

Marl crumbling decrease using geopolymer mixtures: lessons from experimental tests in the vicinity of Fez, Morocco

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Keywords: marl; crumbling; geopolymer mixture; valuation; Fez; Morocco

Abstract: Despite its abundance in many regions of the planet, the marl's use in practical issues engender many problems, in relation to its components' quality, its physical and chemical characteristics and its valorization costs. The crumbling is among its major critical aspects that affect its engineering behavior, its handcraft and other socio-economic uses. This paper apprehends the physical and chemical properties of the Miocene marl in Fez vicinity (Morocco). They are investigated to measure their changes and see how they affect the marl's geotechnical characteristics. Mixed with cellulose and pozzolan at different concentration degrees, the marl samples were exposed to extreme temperatures, observed in the laboratory and interpreted in terms of characteristics reliable to crumbling risk reducing as cohesion and materials' resistance. By adding the cellulose and pozzolan to the raw marl at different percentages (1%, 5%), (2%, 10%), (3%, 15%), (4%, 20%), (5%, 25%), we have tested its characteristics using various techniques and analyses. We mainly used X-ray diffraction, X-ray fluorescence and SEM techniques. This study apprehends the possible production of innovative materials based on marl mixtures. It emphasizes the valorization of the Miocene marls in the region of Fez (Morocco) but its aims are larger as the results may be applied for marl worldwide. The main goal is to identify the best thresholds that are suited to improve the mixed marls' physico-chemical properties and therefore reduce risks of crumbling in marl. The results are concluding as chemical analyses show a slight variation of major chemical elements in the samples' contents. We note that after adding pozzolan and cellulose to marl, the Fe₂O₃ and SiO₂ increase and favors neoformation of clays. New illite and metakaolinite appear. The difference is important when we compare results between characteristics of the raw marl and those of the mixed marls, in particular at temperatures of 900 °C and 1000 °C. The mineralogical analyzes have shown the appearance of well crystallized hematite. In mixed marl, these results were confirmed by the electron microscope analysis. The results converge to confirm the positive impact of geopolymer mixtures as they contribute to decrease the risks of marl crumbling as its cohesion and resistance become intense. These new marl's characteristics are innovator and show a new degree of the material' transformation. They favor the marl's advantageous exploitation in economic and industrial activities. Even locally experimented, the importance of the obtained results may be worldwide.

1. Introduction

The marl and clay are basic materials in craft activities and industries all over the world. They often constitute complex natural mixtures with considerable plasticity. Their major components are clay and calcium carbonate with proportions varying from 30 to

70%. Other minerals with different grain size and physicochemical properties are observed in marl (Strazzera et al., 1997; Kabre et al., 1998; Celik, 2010; Ngun et al., 2011; Njoya et al., 2012). In the ceramic industry for example, these characteristics are important to perform innovative products and less crumbling, more resistant to environmental stresses and less consuming the energy. Good marl and clay quality tiles require a high firing temperature over 1100°C and are therefore more expensive (Sawadogo et al., 2014). The production of environmentally friendly materials and inexpensive used materials are of interest for craft and industry activities (ceramics, cooking pots, tiles, etc.). Several approaches have been reported in the literature concerning the use of certain industrial additives or wastes as cementing agents to improve the properties performance of clay and marl raw materials (Gidley and Sack 1984), clay soils (Kolias et al., 2005, Yong and Ouhadi, 2007, Mesrar et al., 2015), residual soils (Miller and Azad, 2000), fly ash from rice husk (Seco et al., 2011), artificial pozzolan (Al-Rawas et al., 2005) and geopolymers (Akdim et al., 2017; Mesrar et al., 2017).

Several studies focus on the production of bricks based on binary mixtures (Jordán et al., 2005, Liew et al., 2004a, 2004b, Monteiro et al., 2008, Weng et al., 2003) and ternary mixtures (Chiang et al., 2009, Montero et al., 2009; Chaib et al., 2016).

Applied studies of marls and clays' uses in industries underlay the necessity to scientifically investigate clay and marl characteristics to conceive improved materials that may increase economic benefits and technical use (Reeves et al., 2006; Baccour et al., 2011; Harvey et al., 2013; Poirier, 2011; Mesrar et al., 2013). Rakhimova et al. (2018) studied the mixture of marl with a high calcite/ alumino silicates ratio as a geopolymer precursor and limestone as a mineral addition to this geopolymer. The calcination of marl at 800 °C resulting in the formation of reactive Si, Al, and Ca due to the dehydroxylation of clay minerals and decarbonation of calcite makes marl suitable for use as a geopolymer precursor.

Mesrar, 2013 studied the characterization of the marl Miocene of Fez after doping with metal oxides (Al_2O_3 , MnO_2 and Fe_2O_3), the results showed the marl doped acquired the good property mechanical compared of crude marl.

In Morocco, marls and clays are very abundant mainly in the north, and are of great interest in several areas. They are used in building, engineering projects and craft industries (ceramic, cooking pots, pottery, etc.). However, they often suffer from the lacking material's quality, due to its physical and chemical properties, engendering risks of crumbling and problems of materials cohesion and products less durability (Mesrar et al., 2013). Their mixture with polymers and selected minerals is tested under specific conditions, and scientifically studied to find adapted materials that are resistant and less crumbling. However, the crumbling of marls is still a serious risk for ceramic and building activities, mainly in the vicinity of Fez (Figure 1).

The main objective of this study is to analyze geochemically the material, to apprehend its characteristics and observe their influence on the marl's quality. The study focuses on the material's potential crumbling and how it may be reduced by testing different fired mixtures. We aim to identify the best situation where the marl become more resistant and less exposed to the crumbling risk.



Figure 1. The crumbling aspect of Miocene Marl in Fez vicinity (Morocco)

2. The studied area and its geological context

The study area is located in the Saïs plain, at the East of Fez, six kilometers far from the city. It is located in a morphological hilly context on the northern flank of Ben Jallik hill where several quarries are opened for marl exploitation (Figure 2).

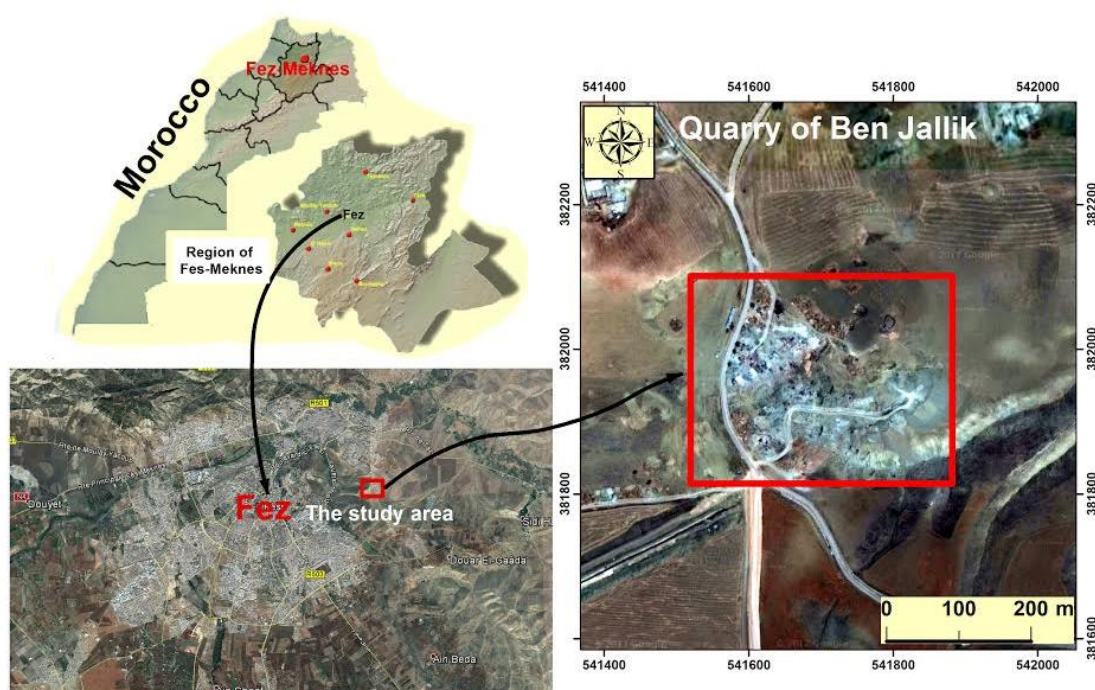


Figure 2. Location of the study area (Quarry of Ben Jallik, Fez vicinity)

The geological profile shows in its upper part marly Miocene marls of various hues and colors (blue, green and whitish). As the blue and green marl are preferred by artisans are most exposed in the quarrels we sampled them for study in three sites. The geological studies related to the Miocene marl, its sedimentary, stratigraphy and tectonic characteristics are numerous (Taltasse, 1953; Ait Brahim and Chotin, 1983; Ahmamou et al., 1989; Chalouan et al., 2014). Their development has been the subject of numerous scientific studies (El Qandil, 2007; Mesrar, 2013) but the geochemical characterization of these marls and their behavior in admixture with polymers and pozzolan in specific thermal conditions remains to be done.

3. Methods

In this study, the crumbling process in three sites is apprehended basing on the field work observation and the laboratory tests. These tests were performed on bulk raw marl and mixtures of marl, pozzolan and cellulose using different dosages and percentages (Table 1). To facilitate the comparison of results obtained elsewhere in the Sais plain and Taza (Morocco), we adopt similar percentages of mixtures studied by Akdim et al., 2017, Mesrar, 2013 and Mesrar et al., 2013. The pozzolan material came from the volcanoes of the Middle Atlas, South – East of Ifrane. The cellulose polymer used in the study consisted of a commercial cellulose powder. The cellulose was selected to complement the pozzolan's promoting effect by improving textural properties (such as porosity and strength) as well as heat resistance of the marl. Each studied sample weighs 100g and had a different initial mixture. The preparatory actions were performed in the laboratory Geo-resources and Environment of the FST (USMBA, Fez) and geochemistry and petrographic analyses were done in the Innovation City of Sidi Mohamed Ben Abdellah University, Fez.

Table 1. The analyzed marl composition and dosage mixtures

Marl sample number	% of pozzolan	% of cellulose
Bj1 raw and bulk marl	0	0
Bj1A	+ 5% pozzolan	+ 1% cellulose
Bj1B	+ 10% pozzolan	+ 2% cellulose
Bj1C	+ 15% pozzolan	+ 3% cellulose
Bj1D	+ 20% pozzolan	+ 4% cellulose

The geochemical characteristics of these materials were studied using the X-ray diffraction and the X-ray fluorescence. The parameters of the marl before and after doping with additives were measured analyzed and interpreted.

The X-ray fluorescence spectrometry (WD-XRF) of marl samples was done using the Axios spectrometer, Panalytical sequential mark. This analysis was carried out after grinding the material and pressing 10 g of each sample.

The bulk mineralogical characterization was done using the X-ray diffraction analyses. They were carried out using a Panalytical X'Pert High Score diffractometer, which is a direct optical coded goniometer with a working wavelength corresponding to copper Cu K α = 1, Å 54060. The XRD patterns were made in the range of from 10 ° to 80 ° with a step width of 0.06682. Errors for the quoted mineral concentrations are around ± 5 % accuracy. The relative abundance of minerals has been estimated from the height of the main peak multiplied by correction factors (Moore and Reynolds, 1997).

The Scanning electron microscope (SEM) was also invested in the study. The observation with a scanning electron microscope (SEM) has the advantage of visualizing crystals' surfaces and reveals details of great fineness, in particular to characterize the inter and intra-granular spaces (first space between the grains and in the liaison phase) and allows to recognize constituent grains. This analysis is done on bricks prepared in the laboratory Geo-resources and environment in the FST, at the University Sidi Mohamed Ben Abdellah in Fez.

The bricks were analyzed at different stages (bulk stage at temperature 0°C and after firing them at different temperatures, 900°C and 1000°C). The objectives were to apprehend their normative composition, their characteristics and their physical behavior depending on defined conditions and interpret them in terms of marl valuation quality and how its crumbling may be reduced.

4. Results and discussion

4.1. Chemical composition

The analyzes performed on the raw marl samples and other samples obtained from blends with pozzolan and cellulose at different percentages show important geochemical changes under high temperatures.

As shown in figures 3 and 4, the geochemical composition of the raw marl shows a wide range of major chemical elements such as the SiO_2 , the Al_2O_3 and Fe_2O_3 . Other elements such as CaO , and MgO are also present even in minor concentrations, often under 10% of the samples' weight. After the firing operations of the marl samples, we observed significant changes in these characteristics depending on the firing temperatures, the dosages and adopted mixtures. They permit a best understanding of the material quality that may reduce its crumbling and offer its valuation opportunities.

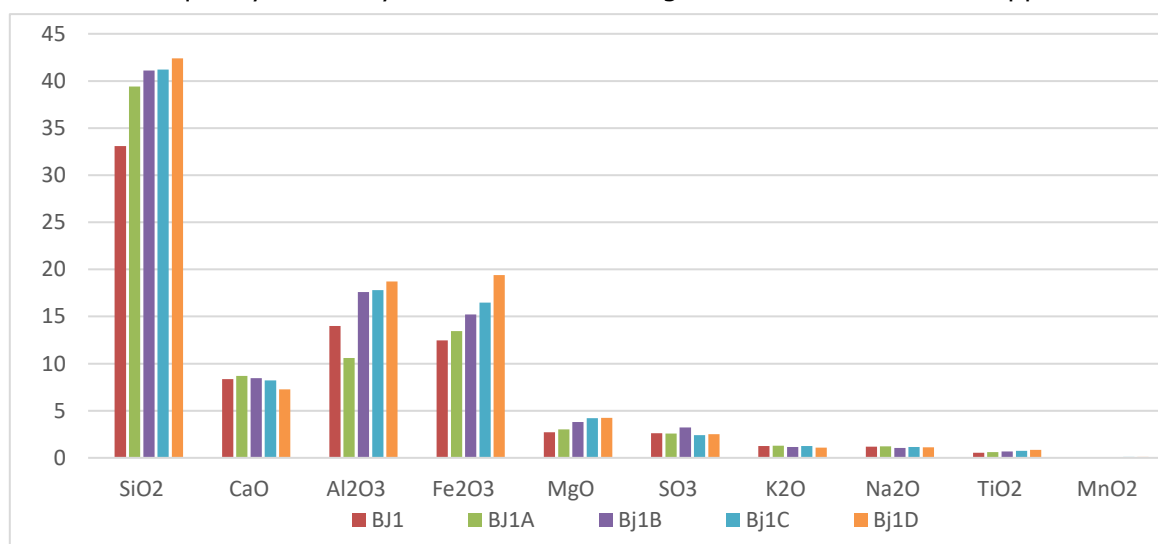


Figure 3. Percentages of the major chemical elements in the Bj1marl and its mixtures with cellulose and pozzolan, fired at 900°C.

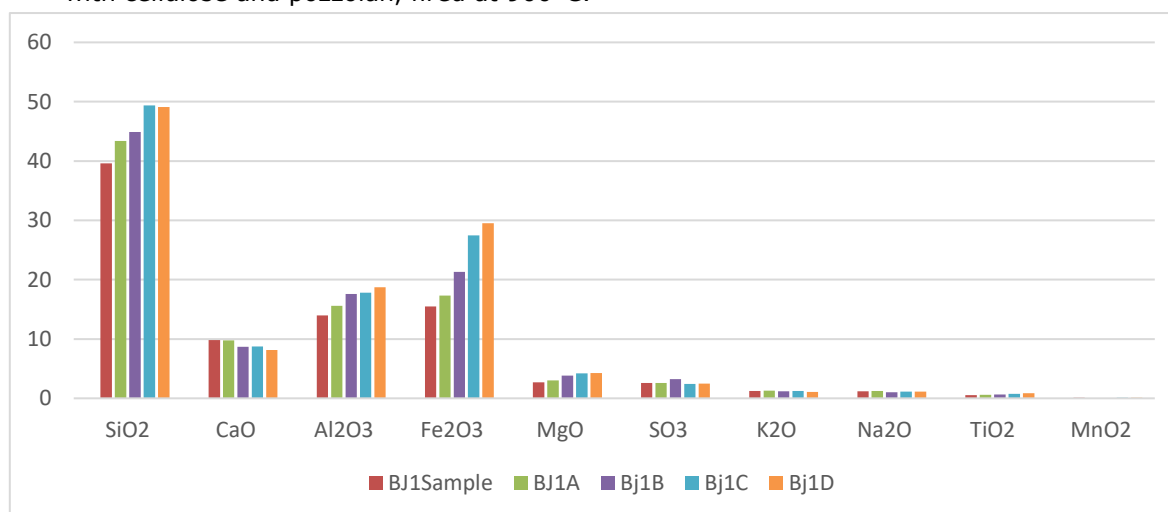


Figure 4. Percentages of the major chemical elements in the Bj1marl and its mixtures with the cellulose and pozzolan, fired at 1000°C.

The analysis of the chemical composition by XRF marl doped with pozzolan and cellulose and firing at different temperature (900°C and 1000°C) reveals a significant enrichment in silica (SiO_2), alumina (Al_2O_3), iron oxide (Fe_2O_3) and titanium oxide (TiO_2). Their concentration increases as the doping level is increasing. However, doping causes a slight decrease in some major elements' remains like CaO, which is varying quantitatively (Figure 3 and 4).

The high percentage of SiO_2 in fired samples reflects the added large quantity of the quartz in this material. The presence of Al_2O_3 is related to the presence of kaolinite. However, as observed in SEM analysis, the presence of Fe_2O_3 is related to the hematite. The Iron oxide (Fe_2O_3) is the main colored component often seen. The contents of Iron oxide are also being sensitive to firing and contribute to produce unexpected results in color and texture of the fired marls. The color caused by iron oxide (Fe_2O_3) in clays depends on the changes in the ratio of CaO.

The X-ray curves (Figure 5 and 6) show an increasing crystallization of quartz, mullite, metakaolinite and dolomite. It is conformed to the increasing percentage of pozzolan and cellulose in the samples and confirm their important in ceramics (Asquini et al., 2008). The results obtained show the predominance of mullite and metakaolinite in the mixed samples, in particular at 800 °C which favors the required properties of ceramics. The illite is well known as a favorable content for the sintering step by its contribution to increase the density of the material, when it allows the formation of a liquid phase during heating.

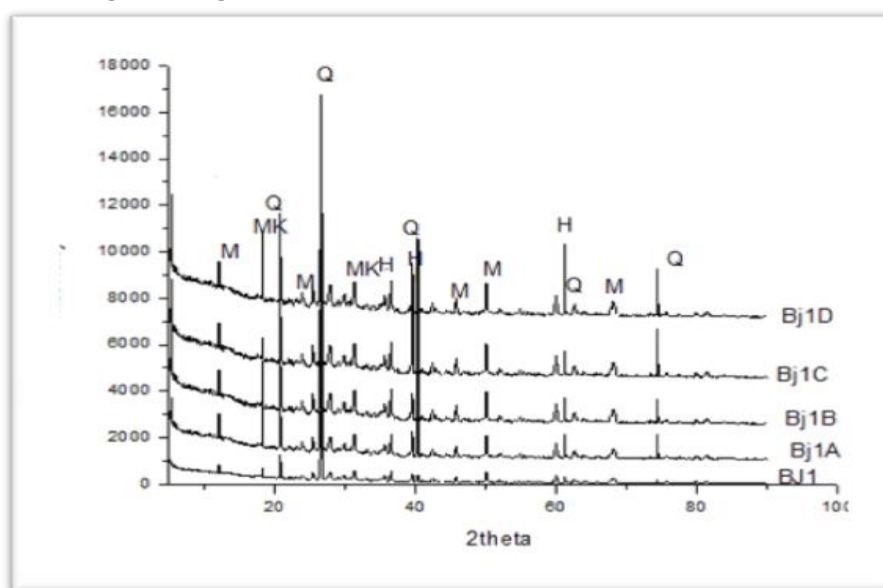


Figure 5. The X-ray analysis of the raw marl Bj1 before and after doping with Pozzolan and cellulose at different percentages (5%, 1%) (10%, 2%) and (15%, 3%) and (20%, 4%), 900 °C. (where, MK = Metakaolinite, Q = quartz M = Mullite, C = calcite, D = dolomite. H: Hematite)

These results show a perfect crystallization of minerals such as quartz, hematite and the transformation of illite and kaolinite into mullite and metakaolinite. From these curves, it is observed that there is an increase in the intensities of the minerals with the addition of pozzolan and cellulose. Quartz (SiO_2), mullite (M) and metakaolinite (MK) and a small peak for CaO and Al_2O_3 are observed. These phases usually occur mainly in cement, ceramics and glass-ceramic materials produced from incinerated waste and glass powder (Asquini et al., 2008). The increments in the cullet content in the samples showed a marked reduction of peaks in CaO and Al_2O_3 , while the presence of amorphous phases can be observed through the analysis of the raw marl sample.

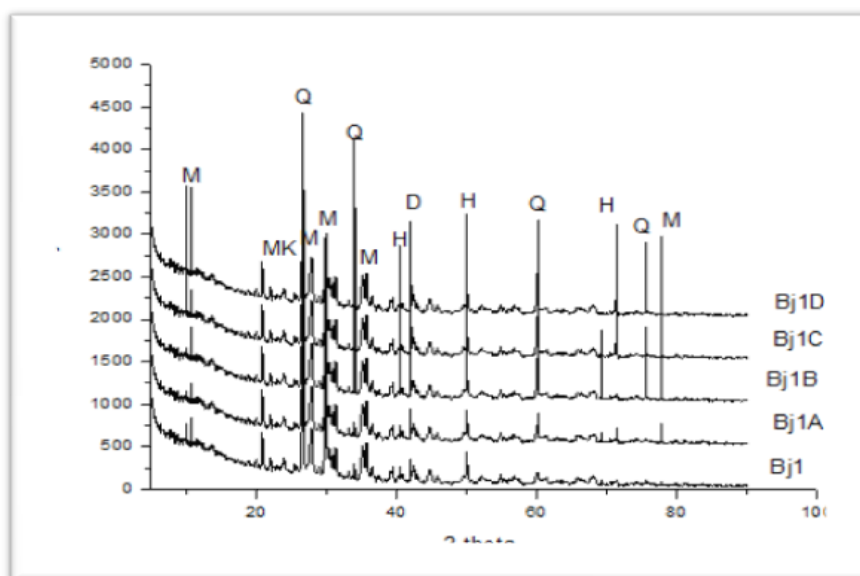


Figure 6. The X-ray analysis of the raw marl Bj1 before and after doping with Pozzolan and cellulose at different percentages (5%, 1%) (10%, 2%) and (15%, 3%) and (20%, 4%), 1000 °C. (where, MK = Metakaolinite, Q = quartz M = Mullite, C = calcite, D = dolomite. H: Hematite)

4.2. Texture changes

The textural observations using SEM-EDX are convergent as the results confirm the perfect crystallization of quartz. The clay fraction of the material is composed of quartz and calcite as the major impurity in the samples, confirming the results of the X-ray fluorescence which shows high proportions of SiO_2 (Quartz).

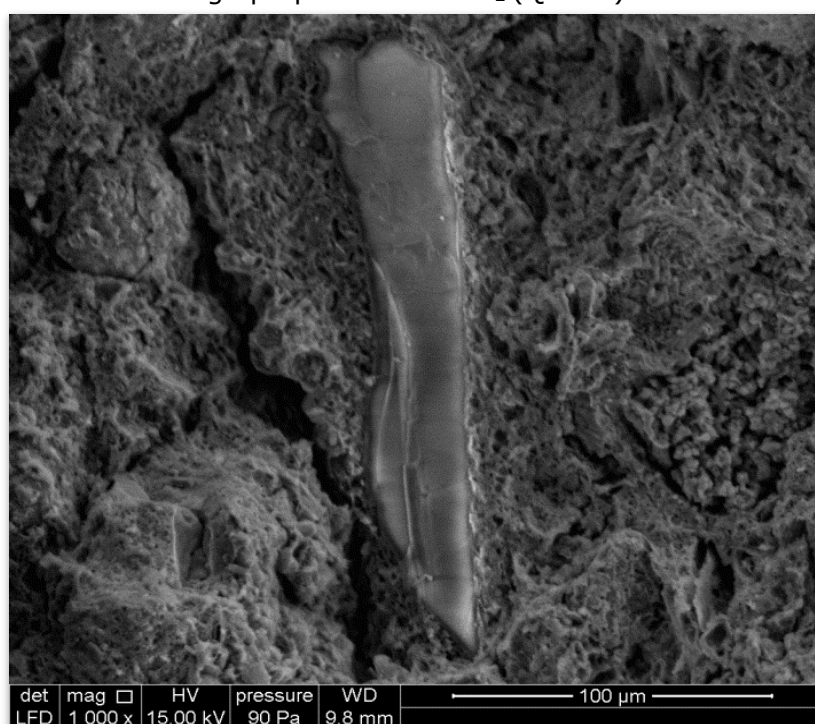


Figure 7. The SEM photomicrograph of the mixed marl (Bj1D) fired at 1000°C.

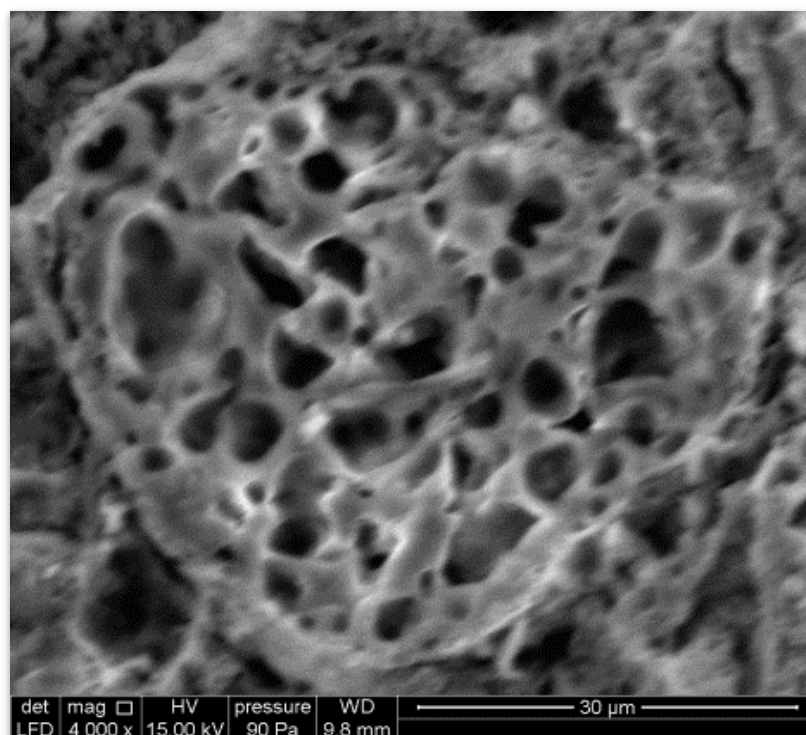


Figure 8. The SEM photomicrograph of the mixed marl (Bj1E) fired at 1000°C

The heterogeneities in textures are observed in the fired materials at high temperatures in the interfaces between the coarse grains and the matrix. The increase in the glassy phase was interpreted by the transformation of SiO_2 and Al_2O_3 into liquid phase at higher temperature (Pauzi, 2011).

4.3. Syntheses and relevance

The observed change in chemical components of burned marl, pozzolan and cellulose mixtures might explain how high CaO contents may turn the marl color to yellowish and pinkish shades. The presence of CaO leads to a high mechanical strength in marl (Mesrar, al., 2015). Mullites and Metakaolinite often develop within the burning process of marl and make it more valuable for economic activities. It reinforces its cohesion properties and reduce its crumbling risks.

Mullites are important minerals in the composition of terracotta clays (bricks, tiles and pottery) because they favor sintering at a relatively low temperature. Metakaolinite is a more refractory mineral, limiting creep at high temperatures. The water of constitution is extracted in the range temperature of 400–800 °C. Its departure causes deformation of the crystal lattice and makes kaolinite less stable. It contributes to the formation of metakaolinite. During the rise in temperature, this crystalline phase degrades progressively to become substantially amorphous at about 950 °C. The metakaolinite has the structural formula $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$. Dehydroxylation may be preceded by a prehydroxylation step by progressive delocalization of the protons (Fripiat and Toussaint, 1960).

As deduced from the textural analysis of samples, these results show a perfect crystallization of minerals and important heterogeneities that are probably playing an important role in the interconnection of pores within the clay matrix. The coalescent particles and the formation of typical clays, such as the metakaolinite are common processes observed in fired marl at 1000°C, mainly in mixed marl. It may be interpreted as a constructive process that increases the marl's resistance to crumbling forces and its counteraction to damaging effects of humidity and temporal thermal changes. As confirmed by Fiori et al., 1989 and Sié et al., 1997, the geochemical characteristics of

marl are influent in the materials' valuation in industry of tiles. Our study shows that these characteristics are certainly contributing to reduce the marl's crumbling and make the material more suitable for sustainable craft and industrial uses.

5. Conclusions

According to these results the physic - chemical characteristics in raw marl samples changed after mixtures with pozzolan and cellulose. The important variations in chemical composition and crystals' texture are observed. Additives and different thermal tests favor change as the progressive importance of the major elements become important in most tests. Adding pozzolan and cellulose to marl contributes to the Fe₂O₃ and SiO₂ increase and favors neoformation of clays, such as illite and metakaolinite. The difference is important comparing results between the raw marl and mixed marl in particular at temperatures of 900 °C and 1000 °C. The mineralogical analyzes have also showed the appearance of Hematite and well crystallized Quartz in doped marl.

These results were confirmed and observed under the electron microscope. The SEM analyses of different samples show a good progressive crystallization and the appearance of the vitreous phase in high temperatures.

The obtained results are significant because the tested mixed marl samples illustrate considerable substantial improvements as clays become homogenous and probably more resistant to crumbling. These characteristics reinforce the marl counteraction to the damaging effects of the humidity and temporal thermal changes. These characteristics are contributing to reduce the marl's crumbling and make the material more suitable for sustainable craft and industrial uses.

Acknowledgments: We thank the Sidi Mohamed Ben Abdellah University of Fez for its administrative and technical support to this study. We used its equipment and experiments' material in the Innovation City and labs of the FST and FSDM.

Funding: No external private funding was given to the study.

Author contributions: Mariam Akdim (the corresponding author) realized the field and laboratory works, interpreted the results and prepared the first draft of the article. Pr. Jabrane Raouf contributed to the scientific works supervision and results presentations. Pr. Mesrar Leila discussed the results in the light of her previous works. Pr. Alami Anouar discussed the article's content, interpreted the results and improved the text. Pr. Selmaoui Sabah's observations and contributions improved the article.

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