



Production and characterization of geopolymer blocks based on hydroxyapatite rich biomass ashes

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Abstract

Vegetal and animal derived biomass ashes were selected to realize new sustainable chemically consolidated ceramics via acid or alkaline activation. Mixed biomass ashes are mainly based on calcium phosphate and secondly on aluminosilicate compounds; they still represent an unexplored source material to be used in alternative ceramics production for building industry by low-cost manufacturing processes. Mixed biomass ashes were used to produce room-temperature hardened materials by chemical activation with solutions of phosphoric acid or potassium poly-silicate. Reactive mineral powders (metakaolin, magnesia and wollastonite) were also added to fasten the consolidation and the formation of a binding phase. The obtained formulations were finalized to maximize the amount of ashes disposed. Microstructural, physical and mechanical properties of the obtained materials were evaluated, along with their water stability. Acid-activated samples, even in presence of secondary reactive precursors, maintained a crystalline structure, while alkali-activated ones were able to form a partially amorphous and well consolidated matrix, with compressive strength of about 20 MPa.

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1. Introduction

Biomass combustion represents up to now a promising alternative for green energy generation. However, this process necessarily involves the production of considerably huge amounts of ashes that have to be disposed or reused with an adequate strategy. Bottom and fly ashes deriving from the incineration of vegetal residues are until now used as soil fertilizers or earmarked for the cement industries [1,2]. Animal-derived biomass ashes, mostly deriving from co-combustion or gasification processes with other secondary fuels, are generally landfilled or used for phosphorous recovery or fertilizers [3,4]. In fact, although biomass ashes have been widely considered as an alternative feed for the cement industry [5,6], their use as a raw material for cement or aggregate replacement actually has only for very limited cases [7].

Indeed, the possibility of reusing biomass ash, either animal or vegetal derived, in the building and construction industry has until now been limited to the addition of low percentages of wastes to ordinary Portland cements or other binding systems [7–11]. The practicability of using these kind of wastes as a primary source to produce new materials or precast elements through the processes of chemical activation is therefore a promising way that nowadays has been explored only to a limited extent [12,13]. The use of bonding materials that chemically react with the waste ashes at low temperatures to form consolidated ceramic-like bodies is undoubtedly attractive. Alternative systems of acidic and alkaline activation include chemically bonded phosphate ceramics, synthesized by acid–base reaction between an acid phosphate and a metal oxide (CaO, MgO, etc.) [14], characterized by a significant content of aluminosilicate bonding phase, both belonging to the wider family of geopolymers [15,16]. Geopolymers are indeed generally synthesized through the activation of an

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aluminosilicate with alkali metal hydroxides, silicates or phosphates at ambient temperature or slightly above, and are characterized by good thermal and acid resistance, low environmental impact and low cost. Alkali-activated geopolymers form SiO_4 and AlO_4 3D tetrahedral linked units by dissolution, reorganization and polymerization reactions, where alkali ions Na and K counterbalance the negative charges of AlO_4 . In the case of phosphoric acid-activated aluminosilicates, when the reaction system contains low-concentrations of SiO_4 and AlO_4 , these units are bound by PO_4 into similar amorphous to semicrystalline structures, forming phospho-aluminates (Al-O-P) and phospho-sialates (P-O-Si-O-Al-O-P) [14].

Starting from the results obtained from a previous reactivity study [17] a proper chemical consolidation step was implemented and chemically consolidated ceramic-like materials were obtained. Therefore, assessed their reactivity potential, mixed biomass ashes were used as primary raw material to cast geopolymers with low environmental impact, good structural properties, temperature stability and a low processing cost. In this investigation chemical bonding among calcium-phosphate based biomass ashes has been promoted, and room-temperature hardened materials have been produced by joint activation of biomass ashes and other secondary precursor (metakaolin, magnesia, and wollastonite), using different compositions and curing conditions.

2. Materials and methods

2.1. Biomass ash

Five different biomass ash types were used to formulate chemically consolidated materials. They all derived from a power plant located in Faenza, RA (Tampieri Energie s.r.l., Italy), from the combustion of residues of both vegetal and animal derived wastes (Fig. 1). Except for fraction S_3 , all biomass ashes have a similar chemical nature and mineralogical composition, being mainly defined by noticeable amounts of Ca and, secondarily, P and crystalline phases such as hydroxyapatite and substituted hydroxyapatite or calcium phosphates. S_3 , differently, is characterized by significant amounts of calcium hydroxide and potassium chloride [17]. The particle size distributions of the ashes differ broadly, depending on the filters of the plant from which they are collected. Two coarse fractions, one dry and one humid (S_2 and S_h respectively), are collected under the grids in a first step of the process: their particle size ranges from $20\ \mu\text{m}$ to up to 3 mm. S_1 and S_4 refer to two finer fractions which settle in the cyclone filters

after the flue gas treatment step and their particle size varies from $0.5\ \mu\text{m}$ to 1 mm. S_3 refers to the finest fraction (particle size from 10 to $3\ \mu\text{m}$), which originates after a second treatment of the flue gases in sleeve filters.

The five ash powders were mixed in the weight ratio of $S_1:S_2:S_3:S_4:S_h=1:1:1:3:5.3$, corresponding to the effective ratio of amounts/year of each ash type produced by the plant, with the aim to maximize the total volume of disposed ashes through chemical activation. After mixing, the obtained ash blend (AB) was oven-dried at $105\ ^\circ\text{C}$ for 1 h, then it was ground and sieved to obtain a particle size $< 100\ \mu\text{m}$. Elemental composition for AB is reported in Table 1. As expected, correspondingly to each ash chemical composition and its relative amount in AB, the ash blend evidenced high amounts of Ca and P, along with lower amounts of alkali metals and Cl. Si and Al were found only in minor quantities. Accordingly, given the direct matching between the ashes and the blend as regards their elemental compositions, mineralogical data are assumed to be correspondent to those of the single ash (as confirmed by XRD data reported hereafter).

2.2. Samples preparation

On the basis of previous studies and chemical composition of each single ash [17] different acidic or alkaline solutions were singled out to activate AB powder. Phosphoric acid solutions have been widely demonstrated to be good activators for aluminosilicates as metakaolin [18,19], $\text{Ca}(\text{OH})_2$, CaO, MgO or Ca-rich phosphate salts [14,18], while KH_2PO_4 aqueous solutions have been successfully used to formulate high strength magnesium phosphate cements [20]. An aqueous solution of H_3PO_4 (5 M) was prepared from H_3PO_4 85% (ACS reagent grade from Sigma-Aldrich, USA), while KH_2PO_4 was chosen to prepare an aqueous solution (coded KHP) ($\geq 99\%$, Sigma-Aldrich, USA) to replace H_3PO_4 , in order to avoid excessive heat release during mixing procedures and use of potentially harmful acidic activators. An aqueous solution of potassium disilicate, used as activator for alkaline consolidation, was hence prepared with a molar ratio of $\text{H}_2\text{O}/\text{K}_2\text{O}=23$ dissolving KOH pellets (85% purity from Sigma-Aldrich, USA) with deionized water in a commercial solution of potassium polysilicate (KSil 35/35 Ingessil s.r.l., Italy).

To promote and improve the activation process of the biomass ashes, readily reactive raw powders were selected and added as secondary precursors for the joint chemical activation of AB in

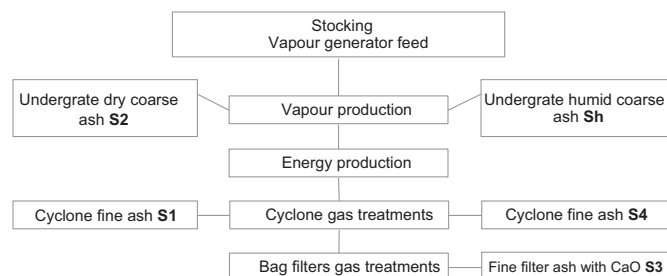


Fig.1. Flow chart of the incineration plant.

Table 1

AB chemical composition ($\pm 5\%$) obtained by ICP – Inductively Coupled Plasma ^(a), CHNS – Total Carbon, Hydrogen, Nitrogen, Sulfur content analysis ^(b) and EDS – Energy Dispersive X-ray Spectroscopy analysis ^(c).

Chemical element (wt%)											
Al ^a	C ^b	Ca ^a	Cl ^c	Fe ^a	K ^a	Mg ^a	N ^b	Na ^a	P ^a	S ^b	Si ^a
0.5	3.4	26.7	1.5	0.3	4.0	1.2	0.0	2.4	12.4	0.6	2.8

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