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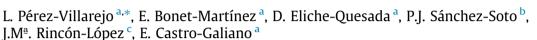
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Biomass fly ash and aluminium industry slags-based geopolymers





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ABSTRACT

Geopolymers are a new class of non-Portland cements produced using an alumino-silicate material and an activating solution, which is mainly composed of sodium or potassium and waterglass to be subsequently cured at relatively low temperatures. Those can be formulated by adding natural minerals, waste and/or industrial by-products. The study investigates the microstructural properties of geopolymers synthesized from metakaolin (MK) and the admixture of fly ash (FBA) and aluminium industry slags (AIS) at different ages of curing. Five different geopolymer compositions were prepared and characterized by XRD, ATR-FTIR and SEM/EDS. The study revealed that geopolymeric gels are identified, which show mainly glassy microstructures, in agreement with the X-ray amorphous diffraction patterns, broad FTIR features and confirmed by SEM/EDS, with promising results prior to an industrial scale.

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1. Introduction

Geopolymers are described as a wide variety of inorganic and composite materials with limited restrictions on alumina and silica content [1]. Chemically geopolymers are formed of tetrahedral alumina and silica units condensed at room temperature, yielding to three-dimensional network structure [2]. Geopolymers are a class of alkali-activated materials that use an aluminosilicate source that produces a cementitious binder. Since the 90's of 20th century, alkali activation research has grown dramatically in all corners of the globe [3]. However, much research has been focused on the development of geopolymers as Portland cement substitute from coal fly ashes, slags and another industrial wastes [4], while investigations regarding the production of geopolymers using biomass ashes have been scarce.

Fly ash from biomass combustion is composed mainly of amorphous silica and alumina, which makes it a suitable material for the production of geopolymers reducing the environmental impact of its production [5].

In the recycling process of the waste generated in the secondary aluminium industry, there is a by-product whose main component is aluminium oxide and whose characteristics closely resemble bauxite.

In this work, metakaolin and biomass fly ash, as precursor of aluminosilicates, and aluminium industry slag (AIS) as source of alumina, were examined as raw materials to obtain geopolymers using NaOH solution and sodium silicate solution (water glass) as activating agent. The geopolymers obtained were studied after curing for 60 days at room temperature, with a previous treatment at 60 °C for 24 h. The samples were subjected to characterization assays by attenuated total reflectance (ATR-FTIR) and X-ray diffraction (XRD). Finally, samples were subjected to microstructural and microchemical SEM/EDS analysis after 28 and 60 days. In this paper, a preliminary advance of the main results is presented.

2. Materials and methods

2.1. Materials

The raw materials used to synthesize geopolymers are meta-kaolin (MK), biomass fly ash (FBA) provided by the Aldebarán Energía del Guadalquivir S.L. located in Andújar (Jaén, Spain), a plant that generates renewable energy using olive-pruning and forest pruning (pine) biomass. Aluminium industry slag (AIS) was supplied by the company Befesa Aluminio S.L. located in Valladolid, Spain. They were used without any pre-treatment. For alkaline activation, a mixture of sodium silicate "water glass" (Panreac S.A.; 8.9 wt% Na₂O, 29.2 wt% SiO₂ and 61.9 wt% H₂O) and NaOH (reactive grade, 98 wt%, Panreac S.A) was used. The NaOH solution (5 M) was prepared in distilled water. The weight

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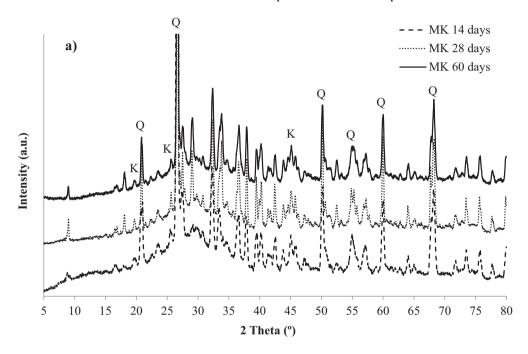
Table 1XRF of raw materials: metakaolin (MK), aluminium industry slags (AlS) and biomass fly ash (FBA).

Oxide content (wt. %)	MK	AIS	FBA
SiO ₂	66.50	8.60	41.2
Al_2O_3	25.66	56.22	8.05
Fe ₂ O ₃	0.54	1.81	2.78
MnO	0.01	0.19	0.18
MgO	0.12	5.75	4.63
CaO	0.14	1.90	22.10
Na ₂ O	0.07	3.88	-
K ₂ O	3.10	1.41	6.78
TiO ₂	0.26	0.54	0.45
P_2O_5	0.02	0.03	1.99
SO ₃	-	0.19	0.28
SrO	-	-	0.05
LOI	0.28	18.47	-

ratio between NaOH solution and water glass (NaOH/water glass) was 0.20 and the ratio between liquid and solid (L/S) used for all geopolymeric mixtures was 2.85.

2.2. Preparation of geopolymers

The geopolymers were prepared with five different compositions: pure MK and four other compositions such that GP1 (50% MK, 25% AIS, 25% FBA with a ratio Si/Al = 2.30), GP2 (50% MK, 33% AIS, 17% FBA, Si/Al = 1.95), GP3 (40% MK, 35% AIS, 25% FBA, Si/Al = 1.85) and GP4 (40% MK, 25% AIS, 35% FBA, Si/Al = 2.35). The synthesis was carried out by mixing the raw materials for 10 min. Subsequently the activating solution was added and stirred for 15 min. Then, the generated slurry was transferred to plastic molds. The samples were cured under controlled conditions



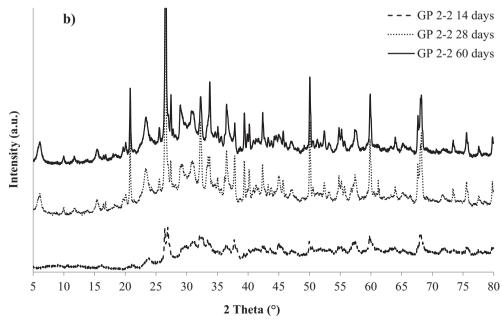


Fig. 1. XRD diffractograms of a) MK; b) GP2; c) GP3 and d) GP4 geopolymers evolution at different curing ages (Q: Quartz; K: Kaolinite).

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