



# Manufacture of sustainable clay ceramic composite with composition $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-CaO-K}_2\text{O}$ materials valuing biomass ash from olive pomace

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## ABSTRACT

Fly ash is a biomass combustion by-product produced by dragging ash from the base of the furnace. Disposing of ash is a growing economic and environmental burden. Based on physical and chemical properties, fly ash could be used in the manufacture of construction materials. This paper investigates the influence of biomass fly ash from olive pomace as additive to manufacture of clay ceramic composite materials. Fired clay brick at 950 °C were prepared containing between 0 and 25 wt% fly ash. Final products are studied by water absorption, bulk density, loss of ignition, linear shrinkage, compressive strength and physisorption  $\text{N}_2$ . The results reveal that the porosity of the materials increases with the level of fly ash replacement (10% up to 25 wt%) resulting in to increased water absorption and decreased compressive strength. Fired clay brick developed in this study can be used for construction materials based on criteria of the current regulations.

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## 1. Introduction

Currently, one of the biggest problems for society and the environment is the incessant increase in the generation of waste and the reduction of the space available for its settlement in landfills. Therefore, it is necessary to plan the start-up to treat them and improve their management. Nowadays the main sources of energy used worldwide are fossil fuels which are very easy and economical to obtain and transport [1]. One of the most promising alternatives is the generation of energy from biomass (burning).

In Spain and especially in Andalusia, the generation of energy from the burning of biomass offers good prospects for the future according to the Plan for the Promotion of Renewable Energies which in the future will imply an increase in waste from the burning of biomass.

The two types of solid waste that are produced in the process of incineration of biomass are fly and bottom ash [2]: fly ash are the particles dragged by the gas stream outside the combustion chamber and that they collect and precipitate in filters and the bottom ashes are the residues of coarse fraction that remain deposited in the grill.

Ceramic products are ideal candidates to accommodate considerable amounts of wastes through the inertization and neutralization by encapsulating in the ceramic matrix [3–5].

There are studies where these biomass ashes have been used as fertilizers [6], and increasingly studies have been carried out on the use of bottom ash from the combustion of olive pomace in the manufacture of clay bricks [7]. The ashes used in the elaboration of the materials were obtained from a biomass plant and are the result of the burning of pomace, a waste from the olive industry.

Subsequently, the pieces were obtained by mixing clays and studied in terms of physical and mechanical properties such as the absorption and suction of water, the linear shrinkage and the loss of mass experienced by the pieces, the bulk density and the compressive strength, comparing the results obtained with those of pure clay and adsorption-desorption of  $\text{N}_2$ .

The purpose of this research work is to provide new knowledge about the use of fly ash as a raw material for the manufacture of ceramic materials.

## 2. Materials and methods

### 2.1. Materials

The materials used in the present study were clay (black, red and yellow) and biomass fly ash. The clay was collected from the

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**Table 1**  
Chemical composition (XRF) of clay and biomass fly ashes (FBA).

Oxide content (%)	Clay	FBA
SiO <sub>2</sub>	54.4	11.7
Al <sub>2</sub> O <sub>3</sub>	12.36	2.51
Fe <sub>2</sub> O <sub>3</sub>	4.58	1.26
CaO	8.76	10.2
MgO	2.46	3.03
MnO	0.03	–
Na <sub>2</sub> O	–	–
K <sub>2</sub> O	3.37	42.66
TiO <sub>2</sub>	0.60	0.11
P <sub>2</sub> O <sub>5</sub>	0.11	2.97
SO <sub>3</sub>	0.68	3.6
ZnO	0.026	–
SrO	0.027	0.0367
ZrO <sub>2</sub>	0.033	–
Cl	–	2.26
LOI	12.51	18.54
Total	99.946	99.005

clay quarries located in Bailén (Spain). The clay mixture used in this research consisted of 40% black, 30% red and 30% yellow clay type. Fly ash, a by-product of combustion in a steam boiler, from the biomass power plant “La Loma”, located in Villanueva del Arzobispo, province of Jaén (Andalusia, Spain). The biomass plant uses as fuel olive pomace. The ash production of the plant is: bottom ash 21.74% (1500 tons/year) and fly ash 78.26% (5400 tons/year). Chemical analysis of clay and fly ash are presented in Table 1.

## 2.2. Preparation of ceramic composite materials

The raw materials, once dried at 110 °C for 24 h, were sieved to obtain a homogeneous particle size of less than 150 µm. The fly ash of biomass was added to the clay in different quantities obtaining series of pure clay, and with 5, 10, 15, 20 and 25 wt% of fly ash. To give the mixture plastic properties, 10 wt% of water, mixed homogeneously in a kneader, was added. The samples were formed in a rectangular matrix of 60 × 30 mm by applying uniaxial loads of 10 MPa on a hydraulic press Mega Model KSC 15. The fired samples were designated as xCVLL, where x denotes the content (wt%) of biomass fly ash in the matrix clay.

After another drying period, the specimens in each series were sintered in a Nabertherm furnace using a heating rate of 2 °C/min up to 950 °C, keeping this temperature for 1 h. Finally, they were allowed to cool until reaching an ambient temperature. To allow

comparative results, six samples per series were prepared to perform the tests.

## 2.3. Characterization of raw materials

The chemical composition was determined by X-ray fluorescence (XRF) using the Philips Magix Pro PW-2440. The crystalline phases present in the clay and fly ash were determined using X-ray powder diffraction in a PANalytical X'Pert PRO MPD diffractometer with CuK1/2 radiation (1.5406 Å) and High Score Plus software. The microstructural analysis was performed by scanning electron microscopy (SEM) using a JEOL SM 840 microscope. Crystalline phases of Biomass fly ash and clay were evaluated using X-ray diffractometry with an X'Pert Pro MPD automated diffractometer (PANalytical), equipped with a Ge (111) primary monochromator, using monochromatic Cu Kα radiation and an X'Celerator detector.

## 2.4. Characterization of ceramic composite

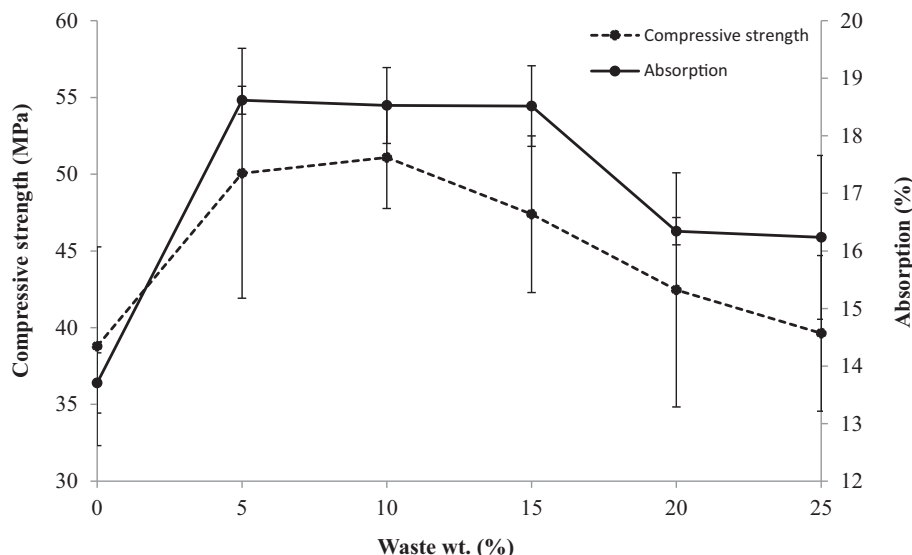
The linear shrinkage (%) was determined from the length of the samples before and after firing using a calliper with a precision of ±0.01 mm. The mass loss on ignition was determined as the mass loss between drying at 110 °C and firing at 950 °C. Standard tests EN 772-13:2001 and UNE 67-027-1984 were used to evaluate the apparent density and water absorption for all of the series. The water suction (Kg/m<sup>2</sup>·min) was determined through capillary action according to the standard procedure UNE 67-031-1985.

Six samples were tested for compressive strength in accordance with Standard Test Method EN 772-1:2011 in a Shimadzu laboratory testing equipment.

The porosity and distribution of the particle size were measured using N<sub>2</sub> adsorption–desorption isotherms at 77 K in a Micromeritics equipment (TriStar II 3020 model), following the BJH method (Barrett et al., 1951).

Crystalline phases of ceramic composite were evaluated as raw materials using X-ray diffractometry with an X'Pert Pro MPD automated diffractometer (PANalytical), equipped with a Ge (111) primary monochromator, using monochromatic Cu Kα radiation and an X'Celerator detector.

Microscopic studies were carried out by FE-SEM by using a JEOL SM 840 microscope, equipped with energy dispersive X-ray (EDX) for microanalysis.



**Fig. 1.** Compressive strength and absorption of water of the fired ceramic composites at 950 °C as function of ash addition.

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