DEVELOPMENT OF RELIABLE BUILDING BRICKS INCORPORATING OLIVE-MILL WASTEWATER FOCUSING ON THERMAL INSULATION AND ENERGY SAVINGS

Xenofon Spiliotis1, Vayos Karayannis2, Stelios Lamprakopoulos1, Stefanos Zaoutsos3, George Papapolymerou1

1 University of Thessaly, Gaiopolis, Larissa, 41110, Greece
2 University of Western Macedonia, Department of Chemical Engineering Kozani, 50100, Greece
E-mail: vkarayan62@gmail.com

ABSTRACT

The present communication reports the development of reliable building bricks with improved thermal insulation behavior through substitution of the water mixed with the standard ceramic clayey raw materials with olive-mill wastewater (OMWW). This endeavor will contribute to circular economy and environmental protection as well as possible energy savings and CO₂ emission reductions. OMWW is an aqueous by-product generated in large amounts during olive oil production processes. It is characterized by high COD values, low biodegradability, high toxicity, acidic pH, a high concentration of potassium and high solid matter content. Therefore, finding eco-friendly and economically viable solutions for OMWW treatment and valorization represents a significant challenge in the oil-producing countries, in order to avoid severe environmental problems from the uncontrolled disposal of this effluent. For that purpose, a series of ceramic brick specimens are fabricated with the introduction of either olive-mill wastewater or water employing a pilot-plant simulation of the industrial brick manufacturing procedures. The brick thermal conductivity and the mechanical performance are evaluated. According to the results obtained the replacement of fresh water by olive-mill wastewater in the ceramic manufacturing provides a production of lighter building bricks of a decreased thermal conductivity (hence increased thermal insulation capability) as well as an acceptable and a reliable mechanical strength. Furthermore, the energy consumption measurements reveal energy savings of as much as 30 % attained during the brick firing.

Keywords: building bricks, olive-mill wastewater, water and energy savings, thermal conductivity, mechanical strength, reliability.

INTRODUCTION

The recent trends in the brick manufacturing industry show an increase of the use of thermal insulation bricks [1 - 5]. Actually, the use of ceramics as thermal insulating materials is one of their main applications. The thermal conductivity of the construction materials in particular is a significant factor that influences the energy associated with buildings heating and cooling. This is of critical importance especially in countries with extreme climatic conditions [6, 7]. Moreover, the mechanical strength reliability is one of the critical factors that may restrict the wider use of brittle materials in various structural applications. In fact, the strength data of quasi-brittle to brittle materials tend to scatter strongly depending on the existence of flaws such as voids, micro-cracks, and impurities in the bulk of the material [8]. This leads to the necessity of a fracture data statistical analysis by applying probabilistic theories in view of the utilization of brittle products such as ceramics in design and manufacturing. Among other distributions examined, the Weibull statistics [9] possesses a strong theoretical basis and has been lately used to estimate the distribution and the reliability of the brittle materials fracture data.
Indeed, the Weibull analysis appears to be of a particular importance to assess the quality of the construction ceramics incorporating solid industrial by-products [10].

On the other hand, olive-mill wastewater (OMWW) or “alpechin” is an aqueous waste by-product of a strong offensive smell generated in large amounts from the extraction processes during olive oil production. Its uncontrolled disposal leads to severe environmental problems concerning especially the natural water reservoirs, the soil fertility and porosity, and generally the whole ecosystem. This is due to OMWW high COD values, low biodegradability, high toxicity, acidic pH, a high concentration of potassium and high solid matter content. The typical characteristics of OMWW are already reported in other studies [11-15]. Therefore, finding eco-friendly and economically viable solutions for OMWW treatment and disposal, such as their valorization into building ceramics, represents a significant challenge in the major oil-producing countries. Moreover, the high energy content of the biomass remaining in OMWW can be introduced to the ceramic clay bodies for fuel savings. Actually, the biomass energy potential is considered to be one of the most promising among the renewable energy sources due to its wide availability worldwide. Generally, the safe management of the industrial effluents as useful secondary resources in the development of value-added products will contribute to environmental protection, symbiosis and an ample coordination in the industrial sector in accordance with current E.U. policies [16, 17].

The present communication reports research data concerning the beneficial utilization of OMWW in ceramic manufacturing instead of the fresh water used to mix with standard clayey raw materials. The latter procedure is typically used by the ceramic industry to form a plastic mass for extrusion of reliable building bricks. The focus is placed on the brick thermal insulation capability (in terms of thermal conductivity) and provision of energy savings in view of the apparent environmental benefits.

Aiming this the ceramic brick specimens are shaped by extrusion under vacuum and fired/sintered at two different peak temperatures (850°C and 950°C) for consolidation. Then, the physico-mechanical properties and the microstructure of the materials produced are thoroughly characterized. The thermal burnout of the solids introduced to the brick body through OMWW is expected to affect the porosity of the ceramic body and consequently the thermal conductivity and thus the thermal insulating behavior of the bricks. The possible energy savings leading to CO₂ emission reductions are also evaluated by carrying out comparative energy consumption measurements upon firing at 950°C of specimens containing either OMWW or fresh water.

**EXPERIMENTAL**

**Brick specimen production**

A series of brick bodies were produced by incorporating each time either water or OMWW (approximately 20 % wt.) in the “Viokeral” clay mixture typically used by the Greek industrial enterprise “Terra S.A.” for standard ceramic manufacturing [18]. A schematic diagram of the proposed method is shown in Fig. 1. Specifically, the clay mixtures were kneaded with either water or OMWW to obtain a proper plastic mass for brick specimen shaping using a pilot-plant vacuum extruder provided with a manual cutter. The rectangular brick cross section bars referred to 80mmX50mmX24.1mm. The extruded test specimens were exposed to natural drying (for 24 h) and then to forced drying at 105°C (for 24 h) until reaching a constant weight. The dried test pieces were fired in a programmable electric chamber furnace following a protocol of a gradual temperature increase up to a peak temperature at which they remained for 15 min in order to minimize the energy consumption. The final firing/sintering temperatures examined were 850°C and 950°C.

![Flowchart of the procedure followed for building bricks production](image_url)
Energy consumption measurements

The energy consumption was measured by a laboratory precision equipment (ADELEQ, Model DRT-301D) connected on-line to the electric furnace. Initially, the thermal inertia of the electric chamber furnace was measured twice (2% mean deviation) following a protocol of a gradual temperature increase by a step of 100°C up to 900°C. The last step was from 900°C up to 950°C, which included also a soaking time of 15 min at the peak temperature of 950°C. Afterwards, the energy consumption of the bricks was measured after subtracting the thermal inertia of the furnace. The energy consumption values per 5 kg of a product were calculated.

Bricks characterization

The determination of the total volume shrinkage, the weight loss upon sintering, the apparent density, the open and total porosity, and also the three-point bending strength was conducted using fired brick specimens according to ASTM C67: Standard Test Methods for Sampling and Testing Brick and Structural Clay Tile. Then, the strength measurement results were analyzed using the Weibull statistics. The thermal conductivity coefficient was measured at 25°C by the Anter Unitherm Model 2022, which applied the guarded heat flow meter method. The tests were carried out in accordance with ASTM E1530: Test Method for Evaluating the Resistance to Thermal Transmission of Thin Specimens of Materials by the Guarded Heat Flow Meter Technique. All results presented here are the mean value of 25 specimen measurements. Scanning Electron Microscopy (SEM) analysis of the ceramic microstructures produced was carried out using a JEOL 6610LV microscope.

RESULTS AND DISCUSSION

The effects of water substitution by OMWW and of sintering temperature on a) the total volume shrinkage (TVS) and the weight loss on sintering (LOS), b) the apparent density (d), c) the open (OP) and total porosity (P), and d) the thermal conductivity coefficient (k) of the ceramic bricks are presented in Fig. 2.

Fig. 2. Effects of olive-mill wastewater (OMWW) substitution for water and of firing temperature on: a) the total volume shrinkage (TVS) and the weight loss on sintering (LOS), b) the apparent density (d), c) the open (OP) and total porosity (P), and d) the thermal conductivity coefficient (k) of bricks.
Bricks appearance and physical properties

The experimental results in Figs. 2a and 2b show that the water substitution by OMWW affects the specimen appearance and the corresponding physical properties as follows:

- it improves the mixture workability during extrusion and produces more pale specimens;
- it promotes the weight loss during sintering by almost 35% at 850°C (from 5.74% to 7.71%) and 52% at 950°C (from 5.12% to 7.8%) due to the combustion of the additional organic matter present in OMWW;
- it restricts the volume shrinkage by 43.2% at 850°C (from 15.7% to 8.9%) and 51% at 950°C (from 17.4 to 8.5%) resulting in more reliable products in respect to their geometry;
- it decreases the bulk density by 10.5% at 850°C and 11.4% at 950°C producing lighter bricks;
- the sintering temperature increase from 850°C to 950°C does not affect the total volume shrinkage (TVS) and the density.

Porosity

Fig. 2c shows that OMWW embodiment increases significantly the open and the total porosity by 41% and 24.5% at 850°C and 48% and 28.5% at 950°C, respectively. This means that the closed pores contribution to the total porosity is decreased. The sintering temperature increase from 850°C to 950°C has in fact a slight effect on the open porosity of the prototype specimens. It decreases by 8%.

Thermal conductivity

Fig. 2d shows that OMWW presence in the clay mixture decreases the thermal conductivity coefficient (k) by 19.5% at 850°C and 14% at 950°C. This finding indicates that the heat transfer resistance of the ceramic material obtained increases considerably resulting in an improved thermal insulating capability. On the other hand, the increase of the sintering temperature from 850°C to 950°C increases k by 4.3% and 11.3% for specimens mixed with water and OMWW, respectively. The previous conclusions can be explained by the porosity variations (see Fig. 2b).

Mechanical strength - Reliability

The Modulus of Rupture (MOR) in MPa upon 3-point bending testing of the fired specimens is calculated on the ground of the following equation:

\[ \text{MOR} = \frac{3PL}{2bw^2} \]  

(1)

where P is the force (MN), L is the opening width (m), b is the width of the specimen (m), while w is the thickness of the specimen (m).

Then, the modulus of rupture (MOR) data is modeled using the Weibull distribution analysis. The Weibull
parameters are estimated and studied in order to assess the quality of the ceramics produced.

Fig. 3 presents the effects of the firing/sintering temperature and the water substitution with OMWW on a) the modulus of rupture (MOR), b) the stress survival probability (Ps) distribution (reliability versus strength diagram), and c) the Weibull parameters, namely (i) the Weibull modulus (m) and (ii) the characteristic strength ($\sigma_0$) of the bricks obtained. It is evident from Fig. 3a that MOR is decreased by 10% at 850°C and by 13.5% at 950°C in case of OMWW embodiment in the clay mixture instead of water. However, as the firing temperature is increased from 850°C up to 950°C, MOR increases by 11.3% and 7% when water and OMWW is introduced, respectively. The results in Fig. 3b show that almost all the Ps distribution curves present relatively similar slopes indicating mechanical strength results of a comparable reliability. The Weibull modulus (m) in Fig. 3c describes the scatter of the data. It should be specified here that small values of m would refer to large strength scatter. In fact, the m values of all specimen sets produced are equal or even higher than 5. Such values are typically found for traditional ceramics. Hence, the strength data remains reliable in all cases considered here. Indeed, the Weibull characteristic strength ($\sigma_0$) appears to be approximately 50% higher for the bricks obtained by substituting water with OMWW than for the prototypes at both firing temperatures examined. This indicates a clear improvement of the strength reliability.

Microstructure

Fig. 4 presents the SEM micrographs of specimens with water or OMWW at firing temperatures of 850°C and 950°C. The micrographs do not reveal significant changes in the microstructure after water substitution by OMWW. The microstructure becomes more compact by increasing the firing temperature from 850°C up to 950°C. An extended diffusion possibly combined with restricted viscous flow phenomena in the ceramic matrices is observed.

Electric power consumption

The water substitution with OMWW (a total solids dry content of 1.65% wt.) in the clay mixture decreases sharply the energy consumption upon firing by almost 34
%. In fact, energy of 3.08 KWh is needed to sinter 5 kg mass of unfired specimens mixed with water, while only 2.04 KWh is consumed in case of OMWW introduction. This decrease is due to the thermal burnout of OMWW solids providing a high energy release. It can be utilized in a continuous ceramic production line.

CONCLUSIONS

The characterization results show that the replacement of fresh water (approx. 20 % of the plastic mass) by olive-mill wastewater in ceramic brick manufacturing results in the production of lighter ceramic bricks of decreased thermal conductivity (thus increased thermal insulation capability) and reliable mechanical performance.

Furthermore, the energy consumption measurements reveal that production energy savings of as much as 30 % are attained during the firing. This is expected to lead to CO₂ emission reductions and to contribute to the alleviation of the intense environmental problems associated with the safe disposal of this industrial effluent.

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REFERENCES

