

THE EFFECT OF PRESSURE ON ORDER/DISORDER IN KAOLINITE UNDER WET AND DRY CONDITIONS

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Abstract—Well ordered kaolinite was isostatically and uniaxially pressurized up to 13,200 kg/cm² for 10 min in dry conditions and the effects of pressure on kaolinite order were determined by analyzing the shapes of two-dimensional diffraction bands on X-ray powder diffraction patterns. Increased pressure decreased the percentage of low-defect kaolinite phase, and isostatic pressure proved to be more effective than uniaxial pressure in increasing disorder, *e.g.* the degree of disorder resulting from 2000 kg/cm² isostatic pressure was equivalent to that caused by a 3200 kg/cm² uniaxial pressure. Also, the effect of high pressure was similar to that obtained with lower pressures applied several times (*e.g.* the effect of applying 8500 kg/cm² pressure for 10 min was comparable to using 3200 kg/cm² pressure five times).

In addition, six kaolinites of different structural order were isostatically pressurized up to 4000 kg/cm² for 10 min, both in dry and wet (water) conditions. Under dry conditions, changes in structurally ordered kaolinite were comparable to those cited above whereas kaolinite pressurized in wet conditions showed a moderate improvement in structural order.

These results may contribute to our understanding of kaolinite behavior during burial diagenesis and low-grade metamorphism. In addition, these results can also be used in industry to improve kaolin technological properties that depend on kaolinite structural order by application of appropriate industrial pressure processes.

Key Words—Experimental Pressure Behavior, Kaolinite Order Disorder.

INTRODUCTION

Variation in the degree of kaolinite structural order can give rise to large changes in the intensity, shape and position of *hkl* reflections. With increasing disorder the 021, 111 reflections become increasingly broadened until only a smooth, asymmetric diffraction band results; the reflections 131, 201, which form two groups of triplets in less disordered kaolinites, become two doublets in disordered kaolinites (Brindley, 1980). The nature of this disorder has been studied for >50 y and yet unanswered questions remain, *e.g.* do natural disordered samples crystallize in a disordered state or have they been disordered by physical action? Might we expect kaolinite that crystallized with a partially disordered stacking sequence to be distinguishable from one that has suffered from natural physical action (Reynolds and Bish, 2002)?

Different models have been proposed to explain the nature of disorder in kaolinites based on: (1) random shifts by $\pm b/3$ (Brindley and Robinson, 1946); (2) rotations by $\pm 120^\circ$ (Murray, 1954); (3) octahedral vacancy displacement (Plançon and Tchoubar, 1977); (4) enantiomorphic B layers (Bookin *et al.* 1989); and (5) enantiomorphic B and some C layers (Plançon and Tchoubar, 1977; Bookin *et al.* 1989). Although the model proposed

by Brindley and Robinson (1946) explained the unaffected $k = 3n$ reflections and deterioration of $k \neq 3n$ reflections in disordered kaolinites, later theoretical consideration suggested that such translations were energetically unfavorable (Giese, 1982), and experimental and theoretical work led to the presently accepted and rather complex model of kaolinite disorder displacement (Plançon and Tchoubar, 1977; Bookin *et al.*, 1989; Plançon and Zacharie, 1990). This model includes three types of defects: defects in the placement of the vacant octahedral site; two possible translation directions between layers of approximately $-a/3$ and $-a/3+b/3$; and modification of these interlayer translations by random shifts (Reynolds and Bish, 2002).

The effects of grinding on kaolinite have been studied widely (Laws and Page, 1946; Gregg *et al.*, 1954; Takahashi, 1959; De Luca and Slaughter, 1985; Gonzalez Garcia *et al.*, 1991; Reynolds and Bish, 2002) and the most important conclusions obtained from these studies can be summarized as follows: (1) grinding changes both the size and shape of particles and pore distribution; (2) grinding affects the physico-chemical properties (specific surface area, cation exchange capacity, water absorption and acidic media solubility); and (3) grinding decreases kaolinite structural order and induces structural changes. Reynolds and Bish (2002) showed that disorder in high-defect kaolinite is caused by the interstratification of normal kaolinite layers with their enantiomorphs, and that grinding of

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Table 1. Description of the kaolins studied.

Kaolins	Origin	References
St. Austell (UK)	Hydrothermal alteration of granite	Bristow (1993)
Lipari (Italy)	Hydrothermal alteration of andesite and rhyolite	Morandi <i>et al.</i> (1992)
Alvaraes (Portugal)	Granite weathering	Gomes <i>et al.</i> (1990)
Montecastelo (Spain)	Granite weathering	E. Galán (pers. comm.)
Poveda de la Sierra (Spain)	Sedimentary (Cretaceous)	Galán and Martín Vivaldi (1973)
Warren (Georgia, USA)	Sedimentary (Tertiary)	Patterson and Murray (1975), van Olphen and Fripiat (1979)

kaolinites does not produce a progressive increase in disorder for all the crystallites present in a sample; instead, grinding apparently creates increased amounts of a disordered kaolinite that coexist with unaffected material.

The decrease of structural order in kaolinite caused by grinding can also be produced by high static pressures. Range *et al.* (1969) showed that static pressures, ranging from 1 to 15 kbar, produced strong disorder in kaolinite that was originally well ordered, but crystal morphology was essentially preserved after the pressure treatment. Later, La Iglesia (1993) showed that pressure can induce both disorder in kaolinite and crystal morphological changes, in the form of fractures, bends and deformation of the sheets. Recently, Johnston *et al.* (2002) showed that high pressures can induce a structural phase transition in dickite, between 2.0 and 2.5 GPa. This phase transition consisted of a shift in the layers, with the stacking sequence and interlayer hydrogen bonding topology changing significantly. Although the space group of the high-pressure form of dickite remained the same, the β angle was reduced from 96.7° at low pressure to 90.4° at high pressure.

The objectives of this investigation were to evaluate the changes produced in kaolinite by uniaxial and

confined pressure and to determine the effect of the presence or absence of water during these experiments. The resulting data may contribute to the interpretation of processes occurring in kaolinite-rich rocks during burial diagenesis and low-grade metamorphism and can potentially be used to improve technological properties of kaolin.

EXPERIMENTAL

Kaolin characterization

Six samples containing kaolinites of different structural order were studied (Table 1). Most were industrial (washed) kaolin samples used in ceramics, paper filler or coating, plastics and paints. Mineralogical analyses of kaolins were performed with a Philips PW1130/90 X-ray diffractometer using Ni-filtered $\text{CuK}\alpha$ radiation and an automatic divergence slit. Bulk quantitative analyses were based on the Schultz (1964) method, after correcting intensities for the automatic slit. Clay minerals were studied in oriented aggregates using standard methods involving drying at room temperature, solvation with ethylene glycol, and heating at 350 and 550°C for 2 h. Phase abundances were semi-quantitatively estimated according to mineral intensity factors

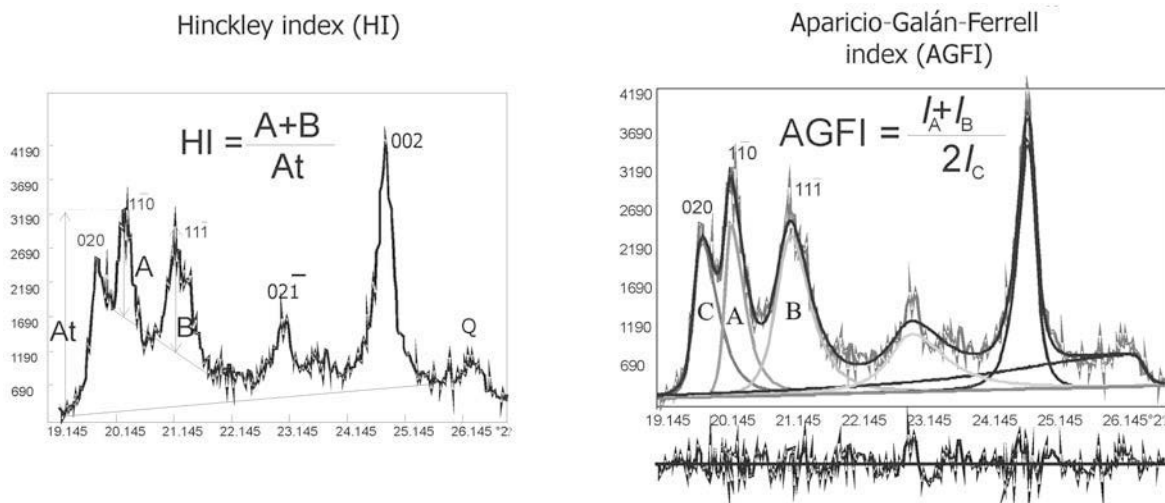


Figure 1. Measurement of the Hinckley index and the Aparicio-Galán-Ferrell index.

Table 2. Mineralogical compositions (wt.%) of the kaolins.

	Kaolinite	Halloysite	Quartz	Illite	Silica gels	Alunite
St. Austell	95	x	x	x	x	—
Lipari	75	—	—	5	x	15
Alvaraes	95	x	<5	—	—	—
Montecastelo	98	—	x	x	—	—
Poveda	95	x	<5	<5	—	—
Warren	98	x	<5	<5	—	—

x: Observed using SEM/TEM

proposed by Martín Pozas *et al.* (1969) and Martín Vivaldi *et al.* (1968). This methodology is similar to that later generalized by Chung (1974). In order to complete the mineralogical characterization, samples were observed by scanning electron microscopy (SEM) using a Jeol JSM-5400 electron microscope and by transmission electron microscopy (TEM) with a Philips CM10 instrument. Chemical composition was determined by atomic absorption/emission spectrometry.

Changes in the diffraction pattern produced by pressure were evaluated using the Hinckley Index (HI) (Hinckley, 1963), the Aparicio-Galán-Ferrell Index (AGFI) (Aparicio *et al.*, 1999, 2001) (Figure 1), and the 'expert system' of Plançon and Zacharie (1990). The last method provides quantitative information on kaolinite structural order-disorder, such as the number of different phases in the sample (1 or 2). For a biphasic system it establishes the percentage of low-defect phase (% ldp), and for single-phase samples the system determines the following parameters: amount of C layers (Wc), variation of interlayer translation about mean values (δ), proportion of translation defect (p), and mean number of layers (M). A side-loaded sample holder was used to minimize preferred orientation, and diffraction measurements were determined in quintuplicate. According to Aparicio and Galán (1999), the HI is influenced by the presence of quartz, feldspar, Fe hydroxide gels, illite, smectite and halloysite. The AGFI is less influenced by associated minerals and X-ray amorphous phases than the HI (Aparicio *et al.*, 1999, 2001).

Kaolinite accounted for 80–98 wt.% of all the samples (Table 2), accompanied by quartz and illite as

minor phases (<5 wt.%, by XRD). The presence of halloysite and silica gels was evaluated by SEM and TEM. Alunite is an impurity (15%) only in the Lipari kaolin. Chemical compositions were consistent with mineralogy for all samples, and the following findings (Table 3) are worth special note: (1) the Fe content in the Alvaraes kaolin (1.19%) can be ascribed to Fe oxide, tested by TEM/EDS; (2) the TiO₂ content in the Georgia kaolin is due to anatase and/or rutile; and (3) the high SiO₂/Al₂O₃ ratio in the St. Austell (1.3), Alvaraes (1.33), and specially in the Lipari kaolin (1.29) is due to the presence of quartz, alunite, silica gels, tested by TEM/EDS, and the low kaolinite content in Lipari kaolin (~80 wt.%).

The 'expert system' of Plançon and Zacharie (1990) suggested that all kaolins consist of two kaolinite phases, a low-defect kaolinite and a disordered kaolinite. The Lipari, Montecastelo, Warren and St. Austell kaolins present a higher percentage of low-defect phase (% ldp) and the Alvaraes and Poveda kaolins have lower values of %ldp (Table 4). According to the AGFI, all kaolins contain low-defect kaolinite, and the HI indicated the presence of low-defect kaolinite, except for the Alvaraes and Warren kaolins which contain medium-defect kaolinite (Table 4).

Experimental design

Poveda kaolin was used during the first part of the experiment to evaluate the effect on kaolinite of three different pressure conditions: (1) confined or isostatic; (2) uniaxial; and (3) uniaxial n times. Isostatic pressure was obtained by introducing a gum bag containing the

Table 3. Chemical compositions (wt.%) of kaolins.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	Loss on ignition	Total	SiO ₂ /Al ₂ O ₃
Theoretical	46.55	39.49							13.96	100	1.179
St. Austell	47	36	0.28	0.15	0.03	0.22	0.21	—	14.74	98.6	1.306
Lipari	45	35	0.47	0.08	0.49	0.17	0.34	—	18.50	100.1	1.286
Alvaraes	46	36	1.19	0.10	0.04	0.15	0.15	—	14.77	100.4	1.333
Montecastelo	46	39	0.56	0.09	0.02	0.09	0.47	—	13.70	99.9	1.179
Poveda	47	39	0.60	0.11	—	0.16	0.24	0.10	13.70	100.9	1.205
Warren	46	39	0.43	0.05	—	0.10	0.14	1.20	13.70	100.6	1.179

Table 4. Results of % low-defect phase (% ldp), Hinckley index (HI) and Aparicio-Galán-Ferrell index (AGFI). \bar{X} : mean value; σ_{n-1} : standard deviation value.

	% ldp		HI		AGFI	
	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}
St. Austell	24	0.933	0.89	0.011	0.97	0.018
Lipari	41	1.030	1.13	0.034	0.98	0.029
Alvaraes	14	0.976	0.79	0.046	0.96	0.051
Montecastelo	29	2.039	1.10	0.021	1.19	0.020
Poveda	23	1.687	0.89	0.034	0.91	0.023
Warren	26	3.200	0.83	0.028	1.05	

sample into a high-pressure container filled with water (Figure 2); in this configuration 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 was 1 min and it was maintained for 10 min. The isostatic pressures used were 500, 1000, 1500 and 2000 kg/cm².

Uniaxial pressure was obtained by introducing the sample between two pistons (Figure 2). The pressure was gradually increased to the maximum, which was maintained for 5 min. The uniaxial pressures reached were 3200, 8500, 10800 and 13200 kg/cm². Uniaxial n times experiments consisted of the application of uniaxial pressure of 3200 kg/cm² 2, 3, 5, 10 and 14 times. The procedure involved pressing, dismounting, grinding, and repeating the cycle n times.

The results obtained with the Poveda kaolin during the three pressure programs indicated that isostatic pressure was more effective than uniaxial pressure in increasing disorder. Therefore this procedure was used during the second part of this study and was applied to the other kaolins (Table 1). In order to determine whether the presence of water can prevent the disordering of kaolinite or promote its evolution to another polytype, a water-kaolin (1:1) suspension was also used in the experiment. The range of pressure during this step varied between 1000 and 4000 kg/cm² for 10 min.

RESULTS AND DISCUSSION

After pressure treatment the Poveda kaolin showed broadening of the 02 l and 11 l reflections (20–23°2 θ CuK α) independently of the pressure system used, whereas the 13 l , 20 l band (35–40°2 θ CuK α) was only affected by uniaxial pressures (Figure 3). According to the ‘expert system’, these changes in XRD patterns are associated with structural changes in kaolinite (Table 5), although it should be noted that when the percentage of low-defect phase is ~10% the expert system is not reproducible, because the results of the five measurements of each sample are not coincident, sometimes detecting two phases and sometimes only one disordered phase.

Isostatic pressure decreased % ldp from 23.3 to 9.7%, but the 13 l , 20 l band was not affected. According to Reynolds and Bish (2002), this type of disorder involves interstratified enantiomorphs or another type of disorder which, because of the symmetry of the layers or their translations, does not affect the $k = 3n$ reflections (Figure 3).

Uniaxial pressure decreased % ldp to 9 at 3200 kg/cm², but under higher pressures only a disordered phase was found, characterized by: (1) the probability of shifts of $-a/3+b/3$ of 0.29; (2) the absence of C layers; (3) a variation of interlayer translation about mean values of 0.03; and (4) a mean number of layers of

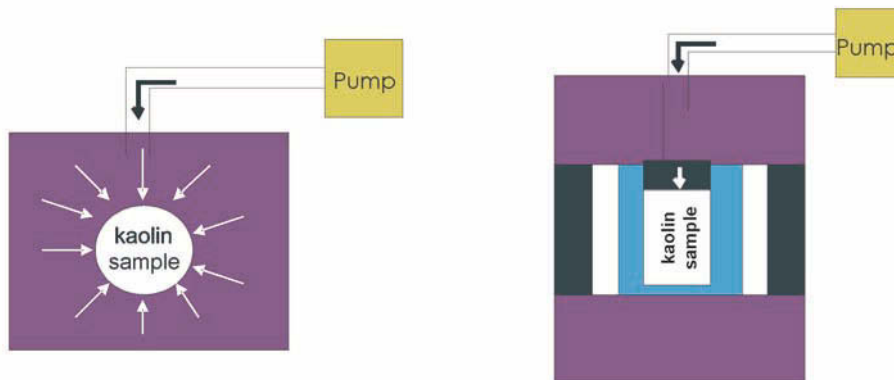


Figure 2. Schematic representation of the two different pressure methods.

Table 5. Results for the Poveda kaolin following application of the expert system of Plançon and Zacharie under isostatic and uniaxial pressure.

	Two kaolinite phases		One disordered kaolinite phase							
	% ldp	σ_{n-1}	p	σ_{n-1}	Wc	σ_{n-1}	δ	σ_{n-1}	M	σ_{n-1}
Poveda kaolin	23.0	1.761								
Isostatic pressure										
500 kg/cm ²	12.89	1.297								
1000 kg/cm ²	10.81	0.626								
1500 kg/cm ²	10.10	1.247								
2000 kg/cm ²	8.67	1.375								
Uniaxial pressure										
3200 kg/cm ²	9.86	1.611								
8500 kg/cm ²			0.29	0.016	0.00	0.00	0.02	0.055	42	0.00
10800 kg/cm ²			0.29	0.027	0.00	0.00	0.03	0.00	42	0.00
13200 kg/cm ²			0.28	0.020	0.00	0.00	0.03	0.00	41	0.00
Uniaxial n times										
2 times	9.68	1.210								
3 times	9.75	0.254								
5 times			0.33	0.023	0.00	0.00	0.03	0.008	41	0.00
10 times			0.33	0.027	0.00	0.00	0.03	0.00	41	0.00
14 times			0.33	0.025	0.00	0.00	0.03	0.005	41	0.00

% ldp = percentage of low-defect phase

p = proportion of translation defect

Wc = amount of C layers

δ = variation of interlayer translation about mean values

M = mean number of layers

σ_{n-1} : standard deviation value.

41. The diffraction pattern showed severe demodulation of the $k = 3n$ peaks and broadening of the 021 and 111 reflections, so the defects involved interstratified enantiomorphs and random translations.

The effect of applying a 8500 kg/cm² pressure for 10 min was comparable to that of applying a 3200 kg/cm² pressure five times, which gave a disordered phase

characterized by the same parameters as if the pressure were 8500 kg/cm², except that the probability of shift by $-a/3 + b/3$ increased slightly (0.33).

The comparison of the two methods of pressurization showed that isostatic pressure is more effective than uniaxial pressure in increasing disorder; the degree of disorder resulting from 2000 kg/cm² isostatic pressure

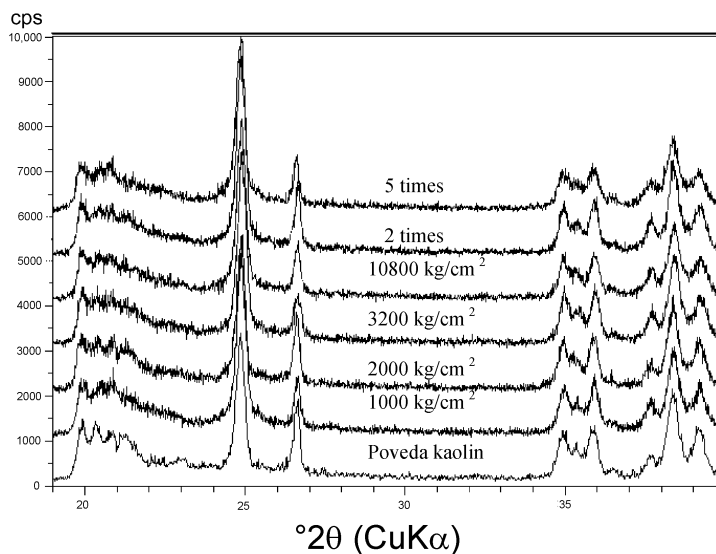


Figure 3. Effect of the isostatic and uniaxial pressure on the XRD pattern of Poveda kaolin.

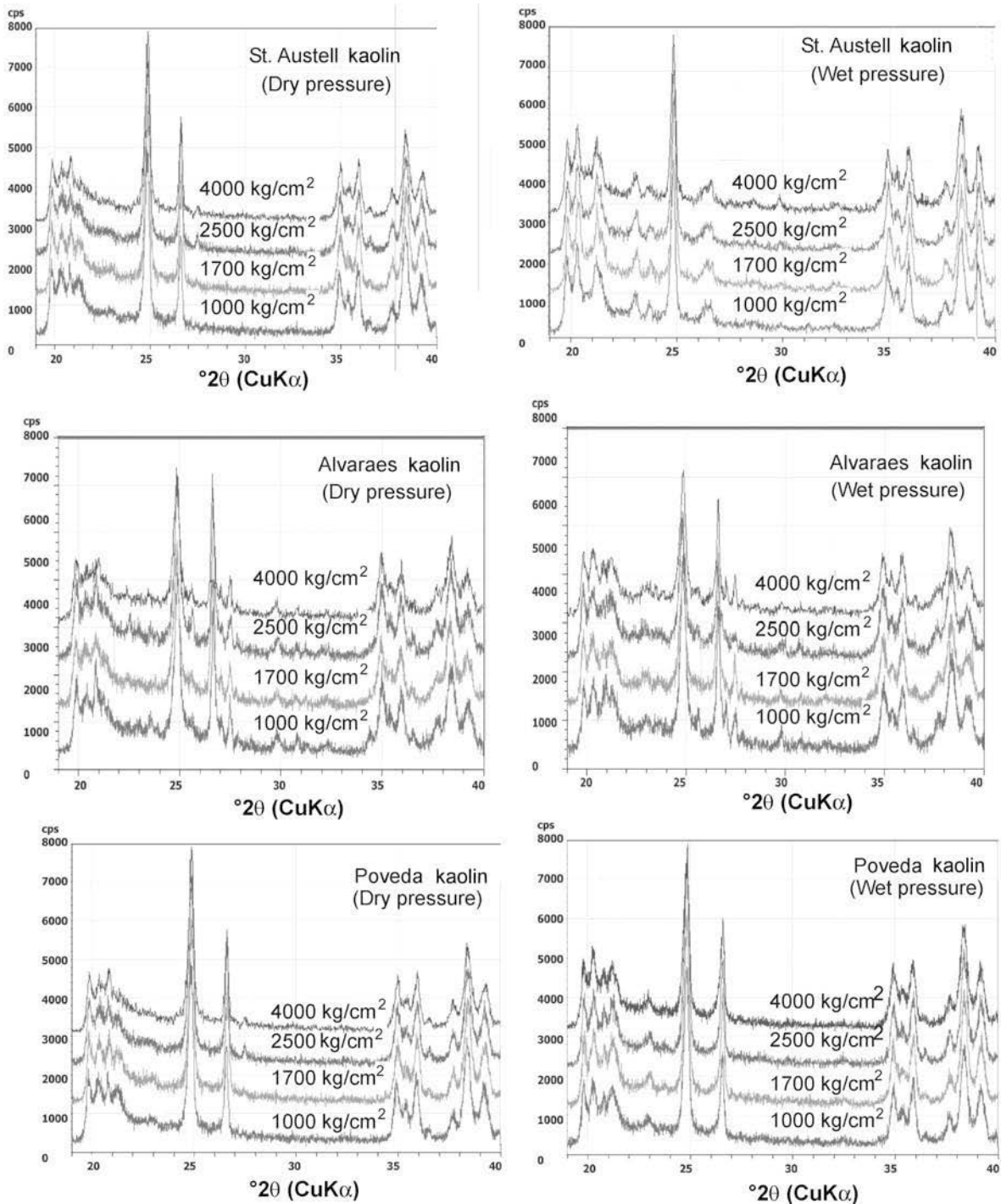


Figure 4. Variations in XRD patterns for three kaolins under dry- and wet-pressure conditions.

was equivalent to that caused by a 3200 kg/cm² uniaxial pressure.

When isotatic pressure was applied under dry conditions to other kaolins, the 02 l and 11 l reflections were mainly affected, but the 13 l , 20 l reflections were only affected at the highest pressure (4000 kg/cm²). On the

contrary, hydrous pressure conditions did not appear to affect the XRD patterns. Figure 4 illustrates the variations of the XRD patterns in three kaolins, both in dry and wet conditions. In all cases, the sequence of reflections most affected by pressure was the 02 l reflection in dry conditions, but this reflection was unaffected in wet conditions.

Table 6. Effects of pressure on kaolinite order deduced by the application of the expert system of Plançon and Zacharie (1990) in dry and wet conditions.

(a) Variation of percentage of low-defect kaolinite phase (%ldp)

	1000 kg/cm ²		1700 kg/cm ²		2500 kg/cm ²		4000 kg/cm ²	
	<i>X</i>	σ_{n-1}	<i>X</i>	σ_{n-1}	<i>X</i>	σ_{n-1}	<i>X</i>	σ_{n-1}
St. Austell dry	13.4	0.7304	11.6	1.3610	12.1	1.0532	9.7	1.8337
St. Austell wet	26.5	1.2040	35.0	1.7502	37.0	1.4117	38.0	2.6305
Lipari dry	30.0	1.2907	23.0	2.2250	20.0	3.8831	1 phase	
Lipari wet	41.0	3.1358	42.0	2.7693	43.0	1.0581	43.1	3.4201
Poveda dry	1 phase		1 phase		1 phase		1 phase	
Poveda wet	10.3	1.2105	11.6	1.7562	12.9	1.4593	13.2	1.5236
Warren dry	13.5	1.7288	10.5	1.1584	10.5	1.9062	1 phase	
Warren wet	20.6	4.4786	22.6	2.1812	24.0	3.8026	24.5	3.4521
Alvaraes dry	1 phase		1 phase		1 phase		1 phase	
Alvaraes wet	9.9	0.7279	10.7	1.2788	11.2	1.3949	22.2	2.8534
Montecastelo dry	1 phase		1 phase		1 phase		1 phase	
Montecastelo wet	40.0	3.1736	42.2	1.0993	43.4	1.4095	43.3	2.8553

X: mean value

σ_{n-1} : standard deviation

(b) Characteristic parameters of disordered kaolinite phase.

		Lipari dry	Poveda dry	Alvaraes dry	Montecastelo dry
1000 kg/cm ²	<i>M</i>		31	25	63
	σ_{n-1}		0.00	0.00	0.00
	<i>Wc</i>		0.00	0.00	0.00
	σ_{n-1}		0.00	0.00	0.00
	δ		0.03	0.03	0.03
	σ_{n-1}		0.0493	0.0034	0.04
	<i>p</i>		0.21	0.31	0.154
1700 kg/cm ²	σ_{n-1}		0.0643	0.0048	0.0055
	<i>M</i>		31	31	63
	σ_{n-1}		0.00	0.00	0.00
	<i>Wc</i>		0.00	0.00	0.00
	σ_{n-1}		0.00	0.00	0.00
	δ		0.03	0.03	0.016
	σ_{n-1}		0.0424	0.00	0.0089
2500 kg/cm ²	<i>p</i>		0.22	0.26	0.178
	σ_{n-1}		0.0052	0.0068	0.0084
	<i>M</i>		31	31	63
	σ_{n-1}		0.00	0.00	0.00
	<i>Wc</i>		0.00	0.00	0.00
	σ_{n-1}		0.00	0.00	0.00
	δ		0.03	0.03	0.022
4000 kg/cm ²	σ_{n-1}		0.0087	0.0043	0.0045
	<i>p</i>		0.29	0.31	0.232
	σ_{n-1}		0.0380	0.0451	0.0084
	<i>M</i>	42	38	36	61
	σ_{n-1}	0.00	0.00	0.00	0.00
	<i>Wc</i>	0.00	0.00	0.07	0.00
	σ_{n-1}	0	0	0	0
	δ	0.0240	0.03	0.032	0.022
	σ_{n-1}	0	0.0565	0.0045	0.0447
	<i>p</i>	0.172	0.26	0.272	0.232
	σ_{n-1}	0.0356	0.0745	0.0521	0.0084

M = mean number of layers

Wc = amount of C layers

δ = variation of interlayer translation about mean values

p = proportion of translation defect

σ_{n-1} : standard deviation value

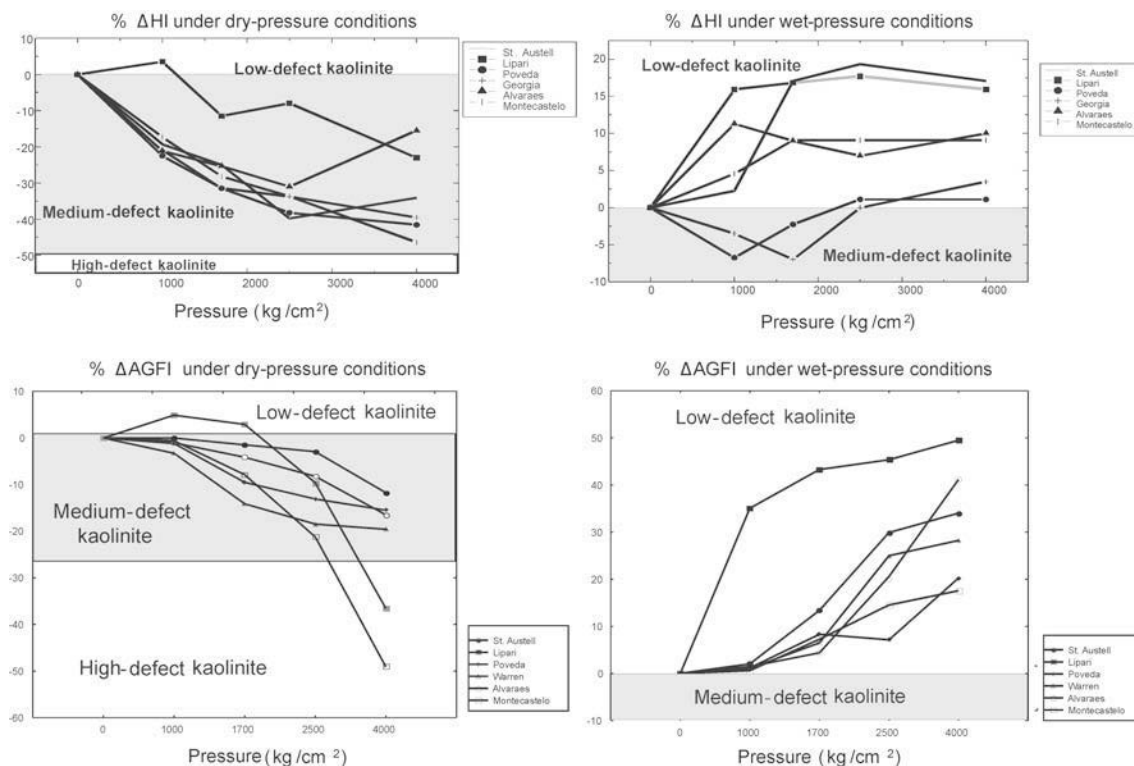


Figure 5. Variation percentages of Hincley index (HI) and Aparicio-Galán-Ferrell index (AGFI) under dry- and wet-pressure conditions.

Under isostatic pressure in dry conditions the percentage of low-defect phase decreased with increasing pressure for all kaolins (Table 6a). A disordered phase also appeared for the Poveda, Alvaraes and Montecastelo kaolins under all pressures used (Table 6b). It is noteworthy that: (1) for the Alvaraes kaolin the mean of the layers increased from $M = 25$ to 36, and C layers were detected at 4000 kg/cm^2 ; (2) for the Montecastelo kaolin the probability of shifts by $-a/3+b/3$ varied slightly from 0.15 to 0.23.

The influence on the XRD patterns is also shown in the variations of HI and AGFI (Table 7), especially for the Lipari and Montecastelo kaolins. The HI and AGFI presented a low standard deviation in comparison with data from the expert system. For this reason and in order to compare the effect of pressure in all the kaolins at the same time, the percentage variation of HI and AGFI was plotted (Figure 5). Under dry conditions, HI and AGFI showed that increasing pressure caused a variation from low-defect kaolinite to medium- (high-) defect kaolinite for all the kaolins tested. On the contrary, under wet conditions, the percentage of low-defect phase increased for the St. Austell, Montecastelo, Alvaraes and Lipari kaolins, and it remained unchanged for the other two kaolins (Table 7). These results are consistent with those obtained using the AGFI, with the Montecastelo and St. Austell kaolins having highest values. However, the HI behavior was not in agreement with expert system results, probably because it is more affected by XRD

pattern variations produced by the effect of the pressure than the AGFI.

These results, showing the results of pressure on kaolinite order/disorder, can contribute to our understanding of the effects of pressure in burial diagenesis and low-grade metamorphic environments, where kaolinite can increase in structural order and transform to dickite, but it also can be destroyed or react to form pyrophyllite, *etc.* (Kossovskaya and Shutov, 1963; Ehrenberg *et al.*, 1993; Ruiz Cruz and Andreo, 1996). These results also show how pressure can disorder kaolinite in a dry environment, and on the contrary, how under wet conditions structural order can be improved or at least maintained. But under diagenetic and low-grade metamorphic conditions, where kaolinite structural order and crystal size generally increase, other factors such as temperature, time, rock porosity and composition, pore-water composition and pH can contribute to modifying kaolinite behavior with increasing pressure. In many environments, these other parameters can well have more influence on kaolinite/dickite transformation than pressure.

In addition, the results obtained in these experiments can be also used in industrial applications to improve or modify certain kaolin technological properties (viscosity, plasticity, brightness, BET, ...), many of which depend on kaolinite structural order. Kaolins containing disordered kaolinite are suitable for ceramics and as fillers for the paper industry, but well-ordered kaolinites

Table 7. Results of Hinckley index (HI) and Aparicio-Galán-Ferrell index (AGFI) under dry and wet isostatic pressure.

(a) HI

	1000 kg/cm ²		1700 kg/cm ²		2500 kg/cm ²		4000 kg/cm ²	
	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}
St. Austell dry	0.71	0.04060	0.66	0.0741	0.53	0.0452	0.58	0.0709
St. Austell wet	0.90	0.00780	1.03	0.0226	1.05	0.0574	1.03	0.0405
Lipari dry	1.17	0.05200	1.00	0.0430	1.04	0.0923	0.87	0.0789
Lipari wet	1.31	0.03540	1.32	0.0846	1.33	0.0333	1.31	0.0565
Poveda dry	0.69	0.02570	0.61	0.0390	0.55	0.0475	0.52	0.0557
Poveda wet	0.83	0.03900	0.87	0.0376	0.90	0.0696	0.92	0.0298
Warren dry	0.68	0.05660	0.59	0.0554	0.57	0.0523	0.52	0.0538
Warren wet	0.83	0.02800	0.80	0.0684	0.83	0.0178	0.89	0.0808
Alvaraes dry	0.56	0.09480	0.53	0.0518	0.49	0.0222	0.60	0.1626
Alvaraes wet	0.79	0.03560	0.80	0.0490	0.73	0.0502	0.92	0.0335
Montecastelo dry	0.91	0.01150	0.79	0.0560	0.73	0.0394	0.59	0.0303
Montecastelo wet	1.15	0.01550	1.20	0.0475	1.20	0.0441	1.99	0.0315

(b) AGFI

	1000 kg/cm ²		1700 kg/cm ²		2500 kg/cm ²		4000 kg/cm ²	
	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}	\bar{X}	σ_{n-1}
St. Austell dry	0.96	0.0458	0.93	0.0659	0.89	0.0923	0.81	0.0340
St. Austell wet	0.99	0.0334	1.10	0.0391	1.26	0.0347	1.30	0.0745
Lipari dry	1.64	0.0384	1.52	0.0495	1.30	0.0449	0.84	0.0338
Lipari wet	1.61	0.0182	1.77	0.0062	1.89	0.0356	1.94	0.0301
Poveda dry	0.83	0.0535	0.76	0.0281	0.73	0.0254	0.71	0.0609
Poveda wet	0.85	0.0398	0.91	0.0219	0.90	0.0309	1.01	0.0328
Warren dry	0.89	0.0165	0.79	0.0283	0.75	0.0171	0.74	0.0510
Warren wet	1.18	0.0159	1.15	0.0756	0.98	0.0374	0.91	0.0322
Alvaraes dry	0.68	0.0318	0.67	0.0205	0.66	0.0292	0.60	0.1845
Alvaraes wet	0.69	0.0176	0.71	0.0527	0.82	0.0227	0.96	0.0514
Montecastelo dry	1.02	0.0199	1.00	0.0168	0.88	0.0274	0.62	0.0481
Montecastelo wet	1.31	0.0304	1.39	0.0207	1.41	0.0393	1.45	0.0316

 \bar{X} : mean value σ_{n-1} : standard deviation value

are more appropriate for coating-grade rubber or paints. Thus, an industrial-pressure process under dry or wet conditions could optimize some of these industrial properties, inducing variations in kaolinite structural order.

CONCLUSIONS

Comparison of the two methods of pressure treatment showed that confined (isostatic) pressure is more effective than uniaxial pressure in increasing kaolinite disorder. For a given time of treatment, the degree of disorder resulting from 2000 kg/cm² isostatic pressure was equivalent to that caused by 3200 kg/cm² uniaxial pressure. Isostatic pressure applied under dry conditions primarily affected the 02l, 11l reflections, so this disorder must involve interstratified enantiomorphs or

other types of disorder that does not affect the $k = 3n$ reflections. Isostatic pressure under dry conditions caused the percentage of low-defect phase to decrease and the HI and AGFI showed 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, the XRD pattern was not affected apparently, and HI and AGFI increased.

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