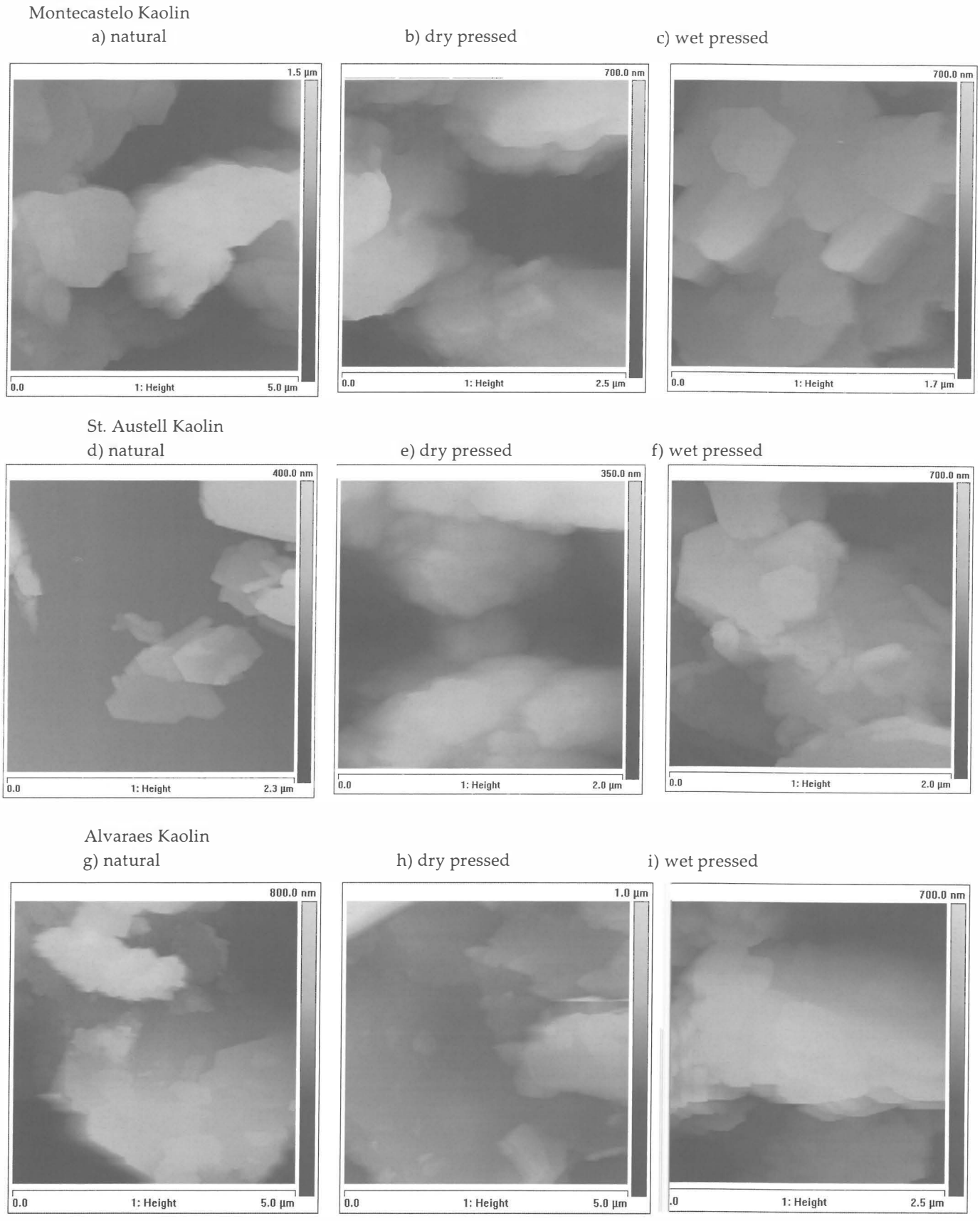

Figure 1: AFM images (height) of natural and pressed kaolins.

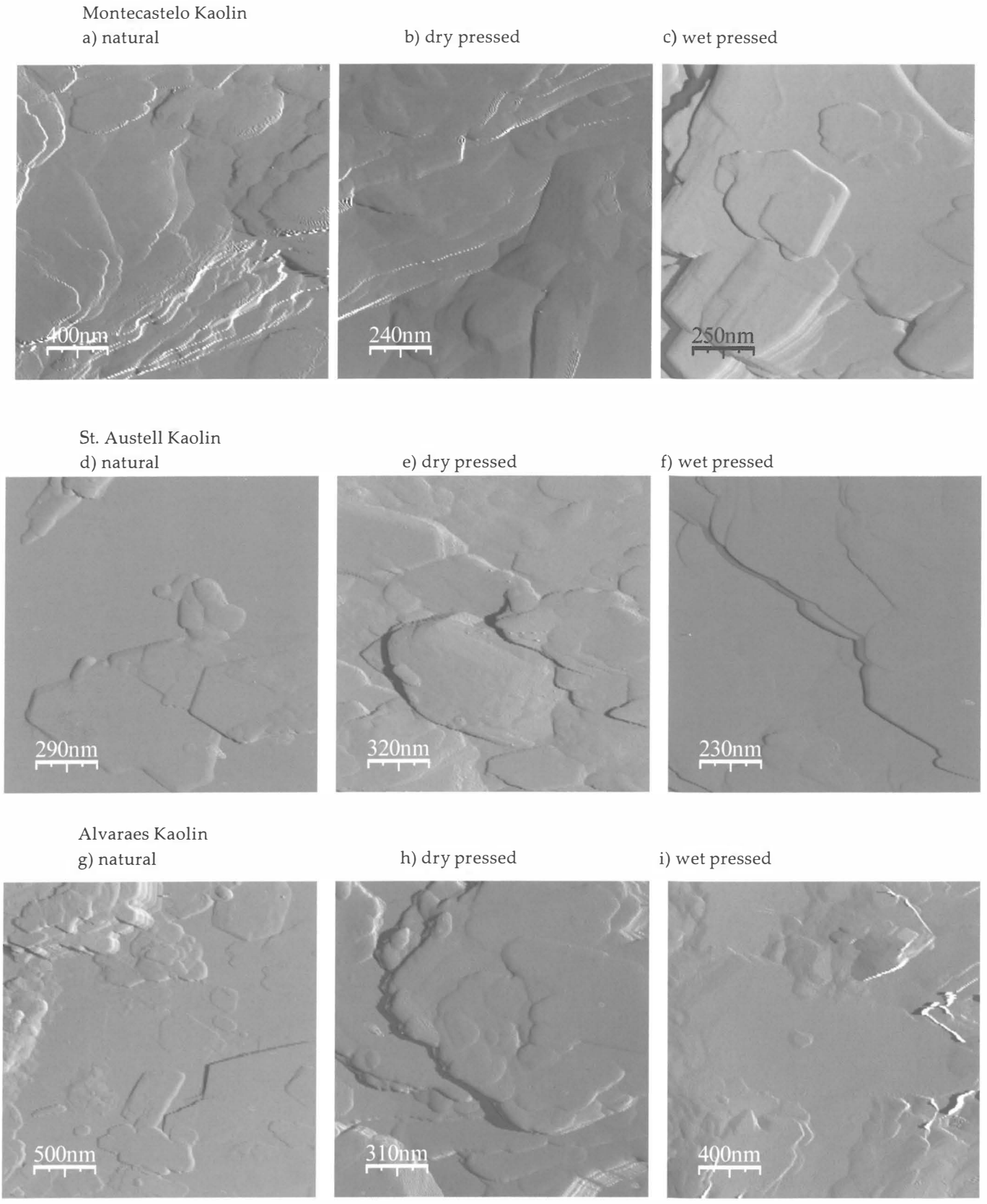

Figure 2: AFM images (amplitude) of natural and pressed kaolins.

water. During dry pressure kaolinite crystals gradually lost their pseudohexagonal morphology, edges were rounded and books and vermicular stacks were fractured and distorted (Figure 2b, 2e and 2h). By the contrary kaolinite morphology did not show apparently any change in wet conditions by SEM and TEM. Crystals maintained their pseudohexagonal morphology and particle-size. AFM study of wet pressed kaolinites shows an increment of edges perfection on kaolinite crystals and a low presence of deformed crystals (Figure 2c, 2f and 2i). Figure 1 reports height data which are quantitative for calculating nanomorphology, whereas Figure 2 reports the amplitude data which are more sensitive to the edge morphology of the crystals. On the other hand, halloysite detected in St. Austell kaolin appears fractured under dry pressed conditions and apparently unaffected under wet conditions.

In general isostatic pressure in dry conditions produces an increase of roughness whereas in wet conditions roughness decreased (Table 1, Figure 1). Alvaraes kaolin presents the biggest roughness increase in dry conditions probably due that this kaolin presents lower kaolinite structural order degree in natural conditions and lower particle size. These roughness changes are very interesting in relation with technological application of kaolin.

The nanomorphology change of kaolinite is inversely correlated with the variation of the degree of kaolinite structural order as determined by X-ray diffraction (Galán et al., 2006) (Table 1). In fact under dry conditions the percentage of low-defect phase (ldp) decreased, showing the Hickley index (HI) and Aparicio-Galán-Ferrell index (AGFI) a variation from low-defect to medium- (high)-defect kaolinite. On the contrary, under wet conditions the percentage of low-defect kaolinite was maintained or increased, and HI and AGFI increased.

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