



Texturation of model clay materials using tape casting and freezing

Gisèle Lecomte-Nana*, Amina Mokrani, Nicolas Tessier-Doyen, Kévin Bousois, Hervé Gouere-Doubi

Groupe d'Etude des Matériaux Hétérogènes, Ecole Nationale Supérieure de Céramique Industrielle (ENSCI), Centre Européen de la Céramique—GEMH, 12 rue, Atlantis, 87065 Limoges Cedex, France

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Abstract

The present work aimed to investigate the processing of textural clay based materials using tape casting together with freezing. Two model raw materials were used, namely: BIP kaolin from France and ABM montmorillonite from Mediterranean region. The mixtures of both clays were studied, whereby, the amount of montmorillonite was 0, 5, 10, 20 or 50 mass%. After tape casting, the as-obtained green bands were frozen into liquid nitrogen, lyophilized and then fired at 1050 °C or 1200 °C.

The amount of montmorillonite appeared as a critical parameter that controls the cohesion of the dry products. For montmorillonite content ≥ 20 mass%, the products exhibited multiple cracks after lyophilisation. With lower montmorillonite content, the cohesion of the dry products was satisfactorily and a macroscopic cross-linked surface texturation was observed. After calcination at 1050 °C or 1200 °C, the texturation appeared well defined. Moreover, calcination at 1200 °C increased the densification of products and the occurrence of a glassy phase was noted.

The combination of both tape casting and freezing (freeze tape casting) is a promising way to develop various clay-based and composites materials exhibiting unique microstructure organization and characteristics with potential application in the field sustainable and environmentally friendly filtration, adsorption or catalysis.

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1. Introduction

Clays can be defined as natural raw materials mainly constituted with clay minerals as major components. They are used for a wide range of application ranging from traditional to advanced ceramic materials as well as for drilling fluids, cosmetics, pharmaceutical and medical applications [1–9]. The main advantage of such readily available resources is their environmentally friendly character in most cases and furthermore their recyclability since they can easily be re-used and therefore match sustainability requirements. The basic knowledge on clay minerals is a key point to clearly understand the level of their tunable characteristics regarding various applications. It is well-established that most clay minerals are layered hydrous magnesium or aluminum silicates, also called

phyllosilicates [10], which impart plasticity to clays and which are divided in two groups:

- 1:1 or Te–Oc phyllosilicates whereby the structural unit is composed of one tetrahedral sheet (silicon cations) that is covalently linked to one octahedral sheet (aluminum or magnesium cations) through apical tetrahedral oxygens. Kaolinite is a typical clay mineral belonging to this group and its ideal structural formula is $[\text{Si}_4\text{O}_{10}\text{Al}_4(\text{OH})_8]$. Their characteristic layer-to-layer distance (d-spacing) is 7.1–7.3 Å and only few cations substitution is observed here.
- The 2:1 or Te–Oc–Te phyllosilicates in which the structural unit contains one central octahedral sheet sandwiched between two tetrahedral sheets. Their d-spacing varies from 10 to 14 Å depending on their interlayer characteristics. Many substitutions can occur in octahedral and tetrahedral sheets, thus leading to charge deficit per unit cell ranging from 0.3 to 1.8. The net negative resulting from these isomorphous

*Corresponding author. Tel.: +33 587 50 25 59; fax: +33 587 50 23 01.

E-mail address: gisele.lecomte@unilim.fr (G. Lecomte-Nana).

substitutions is compensated for by interlayer cations, less or more hydrated (mainly K, Na, Ca and Mg). The latter interlayer species can be easily exchanged since they are loosely held, which is the case for smectites and more precisely montmorillonites having the general formula $[\text{Si}_8\text{O}_{20}\text{Al}_{3.5-2.8-y}\text{Mg}_{0.5-1.2}(\text{OH})_4\text{M}_{0.5-1.2}]$ (M=interlayer cation). Also montmorillonite are swelling minerals because the hydration degree of interlayer is very sensitive toward surrounding moisture. These characteristics can partly explain their higher reactivity compared to 1:1 phyllosilicates.

In ceramic industry, kaolinite (main constituent of kaolins) is considered as a refractory clay mineral with low plasticity compared to montmorillonite [11,12]. In fact, the latter brings more plasticity and ensures a good cohesion in green ceramic bodies; also montmorillonite allows lowering the sintering temperature due to the presence of alkaline and earth-alkaline cations (fluxing agents). But the swelling character of these montmorillonites [13] is very detrimental for ceramic products because during the drying stage, the loss of physisorbed-water is associated with a significant shrinkage that promotes cracks development and propagation. For this reason, the amount of montmorillonite is always reduced in wide diffusion ceramic bodies (less than 3 mass%). Besides, montmorillonites have been widely used to develop textural materials.

Regarding aforementioned points, the present work aims to explore a different processing including montmorillonite amount > 5 mass% with the scope to take advantage of the natural aspect ratio and shape phyllosilicates to develop textural clay-based materials. Kaolin and montmorillonite model raw materials have been used in order to limit interactions with commonly associated minerals or impurities. To our knowledge, such investigation has not been explored in literature.

2. Experimental

2.1. Materials

Two raw model clays are used in the present study: BIP kaolin and ABM montmorillonite. Their chemical and mineralogical compositions are presented in Tables 1 and 2. The BIP kaolin is mainly constituted with kaolinite clay mineral as expected (silica/alumina ratio=1.3) and also contains few amount of associated minerals (micaceous phases and quartz). In the case of ABM montmorillonite, the major clay mineral is an aluminous montmorillonite whereby the interlayer cations are calcium, magnesium as well as potassium ions. The physical characteristics of these model clays are given in Table 3.

Table 1
Chemical composition of the raw clays (mass%).

	SiO ₂	Al ₂ O ₃	TiO ₂	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O
ABM Montmorillonite	56.57	17.90	0.36	4.25	2.65	5.96	1.24	< 0.20
BIP Kaolin	48.10	36.90	< 0.05	0.26	< 0.20	0.17	1.90	< 0.20

2.2. Processing

Tape casting is used for the elaboration of the various samples with the aim to enhance the natural texturation of clay platelets. Since kaolins exhibit less plasticity and cohesion in green bodies in comparison with other clays, a binder and a plasticizer have been used namely PVA (vinyl polyacetate) and PEG (polyethylene glycol). Preliminary tests show that the appropriate amount of each additive is 5.5 mass% (with respect to the clay content). The samples studied in this work are made of BIP kaolin—ABM Montmorillonite mixture with 0, 5, 10, 20 and 50 mass% of montmorillonite labeled respectively KM0, KM5, KM10, KM20 and KM50. The desired amounts of kaolin and montmorillonite are first added in deionized water in order to reach a solid content of 50 mass%. After a vigorous mixing during 5 min, the plasticizer and binder are added, and then the slurry is homogenized for additional 5 min. The as-obtained slurry is maintained under roll-mixing in order to ensure the homogeneity and to prevent bubbles formation (de-airing) within the slurry prior to its use. The tape casting equipment is constituted with a single blade in motion over a table made off with marble. Once the container is filled with the slurry, the casting is performed at previously monitored casting rate (1 cm/s in this case) and desired gap to ensure the final thickness of the green band. The as-obtained band (Fig. 1(b)) is divided in smaller samples, then quenched into liquid nitrogen ($T=-196$ °C). These samples are kept within a freezer at -24 °C before drying through lyophilisation. The lyophilisation apparatus used in this work is from

Table 2
Mineralogical composition of the raw clays (mass%).

	ABM montmorillonite	BIP kaolin
Montmorillonite	90	/
Illite-Mica	< 8	16
Kaolinite	–	> 78
Quartz	< 2	5

Table 3
Physical characteristics of the raw clays.

	BIP kaolin	ABM montmorillonite
BET Specific surface area (m ² /g)	11.10 ± 0.79	107.67 ± 0.79
Grain size (µm)	d ₁₀ =2.82	d ₁₀ =1.64
	d ₅₀ =8.99	d ₅₀ =9.04
	d ₉₀ =27.88	d ₉₀ =31.42
Density (g/cm ³)	2.600 ± 0.004	2.257 ± 0.001

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