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Synthesis and properties of alkali activated borosilicate inorganic polymers based on waste glass

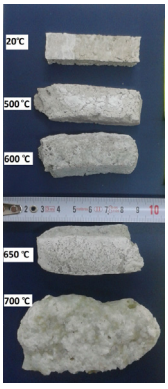
Taha H. Abood Al Saadi [a,b], Alina Ioana Badanoiu [a,*], Adrian Ionut Nicoara [a], Stefania Stoleriu [a], Georgeta Voicu [a]

[a] University Politehnica of Bucharest, Faculty of Applied Chemistry and Materials Science, Dept. Science and Engineering of Oxide Materials and Nanomaterials, Bucharest, Romania
[b] Middle Technical University/Technical Engineering College-Baghdad, Iraq

HIGHLIGHTS

- Inorganic polymers from alkali activation of waste glass with NaOH + borax solution.
- These materials have intumescent behavior.
- Fly ash addition improves their stability in humid environment.

GRAPHICAL ABSTRACT



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ABSTRACT

Alkali activated borosilicate inorganic polymers (AABSIP) can be obtained by the alkali activation of waste glass powder with a NaOH solution in which anhydrous borax was previously dissolved. AABSIPs have the ability to swell during the thermal treatment increasing their volume and apparent porosity. The temperature at which these materials swell is smaller as compared with the one assessed for the inorganic polymers obtained by the alkaline activation with NaOH solution of waste glass powder. The partial substitution of glass powder with fly ash (10%) do not substantially modifies the AABSIP behavior at high temperatures. The presence of fly ash improves the stability in humid environment (moisture) of AABSIP. The intumescent behavior (i.e. swelling as a result of heat/fire exposure) is less affected for the AABSIP paste with fly ash when it's curing take place in humid medium (moisture) as compared to the composition without fly ash.

1. Introduction

Alkali activated inorganic polymers or geopolymers are materials that can be produced from an aluminosilicate source (a wide variety of natural minerals or industrial wastes) and an alkaline activator solution (caustic alkalis or alkaline salts). Among the numerous wastes that can be used as solid or/and liquid component for the synthesis of this type of materials [1–5], our research group selected two i.e. waste glass (cullet) and red mud slurry (waste resulted in bauxite processing industry) [6–9]. The results obtained so far pointed out the possibility to re-use

* Corresponding author.
E-mail address: alina.badanoiu@upb.ro (A.I. Badanoiu).

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silica-soda-lime waste glass powder or mixtures of waste glass and red mud in order to produce binding materials (geopolymers) by alkaline activation with NaOH solution and thermal treatment at 60 °C [6–8]. One interesting property of these materials was their ability to swell, with important increase of volume and porosity, during thermal treatment [7]; therefore, is possible to produce foamed geopolymers by the alkali activation of finely ground soda-lime glasses (with/without red mud addition) and thermal treatment at 600–800 °C, without using any other foaming agents [7,10].

This ability to swell during the thermal treatment is specific for intumescent materials; therefore, other possible application for these materials, obtained by the alkaline activation of waste glass, can be as intumescent materials for passive fire protection. The currently available intumescent materials can be used for door seals, penetration or linear gap sealants, etc. and have, in most cases, lower values for the initiation temperatures (140–200 °C) [11]. Therefore, considering the fluxing ability of boron compounds [12,13] we assess in this study the possibility to obtain alkali activated inorganic polymers (geopolymers) based on waste glass powder with borax addition.

The use of boron compounds in the reagent mixture specific for geopolymers (alkali activated inorganic polymers) synthesis was also reported in the following four studies [14–17].

Nicholson and Fletcher [14] reported the synthesis of a novel geopolymer compositions with structurally incorporated boron; these materials can be obtained from a reagent mixture comprising/including a metal (group I – alkali) silicate, an aluminosilicate and a boron containing compound. In this new type of geopolymer the boron represents an integral part of the structure and it may substitute the tetrahedral aluminium or silicon. According to the authors these materials have as main advantage a longer setting time with respect to aluminosilicate geopolymers based on fly ashes with high calcium content (class C fly ash).

Nazari et al. [15,16] reported also the synthesis of boroaluminosilicate geopolymers by the alkaline activation of fly ash (class F) with a mixture of NaOH solution and anhydrous borax. The compressive strengths of these materials reached 64 MPa, after 90 days of hardening [15]. According to these authors, the presence of B–O bonds in the boroaluminosilicate structure could explain the high values of the compressive strengths; moreover, the microstructure of boroaluminosilicate geopolymers is completely different from the one of traditional aluminosilicate geopolymers [15,16].

Williams and van Riessen [17] obtained a new class of materials with boron content by the alkali activation of silica fume with a mixture of NaOH solution and anhydrous borax; these materials, called alkali activated borosilicate inorganic polymers (AABSIP), have compressive strengths comprised between 45 MPa and 56 MPa. According to these authors, the microstructure of AABSIP is similar to the one of geopolymers i.e. a porous, glassy matrix with some non dissolved silica particles and borax crystals [17].

The above mentioned studies focused on different aspects regarding the properties and application of geopolymers/alkali activated inorganic polymers with boron content i.e. controlled setting time [14], high mechanical strength [15,16] and neutron shielding application [17]; therefore, to the best of our knowledge, we present for the first time in this paper results regarding the thermal behavior of AABSIP based on waste glass.

Previous results obtained on geopolymers based on waste glass powder showed a low hydrolytic stability of these materials due to the presence as main reaction products of soluble sodium silicate (aluminate) hydrates [6,8,9]; therefore, in this study we used also fly ash in some formulation, in the attempt to improve the hydrolytic stability of AABSIP materials.

2. Materials and methods

2.1. Materials

Mixed waste glass powder (MG) was obtained by the grinding of cullet with various colors, from a glass recycling facility.

The main compounds of this soda-lime waste glass powder are: silica (70.45 wt%), alkalis ($Na_2O_{eq} = Na_2O + 0.658K_2O$) 13.25 wt%, calcium oxide (9.75 wt%), aluminium oxide (2.24 wt%) and iron oxide (1.65 wt%). The Blaine specific surface area of MG powder was 3265 cm^2/g.

Fly ash, a by-product resulted in the combustion of pulverized coal in a thermal power plant, was used as solid component for inorganic polymer preparation. The Blaine specific surface area of this material was of 4594 cm^2/g. The oxide and mineralogical compositions of fly ash are presented in Table 1.

The anhydrous borax was obtained by the thermal treatment of borax ($Na_2B_4O_7{\cdot}10H_2O$) at 150 °C for 30 min followed by a second thermal treatment at 300 °C for 15 h [17].

The formulations of AABSIP specimens are based on a recipe proposed by Williams & Van Riessen [17], and are presented in Table 2.

MGB was prepared by mixing the solid component (MG glass powder) with the liquid component i.e. a solution obtained in two steps: i) dissolution of NaOH pellets in water, and ii) addition of anhydrous borax ($Na_2B_4O_7$).

After the borax addition the solution was fast converted into a gel like phase. This solution was mixed with glass powder (MG) or with mixtures of glass powder and fly ash (MGBF). The workability of the paste was initially good but rapidly decreased and the material stiffened in few minutes. In order to obtain more

Table 1
Oxide and mineralogical composition of fly ash (F).

Oxide composition (wt.%)	LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	SiO_2/Al_2O_3
	6.05	40.41	19.02	13.82	12.5	2.59	5.43	2.21
Mineralogical composition	Iron calcium oxide (PDF 74-1860); iron oxide (PDF 84-0307); aluminum silicon oxide (mullite-synthetic) (PDF 82-1237); calcium aluminum silicate (anorthite) (PDF 76-0948); silicon oxide (quartz) (PDF 83-2465)							

Table 2
The composition of studied AABSIP.

Sample	MG wt%	$Na_2B_4O_7$ wt%	Fly ash wt%	NaOH wt%	Distilled water wt%
RB	60	–	–	9.3	30.7
MGB	40.5	19.5	–	9.3	30.7
MGBF	36.45	19.5	4.05	9.3	30.7