

Mineralogical composition and diagenetic processes in the two depositional systems of the Cerro Negro Formation, Buenos Aires, Argentina: industrial application

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ABSTRACT

Clay mineral composition and diagenetic processes of the two depositional systems recognized in the Cerro Negro Formation (Precambrian) were analyzed. The Cerro Negro Formation consist of calcareous and siliciclastic rocks of brown-olive to grey-greenish and grey-reddish colours. Most of the clay minerals recognize a detrital origin but *in situ* transformation and superimposed diagenetic processes were also detected. The clay fraction ($<4 \mu\text{m}$) of the lower depositional system (LDS) is predominantly illitic. The upper depositional system (UDS) also contains chlorite-smectite and smectite and it is separated from the lower depositional system by an undulate paleosurface. Yet, chlorite-smectite and scarce smectite were detected at the top of the lower depositional system. Illite crystallinity (IC) measured in thin deposits (3 mg cm^{-2}) of the $<2 \mu\text{m}$ fraction, situates the sediments of both depositional systems in the diagenetic zone, according to the limits adopted in 1990 ($>0.42^\circ \Delta 2\theta$ at 2°min^{-1} , and $\text{TC}=1\text{s}$). The illitic material is aluminous, with an Intensity Ratio parameter >1 , showing that it is expansive, with $<15\%$ of expansible layers: I+ISII. Differences in the clay mineral composition are attributed here to changes in the paleoenvironmental conditions. The sediments of the lower depositional system were deposited under predominantly subtidal and to a lesser extent intertidal conditions, while subtidal and more intertidal conditions allowed chlorite-smectite to form *in situ* from smectite in the upper depositional system. Superimposed diagenetic processes affected the illitic material which shows the same characteristics in the two depositional systems. Based on technological analyses, all the samples could be suitable for making good red ceramic products of low water absorption, with some vitrification.

Key words. Mineralogy, Illite crystallinity, Paleoenvironments, Diagenesis, Technology, Cerro Negro Formation, Argentina.

RESUMEN

Composición mineralógica y procesos diagenéticos en los dos sistemas deposicionales de la Formación Cerro Negro, Buenos Aires, Argentina: aplicación industrial. La composición mineralógica de las arcillas de la Formación Cerro Negro (Precámbrico) fue analizada en los dos sistemas deposicionales que la componen. Las sedimentitas están constituidas por rocas calcáreas y silicoclásticas de tonalidades castaño-oliva, gris-verdosas y gris-rojizas. Las arcillas reconocen, en su mayor parte, un origen detrítico, aunque se han producido transformaciones y procesos diagenéticos sobrepuestos. La fracción arcilla ($< 4 \mu\text{m}$) del sistema deposicional inferior está compuesta fundamentalmente por material illítico. El sistema deposicional superior, separado del sistema deposicional inferior por una paleosuperficie ondulada contiene, además, clorita-esmectita y esmectita, las cuales comienzan a detectarse en el techo del sistema deposicional inferior. El índice de cristalinidad de la illita (IC), medido sobre depósitos delgados (3 mg cm^{-2}) de la fracción $< 2 \mu\text{m}$, ubica a las sedimentitas de ambos sistemas deposicionales en la zona de diagénesis, de acuerdo a los límites adoptados en 1990 ($> 0,42^\circ \Delta 2\theta$ a 2°min^{-1} , y $\text{TC}=1\text{s}$). El material illítico es aluminoso, con un parámetro de relación de intensidades > 1 , indicando expansibilidad, con $< 15\%$ de capas expansivas y clasificado como I+ISII. El cambio en la composición mineralógica de las arcillas de los dos sistemas deposicionales se atribuye a variaciones en las condiciones ambientales. Los sedimentos del sistema deposicional inferior se depositaron bajo condiciones fundamentalmente submareales y en menor proporción intermareales, mientras que la presencia de clorita-esmectita en el sistema deposicional superior es atribuida a transformación a partir de esmectita en ambientes con mayor influencia de condiciones intermareales. Procesos diagenéticos sobrepuestos afectaron al material illítico, el cual presenta las mismas características en ambos sistemas deposicionales. De acuerdo con los ensayos tecnológicos realizados, las arcillas son adecuadas para su utilización en la obtención de muy buena cerámica roja, con baja absorción de agua y algo de vitrificación.

Palabras claves: Mineralogía, Cristalinidad de la illita, Paleoambientes, Diagénesis, Tecnología, Formación Cerro Negro, Argentina.

INTRODUCTION

Recently, the Proterozoic Cerro Negro Formation (Iñiguez and Zalba, 1974) has been divided into two depositional systems on the basis of stratigraphic and sedimentological evidence (Andreis *et al.*, 1992). Up to the research carried out by Andreis *et al.* (1992) all the previous observations have been made on what was called the upper depositional system.

The aim of this paper is to analyze clay mineral composition and the incidence of diagenetic processes through illite crystallinity on pelitic rocks of both sequences. Furthermore, technological properties of the clay deposits were also analyzed in order to determine their industrial application.

GEOLOGICAL SETTING

According to Andreis *et al.* (1992), the lower depositional system (LDS) crops out in the San Martín-Sierras Bayas area and in the La Providencia quarry (Fig. 1), while the upper depositional system (UDS) is found at the locality of Cerro Negro and the San Martín quarry. Maximum thicknesses of up to 50 and 20 meters respectively, were measured. The above mentioned authors showed that the LDS sequences are composed of siliciclastic, calcareous, siliceous and phosphatic rocks, with predominant reddish colours. The UDS sequences, on the contrary, include muddy, heterolithic and subordinate sandy facies, with greenish

to reddish hues (Figs. 2 and 3). Both, the lower and the upper sequences are separated by a gently undulate paleosurface.

From sedimentological evidence (bedding, lithofacies, cyclicity, paleocurrents, among others), sediments related to the LDS were deposited during two phases of a major transgression event into an epeiric basin, under subtidal conditions. First, reddish phosphate and cherty sediments were deposited in shallow bays, followed by the deposition of calcareous sediments in a rather muddy and warm water sea.



FIG. 1. Location maps.

The UDS sediments were deposited in an epicontinental open sea, probably in near-shore areas related to a renewed sea-level rise. The sequences reflect deposition under subtidal to intertidal conditions, with wave action, periodically intensified by storms. Thin, localized, low-density turbidites are attributed to this storm activity. Nevertheless, other similar turbidites, associated to mass gravity slumps (westward oriented) may have been related to syndepositional tectonic causes, mainly produced by earthquakes along active faults located toward the northern basin border (Iñiguez *et al.*, 1989).

The upper depositional system has been found not only in the Sierras Bayas area, but it also extends regionally up to the Barker area (207 km southeast from Sierras Bayas). Today, we can assume that the olive-gray shales found by Zalba (1981) above the Loma Negra Formation in Barker and correlated with the Cerro Negro Formation, correspond to the upper depositional system. The late Proterozoic age of these deposits was confirmed by radiometric dating (Rb-Sr and K-Ar), showing 723 ± 21 Ma, according to Bonhomme and Cingolani (1980).

These sediments constitute important clay deposits which are largely exploited in the Province of Buenos Aires (Fig. 1), the main non-metallic mining area of the country.

In order to evaluate the industrial application of these illitic materials, some technological tests and particlesize distribution were carried out. These standard tests include refractoriness, plasticity, plastic to dry and dry to heat shrinkage, porosity, mechanical strength and colour after heating at different temperatures.

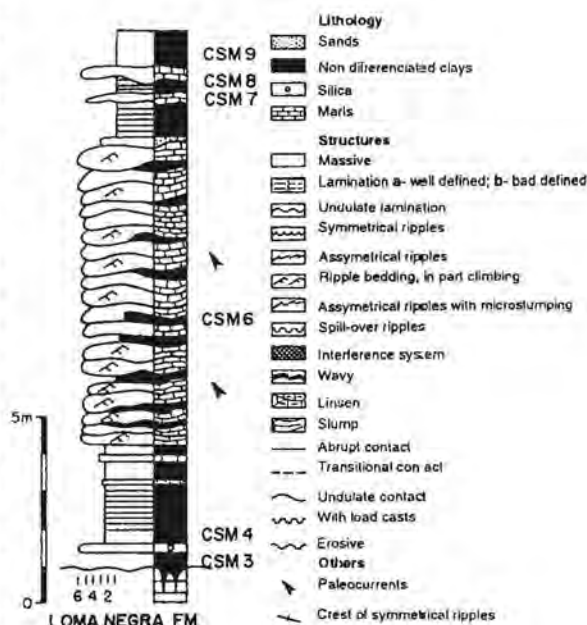


FIG. 2. Lower depositional system at the locality of San Martín, Sierras Bayas.

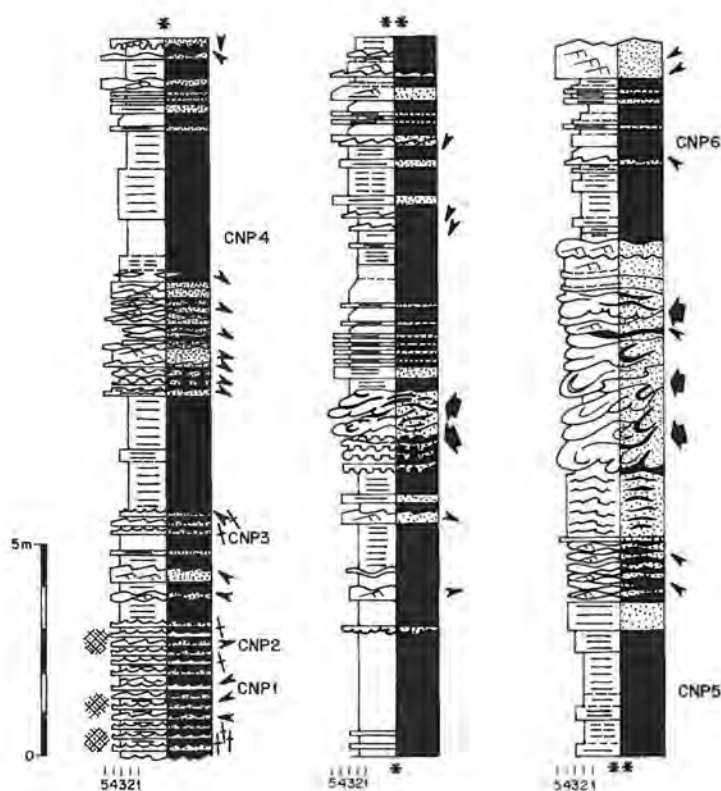


FIG. 3. Upper depositional system at the locality of Cerro Negro (taken from Andreis *et al.*, 1992). Legend as in figure 2.

MATERIALS AND METHODS

Based on detailed stratigraphic profiles established in the Cerro Negro and San Martín quarries and the study of outcrop samples (Figs. 2, 3), clay minerals were analyzed on X-ray diffraction patterns of oriented (size fraction $<4 \mu\text{m}$) and random aggregates for routine analysis (air dried: AD, glycolated: GL and heated: H to 550°C) following Association Internationale pour L'étude des Argiles (AIPEA) Project on the Standardization of Preparation Techniques: Standardization on Preparation Techniques Committee (SPT), (Thorez and Kanda, 1987). Saturation with potassium chloride overnight, solvation with glycerol and heating to 550°C were applied to exclude presence of vermiculite and to confirm the existence of smectites. The Greene-Kelly test (1953) was carried out to identify montmorillonite among other smectite types (L-300 GL). The X-ray diffraction analyses were carried out on a Philips equipment set, at 40 kV and 20 mA, with Ni filter and $\text{CuK}\alpha$ radiation.

Following Kübler (1987), the IGCP 294 Project

Working Group (1990), Kisch (1991) and Krummer and Buggish (1991), illite crystallinity (IC) was determined on the $<2 \mu\text{m}$ fraction and measured on thin preparations (3 mg cm^{-2}). We followed Kübler's limits for the anchizone: $0.42^\circ/0.25^\circ\Delta 2\theta$ at 2° min^{-1} , using a time constant at 1 second which had been adopted by a large number of workers. Values greater than $0.42^\circ\Delta 2\theta$ correspond to the diagenetic zone.

The 'Intensity Ratio' parameter (IR), defined as:

$$\text{IR} = \frac{I(001)/I(003) \text{ air dried}}{I(001)/I(003) \text{ glycolated}}$$

was determined (Šrodon, 1984) to identify expanding components in the illitic material. The $<0.2 \mu\text{m}$ fraction was used to avoid quartz contamination, which would interfere with the (003) illite reflection. Using X-ray techniques, the method developed by Šrodon (1984) was applied in order to differentiate between illitic materials and for the precise identification of the mixed-

layer component. The authors followed the sequence carried out by Šrodoní (1984) and their results are schematically shown in Fig. 4.

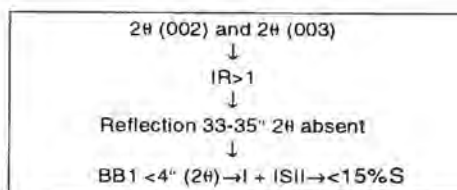


FIG. 4. Results following Šrodoní's sequence (1984) for the two depositional systems, where BB1 is defined as the joint breadth of the (001) illite adjacent I/S reflections, measured in degrees 2θ from where the tails of the peaks join the X-ray background.

Structures resulting from different stacking sequences of layers are termed polytypes, provided that the layer composition is similar. The authors followed Šrodoní's and Eberl's (1984) suggestions to identify the polytype species in all the samples. An empirical parameter α was measured to characterize Md/M structures. This parameter correlates well with

the illite-smectite layer ratio and with the shape of the (20, 13) diffraction bands.

Two composite samples were prepared for the industrial tests. One of them from the Cerro Negro upper depositional system (CNP 1, 2, 4, 5, 6 clays) and the other from the Cerro Negro lower depositional system (CSM 3, 4, 8, 9 clays, Figs. 2, 3, levels of sampling). A small quantity of bentonite was added to the CNP samples to improve their plasticity and green strength.

The powder with 5% moisture was pressed at 250 kgf cm^{-2} to obtain the test bars.

Square section bars were prepared by plastic forming adding a variable amount of water so that the clay could be easily worked. The bars were heated at 950, 1,000 and 1,050°C for two hours.

Pyrometric cone equivalent (IRAM 12507, 1958) and plasticity index (IRAM 10501 and 10502, 1968) were obtained and porosity, water absorption and linear heating shrinkage were measured. Room temperature Modulus of Rupture (MOR) of the clay bars was measured by three-point loading with a span of 40 mm and at a crosshead speed of 4.5 mm min^{-1} . The particle size distribution was obtained by wet sieving and a sedigraph was used to analyze the <44 μm fraction.

RESULTS AND DISCUSSION

Total and clay fraction X-ray diffraction analyses of the two depositional systems show differences in composition. Table 1 shows that the lower depositional system (CSM samples) is mainly composed of illitic material (AD: 10Å; GL: 10Å; H: 10Å), with abundant quartz, no feldspars and the presence of calcite in calcareous caysstones and marls. The upper depositional system (CNP samples) is predominantly illitic, with the systematic presence of random chlorite-smectite (AD: 14Å; GL: 15Å; H: 12Å) and scarce smectite (montmorillonite) (Li -300 GL=10Å), with very abundant quartz and abundant feldspars. It is noticeable that in the whole rock sample, the content of chlorite-smectite increases with respect to fine-grained fractions (4 μm) (Fig. 5). CSM 9 sample has a similar mineralogical composition to the upper depositional system and, probably, reflects a very slow change in the environment already initiated at the end of the first sedimentary cycle.

The IC values of all samples (Table 1) are between 0.60 and 0.70 ($\Delta 2\theta$). The 'Intensity Ratio' parameter

(IR) > 1 in all the samples indicates that expanding layers are always present in the illitic material. The illitic material was characterized as I-ISII with <15% of expanding layers. ISII type of ordering was defined by Reynolds and Hower (1970) as the probability of a smectite layer being followed by three illite layers equaling 1. The polytypes present in the samples are M and Md, according to the α parameter (Table 1).

According to the depositional environments, clay mineralogy and illite crystallinity, the authors assume that the clay minerals generally reflect a detrital origin, with superimposed diagenetic processes. Most of the illite is considered to be of detrital origin (cf. Zalba *et al.*, 1992) but the IC situates these sediments in the diagenetic zone (IC > 0.42), according to Kisch's measurements (1991). The chlorite-smectite is attributed to transformation in subtidal to intertidal subenvironmental conditions, probably from parent smectite-like material. The illitic material shows the same degree of diagenesis in the two depositional systems.

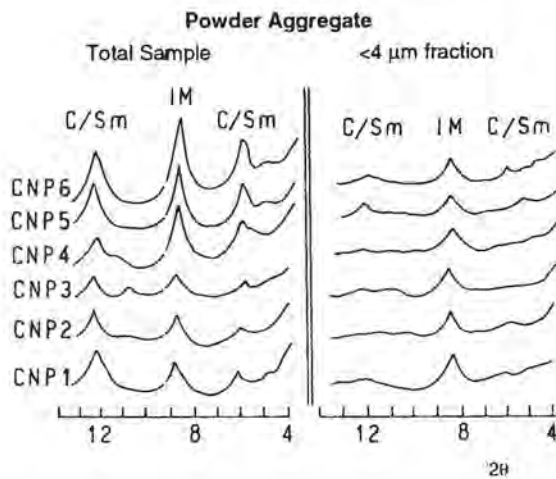


FIG. 5. X-Ray powder diffraction patterns of total and <4 μm fraction of CNP1-CNP6 samples showing (001) and (002) peak intensities of chlorite-smectite.

TABLE 1. MINERALOGICAL CHARACTERIZATION OF THE TWO DEPOSITIONAL SYSTEMS OF THE CERRO NEGRO FORMATION.

	Oriented sampled <4 μm (%)				Other components in total sample			Identification of illitic material			
	Sample	IM	C/Sm	Sm	Q	F	Cal	IC	Ir	TIM	P
Upper Depositional System	CNP6	65	20	15	v.a	a	-	0.70	1.21	I+ISII(S<15%)	Md/M
	CNP5	65	25	10	v.a	a	-	0.70	1.27	"	"
	CNP4	75	20	5	v.a	a	-	0.65	1.21	"	"
	CNP3	70	30	-	v.a	a	v.a	0.65	1.25	"	"
	CNP2	65	30	5	v.a	a	-	0.70	1.25	"	"
	CNP1	70	25	5	v.a.	a	-	0.70	1.29	"	"
Lower Depositional System	CSM9	80	5	15	v.a	-	s	0.65	1.68	I+ISII(S<15%)	Md/M
	CSM8	100	-	-	v.a	-	-	0.70	1.38	"	"
	CSM7	100	-	-	a	-	v.a	0.60	1.28	"	"
	CSM6	100	-	-	v.a	-	v.a	0.70	1.38	"	"
	CSM4	100	-	-	v.a	-	-	0.70	1.18	"	"
	CSM3	100	-	-	v.a	-	-	0.65	1.22	"	"

IM: illitic material; C/Sm: Chlorite-smectite, Sm: smectite; Q: quartz; F: feldspar; Cal: calcite; IC: illite crystallinity ($\Delta^{\circ}2\theta$); Ir: expandability parameter; TIM: type of illitic material; P: polytype; v.a: very abundant; a: abundant; s: scarce.

Composite CNP and CSM samples show differences in composition. XRD analysis of the CNP composite sample indicates the presence of abundant quartz, with minor amounts of feldspars and chlorite-smectite and a very low content of calcite and smectite. In CNP composite sample 55% of the particles is larger than 74 μm equivalent spherical diameter (esd) and 22% is smaller than 2 μm diameter. As was determined by the particle size distribution, the coarse fractions were higher than expected because complete dispersion was not achieved and the clay did

not disaggregate due to its hardness and compaction (shales), consequently, addition of bentonite was necessary to make the clay workable.

The CSM composite sample shows significant amount of quartz and also scarce smectite has been recognized. This sample is more fine-grained than the CNP type. The particle size distribution indicates that the clay is composed of 22% particles larger than 74 μm and 50% smaller than 2 μm diameter, so its plasticity index is higher than the value for the CNP one.

The clays have similar pyrometric cone equivalent

(CPE=15) and red colour after heating. As their main constituent is illite, the associated alkalis favour a low refractoriness (Worrall, 1982). Dried and heated test pieces were characterized by linear shrinkage, porosity, water absorption and bending strength in composite CNP plus bentonite and CSM samples. Results are given in table 2 for semi-dry and plastic formed tests. After drying, the test pieces did not show appreciable differences (see Table 2). The linear shrinkage values were low and therefore they had low green strength. Heated bars showed uniform red colours as well as good appearance, with no cracking or fissures. The linear shrinkage of the CSM samples is higher than the value for the CNP ones at 1,050°C and consequently the water absorption is low.

Similar results were obtained after heating both samples at 1,100°C. Ceramic products were charac-

terized by near 10% linear shrinkage, without deformation. They are dense, with no porosity although they show some signs of vitrification.

Results of these preliminary technological tests indicate that the CNP and CSM clays are suitable for the manufacture of red heated ceramic bodies, with low water absorption.

It is well known that the final properties of the ceramic product can vary considerably depending on the mineralogical composition of the natural clay, the shaping process and the temperature and heating cycle.

Concerning the mineralogical characteristics, recent studies carried out using Italian clays (Fiori *et al.*, 1989) showed that 'red stoneware' or 'red gres' are obtained using illitic-chloritic clays, with quartz, feldspar and some smectite. Those clays did not contain carbonates.

TABLE 2. COMPARATIVE TECHNOLOGICAL PROPERTIES OF THE COMPOSITE SAMPLES OF THE TWO DEPOSITIONAL SYSTEMS OF THE CERRO NEGRO FORMATION.

	Upper depositional System CNP+ Bentonite						Lower depositional System CSM				
	T [°C]	LS [%]	AP [%]	WA [%]	AD [g cm ⁻³]	MOR [kgf cm ⁻²]	LS [%]	AP [%]	WA [%]	AD [g cm ⁻³]	MOR [Kgf cm ⁻²]
Plastic Formed Test	100	3.0	-	-	-	13	4.0	-	-	-	13
	1,000	5.0	25.5	13.0	2.0	147	4.0	-	-	-	13
	1,050	6.8	17.3	6.6	2.1	235	8.9	12.2	5.4	2.2	325
	1,100	11.4	2.6	1.1	2.3	415	10.9	5.0	2.1	2.4	325
Semidry Test	100	0.0	-	-	-	33	0.0	-	-	-	20
	1,000	0.0	19.2	8.7	2.2	214	0.5	20.1	9.6	2.7	265
	1,050	1.0	12.1	5.2	2.3	421	2.9	12.3	5.3	2.3	357
	1,100	2.5	1.6	0.8	2.4	514	4.7	6.0	2.5	2.4	442

T: Temperature; LS: linear shrinkage; AP: apparent porosity; WA: water absorption; AD: apparent density; MOR: modulus of rupture.

CONCLUSIONS

The lower depositional system clay fraction is composed mainly of I+ISII with <15% smectite layers and with Md/M polytypes, while the upper depositional system bears the same I+ISII plus random chlorite-smectite, mainly concentrated in coarser fractions, and scarce smectite. The presence of chlorite-smec-

lite and smectite in the upper part of the lower depositional system is attributed to very slow changes from subtidal-intertidal to more intertidal conditions already initiated at the end of this first depositional cycle.

In the lower depositional system the absence of feldspars is attributed to intrabasinal supply of sed-

iments which were mainly derived from chemical precipitation (calcium carbonate), while in the upper depositional system abundant feldspars are related to predominantly extrabasinal terrigenous supply of sediments (sand and clays).

The regional appearance of smectite and random chlorite-smectite associated to illitic material in the upper depositional system suggests transformation in response to changes *in situ* in the sedimentary environment (subtidal to more intertidal conditions).

Illitic material is considered to be of detrital origin,

with superimposed diagenetic processes and which situate these sediments in the diagenetic zone: $IC=0.60-0.70$ ($\Delta 2\theta$).

CNP and CSM samples could be suitable to make good red ceramic products (Table 2) of low water absorption, with some vitrification. This behavior may be attributed to the adequate content of illite, chlorite, quartz and feldspar as compared with clays used in the Italian ceramic industry. Impurities which are prejudicial like calcite and smectite, are present in small amounts.

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