



Recycled natural wastes in metakaolin based porous geopolymers for insulating applications

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ABSTRACT

The effectiveness of two amorphous silica based wastes, rice husk ash and volcanic ash powders, used to act as structural reinforcement and partial replacement of metakaolin in porous matrices for thermal isolation was assessed. Both wastes produced colloidal phase that contributed to improve the reactivity and homogeneity of the matrices enhancing the mechanical strength. Rounded pores were obtained in relation with significant reduction of the interpore partitions under controlled addition of the blowing agent. The bulk thermal conductivity decreased from 0.6 to less than $0.15 \text{ W m}^{-1} \text{ K}^{-1}$ with the increase of the concentration of blowing agent. The final matrix presented pore size distribution and pore connectivity that allows analytical description and prediction of their effective thermal conductivity. Results demonstrated that clear relations can be established among the viscosity of the pastes, chemical composition, amount of blowing agent and the volume expansion/pore size distribution.

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1. Introduction

In the context of global warming for environment, the role played by the insulating materials for the achievement of the reduction of energy consumption is significant. This explains the continuous increase of interest of researchers and governments in this class of materials especially the low cost ones that are produced with a lower energy consumption and that may contribute to the management of some natural or/and industrial wastes [1,2]. Recent studies have proposed in this direction porous inorganic polymer matrices from clays [3,4] or wastes [5]. The motivation comes from the low values of the thermal conductivity of these materials ($< 1 \text{ W m}^{-1} \text{ K}^{-1}$) and their fire resistance when compared to organic polymer foams. The introduction of volcanic ash or rice husk ash in the system of porous Inorganic Polymer Composites (IPC) would improve the Si/Al ratio in the pore solution allowing the reduction of the amount of sodium silicate necessary to achieve a good porous structure. This also provides the possibility to use only sodium hydroxide as activator. The system NaOH–rice husk ash or NaOH–volcanic ash will develop a viscous

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paste with binding properties capable to act as binder and substitute sodium silicate giving high compactness and mechanical resistance to the material [6–8]. It is recognized that the presence of sodium silicate in the bulk composition of inorganic polymer cements is one of the limitations for this new class of materials to be as “green” [9]. Successful formulations in which the sodium silicate is absent, or reduced, sound promising for the sustainability of the final porous matrices [9]. The processing route appears to be less exigent in terms of energy (room temperature) and materials added for pore formation. Usually the development of capillary porosity through the use of organic matter required important quantity of energy to escape the organic matter at high temperatures [4]. Process which is detrimental for the environment due to the energy cost and CO_2 emitted [10–12].

Experimental results have shown that the aluminum metal is subject to direct dissolution in concentrated alkaline solutions [13]. The direct dissolution is a very intensive process which leads to a high corrosion rate. With elapsed time a film is formed on the metal surface which acts as a barrier for the species transport. Therefore, the corrosion rate is significantly reduced after formation of the film. The direct dissolution of aluminum is due to the following reactions:



Table 1
Bulk chemical composition, particle size distribution and phases of MK, PZ and RHA.

	Oxides/wt%									Granulometry/ μm		
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	Na ₂ O	K ₂ O	MgO	L.O.I	d(0.1)	d(0.5)	d(0.9)
MK	55.21	38.82	1.10	4.26	0.00	0.14	1.32	0.89	0.55	1.922	8.228	33.154
P	45.57	15.94	12.81	3.28	8.97	1.03	2.83	5.76	0.21	1.129	5.439	25.380
R	89.03	2.23	0.66	2.59	0.96	0.17	2.34	0.38	3.11	1.33	4.901	20.520

Note: The three materials contain amorphous phase which is the major, muscovite, illite and quartz for MK; diopside, augite and anorthite for P.



The reactions are highly exothermic, accompanied by a rapid evolution of hydrogen gas. Clearly, the direct aluminum dissolution is accompanied by the production of hydrogen. From experimental studies, gas bubbles have been observed at the aluminum metal surface [3,13,14]. Therefore, the corrosion of pure aluminum in alkaline solution proceeds mainly by water reduction according to (Eqs. (1) and 2): the dissolution rate depending on the concentration of OH⁻ and aluminate ions Al(OH)₄⁻ at the solid/liquid interface. It was concluded that the process of the corrosion of aluminum can be used for the production of porous geopolymers [3–5]. The high alkalinity of the pore solution favors a very high rate of dissolution of aluminum producing enough gases for the optimum expansion of geopolymer pastes [3]. The sodium silicate has been found to be an effective inhibitor of corrosion in aluminum–alkaline systems [13,14]. The inhibition is due to the formation of an amorphous aluminosilicate film on the metal surface. Labbe and Pagetti [14] showed that after an initial drop in the solution potential due to the dissolution of aluminum, the potential increases with time, followed by a series of oscillations that reveal a tendency for the inhibition film to become, alternately, partially or totally inhibiting [14]. Finally, the potential reaches a stabilization value. The larger the silicate concentration is, the greater the stabilization will be. The action of the aluminum to form pores in geopolymers is limited with time and by the concentration of the metal, the degree of reactivity/polycondensation of the geopolymer, the setting step as well as the rheology of the pastes. These parameters affect the process of the design of porous inorganic polymer composites.

The present work has an objective to assess the impact of rice husk ash and volcanic ashes on the processing of porous inorganic polymer and on the properties of the final products. It was expected that the colloidal phase from the dissolution of amorphous content of both wastes will improve the volume expansion, the light weight structure and harmonize the distribution of pores. Moreover the colloidal phase would enhance the tortuosity of the skeleton maintaining at a very low level the flow of the thermal flux through the matrix. The processing route used in this study appears more sustainable as the two wastes are usefully recycled. Their presence reduced the amount of clayey materials to be calcined, hence the energy consumed and CO₂ released. A homogeneous matrix with limited constraints linked to the unreacted particles, like quartz grains [3] and residual alkalis. The homogeneity will harmonize the distribution of the movement of gas produced from the corrosive reaction of metallic powder and help in controlling the pore size distribution. This enables the design of models suitable for the description and prediction of the effective thermal properties of porous inorganic polymer materials [3,4,15–17].

2. Materials and experimental methods

2.1. Materials

Standard kaolin from the Mayouom deposit in Cameroon [18] was used for the preparation of the metakaolin. The white variety as described by Njoya [16] which is a sand-poor kaolin, 76–85% kaolinite and 2–9% quartz with illite, muscovite, anatase and hematite as secondary minerals was selected. The kaolin was sieved to eliminate the residual quartz sand, dried and calcined at 700 °C for 4 h. The volcanic ash was from the Mungo deposit [19] in the Littoral region of Cameroon. The material presents essentially an amorphous matrix in which fine grains of anorthite, entastite-augite, diopside are embedded [19,20]. Rice husk was collected in a rice mill at Ntarikon-Bamenda in the North-West Region of Cameroon. It was considered as a valueless by-product of rice milling and thus burnt in heaps near the mill. Husks were also collected burnt at 700 °C for one hour in an electric furnace at a temperature rise of 3 °C/min for comparison. The bulk chemical composition and the minerals content of the above described materials are presented in Table 1. The aluminum powder (99%, particles size less than 74 μm , density=2.70) and sodium hydroxide (96%) were of laboratory grade from Sigma Aldrich, Italy. The sodium silicate solution (SiO₂/Na₂O=3.0) was provided by Ingessil, Verona, Italy.

2.2. Preparation of the porous inorganic polymer composites

Preliminary investigations (results not show here) permitted to adopt 2.5 volume of metakaolin for 1 volume of volcanic ash or rice husk ash. The above described volume ratio corresponds in mass to 1:1 for the metakaolin:volcanic ash while it is 7:1 for the couple metakaolin and rice husk ash respectively. The relative high amount of volcanic ash in the IPC matrix compared to rice husk ash is due to the fact that the volcanic ash is an aluminosilicate with a relatively high amount of alumina (15 mass% of Al₂O₃) compared to the rice husk ash (2 mass% of Al₂O₃). Moreover, volcanic ash, as others pozzolanic materials (fly ash, slag, etc.) is suitable as 100% solid precursor for the production of inorganic polymer composites [20,21]. Its relatively high density is responsible for the value of its mass 7 times that of rice husk ash for a unit volume. However, this work presents both materials as promising for porous IPC with high potential for the development of frost-like structure in the geopolymerization context.

Metakaolin (MK), rice husk ash (R) and volcanic ash (P) were ground to fine particles down to 100 μm with the surface area of 24.8, 59.3 and 21.9 m²/g respectively.

Appropriate amounts of solid powders were weighed and introduced into an alkaline solution (sodium hydroxide 8 M and sodium silicate – Na₂O:SiO₂=1:3 – in 1:1 volume ratio) for a liquid/solid ratio of ~0.35 (the solid part of NaOH and Na₂SiO₃ is considered). Five formulations of IPC for MK, MK–P and MKR were prepared. MK4, MK6, MK8, MK10 and MK12 were obtained introducing 4, 6, 8, 10 and 12 g of aluminum powder over 10,000 g of

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