



Effect of glass powder on the technological properties and microstructure of clay mixture for porcelain stoneware tiles manufacture

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HIGHLIGHTS

- A new way to produce the porcelain stoneware tiles is proposed.
- Physico-mechanical and microstructural features of samples are investigated.
- This research shows the ultimate use of waste glass in ceramic industry.

ARTICLE INFO

Article history:

Received 18 August 2017

Received in revised form 6 March 2018

Accepted 7 March 2018

Keywords:

Waste glass powder

Microstructure

Mechanical properties

ABSTRACT

The present study focused on the use of waste glass powder with clay mixture, for manufacturing of porcelain stoneware wall and floor tiles. The raw materials were characterized in terms of mineralogical and chemical composition. Different mixtures with various amounts of glass were prepared and fired in the temperature range of 1000–1200 °C, for 2 h. The influences of waste glass content on the technological properties (linear shrinkage, water absorption, bulk density and flexural strength) were determined. Microstructural analysis of the fired samples was carried out by X-ray diffraction and SEM. The experimental results revealed that waste glass contributes to improve physico-mechanical properties of samples up to 30 wt% of glass content at 1150 °C. The final product (R₅₀G₂₀C₃₀) showed weak absorbed water value (0.4%) and higher flexural strength (39.06 MPa). Thus, according to the ISO 13006 standard, it can be classified as porcelain stoneware tile, since it satisfies the standard requirements. The addition of high amounts of glass (>30 wt%) into the ceramic bodies is undesirable due to its adverse action on the physical properties of products. Moreover, the SEM micrographs of tiles specimens fired at 1150 °C and with 30 wt% of glass showed a very compact texture and dense microstructure with a mullite fibre. As a consequence, tiles exhibited noteworthy mechanical properties.

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1. Introduction

The increase in the consumption of disposal and returnable material have increased the volume of residues produced in the cities together with the growth of the population. Recycling is an important environmental and economical alternative for each material class not only in the process of their origin, but also in the other activities sectors like ceramic tiles industry, mainly for those which do not decompose easily neither in nature, nor by atmospheric precipitation for thousand years such as glass. Such

recycling makes it possible to reach some important objectives related to the storage of wastes, conserving limited supplies of natural raw materials and consequently, safeguarding the environment [1]. Different kinds of wastes have been incorporated into tiles and bricks. Important investigations have been carried out on this topic and interesting reviews have been published [2–11].

The elaboration of higher quality ceramics requires large amount of raw materials such as: clay, which provides the plasticity of the mixture; fluxing agent (feldspar, talc, Pyrophyllite, etc.), which produces liquid phase during sintering and inert material (quartz) which favour the dimension control of the product after firing [12]. Consequently, the demand in raw materials increases and some have to be purchased because there are not always

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available. This would create the shortage and the rising cost of raw materials.

For these reasons, there is a requirement to reformulate the mixture composition of ceramic bodies by alternative raw materials, which have to be very cheap and available such as waste glass. Such a solution can be considered viable only if the industrial process essentially remains unchanged and the quality and characteristics of the final products do not collapse [1].

Several benefits are obtained by using glass cullet in ceramic materials: Reduce electrical energy consumption, enhance quality of material, reduction in HF emissions [13] and reduction in CO₂ emissions [5].

Some works in the literature show that waste glass bottle are composed of high amount of silica, SiO₂ (70–74) % and considerable amount of alkaline earth oxides: Na₂O (12–16) %, CaO (5–11) % and MgO (1–3) % [14]. Due to its mineralogical and chemical composition, waste glass shows fluxing properties and as a result, can run in replacement of fluxing agent used in traditional ceramic. Several works have been published for this purpose [15–23].

In Cameroon, there are any factories producing ceramic wall and floor tiles, although there are several exploitable clay deposits. Besides, there are large amount of waste bottle, which cause environmental issue such as ground pollution.

Therefore, it is necessary to solve this problem, by developing a research approach, which could help to valorize these raw materials in traditional ceramic sector such as porcelain stoneware tiles.

Porcelain stoneware tiles are the vitrified ceramic building materials, with exceptional technological performances: Low absorption water (<0.5% according to ISO 13006 standard) and high flexural strength (>35 MPa). They are either used for outdoor or indoor application (pavement and wall recovering).

The development of porcelain stoneware tiles remains a significant challenge, in view of the ever increasing production and market. Intensive research on this topic have been carried out and interesting results are available [24–42].

These previous studies which are relevant focused on the combination of clay with amount of quartz sand, feldspar, and sometimes waste glass. Unfortunately composition prepared from clay and waste glass as only raw materials are rarely proposed. This decreases the use potential of glass. Such a study is needed because, as previously said, the chemical composition of cullet showed the large percentage of SiO₂, Na₂O, CaO and MgO. Thus, cullet can act as inert component in total replacement of quartz sand, and as fluxing agent in total replacement of feldspar.

Thus, the objective of this research is to study the feasibility of using clay and waste glass powder in the elaboration of porcelain stoneware tiles. The effect of firing temperature and cullet content in the clay mixture will be investigated and discussed in terms of physico-mechanical properties and microstructure.

2. Materials and methods

The clay materials: Kaolinite clay (R) and plastic clay (G) used in this work were collected from Koutaba (West-Cameroon). The glass waste (green and colorless bottles) was first washed, crushed into small pieces before being grounded to a relatively fine powder using ball mill. Cullet and clay were sieved using a 100 µm and 125 µm mesh sieve respectively.

The chemical analysis was obtained through X-ray Fluorescence (XRF) spectrometry (Bruker S8 Tiger 4 kW spectrometer, INISMA-CRBIC). The samples were molded in borax (Na₂B₄O₇·10H₂O) and the determination of major elements derived according to calibration with standards. The chemical analysis performed by X-ray fluorescence is presented in Table 1.

The X-ray diffraction (XRD) patterns were obtained with a Bruker D8-Avance Eco 1Kw diffractometer (CuKα radiance, λ = 1.5418 Å, V = 40 125 kV, I = 25 mA) with Lynxeye Xe energy dispersive detector in the Laboratory of "Argiles, Géochimie et Environnements Sédimentaire (AGEs)" at the University of Liège, Belgium. The analysis was carried out on the bulk material (non-oriented powder with grinded particles <125 µm). The XRD patterns were recorded over the 2–70° 2θ angular range for the raw material. The step sizes considered for this analysis

Table 1
Chemical composition of raw materials (wt.%).

Major oxide	Clay samples		Soda lime cullet
	Kaolinite clay (R)	Plastic clay (G)	
SiO ₂	66.78	50.52	73.3
Al ₂ O ₃	15.28	27.80	2.5
Fe ₂ O ₃	7.87	2.91	0.2
TiO ₂	1.51	2.39	0.1
CaO	0.12	0.16	8.7
K ₂ O	0.99	0.70	0.99
Na ₂ O	/	0.14	15.1
MgO	0.52	0.31	3.5
MnO	0.05	0.02	<0.01
Cr ₂ O ₃	0.02	/	0.02
ZnO	0.12	/	/
P ₂ O ₅	0.16	0.14	0.02
L.O.I	6.58	13.72	/
Sum	100.00	98.81	/

was 0.02° 2θ, whereas the time per step chosen was 0.25 s. The first reason is motorized in order to illuminate a fixed sample length whatever the angular position (16 mm for bulk). Qualitative determination of major crystalline phases present in the raw materials (Figs. 1a, 1b and 1c) was carried out using Eva software.

Five mixtures containing proportional amounts of different clay (reddish R and gray G) and cullet (C) were prepared. The details are reported in Table 2. The raw materials were wet mixed in a porcelain pot using a ball mill. The slurry was dried at 105 °C in an oven for 3 days. After drying, the mixtures were then crushed and sieved to pass through 125 µm screen to ensure homogeneity. Specimens of 80 × 40 × 10 mm for flexural strength and linear shrinkage and cylindrical samples of 15 × 7 mm for physical properties were prepared using uniaxial compaction. The pressing strength was fixed at 8 tons for the first samples and 3 tons for the second one. For bare shaped and cylindrical samples, 45 g and 2 g of mixture were respectively used and thoroughly mixed with water as binder. The specimens obtained were first dried in air for 48 Hours, then in an oven at 105 °C for 24 h and later fired in the temperature range of 1000–1200 °C for 2 h, in an electric furnace (Nabertherm, model LH 60/40).

During the firing, the heating rate was kept at 5 °C/min. After the calcination, the fired samples were cooled in the furnace at room temperature (35 °C) and then were subjected to various tests: The linear shrinkage, was determined by direct measurement of specimens lengths before and after the firing. The water absorption values were determined from the weight differences between the sintered and the water saturated samples (immersed for 2 h in boiling water and soak for an additional 24 h at ambient temperature). The bulk density values were determined by the Archimedes' method of immersion in water. The flexural strength was determined using an electromechanical universal testing machine (M & O, type 11.50, and No 21) in three-point bending fixture, 40 mm support span and with crosshead of 3 mm·min⁻¹.

Phase identification of the sintered specimens was performed by X-ray diffraction (XRD) and the microstructures of the fired samples were obtained by observing the fracture surfaces with a scanning electron microscope (SEM).

3. Results and discussions

3.1. Linear firing shrinkage

The shrinkage behavior knowledge of a clay product during sintering is important in ceramic industry. This allow to control the dimensions of final product. A weak linear shrinkage is always privileged, if other physico-mechanical features can be achieved [43]. The shrinkage behavior of different mixtures with different ratios of glass content and various firing temperatures are shown in Fig. 2. There is a tendency for linear shrinkage to increase for all the samples with glass powder content and firing temperature. The higher linear shrinkage means that more thermal changes occurred between clay and glass powder. This behaviour is fostered by raising the temperature. It may be suggested that the presence of high amount of fluxing oxides originated from waste glass, helps the generation of liquid phase which fills the pores, promotes the bringing closer of grains, thus leading to a reduction of linear shrinkage during firing [15]. At 1100 °C, a subsequent decrease in shrinkage value is noticed when the level of powder

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