



Study of the chemical activation of hydroxyapatite rich ashes as raw materials for geopolymers

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Abstract

The behaviour of ashes, deriving from mixed vegetal and animal biomass incineration, was studied in different alkaline and acidic environments, in order to assess their suitability as primary raw material for low-temperature chemical consolidation. 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. Their chemical behaviour was studied as a function of pH and chemical nature of the leaching solutions, to identify the suitable conditions for ashes chemical consolidation. Preliminary results indicated that acidic digestion of ashes, regardless acid counterion nature, is able to determine the complete decomposition of complex calcium-phosphate phases. Then it allows recombination of dissolved phases into new ones, thus promoting chemical consolidation. Alkaline media were found to be less effective, however, biomass ashes might be successfully regarded as partially reactive fillers for alkali-activated materials.

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1. Introduction

Nowadays biomass is representing one of the most promising sustainable energy sources, as its use can have many advantages over conventional energy sources, or even over other green alternatives, such as relatively low costs, the possibility to promote regional economic policies and the creation of alternative income sources for farmers [1] and in general for industry.

Therefore, Europe is planning to achieve by 2020 that half the energy produced from renewable sources (20% of the whole European production) is obtained from biomass sources. However, to ensure that thermal conversion of biomass effectively results in a sustainable viable option to produce green energy, a proper plan for the disposal or valorisation of residual wastes and ashes produced from combustion is needed.

The building industry is actually one of the main field in which biomass ashes and residues can find useful applications, because of the large volume of raw materials and final products that every year are involved in the construction field [2]. However, biomass ashes reconversion and their reuse in concrete mixtures or precast products is tightly limited by current European standards, given their non-coal origin [3]. Indeed, wide amounts of alkalis, sulphates, phosphates and other detrimental oxides for concrete production tend to make biomass ashes undesirable for applications where conventional coal fly ash are widely used.

In particular, ashes derived from the co-combustion of plant or woody materials and other organic sources, such as meat and bone meals or wastes from livestock farming, generally contain considerably high quantities of these phases, so that different new solutions still have to be found for their reconversion.

By now about 18 mln ton/year of livestock wastes, mainly containing calcium and phosphorus compound, are produced in the European countries [4], the most of them being reused for fertilizer substitutes or remaining unprocessed. Therefore, animal-

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derived biomass ashes are still constituting an unexploited resource even for construction industry, which is often the pioneer in the field of recycling/reuse of waste materials as raw building materials.

For example, feedlot biomass ashes demonstrated sufficient reactivity properties so as to be suitable for partial cement replacement in concrete mixtures, in the same way that coal-fly ash are commonly used [5]. Alternatively, calcium-phosphate meat and bone meal ashes have been proposed as a valid alternative to inert silica sand in cementitious mortars, as they generally show physical and mechanical properties comparable to that of siliceous fine fraction used in mortars [6,7]. Few other studies investigated the role of amorphous silica derived from vegetal biomass such as rice husk, both in cementitious and geopolymer binders [8,9]

However until now, besides few investigations that focused on their reuse as Portland cement (OPC) mixture additions, to the author's knowledge calcium-phosphate biomass ashes have never been considered as a potential raw material for geopolymers, the low temperature hardened ceramic-like material.

This preliminary study is hence addressed to evaluate the potential application of various residues deriving from the co-combustion of vegetal and animal biomass for the formulation of OPC-free binding systems. Examples of acidic and alkaline activated alternative systems include [10]: magnesium oxychloride cements [11], obtained by the combination of magnesium oxide with concentrated aqueous magnesium chloride; calcium-sulphate (gypsum) cements and sulfo-aluminate cements in the compositional system of $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{Fe}_2\text{O}_3-\text{SO}_3$; chemically bonded phosphate ceramics synthesised by acid-base reaction between an acid phosphate and a metal oxide, as CaO , MgO , etc. [12]; alkali-activated cements [13,14], a large family of materials characterised by a significant content of aluminosilicate bonding phase. New alkali-bonded materials and relative application fields are more and more studied [15–20], so also increasing the perspective of recycling/reusing of waste materials of very different nature. However, no study reporting the chemical activation of calcium phosphates and aluminosilicates based mixed systems was found by the authors. To this aim, a first feasibility study has been carried out in order to determine whether chemical activation processes, which have long been known for the production of chemically bonded ceramic materials, can be successfully applied. Physical and chemical characterisation and leaching test performed in different acidic or basic solutions were conducted on five different types of mixed vegetal/animal derived biomass ashes, in order to assess the suitability of such waste materials for reuse as raw materials for novel and sustainable binding system.

2. Materials and methods

2.1. Ashes origin

Biomass ashes samples used in the present study were collected from Tampieri Energie Srl power plant, Faenza (RA), Italy, resulting from the co-combustion in a combustion chamber working

at 850 °C of both vegetal and animal-derived wastes. In details, biomass ashes (BA) mainly derive from meat and bone meal, grape seed vegetal flours, dregs and woodchips or other organic massive residues. Five different batches were taken into account, namely S_1 , S_2 , S_3 , S_4 and S_h . S_1 , S_2 and S_4 are collected from cyclone separators where they settle during combustion process, while S_3 , a finer fly ash fraction, is collected from sleeve filters. S_h refers to a coarse fraction that, after treatment through the combustion chamber, does not result as much volatile as the others and falls under the combustion grid. To avoid the formation of any fires, that might originate from residual embers during the combustion, the latters are collected in a hydraulic seal and they get wet, thus acquiring a higher moisture content than the other ones (average initial moisture content of raw ashes thus resulted to be equal to 2.1, 0.7, 0.3, 1.6 and 3.6% for S_1 , S_2 , S_3 , S_4 and S_h respectively).

2.2. Ashes characterisation

All BA used in this study were oven-dried before any characterisation procedure to eliminate residual water and conform S_h relative moisture content to that of the other ashes.

Chemical compositions were evaluated by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Varian liberty 200). To this aim, 50 mg of each fine-milled BA type were digested by microwave oven with 3 ml of an acid solution obtained by mixing HCl , HF and HNO_3 (37%, 40% and 65% ACS reagent grade respectively, from Sigma Aldrich, USA) in specific volume ratio (2:1:6 respectively). The resulting solutions were subsequently complexed by lithium tetraborate, to dissolve insoluble fluoride, before being filtered. Obtained solutions were diluted with deionised water up to 100 ml before being analysed.

Chlorine quantitative analysis and microstructural observations were evaluated by SEM-EDS analysis (Leica Cambridge Stereoscan 360 equipped with EDS INCA Energy 300 microprobe, Oxford Instruments), after coating the samples with carbon. Total carbon, sulphur and nitrogen elemental contents were determined on BA types by CHNS analysis (Perkin Elmer CHNS/O 2400 instrument). Particle size distributions $> 100 \mu\text{m}$ were obtained by hand sieving the ash powders, after being oven dried at 105° for 1 h and quartered, and determining the weight fraction passing different particle size ranges, from 100 μm to 2 mm. Particle size distribution was determined for BA finer powder fractions ($< 100 \mu\text{m}$) by granulometric analysis (Micrometrics Sedigraph 5100 instrument).

Simultaneous thermal analysis (STA) was performed by means of a STA 449 Jupiter (Netzsch Geraetebau, Selb-Germany) instrument up to 1300 °C, with a heating rate of 10 °C/min and in dry air flux. X-ray diffraction patterns were collected (Powder Diffractometer Bruker D8 Advance with $\text{CuK}\alpha$ radiation, Karlsruhe, Germany) on all raw BA types to qualitatively identify their mineralogical and crystallographic composition.

2.3. Releasing test

In order to assess their chemical release and reactivity, each BA was firstly sifted with a 100 μm net sieve. The retained fractions were discarded, while 0.5 g of each passing fraction

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