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Sustainable Ash-Based Geopolymers

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The valorisation of industrial solid by-products into geopolymers, a new class of inorganic polymers, provides two main benefits. Firstly, it solves a significant waste management problem by the transformation of potentially dangerous industrial wastes into safe and value-added products. Secondly, it reduces the carbon footprint of cement concrete. Geopolymers exhibit comparable or even improved physico-mechanical and durability properties to those of standard cement-based concrete, with the potential for use in construction applications. Particularly, geopolymers can use alumina-silicate bearing industrial residues, such as fly and bottom ashes, red mud and slags, as secondary raw materials. In the present study, a comprehensive literature review of recent (last five years) advances in the field of sustainable ash-based geopolymers was conducted, focusing on four main types of geopolymers: a) low-Ca siliceous ash-based, b) high-Ca ash-based ones, c) geopolymers from ash-based mixtures, and d) ash-based geopolymer matrix composites.

1. Introduction

Recent research outcomes show that geopolymers can be used in civil engineering applications instead of standard cement-based concrete. Geopolymers often exhibit improved physico-mechanical properties and chemical inertness. Various alumino-silicate bearing industrial solid by-products, such as fly ashes, agricultural ashes, red mud, and slags, may also be incorporated in geopolymers as secondary resources. At highly alkaline conditions, these aluminosilicates form a geopolymeric binder system that hardens at room temperature similarly to ordinary Portland cement. The benefit of the industrial by-products valorisation into geopolymers is twofold. Firstly, potentially dangerous wastes should be transformed into safe and value-added products (Lakioti et al., 2017). Some works, with particular focus on water stability and leaching behaviour, reported that the geopolymerisation of coal ashes simply occurring via fly ash (FA) activation with alkali increases the ash handling and safe disposal (Miccio et al., 2014). Secondly, another significant benefit is the decrease of concrete carbon footprint (Rudin et al., 2017), as it is widely accepted that heavy industry (Karayannis et al., 2014), and particularly cement industry, contribute substantially to global CO_2 emissions and energy consumption (Loizidou et al., 2016). Many researchers have been motivated towards the development of much cleaner and low-emission technologies for a sustainable built environment (Burdova et al., 2016). Geopolymers from industrial residues activated by an alkaline reagent represent a promising and environmental-friendly solution for low CO_2 footprint cementitious materials (Passuello et al., 2017). This was confirmed by CO_2 footprint estimates for both geopolymer and cement-based concrete, including energy expending activities associated with mining, raw materials transport and concrete construction (Turner and Collins, 2013). Geopolymers can be considered as alternative binders, with significant global warming reduction potential (Heath et al., 2014). On the other hand, in ash-derived geopolymers, the significant physicochemical and mineralogical variations between the different types of FA may lead to notable deviations in the microstructure and properties of the final material. FAs can be categorised as class F (low-Ca siliceous) and class C (high-Ca) FA, depending on their Ca content (ASTM standard C168). In geopolymer production, these two types of FA exhibit a much different behaviour upon alkali activation, which also depends on various processing parameters (Temuujin et al., 2013).

In the present study, an overview of recent progress in sustainable geopolymers formed by the alkaline activation of FAs is presented. The opportunities of their usage as building structural elements and as anti-

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corrosive and thermally resistant materials towards circular economy, is also highlighted. Relevant papers published in the scientific literature in the last five years are reviewed. Research works dealing with geopolymers that are cured both in ambient and elevated temperatures, are discussed. Properties of mortars/concrete made from geopolymeric binders are examined with respect to the processing conditions, in view of an ample implementation of geopolymer technology in the production of construction materials.

2. Sustainable geopolymers based on fly ashes

Various sustainable geopolymers produced from different types of fly and bottom ashes (either siliceous or calcareous) and ash-based mixtures, along with their synthesis parameters (initial mixture composition and particle size distribution of source materials, chemical activator, post fabrication curing procedure), as well as their physico-mechanical properties and durability, are summarised in Table 1 and outlined hereunder.

2.1 Low-Ca siliceous ash-based geopolymers

For the development of geopolymers from FA, the raw material mixture should be appropriately tailored. In particular, the Si/Al ratio in low-Ca siliceous FA activated by an alkaline reagent affected significantly the compressive strength in both geopolymer paste and mortar with 20 – 40 wt% sand. The strength achieved (23.7 – 26.4 MPa after 28 d aging) indicate that these materials may be used in construction applications (Lee et al., 2017). In order to evaluate the suitability of FA for high-strength geopolymers, a comprehensive index combining both physical and glass chemistry factors was proposed and validated in geopolymer pastes, using five typical low-Ca FAs, and exhibiting comparable paste workability (Zhang et al., 2016). For the optimisation of alkali activated FA binder composition/properties, Taguchi design methodology was successfully applied in some geopolymer systems. In case of using Na/Al as starting mixture and Si/NaO in the activation solution, a compressive strength of 43.1 MPa was recorded at the optimum conditions and constituent ratio (Panagiotopoulou et al., 2015). The chemical composition of activator solutions with different Na_2O/Na_2SiO_3 ratios also influences the geopolymerisation process. The higher compression strength obtained with (Brazilian) FA was 28 MPa after 24 h and 48 MPa after 28 d of curing. New zeolitic phases were detected after the geopolymerisation (De Azevedo et al., 2017). Geopolymers were produced from mechanically activated lignite FA (Mucsi et al., 2016). The particle diameter of FA also seems to affect geopolymerisation. Specifically, the compressive strength in geopolymers produced from FA with different particle sizes (at both 27 °C and 60 °C) was influenced by the combined effect of particle size, the SiO_2/Al_2O_3 ratio and the glass content. Higher reaction yield was obtained using finer ash fractions, while with coarser fractions unreacted particles were found and lower reaction yield was recorded (Kumar et al., 2015). The significant effect of grinding fineness of class F FA on geopolymer physical properties was also verified in both a geopolymer paste prepared with $NaOH/Na_2SiO_3$ mixture as alkaline activator and geopolymer foam produced using H_2O_2 as foaming agent. By changing the FA grinding time (5 to 120 min), the paste rheological behaviour changed from plastic to Newtonian liquid (Szabo et al., 2017). Durability testing appears to be necessary to check premature geopolymer deterioration. Geopolymers examined for their durability in aggressive environments were found to be more vulnerable to carbonation and the deterioration due to chloride and sulphate ingress was higher than in ordinary concrete, after 6-year exposure (Pasupathy et al., 2017). Class F FA and coarse aggregate recycled from construction and demolition waste were examined for geopolymer concrete production. Results indicated that the mechanical and durability properties were adversely affected by substituting recycled aggregates for natural ones, following similar trends with those recorded when the substitution takes place in cement concrete (Shaikh, 2016).

2.2 High-Ca ash-based geopolymers

Large quantities of high-Ca FA are available due to the use of low-grade lignite-coal feedstock in power stations. The presence of Ca may improve the mechanical properties of FA-based geopolymers, particularly during ambient curing. Excessive amounts of Ca decrease the workability of geopolymer pastes (Embong et al., 2016). Alkali activated fresh geopolymer concrete may also have short setting time (28 -58 min) due to the existence of high Ca content in FA (Topark-Ngarm et al., 2015). High-Ca FA geopolymer mortar was studied at different curing temperatures (25, 35 and 65 °C) for pedestrian pathway applications. Heat enhanced curing during geopolymerisation, resulting in Si-O-Al network formation in the geopolymer, which exhibited better durability upon immersion in 3 % sulfuric acid (Chindaprasirt and Rattanasak, 2017a). Durability studies were also performed in high-Ca FA-based geopolymer concrete upon 2 % sulfuric acid and 5 % magnesium sulphate exposure for up to 45 d. The strength reduction recorded was up to 12 % for the geopolymer compared to 25 % for ordinary cement concrete, upon 5 % magnesium sulphate exposure for 45 d, while a strength decrease up to 20 % was recorded for pure geopolymer and up to 28 % for cement concrete, upon 2 % sulphuric acid exposure (Lavanya and Jegan, 2015). Durability of high-Ca FA-containing geopolymer

concrete was affected by the mineralogy of FA Ca species. FA containing Ca in highly crystalline forms leads to geopolymer concrete with higher durability, whereas the presence of highly amorphous compounds, such as lime, anhydrite and amorphous Ca, negatively influences the freeze-thaw resistance (Temuujin et al., 2014). Durability to sulphate attack was also achieved for geopolymer mortars from high-Ca lignite bottom ashes. The use of finer bottom ash improved both resistance to sulphate attack and strength, due to a higher reactivity of finer ash and lower population of large pores up to 100 μm (Chotetanorm et al., 2013). Use of recycled concrete aggregate in high-Ca FA-based geopolymer concretes has been reported in several studies. High-Ca FA geopolymer concrete was produced having 7 d compressive strength 30.6 - 38.4 MPa that was slightly lower than that obtained using crushed limestone (Nuaklong et al., 2016). In other work, the mechanical behaviour of recycled concrete aggregate-containing high-Ca FA geopolymer produced upon ambient and heat curing was comparable to that with crushed limestone. For both curing procedures, thermal conductivity and ultra pulse velocity of geopolymers with recycled aggregates were lower than those with crushed limestone. Curing at 60 °C yielded a compressive strength 3 times higher than at ambient conditions (Tho-In et al., 2017). Another study indicated that recycled aggregates from crushed structural concrete members and clay bricks can also be used into high-Ca FA geopolymer concrete with acceptable properties (Sata et al., 2013). Recycled packaging foam was also tested as aggregate in the development of lightweight geopolymer blocks having 1,000 – 1,300 kg/m^3 density, with satisfactory strength and low thermal conductivity (Posi et al., 2015). Partial substitution of class C FA with class F into geopolymer mortars is rather important. Geopolymers with a total of 25 % of their weight replaced by class C FA had the highest setting time and strength, but also an increased shrinkage upon drying (Charoenchai and Chindaprasirt, 2014).

2.3 Geopolymers from ash-based mixtures

Optimal formulation of source aluminosilicate precursors is necessary to produce materials with desired properties for specific applications. Systematic methods have been developed integrating statistical experimental design and optimisation techniques for geopolymer synthesis based on industrial by-product mixtures (Sumabat et al., 2015). Geopolymers production by alkaline activation of FA with cathode ray tube (CRT) glass waste was proposed. Even though the higher values of compressive strength were obtained for reference geopolymers based only on CRT glass, the glass substitution with 25 % of FA improved the geopolymer durability (Badanoiu et al., 2015). The partial substitution of waste glass powder with 10 % FA improved the stability in humid environment of borosilicate inorganic polymers from glass powder. The FA presence, though, did not substantially modify the geopolymer mechanical behaviour at high temperatures (Al-Saadi et al., 2017). Red mud (Bayer's process residue) has also been used synergistically with coal FA into geopolymers. This combination can lead to production of geopolymers with a compressive strength higher than 40 MPa. Geopolymerisation upon ambient curing is feasible to facilitate the potential large-scale utilisation (Jamieson et al., 2016). Geopolymers from red mud and class F FA (ambient conditions) had 11.3 - 21.3 MPa 28 d compressive strength, and an optimum composition of raw materials and activator solution was suggested (Zhang et al., 2014). Paving blocks using 10 % and 20 % red mud in FA geopolymers can meet the leaching standards, and the development of mechanical properties is attributed to a compact microstructure (Kumar and Kumar, 2013). Waste from copper floatation was investigated with FA and coal power station pond ashes (Mongolia) for the production of geopolymer-type mortar and "light-weight" concrete. The ash could be alkali activated leading to binders of suitable compressive strength, whereas the waste could be used as a full substitute of construction sand. A type of light-weight concrete was prepared using an in-situ hydrogen release reaction with the addition of Al (Temuujin et al., 2016). FA can also be used to delay setting of both pure metakaolin and slag, in blended alkali-activated materials. Twenty-six different mixtures of these precursors were studied in a ternary representation, to compare the performance of pure (one precursor) and blended geopolymers (two precursors) (Samson et al., 2017). Another hybridisation approach using class F FA and high-Ca wood ash was proposed: a pressurised forming technique was applied for the fabrication of a low alkalinity geopolymer mortar, with a hardened binder phase consisting of potassium polysialate and gehlenite, and adequate mechanical and durability performance as load bearing block for constructions (Cheah et al., 2015). The hybridisation of FA, high-Ca wood ash and ground granulated blast furnace slag yielded geopolymer mortars with adequate mechanical performance for industrial applications (Cheah et al., 2017).

2.4 Ash-based geopolymer matrix composites

Lately, fibre-reinforced geopolymer matrix composites are being developed. Their brittle behaviour represents a limiting factor to use in structural applications. Reduction in brittleness and toughness increase can be achieved through careful tailoring of the fibre/matrix interface, to create crack dissipating mechanisms for preventing a premature composite failure. In order to avoid possible undesirable losses in modulus as a result of a poor geopolymeric matrix, the effect of initial cure time appears to be significant for toughness

preservation (Jackson and Radford, 2017). Particularly, the incorporation of polypropylene fibres into high-Ca FA geopolymers (cured outdoor for energy savings) resulted in a strong fibre-reinforced matrix, leading to improved tensile strength, crack control and resistance to acid solutions (Chindaprasirt and Rattanasak, 2017b). Basalt fibres were also suggested as potential candidate reinforcement for geopolymer composites. Their addition up to 10 % into FA-based geopolymers, increased the compressive strength by 37 %. This result can be associated with the specific microstructure obtained, composed of FA particles and basalt fibres embedded in a dense alumino-silicate matrix. Higher compressive strength was determined in samples with high Ca/Si ratio (Timakul et al., 2016). The addition of porous biomass (Novais et al., 2016) and rubber particles (Park et al., 2016) in FA-based geopolymers also enhanced the composite properties.

Table 1: Sustainable ash-based geopolymers

Composition	Synthesis Parameters	Physicochemical Properties	References
Class F	Si/Al ratio, alkali activator	Compressive strength	Lee et al., 2017
(5) Australian FAs	Activator/ash ratio, activ.conc.	Mechanical properties	Zhang et al., 2016
Class F, normal pozzolan	R/Al, Na/(Na+K), [Si]/R_2O	Highest strength	Panagiotopoulou et al., 2015
Class F	Activator solution, curing t	Water absor. & permeability	De Azevedo et al., 2017
Activated lignite FA	Alkaline activator solution	Compressive strength	Mucsi et al., 2016
FA: (4) size fractions	T, SiO_2/Al_2O_3, p.s., glass cont	Compressive strength	Kumar et al., 2015
Class F	P.s., viscosity, pore structure	Density, compr. strength	Szabó et al., 2017
Class F	NaOH/Na_2SiO_3	Durability, porosity	Pasupathy et al., 2017
Class F	Limestone/oil palm shell ratio	Compressive strength	Embong et al., 2016
High-Ca FA	Alkali activators	Setting time, compr.strength	Topark-Ngarm et al., 2015
Lignite FA	Curing time	Compressive strength	Chindaprasirt et al., 2017a
Class C	Activator solution, curing time	Surface prop., density	Lavanya and Jegan, 2015
Class F & Class C FAs	NaOH conc., NaOH/Na_2SiO_3, Si/Al, Na/Al	Compressive strength, durability	Temuujin et al. 2014
Ground lignite BAs	NaOH, sodium silicate & curing T	Resistance sulphate attack, compr. strength, sorptivity	Chotetanorm et al., 2013
High-Ca FA	Alkali activator concentration	Compressive, tensile & flex. strength, abrasion resist.	Nuaklong et al., 2016
Coarse aggreg.: crushed limestone & rec.concrete	Temperature curing	Compr. strength, thermal conductivity & ultra pulse velocity	Tho-In et al., 2017
High-Ca FA (class C), (2) types of recycl.aggr.	Aggregate FA/RA ratio (wt.)	Compressive tensile strength, water permeability	Sata et al., 2013
Recycled packaging foam	Na_2SiO_3/NaOH & alkaline/ash ratios, curing T, foam cont.	Compressive strength	Posi et al., 2015
Class F and class C	Na_2SiO_3 and NaOH	Drying shrinkage, compressive strength	Charoenchai and Chindaprasirt, 2014
Industrial byproduct mix	Alkali activator	Mech., therm, & sust. prop.	Sumabat et al., 2015
FA-based mixture	Alkali activator, curing cond.	Mechanical properties	Al-Saadi et al., 2017
Bayer liquor and FA	P.s., surface area, curing t	Compressive strength	Jamieson et al., 2016
Red mud and class F FA	Na/Al, Si/Al molar ratio, cure t	Mechanical properties	Zhang et al., 2014
Red mud & class F FA	Red mud/FA, alkali activator	Physical properties	Kumar and Kumar, 2013
FA &Erdenet float.waste	Alkali activator concentration,	Compressive strength	Temuujin et al., 2016
Metakaolin, FA & slag	Alkali activation rate	Performances	Samson et al., 2017
High-Ca wood ash & class-F pulverised ash	Ashes & water/binder ratios	Compressive, flexural strength & secant modulus	Cheah et al., 2015
Blast furnace slag, high-Ca wood ash & FA	Alkaline activator ratio	Compressive and flexural strength, velocity	Cheah et al., 2017
Nextel 610/MEYEB	Curing time	Modulus and strength	Jackson and Radford, 2017
PP fibre/FA geopolymer, lignite coal high-Ca FA	NaOH & water glass, curing t	Tensile, crack control & acid resistance	Chindaprasirt and Rattanasak, 2017b
Class C FA Basalt fibres	FA/ Basalt Fibres, Ca/ Si ratio	Compressive strength	Timakul et al., 2016

3. Concluding remarks

Various aspects of ash-based geopolymers were presented. Geopolymer binders are mostly eco-friendly products, contributing to the sustainable development. Major factors influencing the microstructure, physico-mechanical properties and durability of geopolymer concretes are the initial mixture composition and particle size distribution of source materials, chemical activator, post fabrication curing procedure and durability in aggressive environment. Particularly, the ash-based geopolymer properties also depend on the type of the FA and/or the reinforcement used. The material and production parameters should be further optimised in order to achieve significant understanding on this novel geopolymer system.

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