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Valorization of olive mill wastewater by its incorporation in building bricks

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Abstract

This investigation deals with the possibility of incorporating the effluent resulting from olive oil extraction activity, known as olive oil mill wastewater (OMW), in the brick-making process. It was undertaken at an important Tunisian brickworks company. In this study, the OMW was mixed with clays following the same brick-making procedure used at the collaborative brickworks in Tunisia. The samples containing OMW were found to be comparable in forming/extrusion performance to a control product that used fresh water. The experimental products produced were tested for their comparative physical properties (volume shrinkage, water absorption, tensile strength of bricks, after firing at 920 °C and paste plasticity) in the unfired and fired states against a control representing the commercial product in the same factory. The results showed a significant increase in the volume shrinkage (10%) and the water absorption (12%), while the tensile strength remained constant. The maximum plasticity index value was found when incorporating 23% of OMW. This rate either maintained the physical and mechanical properties of bricks or improved them. The incorporation of OMW in bricks can represent a promising way to valorize this effluent, to rid the environment of a highly polluting wastewater and to save huge and precious amounts of water in a country where water shortage is a serious problem. This newly-prepared material has a double positive impact: it protects the environment and allows water economy.

Keywords: Olive oil mill wastewater (OMW); Wastewater valorization; Bricks-making incorporating OMW; Paste plasticity; Physical and mechanical bricks properties

1. Introduction

Throughout the Mediterranean area, the production of olive oil generates about 30 million t of wastewater yearly [1]. While processing olive oil, the olive fruit is pressed and an amount of water is added. This amount depends on the process used. In the classical discontinuous process, this quantity is relatively low (about 40%), but in the continuous system, it ranges from 70 to 110% [2]. After this first step, the pasty mix including oil, vegetation and added waters as well as solids from the original pressed olives are processed under pressure to separate the two phases: the first is liquid made up of oil and water and the second is solid usually named olive mill sludge. It is well known that this effluent has a heavy load of organic matter, especially the polyphenols that are considered as environmentally harmful substances and which require removal and treatment.

The possibility of recycling a given process residue would reduce the environmental impact of this effluent. Therefore, recycling any material, rejected as a by-product of a process, would reduce environmental pollution and cost production. Considering the development of technologies and their application in the building sector, the conception of a new material incorporating by-products would be a partial solution to the olive oil processing wastewater [3].

A close look at the composition of OMW shows that the phenols and the organic substances, which are responsible for the high COD value, are problematic for a conventional treatment of this wastewater [4]. For this reason, research has attempted to find new applications to recycle this effluent in the building industry.

Abbreviations: OMW, olive mill wastewater; COD, chemical oxygen demand; C.G.B., General Company of Bricks in Sfax (Tunisia); LOI, loss ignition; OM, organic matter; TOC, total organic carbon; MM, mineral material; W.A., mater absorption; W.D.S., wet dry shrinkage; W.F.S., wet fired shrinkage; D.F.S., dry fired shrinkage.

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Table 1
Particle size distribution of the CGB brickworks feedstock shown as % weight retention within specific size-fraction categories (analyzed by laser technique)

Size particle category (μm)	Distribution (%)
Fine sand 62.5–250	37.79
Silt 2.5–62.5	50.90
Clay <2.5	11.31

In 1986, the possibility of building roofs of houses, using made bricks by the incorporation of OMW, was studied [5]. However, the composition of OMW may affect negatively the physical and chemical properties of the bricks because of their solid content. Later, the ash from sewage sludge was used to make brick material [6,7]. Recently, the olive cake was utilized to prepare bricks whose physical durability and stability were confirmed [8].

In this study, the incorporation of the OMW in brick manufacturing as a substitute for water was investigated. The preliminary research established that this by-product could be introduced at a moderate level into experimental discs of three cm in diameter by 1 cm in thickness [9]. These encouraging results already suggested the need for further development of the research to understand and predict the behaviour of the bricks at an industrial stage. The raw material used to make bricks as well as the processing technology adopted were the same as those used in brick factories in the world.

The current paper describes the impact of mixing clay with OMW on the physical and mechanical properties of the bricks used in building.

2. Materials and methods

2.1. Materials

2.1.1. Clay

In this work three types of clay were used. Clay A is brown in color with a fine granulometry and a high plasticity. Clay B is yellow with a high porosity. The basic component of these two types of clay is montmorillonite, but they also contain a high percentage of kaolinite and illite. Clay S is sandy (70%, w/w, of sand), with a grain size lower than 1.25 mm. The particle-size distribution of the different samples was examined using a malvern 'Mastersizer' laser analyzer [10]. The results are given in Table 1. The different physical and chemical properties of the types of clay used are presented in Tables 2 and 3.

The samples were collected from the area of Agareb (Tunisia), near the factory making bricks.

Table 3
Physico-chemical properties of the three raw materials used for mix-design

Parameters	Clay A	Clay B	Sandy-clay S
Physical characters			
Density	2.49	2.55	2.59
Porosity (%)	32.13	32.55	28.60
Oxide content (%)			
SiO_2	62.31	58.09	82.22
Al_2O_3	12.79	12.73	5.40
CaO	5.52	6.75	3.75
K_2O	2.08	2.02	1.33
MgO	1.70	1.87	0.53
Na_2O	0.31	0.46	0.20
P_2O_5	0.17	0.17	0.15
Fe_2O_3	0.13	0.12	0.24
TiO_2	0.51	0.58	0.50
Cr_2O_2	0.01	0.01	0.01
LOI	10.17	12.62	4.65

LOI: loss ignition.

A light microscope wet screening was used to determine the size of different particles and their composition in the fractions according to color. To wet sieve, water was used to help washing the particles through the screen used in the wet screening. The particles were washed off the screen into a pan and then dried.

The three samples of clay were sieved down 600 and 63 μm. To determine both the mineralogical composition and the amount of such fractions, the light microscope CARI ZEISS and optical microscope SHOTT KL 1500 type were used.

A Leitz heating microscope was used to identify the overall fusion behaviour of the individual clay samples, allowing the behaviour of the different materials to be compared (Table 4). The materials were analyzed separately and then mixed together. Each sample was first mixed with water and then molded into a cylindrical test pellet by tamping the mix to 1.5 mm diameter and 2.5 mm high metal die. The heating rate for the test pellet temperature was programmed to rise by approximately 7 °C/min [11].

The dilatometer curve clearly showed the point of incipient vitrification, recording the length of a brick specimen, and easily observed on a dilatometer trace heated [12]. The dilatometer instrument system used was a BCRA/Doulton dilatometer, with length charge up to 1000 °C.

2.1.2. OMW characterization

The effluent from olive oil processing has a black-brown color with an unpleasant smell of fermented organic matters. Its composition depends on the process used while extracting oil. However, some variations may occur depending on the type

Table 2
Physical characterisation of the three types of used clays

Clay	Particle size	Melting point (°C)	Mineralogical characterization (light microscope and XRD)
A	Fine	1440	Quartz, calcite, gypsum, montmorillonite, kaolinite, illite
B	Slightly bigger	1650	
Sandy S	<1.25 mm	ND	Quartz, calcite, albite, gypsum, illite, dickite, microline

ND: not determined.

Table 4
Characteristic temperatures (°C) describing fusion behavior of the different clays

State of fusion	Starting point	Softening point	Intermediate state	Hemispherical point	Liquefaction point
Clay A	25	1240	1270–1305	1360	1440
Clay B	25	1250	1350–1400	1450	1650
Clay A + B	25	1250	1300–1350	1400	1500

Table 5
Physico-chemical characterization of the olive oil mill wastewater (OMW)

Parameters	pH	Density	Electrical conductivity (mS cm^{-1})	Humidity (%)	COD (g dm^{-3})	OM (%)	TOC (g dm^{-3})	Phenol (g dm^{-3})	MM (g dm^{-3})
OMW	5.00	1.04	10.50	94.00	120.00	92.42	37.60	3.07	15.80

OM: organic matter; TOC: total organic carbon; MM: mineral material.

of olives processed, the area where they are cultivated and their degree of ripeness. Some special features of OMW and their components are summarized in Table 5. The OMW is characterized by an intensive violet-dark brown up to black color, a strong specific olive oil smell, a slightly acidic pH and a high load of organic pollution including polyphenols.

The difficulty to reduce the COD to an acceptable value as required by the standards allowing its discharge in the environment gives rise to a serious problem since the process to be used for the treatment of this effluent will be costly and inefficient. While considering the composition of OMW, the phenols and the organic substances resulting in high COD value, are at the origin of the difficulties in the treatment of the wastewater [11]. In this study, the OMW was selected to be used for brick making instead of water as its treatment by conventional processes is very difficult. With such high rate of water (>85%), this effluent could be used to mix clays when preparing paste for bricks. As a result, an environmental problem will be solved.

2.1.3. Bricks

The different properties of the new building materials were studied on rods (0.12 m in length and 0.01 m in width) and on small bricks (65 mm × 45 mm × 20 mm). Both tested materials were made according to the process used in one of the most important brickworks in Tunisia, the General Company of Bricks (C.G.B.) in Sfax, where first 75% of clay A and 25% of clay B were mixed. Then, 85% of this initial mixture was combined with 15% of clay S and 19.5% of water or effluent (OMW). The molded paste was finally cured for 5 days. After being processed, a series of physical measurements were then carried out using established ceramic procedure on representative batches of each of the dry sets of unfired bricks: bulk density (by mercury displacement), percentage of linear shrinkage (wet/dry), water absorption by boiled water and strength by three point modulus of rupture [13]. Further samples of these same sets of bricks were replicated using the kiln firing schedule in operation at C.G.B. brickworks [10]. Then, the fired bricks underwent further ceramics testing procedures (Table 6).

Considering exhausted gas, the bricks were dried and then fired at 920 °C, a temperature at which all organic matter usually undergoes an oxidation into CO and CO_2. Since OMW contains relatively insignificant quantities of chlorine, the possibility of the formation of hazardous substances such as dioxins or furans was neglected. Besides, while standards for discharged wastewater are well established, there is no regulation available regarding hazardous substances, especially dioxins and furans in Tunisia.

Electronic microscopy photos of bricks made by mixing the different types of clay with water then with OMW were taken by scanning electronic microscope Philips XL 30.

2.2. Plasticity

Plasticity is a highly complex property characterizing the clay deformation. It depends on the flow of the particles over each other, keeping the shape and thereby determining a resistance to flowing. However, as an important feature, plasticity cannot be properly defined or measured. The first way to assess this parameter is to measure the plasticity index using the Atterberg limit method [9]. The second way to determine the plasticity is to measure an applied pressure on the clay, which can deform the

Table 6
Comparative ceramic properties of control and experimental clay products including OMW

Material product	Mix	Unfired bulk density (g/cm^3)	Fired bulk density (g/cm^3)	Wet-dry linear shrinkage (%)	Dry-fired linear shrinkage (%)	Water absorption (boiled) (%)	Mass loss on firing (%)
Rod	Control	0.92 ± 0.10	0.83 ± 0.30	9.30 ± 0.20	1.70 ± 0.04	12.0 ± 0.1	N.D.
	OMW	0.93 ± 0.1	0.84 ± 0.20	9.90 ± 0.20	1.90 ± 0.10	13.6 ± 0.2	N.D.
Brick	Control	2.09 ± 0.20	1.97 ± 0.20	7.30 ± 0.10	1.08 ± 0.10	11.5 ± 0.2	7.34 ± 0.30
	OMW	2.05 ± 0.20	1.92 ± 0.20	7.50 ± 0.20	1.20 ± 0.10	14.8 ± 0.2	8.43 ± 0.20

N.D.: none determined.

material without leading to its rupture. After drying the clay, its deformability is gradually lost and the clay becomes relatively hard and brittle [14,15].

Two types of paste with different rates of water and OMW were experimented. The plasticity index evolution was daily determined during a week. The paste included a variable range of effluent (23–32%).

The plasticity was tested by a plastometer F. Malkin and Coltd on samples sieved to below 850 μm. An axial pressure was exerted on cylindrical specimens placed between parallel tries. During the trials, the speed was kept constant.

2.3. Brick-making process

Three kinds of clay coming from the south of Tunisia (Agareb area in Sfax city) were used. During the preparation, the clays were crushed to break up large chunks, grinding and mixing the raw materials, as in the C.G.B. brick factory. Then, the obtained material was sieved (0.8–1.0 mm) through inclined vibrating screens to remove the stones.

The first step in the forming process, known as the "tempering stage", produced a homogeneous and elastic mass, ready for molding. This is most commonly achieved by adding water to the clay in a pu mill, which contains one or more revolving shafts with blades.

In the stiff-mud process, dried clays are mixed only with a predetermined quantity of water, varying from 23 to 24% (w/w) to provide the plasticity of the paste. Next, the clays were extruded through a die to produce a column of clays. Then, the column was subsequently separated into individual bricks having a moisture rate ranging from 20 to 24%, by a wire-cutter which slices vertically through the column.

Before the firing process, most of this water was evaporated in dryer chambers. Although heat may be generated especially for dryer chambers, heat and humidity had to be precisely regulated to avoid excessive cracking in the brick while heating (Fig. 1).

Drying and firing lasted 48 h. The dried bricks were carried in cars through a tunnel kiln for a 24-h-firing cycle; the maximum temperature reached was 920 °C. After this stage, the bricks exiting the kiln were stored then dispatched.

3. Results and discussion

3.1. Dilatometer curve

In the case of brick clay, the point of incipient vitrification is usually seen at approximately the point of red heat in the kiln at a temperature of about 950 °C. It is noticeable that a very small amount of liquid is formed at lower temperatures because of the presented K_2O pressure. However, the incipient vitrification is also the state where the product begins to exhibit an easily-measured shrinkage [12]. The physical and chemical transformations responsible for the shrinkage are the specimen vitrification resulting from the decomposition of clay and the calcium oxide present as the calcite [16].

The two types of clay referred to as A and B had the same general trends as the mix used in the C.G.B. (Fig. 2).

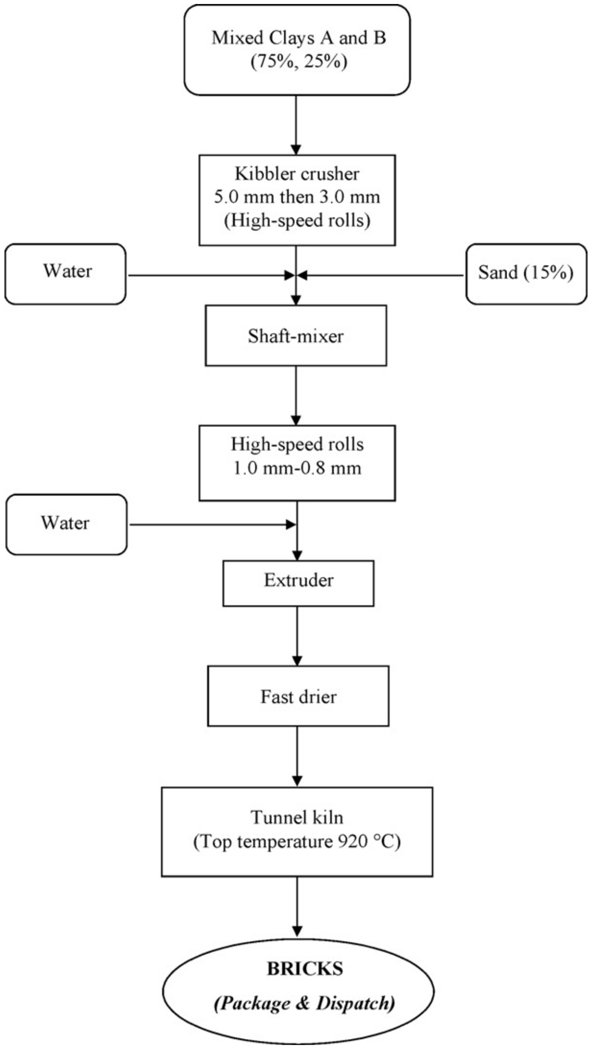

Fig. 1. Flow diagram illustrating the process-line at C.G.B. brickworks.

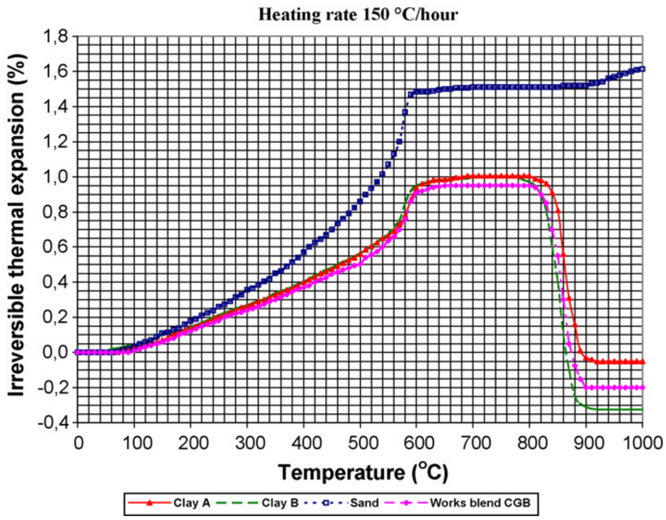

Fig. 2. Dilatometer trace on heating clay brick. Works blend C.G.B. is a mixture of the 3 clays.

The curve representing the irreversible thermal expansion in relation to the temperature showed different steps. In the first one, a linear expansion occurred below 500 °C. Then a permanent expansion in the area of 500–600 °C took place because of the quartz inversion: $\alpha \rightleftarrows \beta$.

The expansion between 600 and 800 °C remained constant because the shrinkage began at the same time as the thermal expansion. This would be explained by the pressure exerted by the carbonate decomposition. At the melting point of the mica (850 °C), the clay underwent a contraction. At about 880 °C, the liquid mica with the calcium oxide resulting from the decomposition of limestone ($CaCO_3$) reacted to give a solid compound including calcium silicate, calcium feldspar or alumino-silicates, following the reaction mentioned below [17]:

$$CaCO_3(\text{solid}) \rightarrow CaO(\text{solid}) + CO_2(\text{gas})$$

It could be concluded that 880 °C was the most suitable temperature for firing such bricks giving the product its optimum strength. As was shown in previous works for clay having high content of $CaCO_3$, the product shrinkage and the dimension variations during firing (800–880 °C) are related to physico-chemical transformations which are linked to synthesis of amorphous phase originating from the clay minerals decomposition and the calcium oxide resulting from calcite [16].

However, all the reactions, which may occur, depend greatly on the firing conditions and the gas atmosphere in the kiln. Moreover, inside the ware, other parameters such as porosity as well as mineralogical and chemical clay composition have a great effect on these reactions [17].

3.2. *Light microscope*

Optical microscopic observations of different particle sizes of wet sieved clays A, B and S showed that the fractions under 600 μm have both gypsum and brown colored agglomerated particles. Quartz is also found among those materials (Fig. 3).

The fraction under 63 μm contained dolomite, limonite, gypsum and quartz in the case of clay A. In clay B, there were black spots of carbonate mixed with dolomite, gypsum, quartz, muscovite and a slight quantity of feldspar.

For sandy-clay S, the microscopic observation exhibited sand, quartz, carbonate, gypsum and a slight quantity of tounaline.

3.3. *Heating microscope*

The fusion behaviour [11] events of separated and mixed clays shown by heat microscopy exalted some transition temperature during firing. Initially, a photograph of the clay sample was taken before heating and subsequently, at each of the event points noted in Fig. 4 and Table 4. The recorded event points were the softening point describing the first confirmation signs of the pellet profile (Fig. 4 and Table 4). It was found that the first signs of softening the three samples of clays (A, B and mixture of A and B) began to visibly take place at approximately 1250 °C. Moreover, the samples progressively continued to soften further with increasing temperature to reach 1350 °C.

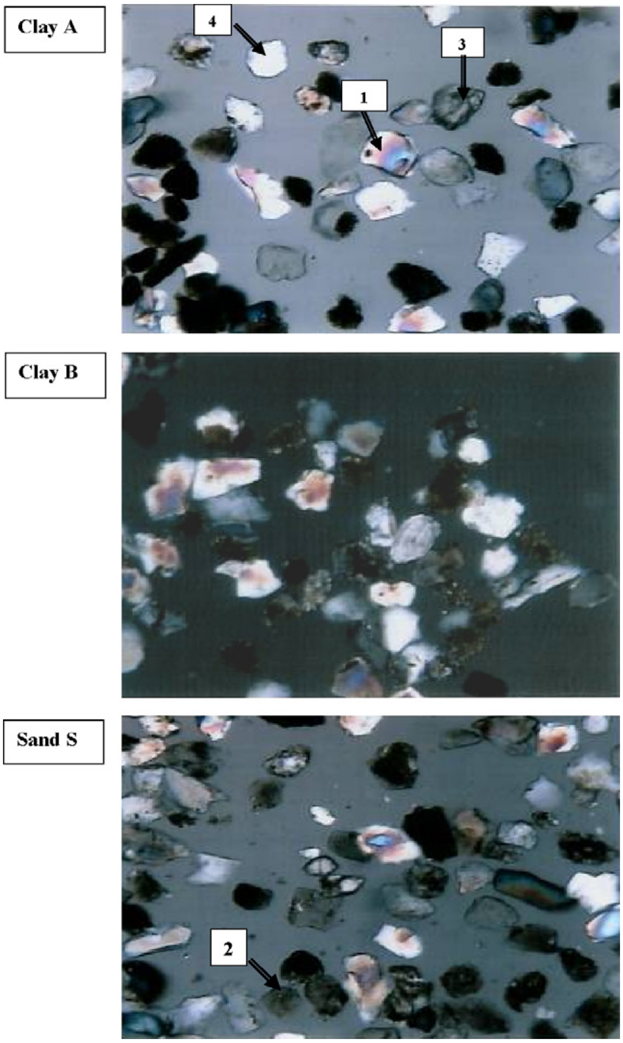

Fig. 3. Optical microscope photos of the three samples A, B and S under 63 μm. 1: Gypsum; 2: calcium carbonate; 3: dolomite; 4: quartz.

The hemispherical point occurred when the pellet was completely in the molten state, and the surface tension strength on the molten liquid produced a symmetrically curved surface. Melting (hemispherical profile) occurred at approximately 1360–1450 °C. The liquefaction point is the stage at which the surface tension influence was overcome and the sample collapsed then flowed to form a melted pool of glass, on the base of the ceramic platen. This occurred at 1440–1650 °C. One explanation of these noticeable differences in fusion behaviour might lie with fluxing components level contained in the clays, such as calcium and iron. Indeed, the results of the chemical analysis confirmed the presence of clay impurities in the tested clays (Table 3). The goal of heating microscopy was to determine the optimum temperature for firing bricks made with OMW.

The mixture of both clays A and B, at rates of 75 and 25%, respectively, had an intermediate melting point at 1500 °C. Since glazing of clay A is easily achieved and the ultimate organic matter oxidation occurs at temperatures inferior to 920 °C, firing of the mix at such rates should avoid the vitrification. Therefore, a temperature around 920 °C was suitable for brick-firing.

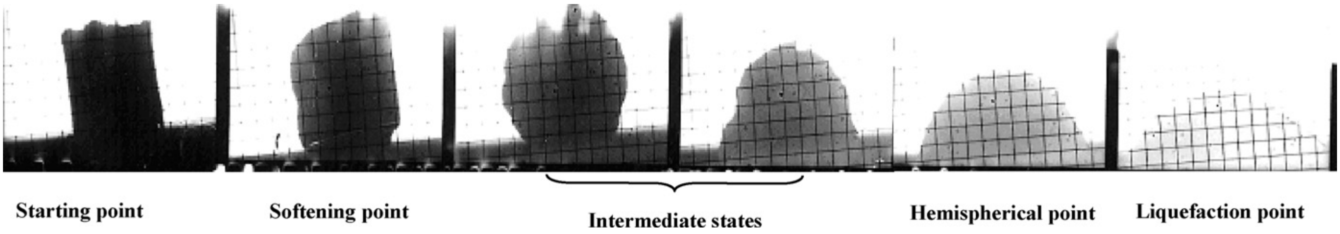

Fig. 4. Successive clay photographs illustrating the fusion behavior event taking place under the Leitz heating microscope during evolution.

Keeping this temperature for firing would not affect the final structure of the manufactured bricks. The heating microscopy photos were taken only for clays mixed with water to confirm the performance of the bricks firing temperature used at the factory. Hence, globally the same behaviour would be found in the clays mixed with OMW, and leading to the same shape of bricks.

3.4. *Impact of OMW incorporation on building material*

3.4.1. *Plasticity*

At the C.G.B. brickworks company, the percentage of water used to prepare the bricks was 24% (w/w).

The variation of the plasticity index during a week is shown in Fig. 5. After a 3-day storage (maturation), the plasticity index tended to stabilize. The curves showed that the maximum value of the plasticity index was found for a paste made of 23–26% water. This result confirms the composition mix used at the C.G.B. brick manufactory (24%).

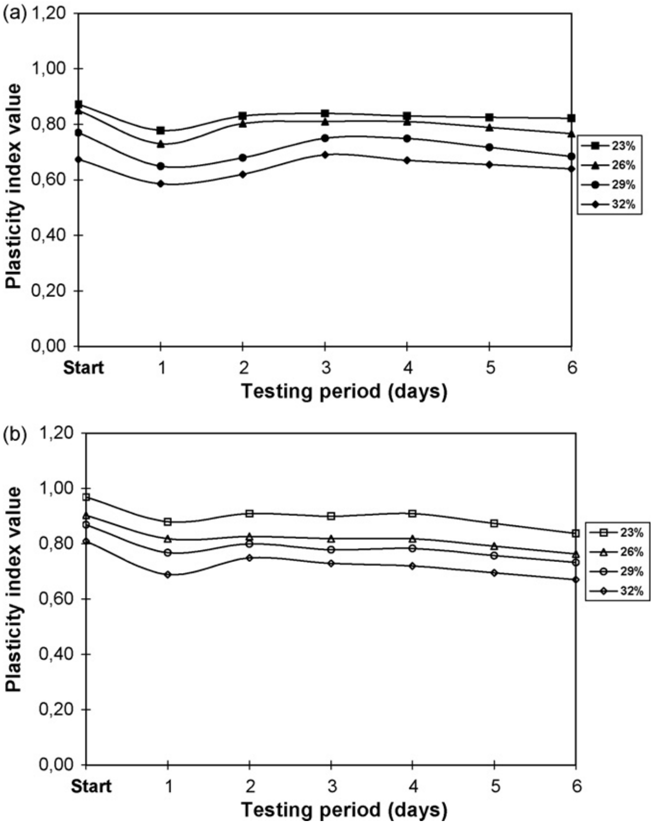

Fig. 5. Plasticity index of pastes containing different amounts of OMW. (a) Water and (b) OMW.

However, the paste mixed with OMW showed a different behaviour. The maximum value of plasticity index was found in a percentage of effluent equal to 23%, and then a decrease of the initial plasticity index was noted. After a 3-day storage, the plasticity reached a constant value that can be explained by the stability of the material. The presence of montmorillonite in the clay generated a non-stability of the paste during the first days. This can be due to a swelling phenomenon, which caused the decrease of the paste plasticity [10].

These comparative results demonstrated that the volume of the OMW amount needed to produce the optimum extrusion performance for the clay body is smaller than that of water. This would be attributed to an additional “lubrificating” effect performed by the small-added volume of OMW. A similar result was previously found in the extrusion effect when adding OMW [10]. The existence of small amounts of olive oil in OMW can promote a usual surface active property [18].

3.4.2. *Laboratory phase results*

The results obtained from the above laboratory testing programme are shown in Table 6; they are evaluated comparatively and explained as follows:

- *Bulk density*: The bulk densities of the control unfired and fired experimental rods are the same as the product including OMW. The drying out of the water surrounding the rod allowed a contraction and close physical contact of packed particles.
- The fired values are lower than those of the unfired counterparts. This fact is due, first to a low level of firing contraction; second to the dissociation of calcareous minerals occurring during firing.
- *Percent linear shrinkage*: The linear drying shrinkages recorded showed a slight increase of 6% with rod prepared with OMW. After firing, the linear shrinkage was reduced because of water evaporation and the body including OMW had a higher percentage [10.5%].
- *Water absorption*: This value is a measurement of open pores to be filled with water, likely remaining within the fired products. The comparative results confirmed that the OMW product is significantly more porous than the control by using boiling water test. The percentage of increase of 12% was observed in rods. This is due to the burning-out of its combustible matter included in OMW.

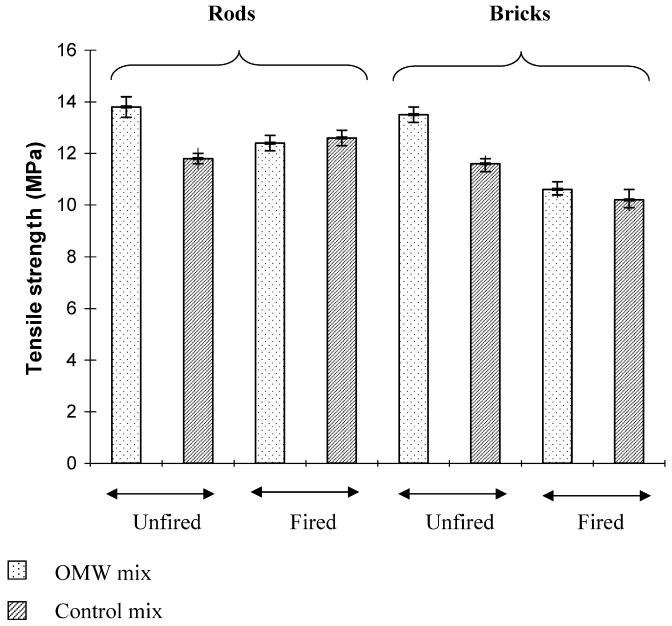

Fig. 6. Impact of OMW incorporation on tensile strength.

- *Unfired tensile strength*: A noticeable tensile strength value was exhibited after the incorporation of the OMW. Indeed, in the studied rods, the tensile strength increased in the unfired material containing OMW (Fig. 6). Hence, it could be concluded that the effluent played rather the role of a plastifiant. These results confirm previous findings [5].

3.4.3. Factory proving trial results

The successful outcomes from the laboratory results confirmed the technical feasibility of introducing OMW into the bricks. After these preliminary interesting results, trials at industrial scale were conducted and 8 ordinary whole bricks were made.

The unfired and fired control and experimental products evaluated comparatively are shown in Table 6 and Fig. 6.

- *Percent mass loss on firing*: The main decrease in mass recorded for fired products can be attributed to habitual ceramic firing reaction. The high level of combustible matter in OMW product has resulted in a higher weight-loss (13%) during firing compared to that of the control. This results from the additional oxidant and the removal of organic matter during firing [19].
- *Bulk density*: The unfired and fired bulk density values of the control and experimental product remained constant (Table 6). After firing, the relatively low mineral concentrations that remained in OMW explain the similarity of the bulk density found in both bodies (control and experimental).
- *Percent linear shrinkage*: The dry-fired shrinkage is comparable to that of rods. Indeed, an increase of 10% was measured in brick including OMW.
- *Water absorption* (%): The experimental product was significantly more porous than the control (Fig. 7). This is due to the creation of additional void sites through the burning out of the combustible matter present in their forming liquid.

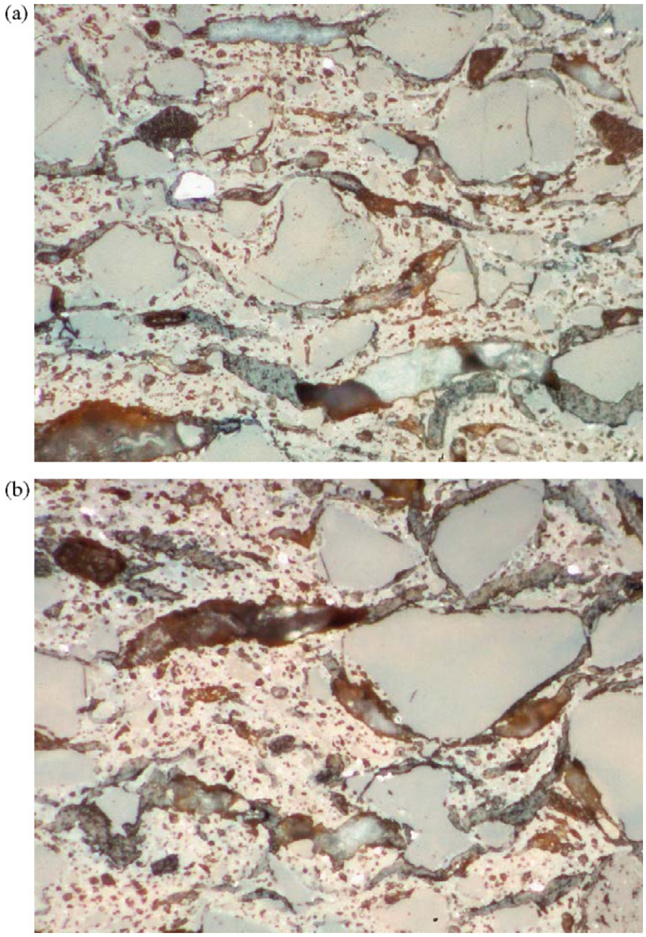

Fig. 7. Scanning electronic microscope photos of fired ordinary brick (Magnitude ×16). (a) Paste mixed with water, (b) paste mixed with the effluent (OMW).

- *Fired tensile strength*: The fired strength of the OMW product is shown to be approximately 9% lower when compared with that of the control, this decrease is due to the increase of the porosity in the internal microstructure.

In spite of these modifications occurring in bricks including OMW, the specific values of the different measured parameters respected the French Standards characterizing ordinary bricks [13].

In some Mediterranean countries where this building material is sometimes used raw unfired, it is interesting to use this unfired experimental product [5]. Furthermore, firing bricks containing OMW did not seem to highly affect their tensile strength.

The current study did not show any inconvenience regarding the long-term use of the experimental brick products. Indeed, no leaching behaviour of the material was noticed as it was observed while using the same material mixed with OMW and unfired.

Also a long-term stability of the material was noticed after two years. This behavior could be easily explained since the product was fired, and the stored bricks made did not show any degradation of residual organic matter overtime.

4. Conclusion

OMW represents a highly polluting effluent in most olive oil producing countries around the Mediterranean basin. The valorization and the recycling of this wastewater could be a partial solution to the negative environmental impact of OMW. Its incorporation in brick-making industry can contribute to this solution.

In this study, our main concern was to assess the impact of OMW incorporation in unfired and fired bricks. For this, the tensile strength, the shrinkage and the water absorption as well as the paste plasticity index of new materials were considered in these laboratory experiments achieved on rods and industrial trials held on bricks.

This work attempted to show that in spite of the solid contents of the OMW, its substitution for the water currently used in brick making did not have any negative effect on extrusion performance. The most suitable firing temperature for such bricks was 880 °C. This differs from the firing temperature used for bricks where water is added during the manufacturing process by the C.G.B. Company (920 °C). This finding can save water and energy as required by the Tunisian Agency of Energy Conservation.

The new materials (bricks and rods), prepared according to the same protocol adopted by the C.G.B. brick company, confirm that the experimental products including the wastewater had increased their water absorption compared to the factory product. This allows a higher level of open porosity and consequently lower density than those of the control.

Furthermore, an increase of the dry-fired shrinkage was noticed because of the presence of organic matter included in the effluent. Nevertheless, the fired tensile strength remained constant for the new material.

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