



Metakaolin-based inorganic polymer composite: Effects of fine aggregate composition and structure on porosity evolution, microstructure and mechanical properties



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ABSTRACT

This paper examines the phase transformation, pore evolution, microstructural and mechanical changes that occur in inorganic polymer cement (IPC) in the presence of three different grade of fine aggregates ($\phi < 100 \mu\text{m}$) of ladle slag, nepheline syenite and quartz sand. Experimental results indicate that polycondensation was enhanced in nepheline syenite based specimens, compared to quartz sand, due to the increase in H–M–A–S phases in relation to the dissolution and interaction of amorphous/disordered fraction of aggregates. H–C–S and H–C–A–S with H–M–A–S phases were identified in the ladle slag based specimens. The formation of these new phases reduced both the cumulative pore volume and pores size. The apparent increase in volume of capillary pores in ladle slag based specimens was explained by the residual bubbles from the carbonates included in raw slag. The flexural strength of the inorganic polymer cement increases from 4 MPa to 4.2, 4.8 and 6.8 MPa with the addition of 20 wt% of quartz sand, nepheline syenite and ladle slag respectively. These values increase significantly between 28 and 180 days of curing (9.1 MPa for ladle slag and 10.0 MPa for nepheline syenite). It was concluded that fines can be used to remove the HM and poorly bounded alumina oligomers in metakaolin based inorganic polymer matrices and improve the interfacial zone for the design of an optimum grade and high-performance composites.

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1. Introduction

Reactions between strong alkaline solution and amorphous to metastable aluminosilicate materials produce an inorganic binder, which is also known as inorganic polymer cement (IPC) or geopolymers [1]. These reactions have been investigated for the development of alternative materials to conventional cements and concretes based on klinker. Researchers and industries are motivated by the global warming concerning the carbon dioxide emission derived from klinkerization process combined to its high energy consumption [1]. Metakaolin [2], industrial waste, industrial by-products, including coal fly ash and metallurgical slag [3], and natural volcanic ash [4] based IPC have been projected as

alternative binders due to the amorphous content of the respective aluminosilicate easily dissolvable in high alkaline solution. An ideal binder should have a pH, reactivity and microstructure that enable the design of dense, stable and high strength mortar and concrete.

White et al. [5] indicated that the subtle structural changes, observed up to 17 h after the mix of solid aluminosilicate with alkaline solution, are predominantly related to the dissolution of the initial metakaolin precursor prior to the formation of gel. When considering IPC and concretes, this dissolution would include not only metakaolin (or fly ash, ...) but also the amorphous fraction of aggregates used. In the presence of soluble silica, reactive aluminum is able to substitute for silicon in many of the oligomers anions that occurs and displays more polymerized structures [2]. Aggregates have in their structure various allotropic forms of silica that directly affect their behavior in mortar or concrete. In a highly

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Nomenclature

H	H ₂ O	S	SiO ₂
M	Na or K	C	CaO
A	Al ₂ O ₃		

concentrated alkali media (case of the pore solution), the amorphous silica is highly reactive compared to metastable silica, β and α -cristobalite and quartz. From the foregoing, it is clear that the alkali content of the binder as well as the amount of aggregates, the nature of the silica in the aggregate and the total proportion of the amorphous or metastable phases as their distribution will govern the interface behavior between the IPC paste and the aggregate and by the way the design of the durable and high strength mortar and concrete. Moreover, It is recognized that the presence of sodium silicate in the bulk composition of inorganic polymer cements is one of the preoccupation for this new class of materials to be defined as “green” [6,7]. Successful formulations can be design with fine aggregates capable to easily react with alkalis and form gel type binder which can completely or partially replace the sodium silicate and improve the sustainability of the final porous matrices [6,7].

During the dissolution/polycondensation the cross-linking and densification is enhanced by the residual amorphous silica present in the matrix and its transformation in H–M–A–S gel phase, acting as bonds. This gel is characterized by colloidal-sized, globular units closely bonded together at their surfaces, whereas the high silica concentration in a silicate-activated system enables a more homogeneous gelation process throughout the interparticle volume [8,9].

The silica to alumina ratio (in the amorphous or reactive fraction of solid precursors) and the alkali to alumina ratio (in the pore solution) directly affect the engineering properties as the stability of geopolymer cement. It is then evident that several groups of geopolymer systems should be considered. Sagoe-Crentsil et al. [10,11] classified geopolymer pastes with low and high Si/Al. When the Si/Al is low (<1), it is expected that the excess of aluminate oligomers formed will combine with the soluble silica at the surface of aggregate. New H–M–A–S phases should be formed alongside the alkaline silicate gels resulting from the reaction of the residual alkali with silica from aggregate. The additional H–M–A–S phases and the alkaline silicate gel formed could promote efficient packing and crosslinking between IPC and aggregate, developing a continuous and homogeneous strengthening phase at the interfacial zone. Dissolved silica from the surface of aggregates can then react easily with the gel modifying their structure and probably acting on the existing pores (meso and macro) and phases. Larger capillary pores from the processing of inorganic polymer cement are generally filled with residual alkali, hence the presence/addition of fine aggregates, also known as fines, will provide materials to form binder phases capable to close those pores. The grow up of additional phases, able to pack more closely against the aggregate surface, will reduce the width between the paste and will contribute to better stress transfer between aggregate and inorganic polymer cement paste.

It is our main objective to study the properties of inorganic polymer cement with ground aggregates (fines) with the aim to see the effectiveness of the addition of fines to improve the mechanical properties of IPC composites. Grounded silica sand, nepheline syenite and calcium-rich aluminosilicate-namely ladle slag from steel industry-aggregates have been here used with particles size below 100 μm . The aggregates content was varied so that to understand the extent of their dissolution and polyconden-

sation in inorganic polymer matrix. The calcium hydrate is the major binding phase within Portland cement system that could be compared to H–M–A–S of inorganic polymer cement system. Further comparison present M–H to substitute C–H since M–H is generally observed as residual product of hydration of geopolymer. When in contact with aggregate, the geopolymer paste will firstly be available with its fluid highly concentrated in M–H. It is expected that the behavior of the system M–H and fine aggregate will provide more homogeneous and stable matrix capable to improve the interface behavior of concrete or mortar. The interfacial zone in binders, mortars and concretes can be improved by monitoring the bulk chemical composition and physico-mechanical parameters (particles size and distribution, compressive compaction and diffusion phenomenon).

These investigations contribute to a comprehensive understanding of the possibility to improve the interfacial zone between inorganic polymer cement and aggregates, which is important for the design and production of dense, resistant and durable composites (mortars and concretes). Determination of the effects of phases transformation on the volume and spatial distribution of pores, the mechanical properties and microstructure constitute the main objective of the study. The empirical data were generated using mechanical testing, Environmental Scanning Electronic Microscopy (ESEM), Mercury Intrusion Porosimetry and Microtomography. Improved understanding of IPC-Fines systems is essential for their use in developing high performance binders, mortars and concretes.

2. Materials and experimental procedures

2.1. Materials and preparation

Metakaolin (MK) was used as amorphous aluminosilicate for the production of inorganic polymer cement, obtained by the thermal treatment at 700 °C for 6 h of a standard kaolin from Cameroon [12]. MK was milled to obtain particle with size below 60 μm ; the surface area value of the resultant powder was 37 m²/g (Brunauer–Emmet–Teller, method by nitrogen absorption on a Micrometitics GEMINI 2360 instrument).

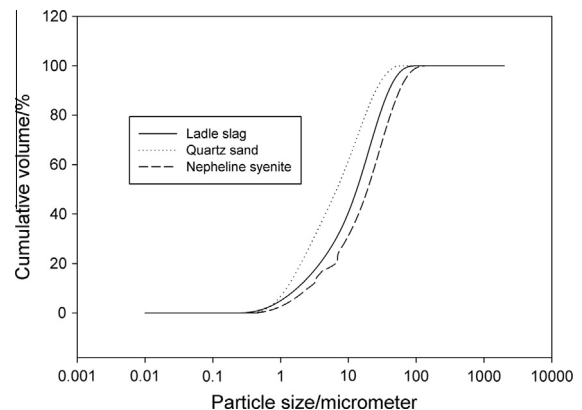


Fig. 1. Particle size distribution of the 3 different fines: ladle slag, quartz sand, nepheline syenite.

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