



Flaws linked to lime in pottery of Marrakech (Morocco).

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Abstract

The studied clayey materials come from soil of Marrakech Haouz Plain. We discussed the influence of grain size, mineralogical and chemical composition of these raw materials on the lime behaviour in pottery shards. In this paper we propose some recommendations to inhibit lime blowing and the efflorescences responsible for flaws. The application of such recommendations will improve the quality of produced pottery and the income of potters in the region.

Key words: Pottery, Lime blowing, Ceramic, Marrakech.

Résumé : Défauts liés à la chaux dans la poterie de Marrakech (Maroc)

Les matériaux argileux étudiés proviennent du sol de la plaine du Haouz de Marrakech. Nous avons discuté l'influence de la granulométrie, la composition minéralogique et chimique de ces matériaux sur le comportement de la chaux dans les tessons de poterie. Dans ce manuscrit nous proposons quelques recommandations pour inhiber le gonflement de la chaux et les efflorescences responsables de défauts dans la poterie. L'application de telles recommandations va améliorer la qualité de la poterie produite et le revenu des potiers de la région.

Mots clés : Poterie, gonflement de la chaux, Céramique, Marrakech

Introduction

The production of ceramics has experienced a large and long evolution throughout the human history. The old classification of ceramics bases itself on the hardness and the surface treatment especially on glazed or no glazed bodies [1]. Later, the grain size, the porosity and the color have been taken into account [2]. Here, we must take in the account the high influence of the chemical reaction of carbonates upon the color during firing especially if salt (NaCl) is present in the mixing water or in the raw material [3–5]. If the reaction is not complete between carbonates and other components of ceramic paste, lime spalls emerge by the cold reactivity of lime (CaO) and efflorescences by the dissolution of CaO in the water and his deposit in the surface [6–8]. Nowadays, the temperature of firing [9] as well as the mechanical behavior [10,11] have been introduced in ceramic classification, together with the introduction of advanced technical ceramics [12].

According to the final ceramics product, the contents of the diverse constituents of raw materials vary in pottery production. These constituents range from clay minerals as plastic and binder components to siliceous sand as skeletal components and finally feldspars and fine carbonates as fluxing agents. All the components react with each other during the firing process. The formation of new mineral phases as mullite, cristobalite, diopside, etc. depends of the initial chemical composition, the firing temperature, the firing rate, soaking time and the shaping method [13–15].

In Morocco, the ceramic industry covers a considerable economic sector. In the Marrakech area (central region of Morocco), the clays are essentially used in earthenware. There are hundreds of small earthenware production units all over the region that use local clays as raw materials. But the traditional production process applied does not take into account chemical, mineralogical and technological characteristics of starting, intermediate and final product, a practice that leads to low quality ceramics. Research works done so far in the field of traditional ceramics have been focused on the characterization of unexploited clay deposits [15–21] and those under exploitation [13,14,22–24] but have scarcely dealt with the improvement of the quality of final products [25–27].

For economic reasons, the ceramic industry has to use clayey materials from nearby deposits. Moreover, a specific deposit may have distinct beds associated with different clayey materials. Hence, local ceramic plants tend to mix proportions of the clayey materials in order to improve the properties of both the unfired ceramic body and the corresponding final product [25]. In the lowlands of Marrakech region, clay deposits are generally far from the potter sites. Being located in a semi-arid climate region, and because of the drought prevailing these last decades, only a marginal part of the population can afford using modern irrigation methods for economic reasons. Hence, part of this land is being used by an alternative income-generating activity – the production of potteries. From then, many villagers have acquired and used this know-how to generate income, but still persistent is the issue linked to the quality of the final products ; cracks, lime blows, melting of pieces and heterogeneous coloration due to the non-uniform distribution of heat in the traditional ovens. In this study, we will discuss the flaws linked to lime, that is lime spalls and efflorescences. The aim is to improve the quality of production in this region zone.

1. Material and methods

1.1. Material

One ceramic pastes of each site [Agafay (Ag) and Tamsloht (Tms)] has been collected from potters. The raw materials are provided for these sites from the Haouz Plain. They consist of quaternary soil. Potters clear the A horizon (approximately the upper 30 cm of soil) to reach the B horizon and then they exploit their raw material in a layer of about 50 cm of thick. This clearance favours the decrease in organic matter and coarse particles contents in the material. The raw materials from Agafay is used to produce plates locally called “Gasriyas”, while those from Tamsloht are used to product nursery vase, and large and small decoration pieces.

The production process adopted by artisans begins with a manual raw material preparation during which the clay is ground using a wooden stick, followed by addition of water and mixing using hands and feet in order to obtain a homogeneous water distribution in the paste with limited air-bubbles. The paste is then separated into blocks and shaped using the potter’s wheel, or hand carved in the case of some decoration pieces of Tamsloht.

Then the shaped green pottery products undergo in the case of Agafay a sun-drying during 4 hours followed by shade-drying during 24 hours before a second sun-drying. While in the case of Tamsloht, the products undergo firstly a shade-drying during 2 to 4 days, followed by sun-drying during 3 hours. The dried products are then fired in traditional ovens, with resulting ceramic products presenting several defects as lime spalls and efflorescences.

1.2. Methods

The particle size distribution of the raw samples has been determined by wet sieving for fraction higher than 40 μm and by a Horiba LA-300 laser diffraction analyser for fraction less than 40 μm at Geosciences and Environment laboratory (LGSE) in the Cadi Ayyad University of Marrakech (Morocco).

The identification of the mineralogical composition of raw materials and neofomed phases after firing has been carried out by X-ray diffraction (XRD) using a Bruker D8-Advance diffractometer with $\text{CuK}\alpha$ radiations (scan step size: 0.02°; time/step: 0.6 s; anode: copper [$\text{K}\alpha=1.5418 \text{ \AA}$]) (AGEs, University of Liege, Belgium). Qualitative identification has been based on Holtzapffel [28] and

Moore & Reynolds [29] by identifying the position of diagnostic peak of each mineral and its harmonics. Methods used for semi-quantitative identification have been described in Cook & al. [30] and in Boski & al. [31] for bulk sample, and in Fagel et al. [32] for total clay and clay fraction. The intensity of each peak has been multiplied by a corrective factor. The intensity of clay fraction has been measured on ethylene glycol spectra.

The major elements (Si, Ti, Al, Fe, Ca, Mn, Mg) have been analysed at the Petrology and Geochemistry unit in the University of Liege (Belgium) by X-Ray Fluorescence (XRF) on lithium–borate fused glass following the method of Duchesne & Bologne [33]. Other elements like S could be present as detected by EDX analysis (ULg). The volatiles content (combined water, organic and inorganic matter) has been determined at LGSE laboratory by Loss-On-Ignition (LOI) on dried samples respectively at 550°C for 4h and 950°C for 2h [34].

A small (3mm long) fragment of fired bricks was carbon coated to observe the lime spall and to control their chemical composition by Scanning Electron Microscopy (SEM) coupled with EDX microanalysis (Chemical department, University of Liege).

2. Results and Discussion

3.1. Grains size distribution, mineralogical & chemical composition

The particle size distribution (Table 1) varies from both sites. Agafay sample displays the highest clay content (51%) while Tamsloht sample display the highest silt content (43%). According to Shepard [35] the two samples can be classified as sand-silt-clay.

Table 1: Grain size composition of samples from Agafay and Tamsloht sites

	Sand	Silt	Clay
Ag	21	28	51
Tms	23	43	34

Table 2: Mineralogical composition of studied samples. C.F. (Corrective factor from literature [30–32] ; EG = Ethylene Glycol

	Ag	Tms	d(001) (Å)	C.F.	
Bulk fraction	Quartz	20	27	3.34	1
	K-feldspar	5	7	3.25	4.3
	Plagioclase	9	7	3.19	2.8
	Calcite	6	15	3.04	1.65
	Dolomite	4	1	2.89	1.53
	Hematite	2	2	2.69	3.3
	Goethite	4	-	4.18	7
	Total Clay	50	42	4.46	20
Clay fraction	Illite	38	29	10	1
	Chlorite	3	2	14	0.4
	Kaolinite	3	2	7	0.7
	Vermiculite	2	1	14	0.34
	Smectite	2	1	17 (EG)	0.25
	Mixed Layers	2	3	12	0.4
Pyrophyllite-talc	-	4	9.2-9.35	2.56	

The raw clay materials consist of quartz, K-feldspar, plagioclase, carbonates, and clay minerals (Table 2). Hematite and goethite are present in low abundance (2% and 0-4% respectively). The clay fraction

principally consists of illite. Chlorite, kaolinite, vermiculite, smectite and mixed layers are present but only in trace (1-3%).

The chemical composition and the Loss on Ignition of the samples are listed in **Erreur ! Source du renvoi introuvable.** The most abundant oxides are SiO₂ and Al₂O₃. MgO is more abundant in Ag due to the presence of dolomite, whereas the CaO is more abundant in Tms sample due to calcite. K₂O and Fe₂O₃ are slightly more abundant in Ag sample, probably due to the higher content on illite clay species and iron oxide minerals respectively in this paste. Na₂O, TiO₂, P₂O₅ and MnO are present only in much lower quantities. The volatiles (loss on ignition) vary from 9 to 13%. The volatiles calculated at 550°C include more than organic matter, i.e. adsorbed and structural water of clay minerals. The LOI at 950°C includes the disintegration of carbonates.

Table 3: Chemical composition and Loss on ignition (LOI) of samples
 * LOI at 950°C indicates the Loss on ignition between 550 and 950°C

	Ag	Tms
SiO ₂	58.0	63.8
TiO ₂	0.9	0.8
Al ₂ O ₃	19.8	14.3
Fe ₂ O ₃	7.7	6.2
MnO	0.2	0.1
MgO	4.3	2.6
CaO	5.1	8.9
Na ₂ O	1.1	0.9
K ₂ O	4.1	2.8
P ₂ O ₅	0.2	0.2
LOI 550	7.0	6.2
LOI 950	4.4	5.0

3.2. Influence of chemical composition

The predominant oxides (SiO₂ and Al₂O₃) are mainly associated with the clay minerals but SiO₂ content is also associated with quartz particles. The content of iron oxide is sensitive to the firing conditions. It often produces unexpected results in colour and texture of the fired clays [36]. All the studied samples have a comparatively high amount of Fe₂O₃ (6-8%), which confers a reddish colour after firing [37]. The relatively high amount of K₂O (3-4%) contributes as the main flux agent of clayey materials [37,38]. In the studied samples, it is related to the high content of illite in the clay fraction. The value of total loss on ignition (LOI) (about 11%) is related to the dehydroxylation of the clay minerals, the burning of the organic matter and the decomposition of carbonates [39,40]. The raw materials are poor in organic matter; the LOI at 550°C does not exceed 7% and is especially related to the dehydroxylation of clay minerals and the burning of organic matter. However OM facilitates portability during transportation of heavy pieces [41] due to the porosity increase, that improves thermal regulation capacity up to a limit from which the product starts to present poor mechanical characteristics [41,42].

3.3. Influence of carbonates

The rate of total carbonates (i.e. calcite + dolomite) ranges between 10 and 16% in the two pastes. During firing, the carbonates decompose at temperature inferior to 950°C [43]. The products are made up of clays with some coarse limestone grains, then lime blowing is generated with time (Figure 1a). Lime blowing is designated in literature by other terms like "lime spalling", "blowouts", "lime popping" [6-8]. The pieces can flake and disintegrate if the calcite is abundant, which is not the case in our samples. According to Kornmann [42], the pressure exerted by the crystallisation of some

particles present in very small pores of diameter 0.005 μm is of the order of 39 MPa. These pressures exerted are inversely proportional to the pore's diameter, the latter being fortunately bigger in earthenware. The large grain of lime blowing (~2mm in diameter) (Figure 1a-b) in fired shard corresponds to an hydraulic lime due to the presence of Si and Al (EDX analysis) and that we can recognize by its slight hardness [44]. The glassy phase from 1050°C appears to be not sufficient to neutralize a small grain (~10 μm in diameter) of magnesia (**Erreur ! Source du renvoi introuvable.**) due to the lack of fluxing agents. Ca and Mg of carbonates may act as fluxing agents [45,46]. So melting can even start at about 800°C when carbonates are present [47]. But a great abundance of carbonates in a material limits the extent of vitrification at temperatures higher than 1000°C compared to material with low carbonates content [48,49]. The presence of Fe₂O₃ and organic matter in the clay may form a very viscous melt from 900°C, and the escape of CO₂ from CaCO₃ might cause a slight bloating [50] and produce large flaws in the products.

3.4. Inhibition of flaws linked to the lime blowing and to efflorescences

Various solutions are proposed in literature to prevent lime blowing. Kornmann [42] said that a wet sieving at 0.2 mm mesh did not generate this phenomenon. Laird & Worcester [7] rather proposed soaking fired clay bodies in cold water (i.e., a process termed “docking” after Ramachandran et al. [50]) immediately after firing to dissolve the lime or the portlandite. These authors demonstrate that it is unlikely that the solubility would be a major factor governing prevention of lime blowing by docking. The rapid absorption of water would bring immediate slaking of lime and would give less apparent volume. No disruptive force will be developed within the piece with prevention of lime blowing. A lime arising from firing of a clayey limestone contains silicates and aluminates and it is called hydraulic lime because it hardens in presence of water [44]. Another solution is to add up to 0.5% of NaCl when sample contains 10% of limestone. This inhibits the transition of calcite to calcium oxide and eliminates spalling and even efflorescences [5,7,8,51–54]. Fabbri & Fiori [55] note that NaCl disappears from 500°C and HCl is formed during the dehydration of clay minerals, and at the same time the decomposition of CaCO₃ begins. Bearat et al [5] proposes that CaO reacts with HCl to give CaCl₂ between 500 and 600°C, the latter reacts with clay minerals to give more stable calcium silicates at a relatively low temperature.

Fluxing agents are also to suggest. The EDX analysis of a grain of lime blowing shows that the silicification is only partial (**Erreur ! Source du renvoi introuvable.**). Results obtained by Cultrone et al. [46] using the Electron Microprobe Analyser (EMPA) have shown that the Ca content in the lime plus periclase mixture decreases significantly at high temperature due to the incorporation of this element into newly formed high-temperature silicate phases as gehlenite, anorthite and diopside, and prohibits the reaction with the air like in his powder form. An addition of fluxing phases can also help to neutralize all the lime by silicification [42], provided the lime grains are small otherwise the silicification will only take place at their surface.

3.5. Impact of NaCl addition

The addition of NaCl is a good solution for flaws linked to lime blowing and to efflorescences. However this addition changes the surface colour to a light or white colour [3–5] at lower temperature than that necessary for a calcareous paste without salt [54,56]. For instance some Tunisian potters use sea-water for white-colored pottery and well water for red-colored pottery [57–59]. Several other regions (Italy, Middle East & Pakistan) have applied this method [3,59–61]. This discoloration only occurs when the raw clay is Ca-rich [5,59], and when the firing temperature is between 750 and 900°C.

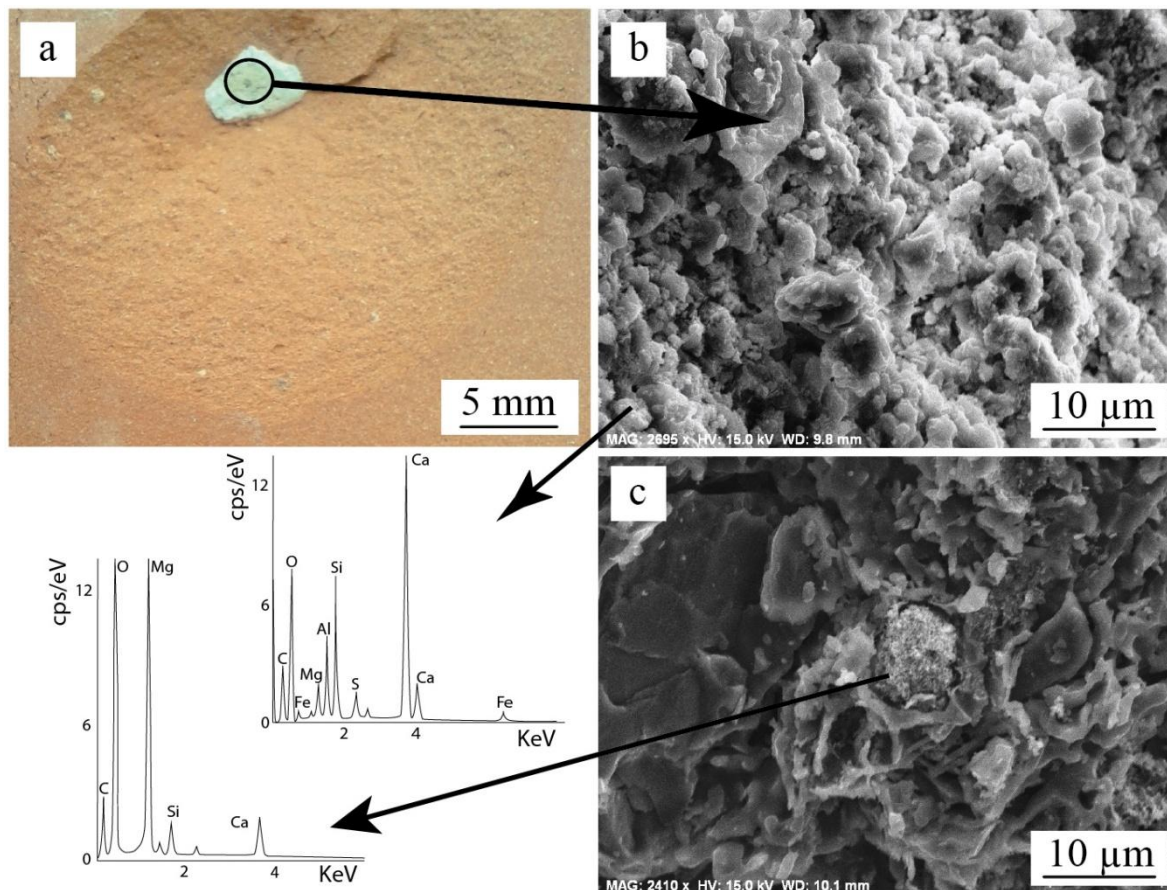


Figure 1: (a) “Lime blowing” manifested in a fired sherd at 950°C ; (b) & (c) SEM micrographs of a partially silicified lime grain and of a grain of magnesia (fired at 1050°C) with EDX analysis of each of them

The presence of NaCl and CaO into a paste causes partial volatilization of K in the form of K_2Cl_2 during firing, especially between 800 and 900°C [59,62]. The amount of neoformed K-feldspar will thus be affected.

The formation of gehlenite, anorthite or wollastonite is catalysed by salt because it increases the reactivity between clays and Ca [5]. From 900 to 1000°C, the colour changes to yellow due to the trapping of Fe^{3+} in the crystal lattice of precipitated calcium aluminosilicates, by substitution of Al^{3+} or Ca^{2+} [5,63–68].

Conclusion

The problem of lime blowing can be easily prevented by a wet sieving at 0.2 mm mesh and by adding some fluxing agent in order to obtain a significant melted phase that will digest the lime powder and will prevent its cold reactivity. The addition of NaCl to the paste is another alternative but it will affect the colour of the product. It should be noted that the presence of limestone is not always a disadvantage. According to Hoard et al. [51], coarse limestone temper grains are suitable for large pieces if the spalling is controlled. The paste is more workable and allows thinner and more globular vessel, and the fired clay body is more resistant to mechanical and thermal stresses. Moreover, the carbonates can be used to change the colour of potteries, especially when we add salt, from red, white to yellow. The implementation of such recommendations by the potters of Marrakech region will promote their production.

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