

Review article

Fly ash-based geopolymers containing added silicate waste. A review



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ABSTRACT

This review summarizes different types of industrial wastes such as biomass ash, red mud, recycled glass and heavy metals waste, in their application for geopolymer production. These wastes, which are currently abundant and urgent to dispose of, cannot be used alone in the geopolymer process because they do not provide a suitable $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio for this technology. For this reason, these by-products are commonly used in addition to other aluminosilicate sources such as fly ash or metakaolin. Important parameters which affect the properties and performance of fly ash based geopolymers with addition of a variety of wastes are discussed based on a comprehensive literature review.

1. Introduction

Concrete is the most produced and used construction material in the world. It is cheap and strong but it has environmental drawbacks. The cement production industry notably contributes to CO_2 emissions [1]. Cement manufacture requires high temperatures, around 1400 °C, with consequent high energy dispersion and emissions. The amount of carbon dioxide released during the limestone calcination and fossil fuel combustion is about one ton for each of cement production [2,3]. Therefore there is a need to find an ecofriendly substitute to concrete in order to reduce greenhouse gas emissions [4].

Geopolymers are a relatively new class of construction materials [4]. Geopolymers do not require calcium-silicate-hydrate gel but utilize the polycondensation of silica and alumina precursors to achieve a superior strength level [5]. Geopolymer materials are usually synthesized using an aluminosilicate raw material and an activating solution which is mainly composed of alkalis of sodium or potassium and waterglass [6].

The innovation of geopolymers relates to the possibility to harden at room temperature without high treatment temperature and consequently reduced CO_2 emissions, representing an eco-friendly innovative alternative to cement [7]. Thanks to these attractive properties this technology is receiving increasing attention in different application fields like refractory filters, lightweight panels for thermal and acoustic isolation, low cost ceramics and fire protection structures.

Portland cement furthermore normally requests a high amount of virgin resources such as limestone, clay and water in the manufacturing process [8]. On the contrary, low cost materials can be used in the geopolymerization process, including waste materials that are not currently reused in other industrial sectors but are abundant and urgent to dispose of.

Through geopolymerization it is possible to use large amounts of hazardous and non-hazardous wastes and to fabricate new products as well as to minimize environmental impact [9]. Indeed geopolymer technology can transform industrial solid waste containing aluminosilicates into useful products thanks to the flexibility and capability to immobilize and stabilize the wastes inside the geopolymer network [10]. In principle, any waste material containing proper amount of silica and alumina can serve as precursor for geopolymerization [11].

Fly ash (FA) is the most used and suitable waste material in geopolymerization due to the huge amount produced worldwide, estimated to be around 780 million tons annually and its great workability [2,12]. Fly ash is a by-product derived from the combustion of coal powders and collected by mechanical and electrostatic separators from the fuel gases of the power plants [13].

Fly ash is composed mainly of amorphous silica and alumina [14] with a favorable shape and size that improve the workability and make this material suitable for geopolymer production [15]. In the last years different studies have evaluated the possibility of using different types of wastes mixed with fly ash as raw materials. The choice of the materials to produce geopolymers depends on factors such as availability, disposal urgency, difficulty for recycling and final applications [16].

This review paper focuses on geopolymer systems that incorporate fly ash in combination with other wastes to complete the geopolymer formulation (Fig. 1).

2. Biomass ash

In the world vegetable oil market, palm oil is one of the most important sources [17]. The biomass produced in this industry is

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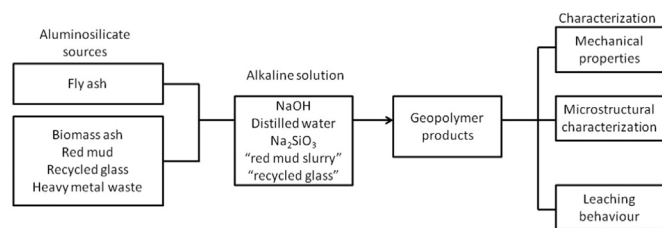


Fig. 1. Schematic diagram of geopolymer production, using inorganic waste, in addition to fly ash, and characterization methods, discussed in this review.

creating environmental problems [18].

The oil is extracted from fresh palm fruit, palm fronds, palm pressed fibers, palm kernel shells and empty fruit. After extraction the wastes are burnt at 800–1000 °C as boiler fuel to produce steam, which is then used in turbines generating electrical energy [19]. The result of the burning is palm oil fuel ash (POFA) and the amount of this waste is increasing annually. In fact it has been reported that only in Malaysia POFA is produced at a rate of 4 million ton/year [20]. Since palm oil is one of the most used raw materials in bio-diesel production, it is expected that the production of POFA will increase every year. This underlines the necessity to find a way to dispose this waste, which is so far mainly landfilled [21].

Another biomass ash is rice husk ash (RHA), a residue generated by biomass electric power plants using rice husk with a world production of about 20 million tons annually [22,23].

Considering the growing quantities of biomass ashes produced and the associated environmental issues, there are increasing research efforts to use this waste to develop sustainable and ecofriendly construction materials. Thanks to the high quantity of SiO₂, ~ 67% in palm oil ash and ~ 90% in rice husk in amorphous form, they can be suitable activated materials to develop geopolymers [21].

In addition to the use of ashes, research is also focused on utilizing palm shells as aggregate and rice husk to obtain water glass [24–27].

To obtain a geopolymer product with attractive mechanical properties, it is necessary to grind, sieve and heat the palm oil fuel ash to increase the reactivity of the material when it is mixed with the alkaline solution. For this reason, different combinations of sieved material and temperatures have been used to obtain suitable raw materials for geopolymerization. The most common pretreatment approaches investigated so far are summarized in Table 1.

The rice husk is usually burnt at 600–700 °C for 6 h to increase the amount of silicon oxide by reduction of carbonaceous materials [32,33]. Sieving the ash to a particle size of ≈ 300 μm is necessary to separate the incompletely combusted fibers and shells.

From a structural point of view POFA is composed mostly of spherical particles with a small amount of plerospheres and irregularly shaped particles while rice husk ash is composed mainly of irregular particles [34,35]. It is known that the shape of the particles is important to determine the amount of alkaline solution necessary to obtain a workable mixture for geopolymerization.

The proportion fly ash:biomass ash to obtain a geopolymer with relatively high mechanical strength (20–50 MPa) was found to be around 65%:35% [36–39].

To harden the raw source composed of POFA or RHA and FA a solution of NaOH and Na₂SiO₃ is usually applied. The best proportion for the molar ratio between water glass and sodium hydroxide was

found to be 2.5, indeed increasing this molar ratio led to the increase of mechanical properties. This correspondence is valid just up to the mentioned value (2.5). At higher values the mechanical properties decrease. This fact is attributed to the higher presence of alkaline solution that hinders the geopolymerization reaction [40].

The weight ratio between alkali solution and aluminosilicate powder is usually between 0.2 and 0.5 [19,36]. This value depends on the ratio between the content of fly ash and palm oil fuel ash, in fact the high specific surface of POFA and RHA requires a higher amount of liquid compared with the quantity necessary in a system composed only of fly ash [13,19,41]. The molarity of the NaOH solution is fundamental to achieve the right geopolymerization reaction. Different researches have used different molarity values, between 8 and 16. Increase of the alkalinity of the solution usually improves compressive strength. However over 10 M the increase of the mechanical property is not significant. For economic reasons it is not convenient to use a molarity over 10 for the NaOH solution [36,39]. Geopolymers based on ashes could be cured at room temperature however there is agreement that the best treatment to obtain superior properties is to cure the specimens for 24 h at 60 °C [42,43].

Ranjbar et al. [38] tested the behavior of geopolymers based on POFA plus FA at high temperature to assess the suitability of these materials for high temperature applications. The samples with higher amount of POFA showed higher deformation at elevated temperatures. As mentioned before, palm oil ash requires a high amount of water to achieve a good workability, which leads to relatively high shrinkage when subjected to high temperature. High quantity of water is undesirable in high temperature applications because it increases the hydration shrinkage of samples when heated [38]. For this motivation POFA is not considered a suitable raw material for geopolymers that require use at high temperatures.

On the contrary, the use of POFA shows good results to realize lightweight construction materials with high flexural strength compared to OPA concrete of similar density. Furthermore this geopolymer exhibits a 39% reduction of thermal conductivity compared to traditional brick [19,44].

The superior thermal resistivity, compared to conventional materials, associated with good mechanical strength, around 30 MPa after 28 days, highlights the possibility to use this waste material as an aluminosilicate source for geopolymer production.

3. Red mud

Red mud, also known as bauxite residue, is a by-product generated during the Bayer's process in alumina production. It is estimated that for each ton of alumina, 1.5–1.6 t of red mud waste are produced [45,46]. Furthermore it is estimated that 2.7 billion tons of red mud were generated in 120 years of alumina production, with an increase to 120 million tons per year currently [47,48]. This huge quantity of waste is classified as a toxic waste for the high basicity and leaching potential, for these reasons red mud is difficult to dispose of [47]. Red mud is usually disposed into on-site waste lakes for further dewatering, caused by the high water content and alkalinity with an average pH value of 11.3, consolidation and storage [49]. Serious environmental pollution can happen if the mud is leaked into the surrounding environment [45,50]. Red mud is composed mainly of oxides and hydroxides of Fe, Al and Si with minor quantities of CaO and TiO₂. Those oxides could be incorporated in geopolymer networks to minimize the leaching [45]. Red mud is a potential candidate in geopolymer technology due to the high quantity of Al₂O₃, SiO₂ and caustic soda it contains.

The SiO₂/Al₂O₃ molar ratio and the amorphous phase inside red mud are too low to achieve a geopolymer using just this waste as the only aluminosilicate source. To resolve this drawback, red mud is used in combination with fly ash, considering that most plants generating aluminum usually generate also fly ash from their power plant [45].

To improve the solubility of aluminosilicate materials in red mud

Table 1
Commonly used POFA treatments before geopolymerization.

	Method 1 [28,29]	Method 2 [29–31]:
Step 1	Sieved using 300 μm	Heat treatment at 105 °C for 24 h
Step 2	Ground to particle size of 45 μm	Sieved using 300 μm
Step 3	Heat treatment at 500 °C for 1 h	Sieved using 45 μm

and to obtain the highest compressive strength in the final geopolymer, Kaya et al. [51] suggested to calcinate it before use. When red mud is calcinated at 800 °C the dissolution of alumina and silica in alkaline solution reaches a maximum because different phases such as muscovite, hibschite and cancrisilite transform into phases that are easier to dissolve [51]. The dissolution efficiency was seen to increase from 200 °C to 800 °C and reached a maximum at approximately 800 °C, suggesting that the optimum calcination treatment to obtain the highest dissolution rate is 800 °C for 3 h [47].

When red mud is used substituting fly ash an increment in the mechanical properties of the geopolymer until a maximum of 15% by mass for the dry mass of fly ash is found. Exceeding this amount of red mud, the mechanical properties decrease due the reduction of the amorphous content in the geopolymer [52]. XRD patterns of red mud containing geopolymers showed that with systematic addition of red mud the amorphous content reduces, while at the same time the iron phases increase in intensity. The geopolymerization degree has been shown to decrease with the addition of red mud and the activation of fly ash was inhibited [51]. In addition XRD and FTIR analyses indicated a decreased of raw material dissolution with increasing red mud content [51].

Increasing the solution/solid ratio brings a positive effect on the mechanical properties of red mud containing geopolymers [53]. According to Duxson et al. [54] this correlates with the increase of the reactive silica, which implies a higher amount of water and sodium in the final product. However this is not beneficial from an environmental and economical point of view. For this reason a low solution/solid ratio of around 0.5 is used [55].

The mechanical properties of red mud based geopolymers, usually cured at room temperature, depend heavily on curing time. To complete the curing samples need at list 21 days, contrary to the fly ash based geopolymers that require just few days [56].

In alumina production not only solid waste composed of red mud is generated, but also a separate sodium aluminate slurry is produced as residue. The use of Bayer liquor instead of sodium silicate, normally used in geopolymerization, has been studied to produce geopolymer cements and geopolymer foams [56–58]. From the results emerges that using bauxite liquid waste instead of sodium hydroxide implies a negligible lost in mechanical properties, at the same time the contaminants inside the liquor do not have an impact on the geopolymer process [58].

Badanoiu et al. developed a foamed geopolymer using red mud filtrate and recycled glass [57]. Geopolymers with 25% of red mud and activated with red mud filtrate which were thermally treated at 700 °C exhibited a contraction phenomenon with a corresponding decrease of open porosity and an important increase of compressive strength (25 MPa). An increase in the temperature above 800 °C was shown to lead to paste expansion with an increase in the open porosity and a related decrease in compressive strength to 8.6 MPa, which is still relatively high for foams [57].

In geopolymers incorporating fly ash, the structure is likely composed of a Fe-silicate chain where Fe atoms are in tetrahedral coordination [59]. Zhang et al., [60] tested the leaching behavior of geopolymers with red mud and fly ash. The samples were immersed in sulfuric acid solution and deionizer water. After 120 days the properties decreased significantly, namely reduction of 30% in compressive strength, 45% for flexural strength and 70% for the Young's modulus. The concentration of heavy metal ions was found to be lower than the respective contaminant limit set by US EPA standard, which confirmed the efficient encapsulation of heavy metals coming from red mud in geopolymers [48].

4. Recycled glass

In the last years investigators have focused on using different types of waste glass in geopolymer production, thanks to the amorphous

nature and pozzolanic properties of this residue [61]. Every year millions tons of glass are generated in the municipal waste and due to impurities and mixed colors only a proportion of this glass waste can be recycled. Thanks to the high contents of alkali and silicate, using recycled glass as raw material for making geopolymers is a convenient alternative, and the required amounts of waterglass and sodium hydroxide can be limited or eliminated completely. This result is favorable from economic and environmental points of view since the alkaline solution is the most expensive component and the principal source of greenhouse gas emission [62].

Different types of glasses can be used in this field including municipal recycled glass, industrial recycled glass, glass derived from lighting equipment, borosilicate glass from pharmaceutical package, fluorescent lamps, solar panel waste glasses, glass produced by DC plasma treatment of waste and even thin-film transistor liquid display panels (TFT-LCD) [63–67]. Also glasses derived from the vitrification of municipal residues, e.g. air pollution control (APC) residues, have been studied for production of geopolymers providing an alternative to the reuse of such residues [68,69].

To avoid chemical and physical interaction the glass is usually separated from contaminants such as metal, plastic and paper and subsequently granulated to increase the reactivity. Different parameters have been analyzed to improve the connectivity between the glass and the other raw materials inside the geopolymer network.

According to Chen und Chiu [70] a continuous increase of the alkalinity of the solution does not increase the mechanical properties. However a too high concentration of alkaline ions in the framework could weaken the structure. When NaOH is in excess the precipitation is too fast and the polycondensation is hindered, which leads to weakening of the end product. On the other hand, a too low amount of NaOH is insufficient to attack the raw materials and to start the geopolymerization process [71].

Following this assumption, a NaOH molarity between 6 and 10 is used and the alkaline solution to solid ratio is modulated between 0.4 and 0.6 to optimize the paste flowability. Decreasing the waterglass to sodium hydroxide ratio, the amount of liquid required for geopolymerisation decreases. A combination of 70% waterglass and 30% sodium hydroxide was considered by Arulrajah et al. [71], who found the most efficient approach to provide an acceptable value of compression strength for masonry use [72]. In this case the weight ratio fly ash to recycled glass was fixed at 30:70. Usually the degree of polymerization of the activated mixtures decreases as the amount of glass inside the structure increases [73]. The decrease of mechanical properties with the increase of glass content is associated with the defect density that results from the unreacted fraction of the raw materials [73].

Redden et al. [61] showed that a mechanical strength of 30 MPa can be achieved using only recycled glass as source material. However the problem with this geopolymer was indicated to be the lack of hydrolytic stability of the sodium silicate gel and, as a consequence, a drastic strength loss under moist conditions. To stabilize the structure it is necessary to introduce in the geopolymer network tetrahedral aluminum: the addition even in small amount results in the formation of a polymeric phase [74]. For this purpose, the glass is usually mixed with fly ash to compensate the usually low content of Al₂O₃ in waste glass chemical compositions. Taveri et al. [67] studied recently the effect of boron coming from B-silicate glass in geopolymerisation. FTIR and NMR analyses confirmed that B takes part in the polymeric chain substituting aluminum in the tetrahedron position [67].

Different studies proved that it is also possible to use waste glass to produce an alkaline solution as alternative to water glass [75–77]. Water glass, whose synthesis entails high energy and environmental costs, promotes the formation of stronger and more durable geopolymers. Glass waste with a chemical composition consisting primarily of SiO₂ (65–75%) and Na₂O (12–15%) is a potential water glass substitute [78].

The quality of the solution depends on the type of glass, time,

temperature, size of glass particles and pH of the solution [79]. For example powder glass stirred in NaOH/Na₂CO₃ solution at 80 °C for 6 h and subsequent filtering was reported to give the best solution in terms of mechanical properties [76].

The amount of constituent, in this case silicon, released by a glass is proportional to the surface area. Particle sizes lower than 45 μm show that silicon in the waste glass induces the same effect as the silicon in waterglass [76,79]. In fact, fine granulometry gives higher pozzolanic reactivity, in addition an increase in curing temperature accelerates the activation of both recycled glass and fly ash [80].

The best procedure involves mixing the glass in NaOH/Na₂CO₃ solution at 80 °C for 6 h and subsequent filtering to obtain an alkaline solution ready to be mixed with raw materials. However more studies must be conducted with this waste material in order to gain understanding about the operational parameters that valorize the potential of different types of waste glasses in geopolymer production.

5. Heavy metal containing waste

Heavy metals are components of many industrial residues as mine tailings, electric furnace slag, electroplating sludge and municipal solid waste incineration fly ash [81]. This type of wastes are rich in heavy metals and should not be let to infiltrate surface or ground water, because they have long life in soil and can lead to environmental problems [82].

Fly ash based geopolymers provide a satisfactory method for heavy metal immobilization with low permeability, good resistance to acid and chloride attack and long durability [83]. Geopolymers are reported to stabilize hazardous and radioactive wastes better than Portland cement [84].

Metals such as Co, Cu, Pb, Cd, Ni, Zn, Pd, As, Ra and U can be incorporated in the 3 dimensional geopolymer network decreasing the mobility of the heavy ions through metal hydroxide precipitation, ion substitution or physical encapsulation [85–87].

The amount of ions that a matrix can incorporate without losing structural integrity is limited and determined by the chemical and physical properties of each element [88].

In most cases the metals are not incorporated in the crystalline matrix but in the amorphous one [89]. Metal ions influence the geopolymer structure differently, mainly because of differences in the atomic ratio, electronegativity and reactivity [90]. Heavy metals can also react with Al- and Si- species dissolved from Al source forming new phases [91].

Different geopolymer reagents as sodium or potassium hydroxide and sodium or potassium silicate in the right quantity are used during geopolymerization. The ion dissolution is strongly affected by the pH of the alkali solution used during geopolymer synthesis. An increase in alkali solution pH improves the ion dissolution and consequently its immobilization inside the matrix [92].

Research results indicate that NaOH comparing to Na-silicate has more effective activation properties regarding ion immobilization. This is correlated with the higher alkalinity of NaOH which favors minerals dissolution and consequently creation of new phases with ions inside the structure [91].

Also the Si/Al molar ratio is important for the ion immobilization, for example, it was shown that leaching of Cr and Cu decreased as the Si/Al ratio increased, while Zn reached its lowest point at an intermediate Si/Al ratio [93,94]. In addition, a Si/Al ratio near 2 was found to be more suitable for binding Pb in the geopolymer gel [95].

A powerful tool used to determine the efficiency on how different ions with various concentrations are immobilized in the geopolymer matrix is the leaching test [96].

Geopolymer samples are crushed and left for a certain amount of time in contact with a solution, usually water, acid solution or Q-brine [97]. After filtration by inductively coupled plasma, it is possible to determine the ions concentration in the solutions by atomic absorption

spectrometry or inductively coupled plasma mass spectrometry.

Leach resistance strongly depends on the type of heavy metals, the matrix composition and the leach solution used for the test [98,99]. Depending on the final structure and stability, the leaching can be controlled by pore diffusion or boundary layer diffusion [90]. The immobilization efficiency is strongly related to the binder microstructure as pore size distribution, pore shape and total porosity [99].

Ogundiran et al. [100] examined the Pb immobilization of lead smelting slag. Pb in fly ash based geopolymers could be immobilized as network modifier, charge supplier and insoluble precipitate encapsulated in the structure. Formation of Pb₃SiO₅ allowed a reduction in Pb leaching. An important mode of immobilization is also the replacing of Na or K by Pb in the structure [101]. Different studies have shown that Pb achieves excellent immobilization efficiency in many condition, being better than that of other ions as Cu and Cd [88,91,96,102].

The compressive strength of geopolymeric materials changes with addition of heavy metal ions. For example, Zn²⁺ addition in fly ash based geopolymers resulted in a decreased in mechanical properties while Cr⁶⁺ and Pb²⁺ increased the mechanical properties [99].

The oxidation number is also an important parameter, Zheng et al. [94] demonstrated that Cr added as Cr(VI) is highly leachable, but reduction of the Cr to Cr (III) improves its immobilization efficiency.

From the results obtained so far it is apparent that heavy metals can be effectively immobilized in geopolymer matrices due to the participation of heavy metal cations in the balance of negative charge of Al in the frameworks [83], indicating an important application of geopolymers in the management of hazardous heavy metal containing wastes.

6. Final considerations and conclusions

The present review has summarized key studies related to the synthesis of geopolymers incorporating wastes, focusing on such problematic wastes that continue to grow and are abundant and urgent to dispose of. The combination of different wastes with fly ash is described in terms of parameters and properties, according to the available literature.

The synthesis of geopolymers using biomass ashes, red mud, recycled glass and heavy metals containing wastes is a potential way to dispose and reuse such wastes which are otherwise hardly recycled. Several innovations in geopolymer production are possible when using such wastes. For example, the use of red mud can have an additional benefit; thanks to the high alkalinity of this residue less alkali activator is necessary, thus reducing the most expensive material used during geopolymer synthesis. At the same time recycled glass with high amount of amorphous silica can be treated to obtain an ecofriendly water glass useful for the geopolymerization process.

Table 2 summarizes the maximum amount of POFA, red mud and waste glass that is possible to substitute for fly ash in geopolymers without affecting the mechanical properties.

From the table it is clear that fly ash can be replaced by POFA and recycled glass in large amount without affecting mechanical properties. On the other hand, the incorporation of red mud in fly ash based geopolymers requires more studies to optimize the chemical composition and relative mechanical properties.

Analysing the available literature, it becomes apparent that using the mentioned wastes, although in lower amount compared to fly ash, there is a significant consumption of such residues with the consequent

Table 2
Maximum amount of POFA, red mud, recycled glass used in fly ash based geopolymers and the relative compressive strength.

	POFA	Red mud	Recycled glass
Raw material:FA	60:40	5:95	70:30
Compressive strength [MPa]	56	28	44

preservation of the environment. In addition, incorporating the right quantity of each waste can improve the based fly ash geopolymer performance, making the geopolymer suitable for different applications. In relation to heavy metal containing waste from the different studies carried out with ions or sludge it is possible to confirm that 0.5 wt% of Cu and Pb can be incorporated inside the geopolymeric matrix. In this case the matrix is not only composed of fly ash but often also metakaolin or pure alumina are incorporated to reinforce the ion immobilization.

Geopolymerization is thus proposed as a realistic technology for the management of large volumes of potentially hazardous or abundant wastes. Expansion of geopolymer based waste management should contribute to considerable saving in disposal cost with a substantial reduction of greenhouse gas emissions. Indeed, extensive research is being carried out to decrease the amount of chemical reagents used during geopolymerization, to study the feasibility of using other types of wastes and to explore different fields of application of this technology.

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