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Rheological properties and microstructure of binary waste red brick powder/metakaolin geopolymer

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HIGHLIGHTS

- Addition of waste red brick powder causes a decrease in plastic viscosity of metakaolin-based geopolymer.
- Thixotropic behavior of metakaolin diminished when more than 75% of waste red brick powder was used in geopolymer mixture.
- Part of the crystalline phase in waste red brick takes part in the geopolymerization process.
- Waste red brick powder and metakaolin show a synergic effect on the compressive strength of blended geopolymer.

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ABSTRACT

Waste brick powder is a material with sufficient pozzolanic properties to be used as a supplementary cementing material in Portland cement-based concrete or as a precursor for the production of geopolymers. This study aimed to investigate a binary geopolymer system composed of metakaolin (MK), waste red brick (WRB) powder and sodium silicate solution as an alkaline activator. Five different mixtures were prepared where 0, 25, 50, 75, and 100% of the MK was substituted with WRB. The rheological parameters of the fresh pastes were correlated with a structural evaluation of the reaction process.

The findings showed that the yield stress of all mixtures was very low, but it increased with time as the geopolymerization reaction proceeded. The more MK was used in the mixture, the faster the yield stress developed. On the other hand, the plastic viscosity strongly depended on the composition of the mixtures and dropped with increasing WRB/MK ratio. The mixtures containing more than 50% MK exhibited a decrease in viscosity with time due to higher reactivity and the faster dissolution of the MK particles.

1. Introduction

Ceramic waste is generated all over the world in large quantities. The ceramic industry itself widely contributes to its production in the form of scrap. For instance, in Europe, the amount of waste produced in the different production stages of the ceramic industry reaches 3–7% of global production [1]. Current trends in the building sector are for the utilization of thin-joint technology in the construction of building envelopes. Since current hollow brick production techniques do not provide sufficiently smooth and even surfaces, the brick blocks have to be surface ground, generating a significant amount of waste ceramic dust which is mostly landfilled, although it has both the composition and granulometry needed to be suitable for application in the building industry.

Waste red brick (WRB) powder is a material with pozzolanic properties. Pozzolanicity is dependent on several characteristics, of which the main ones are the amount of amorphous phase, particle size distribution, and specific surface area. The pozzolanicity of burned clays was determined by Baronio and Binda [2] in 1997. The authors concluded that pozzolanic properties are dependent on the temperature and burning time of the brick body. Böke et al. [3] determined the pozzolanic activity of brick aggregate in old mortars and plasters via electrical conductivity measurements and showed that pozzolanic properties are consistent with amorphous phase content. Pozzolanic activity allows the use of WRB powder as a supplementary cementing material in concrete [4,5], an application which has been known since antiquity in Rome,

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Greece and Mesopotamia. It also enables such powder to be employed as a component of modified lime mortars and plasters, in which manner it has been used since the Middle Ages [6–8].

WRB powder can be successfully used as a precursor in alkali-activated materials or geopolymers [9]. These are generally composed of aluminosilicates with specific properties and an alkaline solution as an activator. Waste-based materials and by-products used in building industry are considered to be environmentally friendly, hence common aluminosilicate precursors are fly ashes from power plants, palm oil fuel ash, ash from biomass, slag, waste brick powder, waste glass, stone dust, incineration products of sludges, and similar materials containing amorphous silica and/or aluminosilicates [10]. Another precursor utilized for geopolymer production is metakaolin (MK), which is manufactured by the calcination of kaolinite containing rocks at 750 °C [11,12]. Since its production is quite expensive, energy consuming process with strong environmental impact, WRB powder can be used as a reasonable alternative to this material.

The course of the alkaline activation of WRB powder is influenced by several factors: the amorphous phase content, particle size, the composition and dosage of the alkaline activator, and temperature. The amorphous phase content is associated with the composition of the raw material for brick making and the burning temperature. These parameters are given and cannot be influenced; therefore, it is necessary to determine the pozzolanic activity of the WRB powder and the amorphous phase content by means of quantitative XRD analysis. The pozzolanic activity determined by the Chapelle test is given in mg of $Ca(OH)_2$ per 1 g of pozzolan and ranges from 300 to 500 mg $Ca(OH)_2$ for common RB powders [13].

The composition and amount of alkaline solution play an important role during the formation of geopolymer products. Sodium/potassium silicate solution modified by sodium/potassium hydroxide is the most commonly used alkaline activator. The effect of the amount and composition of alkaline activator has been investigated by several authors. Tuyan et al. [14] found the optimum activator composition had SiO_2/Na_2O = 1.6 and a concentration of 10% Na_2O. Maximum compressive strength was obtained upon curing at 90 °C for 5 days, but in terms of energy consumption, the optimum treatment was at 80 °C for 5 days. Similar results were also obtained by Reig et al. [15], who studied the geopolymerization of WRB powder at a constant water/binder ratio and a curing temperature of 65 °C.

The above-mentioned studies indicate that WRB powder is mostly activated at higher temperatures, usually above 60 °C, in order to attain sufficient mechanical properties. For this reason, it is preferred to use WRB powder in a blend with other reactive material, e.g. metakaolin, fly ash or slag, in order to maintain effective curing under laboratory conditions. Rakhimova [16] investigated the effect of the addition of waste brick powder in various amounts, with a variety of compositions and from different sources on the properties of pastes and mortars made of alkali-activated slag. Mixtures where 20 and 40% of the slag was replaced by waste brick reached the highest compressive strength (in the range of 90–120 MPa), and the reaction rate dramatically decreased with the increasing specific surface area of the brick powder.

Only few research works concerning the rheology of fresh geopolymer mixtures have been published so far. The rheological properties of geopolymers depend on several factors and cannot be easily generalized as this group of materials involves several different types of aluminosilicate precursors as well as various alkaline activator types and concentrations. Kashani at al. [17] reported that the use of alkali hydroxide causes a significant increase in the yield stress of an activated slag paste, whereas sodium silicate gives a lower yield stress due to the plasticizing and deflocculating effects of the silicate ions. Alonso et al. [18] compared the yield stress for mixtures made from Portland cement, alkali-activated blast furnace slag and fly ash with various liquid/binder ratios and found that the workability of alkali-activated binders is more sensitive to changes in the composition of the mixture than Portland cement. Favier et al. [19,20] reported that the low yield stress of MK-based geopolymer compared to Portland cement paste is caused by negligible and only low energy interactions between MK grains. The high viscosity of MK-based mixture is then caused by hydrodynamic viscous dissipation in the highly viscous sodium silicate solution. Poulesquen [21] studied mixtures of metakaolin and alkaline activator prepared from two types of amorphous silica (Tixosil 331 or Aerosil) and sodium/potassium hydroxide. The viscoelastic parameters of various geopolymers were evaluated using dynamic rheology and showed there is a relation between the formation and size of oligomers and the type of alkaline solution. The authors found faster geopolymerization kinetics with sodium hydroxide but with weaker interaction between components, and hence the limited formation of oligomers, whereas potassium alkaline solution preferably formed a three-dimensional structure.

The present study deals with the properties of fresh mix and hardened geopolymers prepared from metakaolin, WRB powder and modified sodium silicate solution. Rheological properties such as the plastic viscosity and yield stress of fresh mixes were determined and the dissolution/reaction process was evaluated using XRD, FTIR and NMR analysis.

2. Materials and sample preparation

Geopolymer mixtures were prepared from MK and WRB powder as aluminosilicate precursors, and modified sodium water glass was used as an alkaline activator. The metakaolin was supplied by ČLUZ company, Czech Republic. The red brick powder was a waste material obtained from the grinding of hollow red-clay bricks from Heluz company. The activating solution was prepared by mixing 1000 g of commercial sodium water glass with an SiO_2/Na_2O ratio of 1.6 and 37 g of NaOH p.a. in order to adjust the SiO_2/Na_2O value to 1.4. The chemical composition of both the aluminosilicates and the water glass is presented in Table 1.

The particle size distribution of both aluminosilicate precursors was determined by laser granulometry measurement and the distribution curves are presented in Fig. 1. The mean particle size of the WRB powder (d_{50} = 8.5 μm) was very similar to that of the MK (d_{50} = 6.3 μm). However, the WRB powder exhibited a much wider range of particle size than metakaolin. The d_{90} value, which indicates that 90% of all grains are smaller than a given value, was 56.5 μm for the WRB, whereas it was only 11.6 μm for the MK.

Rheological properties were determined for the pastes, which were composed of 40 g of aluminosilicate precursor, 36 g of alkaline activator and 20 mL of water. The composition of the alumi-

Table 1
Chemical composition of metakaolin, red brick powder and water glass (wt%).

Component	Metakaolin	Red brick powder	Water glass
SiO_2	58.70	52.37	26.43
Al_2O_3	38.50	12.50	–
Fe_2O_3	0.72	2.41	–
CaO	0.20	22.83	–
MgO	0.38	1.44	–
Na_2O	0.04	0.24	16.61
K_2O	0.85	2.46	–
SO_3	0.02	3.71	–
TiO_2	0.49	0.40	–
LOI	1.67	0.00	–
Pozzolanic activity (mg $Ca(OH)_2$/g)	1120	467	–