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Natural fibers as reinforcement additives for geopolymers – A review of potential eco-friendly applications to the construction industry

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ABSTRACT

The construction industry is responsible not only for the consumption of huge amounts of natural resources but also for the emission of large quantities of CO_2. Geopolymers have emerged as an environmentally friendly alternative for conventional construction materials since they can be produced from industrial wastes. Similarly to ordinary concrete, geopolymers can also improve their mechanical properties when reinforced with fibers. This paper presents a review of recent advances in the production of natural fiber-reinforced geopolymers produced from industrial by-products and waste materials as promising sustainable construction materials. Regarding the use of industrial wastes, this paper reports the use of fly ash, ground granulated blast furnace slag, construction and demolition wastes and mine tailings for the production of high strength geopolymers. At the same time, a survey of successful reinforcement with natural fibers (from plants such as pineapple leaf, sisal, linen, flax, sweet sorghum, and cotton) is also reported. In this respect, it has been found that the type of fiber, dimensions, amount and pretreatment of fibers affect the final properties of the resulting composites. Moreover, layer reinforcement using woven and non-woven layers of natural fibers seem to be more effective than short fibers randomly oriented.

Contents

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1. Introduction

The search for alternative building materials has gained great attention due to the unsustainability of the modern construction industry. This industry accounts for 30% of global carbon dioxide (CO_2) emissions and consumes more raw materials than any other industry, nearly 50% (wt%) [66]. Ordinary Portland cement (OPC) is widely used for the conventional mortar and concrete productions due to their versatility and highly reliable performance, widespread availability, comparatively low cost of raw materials and processing technologies [55,116]. OPC production not only requires enormous amounts of energy but also releases huge quantities of greenhouse gases mainly because of the calcination of limestone and fuel combustion in addition to mining, grinding and transportation [34]. Due to the high CO_2 emissions and energy consumption, alternative construction materials are been investigated.

In this regard, geopolymers, which can be made from locally available minerals or recycled or waste materials that are generated from industries, agriculture and domestic sources, are perfect candidates that have attracted the construction industry interest [26,41] and the substitution of OPC with geopolymers could result in an 89% decrease in CO_2 emissions for each ton of OPC [27]. The raw materials for geopolymer production are rich in silico-aluminates, which is an advantage since >65% of the Earth's crust consists of Al—Si minerals (NPCS Board of Consultants & Engineers [60]). Davidovits presented the attractive characteristics of geopolymers [27] and the silicon and aluminum-containing minerals can react chemically in alkaline conditions and form polymeric chains and cross-linked networks consisting of Si—O—Al—O bonds [28].

According to Silva et al. [89], the geopolymerisation process can be divided into three main phases: (i) dissolution of the oxidized minerals present in the raw material (usually silica and alumina) under highly alkaline conditions; (ii) transportation/orientation of the dissolved oxidized minerals followed by coagulation and gel formation; and (iii) polycondensation to form a three dimensional network of aluminosilicate structures. Theoretically, any pozzolanic compound or material with a high content of alumina and silica is suitable for geopolymer synthesis in strongly alkaline conditions. However, many factors might be considered for the geopolymerisation reaction. In this respect, one of the most important factors is the determination of the physical characteristics and chemical composition of the raw material, as it determines the alkaline degree of the activator. Since raw materials are diverse and could be different from batch to batch (mineral or waste materials, for example), it is important to fully characterize the samples and, according to this, optimize the composition and amount of the activating solution and curing conditions [18,86,103,106].

The industrial by-products such as fly ash (FA), ground granulated blast furnace slag (GGBFS), rice husk ash (RHA), palm oil fuel ash (POFA), among others [20–22,37,83] have been researched for potential raw materials for geopolymer preparation. In this way, a value is given to otherwise waste and potentially problematic materials. Furthermore, several studies have been reported on the production of geopolymers based on waste materials as copper and tungsten mine tailings (MT), concrete demolition waste and fired clay brick powder [1,2,63,101,113].

Geopolymers presented superior mechanical properties and better resistance to fire, sulfates, and acids compared to OPC-based materials [92]. However, as OPC products, geopolymers show brittle failure due to their low tensile strength that could impose several constraints and limitations in possible structural functions. Traditionally, OPC concretes are reinforced through the addition of steel bars producing a composite material with ductile behavior. However, steel reinforcement has several disadvantages: steel bar corrosion is the main reason of structure deterioration, is highly expensive, and its production is responsible for the 31% of the CO_2 emission of reinforced concrete [35].

Although several review articles have been published on the research area of geopolymers composites, they are focused on presenting research works regarding cellulosic fiber [110], synthetic fiber [88], and fabric-reinforced geopolymers composites [82] for general applications. The objective of this review paper is to compile up-to-date research information about available geopolymer sources and natural fibers in the context of the production of reinforced eco-friendly building materials. For this, recently published research in SCi and Scopus databases in the areas of materials science, construction materials, and composites were reviewed and systematically summarized in two sections. The first part (Section 2) presents a comparative study about the production conditions and reports the mechanical properties of matrices that used different silico-aluminate resources for their fabrication while the second part (Section 3) reports the effectivity in terms of improvement of mechanical properties of different alternatives of natural fibers incorporated as reinforcement of geopolymer composites.

2. Industrial by-products and waste materials for geopolymer production

2.1. Fly ash

Fly ash is a finely-grained inorganic powder produced during the combustion of pulverized coal in thermal power plants for electricity production [31,74]. As global electricity production shows an important dependency on coal sources, enormous quantities of fly ash are generated around the world, e.g. the US alone produced 51 million tons of fly ash in 2014 [11]. There are some applications for fly ash, however, the percentage of fly ash consumed is very low, so most parts of these ashes are dumped in landfills and storage lagoons resulting in potential land, environmental and human health issues [30]. In general, the shape of fly ash particles is spherical with a particle size ranging from <1 μm up to 150 μm [97]. ASTM standards regarding fly ash [12] classifies this material based on its chemical composition resulting in two types of fly ashes: Class F and Class C. The chemical composition of fly ash varies depending on the coal source and the combustion process, but is mainly composed by SiO_2 and Al_2O_3 [30]. Class F fly ash results typically from burning anthracite or bituminous coals, while Class C fly ash is produced when lignite and sub-bituminous coals are combusted [12]. The main difference between both types of fly ashes is that Class C fly ash has greater amounts of total calcium compounds than Class F fly ash [12]. In regards to geopolymer production, fly ash is an excellent raw material since it contains high amounts of silica and alumina. According to Fernández-Jiménez and Palomo [32], there are some characteristics of fly ash that produce a geopolymer with good binding properties: low CaO content i.e. fly ash Class F, <5% of unburned material; <10% Fe_2O_3 content; between 40 and 50% of reactive silica; 80–90% of particles finer than 45 μm, as well as, having a high content of vitreous phases. However, it has been reported that geopolymers with high compressive strengths can also be produced with fly ash with a high content of CaO.

Table 1 summarizes the parameters studied to produce fly ash-based geopolymers and their compressive strengths that have been reported in the literature. A fly ash-based geopolymer paste with a compressive strength of 59 MPa was developed by Kong et al. [45] by mixing class F fly ash with an alkaline solution made by sodium silicate and potassium hydroxide with a silica modulus of 1.2 ($Ms = SiO_2/M_2O$). The curing regime consisted of leaving sealed samples for 24 h at room temperature and then heating them at 80 °C for another 24 h after which the samples were removed from their molds. Plastic films to seal geopolymer pastes to avoid moisture loss during curing were also used by Guo et al. [36] who used vinyl films and made geopolymers from class C fly ash with a 28-day compressive strength of 63 MPa. They used an alkaline solution with Ms. = 1.5 and cured the samples at 75 °C for 8 h, samples were then left at 23 °C until mechanical testing. On the other hand, Palomo et al. [67] used sealed containers to maintain the paste at a relative humidity close to 100%. They reported a fly ash-based geopolymer that showed a compressive strength of 68.7 MPa only after one day of casting. Therefore, it seems that keeping a high relative humidity using sealed molds or covering samples with films might

Table 1
Reported mixtures for fly ash-based geopolymer pastes and mortars.

Fly ash type (SiO_2/Al_2O_3, molar ratio)	Alkaline solution	Liquid/binder (weight ratio)	Compressive strength (MPa) [age]	Curing conditions	Reference
Class F (2.55)	Na_2SiO_3 + KOH(Ms = 1.2)	0.33	59 [5d]	24 h at room temperature + 24 h at 80 °C	[45]
Class C(3.40)	Na_2SiO_3 + NaOH (Ms = 1.5, Na_2O = 10% by weight)	0.40[a]	63.4 [28d]	8 h at 75 °C + 23 °C for 28 d(using vinyl film)	[36]
Class C(3.48)	Na_2SiO_3 + NaOH(Ms = 1.23)	0.30	68.7 [1d]	24 h at 85 °C(sealed container)	[67]
N/A(3.04)	9–14 M NaOH	0.30	20–23 [60d]	At room temperature(Controlled chamber)	[93]
N/A(3.71)	Na_2SiO_3 + 14 M NaOH (Si/Al = 2.3, Na/Al = 0.88)	0.25[a]	45 [28d]	At room temperature	[96]
Class F(5.38)	NaOH(Na = 14%, weight basis)	0.33[a]	120 [1d](with a sand/ash ratio of 3)	24 h at 115 °C	[15]
Class C(3.23)	Na_2SiO_3 + 10 M NaOH (Na_2SiO_3/NaOH mass ratio = 1)	0.20	86 [28d](with a sand/ash ratio of 3.23)	1 h of delay time + 3 d at 75 °C	[23]
N/A (3.44)	Na_2SiO_3 + 15 M NaOH (Na_2SiO_3/NaOH mass ratio = 1)	0.67	70 [2d] (with a sand/ash ratio of 2.75)	48 h at 65 °C (cling film)	[76]
N/A (3.16)	Na_2SiO_3 + 10 M NaOH (Na_2SiO_3/NaOH mass ratio = 1.5)	0.67	35 [2d] (with a sand/ash ratio of 2)	48 h at 65 °C (cling film)	[24]
Class F (2.79)	Na_2SiO_3 + NaOH (Ms = 1–1.5)	0.5–0.7	50–60 [3d] (with a ash/ sand ratio of 0.47)	3 d at 40 °C (humid atmosphere)	[72]

[a] Water to dry binder weight ratio.

be important and should be taken into account during curing of geopolymer pastes. Somna et al. [93] reported geopolymers made of fly ash with a 60-day compressive strength ranging from 20 to 23 MPa using only NaOH solution with concentrations ranging from 9 to 14 M and curing at low temperatures. For curing, they kept the samples in a controlled chamber at 25–28 °C until mechanical testing. Temuujin et al. [96] also reported the development of a geopolymer paste based on fly ash cured at low temperatures, in this case at ambient temperature. They produced a geopolymer with a 28-day compressive strength of 45 MPa from milled fly ash. The alkaline activation of fly ash at low temperatures performed by Somna et al. [93] and Temuujin et al. [96] reached relatively good compressive strengths possibly due to an increase of fly ash reactivity by decreasing particle size, their samples were milled down to d_{50} of 10.5 and 6.8 um, respectively. Production of mortars based on fly ash-based geopolymers has also been widely reported. Atiş et al. [15] prepared a geopolymer mortar with a 1-day compressive strength as high as 120 MPa made with a mixture of sand and fly ash in a ratio of 3:1, activated with NaOH (Na = 14% wt%) and cured at 115 °C for 24 h. Other high-strength geopolymer mortars were produced with fine class C fly ash (d_{50} = 9 μm) by Chindaprasirt and Chareerat [23], who made a geopolymer mortar with a 28-day compressive strength of 86 MPa by an alkaline activation using a mixture of sodium silicate and 10 M sodium hydroxide, in a mass ratio of 1 to 1. Rattanasak and Chindaprasirt [76] also used a Na_2SiO_3/NaOH mass ratio of 1 but with 15 M sodium hydroxide and produced a geopolymer mortar with a compressive strength of 70 MPa after curing for two days at 65 °C. The same curing conditions were used by Chindaprasirt et al. [24] who obtained a fly ash based-geopolymer mortar with a 2-day compressive strength of 35 MPa. Provis et al. [72] did an extensive study varying the liquid/binder ratio and Ms. in the total solution and found that the best compressive strength was obtained for Ms. values from 1 to 1.5 and a liquid/binder ratio ranging from 0.5 to 0.7.

2.2. Ground granulated blast furnace slag

Iron slag is a by-product of the manufacturing process of crude iron in blast furnaces. According to Van Oss [100], iron slag, also known as blast furnace slag (BFS), is generated by the combination of impurities and flux agents removed during the formation of crude iron at high temperatures. The production of BFS per ton of crude iron depends highly on the grade of the iron ore, with a ratio of 1.2 tons of slag per ton of crude iron in low grades ores. BFS is produced in large quantities, the US accounts for 18 million tons in 2017 according to USGS data [99]. Van Oss [100] pointed out that three main types of BFS can be formed depending on the cooling process during crude iron production, each one can be used for a different purpose. BFS cooled at ambient conditions, is mostly used as an aggregate for road metal, concrete, and asphalt. Foamed BFS, which is obtained when cooled by a water jet stream, is used for the production of lightweight concrete. Finally, BFS cooled in water, also known as GGBF, is utilized primarily, after a grinding process, as partial substitution of OPC due to its moderate hydraulic cementitious properties. This blended cement develops a low initial strength making its use unsuitable for applications where high initial resistance is needed. However, GGBFS based geopolymers show good mechanical strength even at the initial stage. Chemical composition analysis of GGBFA shows that it is made primarily of CaO, SiO_2, and Al_2O_3 in decreasing amounts (e.g. GGBFS used by Oh et al. [61] contained 41.78% of CaO, 33.04% of SiO_2 and 13.35% of Al_2O_3).

Table 2 presents the optimal mixtures for the GGFBS-based geopolymers and their compressive strengths reported in the scientific literature. Even though some studies have shown that high contents of Ca in raw materials like fly ash diminish the mechanical properties of the resulting geopolymer, GGBFS based geopolymers, which have high amounts of Ca showed great initial and final strengths (up to 50 MPa) when 10 M sodium hydroxide was used as an activator [61]. The authors suggest that GGBFS and fly ash class C have different chemical forms of calcium. While Ca stays unreacted in class C fly ash reducing the mechanical strength, Ca in GGBFS can form C-S-H bonds that seem to increase the final mechanical strength [61]. In the same direction, Cheng & Chiu [22] also produced a geopolymer with high compressive strengths from GGBFS activated with sodium silicate and 10 M potassium hydroxide. Schilling et al. [85] produced GGBFS-based geopolymers with a 28-day compressive strength of 38 MPa which were cured at 23 °C in a saturated atmosphere. There are also studies that report the production of mortars based on alkaline activation of GGBFS, for instance, Omer et al. [62] presented a GGBFS-based geopolymer mortar with a sand/GGBFS ratio of 2.75, that had a 7-day compressive strength of 47 MPa using an activating solution of sodium silicate and 8 M NaOH with a mass ratio of 2.5. Meanwhile, a geopolymer mortar from GGBFS (sand/GGBFS ratio = 4) was produced by Islam [42] with a compressive strength of 60 MPa at 3 days after casting.

Table 2
Reported mixtures for GGFBS-based geopolymer pastes and mortars.

SiO_2/Al_2O_3 molar ratio	Alkaline solution	Liquid/binder (weight ratio)	Compressive Strength (MPa) [age]	Curing Conditions	Reference
4.04	Na_2SiO_3 + 10 M KOH (SiO_2/Al_2O_3 = 3.36, SiO_2/K_2O = 1.32)	N/A	70 [1d]	3 h at 60 °C (sealed molds)	[22]
4.21	10 M NaOH	0.4	50 [14d]	7 d at 80 °C (water bath)	[61]
6.26	5 M NaOH	0.4[a]	38 [28d]	28 d at 23 °C (>95% RH)	[85]
4.11	Na_2SiO_3 + 8 M NaOH (Na_2SiO_3/NaOH mass ratio = 2.5)	0.4	47.3 [7d] (sand/GGBFS ratio of 2.75)	24 h at 60 °C (with plastic bags)	[62]
4.03	Na_2SiO_3 + 12 M NaOH (Na_2SiO_3/NaOH mass ratio = 2.5)	0.4	60 [3d] (sand/GGBFS ratio of 4)	24 h at 65 °C (with plastic films)	[42]

[a] Water to dry binder weight ratio.

2.3. Construction and demolition waste

The amount of construction and demolition wastes (C&D) is enormous and keeps growing in the world. According to the United States Environmental Protection Agency (EPA), 534 million tons of C&D was generated by the US alone in 2014 [98], while in Europe around 970 million were produced in 2006 [57]. Even though C&D are considered to be harmless materials, it still requires a place for disposal and improper management can cause serious effects on the environment and human health [54]. Unfortunately, waste management systems in less developed countries are inefficient and operate either with low or without standards [104], resulting in wastes disposed of in uncontrolled landfills and dumps [33]. Therefore, recycling C&D to produce new building materials is beneficial, not only for reducing energy and raw materials but also to control the decrease of available land for mining resources and landfills [3]. Moreover, if the amounts of C&D and fly ash produced per year in the U.S. are used as a reference, the potential and interest should increase in the future towards the former material since its generation is about 10 times larger than fly ash.

2.3.1. Clay brick powder

Clay brick powder, a C&D product, has shown great potential as raw material for geopolymer production. Baronio and Binda [16] claimed that clay brick powder has potential pozzolanic activity due to the destruction of the crystalline network when the hydroxyl groups in clay minerals are lost when subjected to high-temperature conditions during production. Chemical composition of clay brick powder is mainly SiO_2 and Al_2O_3, however, it also contains important amounts of CaO, Fe_2O_3 and MgO [3,16,44,75,77,78,113].

A compilation of research results concerning the formulation of the alkaline activating solution for clay brick powder to produce geopolymer binders and mortars is presented in Table 3. Allahverdi and Najafi [3] developed a binder with a 28-day compressive strength of 40 MPa by activation of clay brick powder (SiO_2 = 53.4%, Al_2O_3 = 10.5%) with an alkaline solution of Ms. = 0.6 and with a Na_2O content of 8% (by weight of dry binder). In this study, samples were cured at ambient temperature and high relative humidity (95%). They also found that the addition of waste concrete powder to the mixture is detrimental for the compressive strength e.g. a replacement of 40% (wt%) of brick powder with concrete waste powder caused a decrease in the compressive strength from 40 MPa to 16.5 MPa [3]. On the other hand, Komnitsas et al. [44] studied the geopolymerisation of three types of C&D and found that the best formulation for the alkaline activating solution for their brick powder (SiO_2 = 57.8%, Al_2O_3 = 14.95%) was a mixture of Na_2SiO_3 and 8 M NaOH and the optimal curing temperature was 90 °C, for 7 days using plastic bags to avoid water evaporation. Furthermore, they demonstrated that the compressive strength of brick powder-based geopolymers increased from 8 up to 35 MPa when particle size was reduced from d_{50} of 35 to 6.6 μm. These results are in agreement with data reported by Pathak et al. [68], who studied the effect of particle size in the compressive strength of clay brick powder-based geopolymers. They concluded that finer particles of the brick powder obtained by a longer milling process formed geopolymer binders with better compressive strength [68]. Silva et al. [87] presented an optimization analysis aimed to determine the best alkaline activating solution and curing conditions for fired clay brick powder-based geopolymers production. Their analysis was based on five key parameters: Ms., Na_2O content, water/binder ratio, oven-curing temperature and oven-curing time. Through this optimization process, clay brick powder-

Table 3
Reported mixtures for clay brick powder-based geopolymer pastes and mortars.

SiO_2/Al_2O_3 molar ratio	Alkaline solution	Liquid/binder (weight ratio)	Compressive strength (MPa) [age]	Curing conditions	Reference
8.66	Na_2SiO_3 + NaOH (Ms = 0.6, Na_2O = 8% by weight)	0.30[a]	40 [28d]	At room temperature (humid bath)	[3]
6.57	Na_2SiO_3 + 8 M NaOH (Na_2SiO_3 = 6%, H_2O = 16%)	0.38	49.5 [7d]	7 d at 90 °C (covered with plastic bags)	[44]
5.34	Na_2SiO_3 + 8 M NaOH (Na_2SiO_3/NaOH volume ratio = 2.5)	0.30	15 [90d]	At room temperature (covered with polythene sheet)	[113]
4.42	Na_2SiO_3 + NaOH (Ms = 0.6, Na_2O = 8% by weigth)	0.27[a]	36 [7d]	7d at 65–80 °C	[87]
5.34	Na_2SiO_3 + 8 M NaOH (Na_2SiO_3/NaOH volume ratio = 2.5)	0.30	83 [90d] (with GGBFS replacement of 60%)	At room temperature (covered with polythene sheet)	[113]
4.15	Na_2SiO_3 + NaOH (Ms = 1.5, Na_2O = 5% by weigth)	N/A	120 [28d] (with GGBFS replacement of 60%)	At room temperature (RH = 95%)	[75]
5.12	Na_2SiO_3 + NaOH (Ms = 1.6, Na molality = 8)	0.35[a]	41 [7d] (sand/brick powder ratio of 3)	7 d at 65 °C (controlled bath)	[80]
5.11	Na_2SiO_3 + NaOH (Ms = 2, Na molality = 7)	0.30[a]	50 [7d] (sand/brick powder ratio of 2)	7 d at 65 °C (controlled bath)	[78]
5.11	Na_2SiO_3 + NaOH (Ms = 1.6)	0.40[a]	80 [28d] (sand/brick powder ratio of 3 + CAC replacement of 40%)	28 d at 20 °C (RH = 96%)	[77]

[a] Water to dry binder weight ratio.

based geopolymers with a 7-day compressive strength of 36 MPa were developed using an alkaline solution consisted of Ms. = 0.60, Na_2O content of 8%, water/binder ratio = 0.27 with oven curing conditions between 65 and 80 °C for 7 days. In this line, pure waste fired clay brick powder (SiO_2 = 50.16%, Al_2O_3 = 15.95%) were activated by Zawrah et al. [113] using Na_2SiO_3 and 8 M NaOH with a Na_2SiO_3/NaOH volume ratio of 2.5, to produce a paste with a 90-day compressive strength of 15 MPa. This 90-day compressive strength increased up to 83 MPa by replacing 60% of clay brick powder with GGBFS, maintaining same alkaline and curing conditions. Suitability of clay brick powder and GGBFS blending was also demonstrated by Rakhimova & Rakhimov [75], who manufactured an alkali-activated paste with a 28-day compressive strength of 120 MPa from a precursor material consisting of 40% clay brick powder and 60% GGBFS. Similarly, Reig et al. [80] produced a geopolymer mortar made of brick powder and sand (sand/brick ratio = 3) by mixing the solids with an alkaline solution containing Na_2SiO_3 and NaOH (Ms = 1.6), the product showed a 7-day compressive strength of 41 MPa. A clay brick-based geopolymer mortar was also elaborated by Reig et al. [80] with a 7-day compressive strength of 50 MPa mixing sand and brick in a mass ratio of 2 and Ms. of 2. Furthermore, there are also reports that focus on blended geopolymers, for example, Reig et al. [77] studied the influence of the addition of calcium aluminate cement on alkaline-activated mortars based on clay brick powder. They found that a replacement of 40% of clay brick powder by calcium aluminate cement (CAC) could increase the compressive strength of the mortar from 10 to 80 MPa when cured at 20 °C with an RH of 96%.

2.3.2. Concrete waste powder

Concrete waste powder (CWP) has a high content of SiO_2 and CaO with a lower presence of Al_2O_3 and Fe_2O_3. Nevertheless, a pure CWP-based geopolymer with a 7-day compressive strength of 13 MPa was reported by Komnitsas et al. [44]. Another study has reported the production of geopolymers based on CWP blended with alumino-silicate materials as is shown in Table 4. Vásquez et al. [101] pointed out that CWP needs the addition of a reactive alumina source to develop high mechanical strength materials due to its semi-crystalline nature. These authors showed that the addition of 10% of metakaolin increased the compressive strength from 25.6 MPa (pure CDW geopolymer) to 46.4 MPa under the same conditions. Ahmari et al. [1,2] confirmed this by showing that pure CWP based geopolymers achieved low resistances, while CWP blended with fly ash (in a 1:1 ratio) produced hybrid geopolymers with good mechanical properties. This hybrid geopolymer, activated with NaOH (5-10 M) and sodium silicate (sodium silicate/NaOH ratio of 1:2), resulting in even better mechanical properties than pure fly ash geopolymers activated with the same alkaline conditions. Hence, the addition of CWP could improve the mechanical properties of fly ash geopolymers [1,2]. However, mixing red clay brick powder and CWP for geopolymer production did not show good results as in the case of metakaolin or fly ash blending. This could suggest that there is not enough Al content to form sufficient polysialate units. In this regard, Allahverdi and Najafi [3] have reported that replacing 40% of CWP by red clay brick powder caused a decrease of the compressive strength from 49.5 MPa (pure brick powder-based geopolymer) to 16.5 MPa at 28 days of curing time.

2.4. Mine tailings

Mine tailings (MT) are a residual material from mine operations and are mainly composed of finely-ground sand to silt-sized rock particles, water, and processing reagents used to extract valuable minerals from the ore (Natural Resources [59]). With the number of minerals extracted and processed each year around the world by the mining industry, the volume of MT is enormous, e.g. worldwide MT generation is estimated to be >7 billion tons per year. Therefore, this matter should be of concern in metal producer countries since the proper disposal of MT requires not only a lot of lands but can also constitute a hazardous environmental problem as dangerous heavy metals can reach aquatic ecosystems [84]. As MT disposal is potentially toxic in some cases and could involve an elevated cost to satisfy environmental regulations, its potential use as raw material for geopolymerisation has generated great interest.

As shown in Table 5, copper and tungsten MT have already been widely studied as starting materials for geopolymer production. Pacheco-Torgal et al. [63] evaluated the use of tungsten MT that was composed mainly by SiO_2, Al_2O_3, Fe_2O_3 and K_2O. They used a mixture of NaOH 24 M and sodium silicate (Ms = 1.34) as the activating solution and cured their samples at room temperature, the geopolymers formed showed a 56-day compressive strength of 45.5 MPa. The same curing conditions were employed by Pacheco-Torgal & Jalali [65] to obtain a tungsten MT geopolymer paste with a 28-day compressive strength of 40 MPa using an alkaline solution with a Na_2SiO_3/NaOH mass ratio of 2.5. On the other hand, Silva et al. [91] employed a curing process consisting of two stages to produce a binder with compressive strength up to 24 MPa. In the first stage, specimens were left at room temperature and then were heated at 80 °C in the second stage. Regarding the use of copper MT, Ahmari et al. [1,2] studied the effect of curing temperatures and alkaline solutions in the activation and resulting compressive strength of geopolymers. The chemical composition of cooper MT used by Ahmari et al. [1,2] was mainly SiO_2 and Al_2O_3 with a substantial presence of CaO and Fe_2O_3. They found that an alkaline solution of sodium aluminate and 10 M NaOH at a mass ratio of 1.25 and a curing temperature of 90 °C for 7 days produced a cooper MT-based geopolymer with a compressive strength of 17 MPa. Besides copper and tungsten MT, iron MT has also been used as raw material for geopolymers production. For instance, Kuranchie et al. [50] developed iron MT geopolymers with a 7-day compressive strength of 50 MPa and water absorption of 9% meeting ASTM requirements for the specification of bricks. The formulation of this geopolymer matrix consisted of an activator content of 31% and curing conditions of 7 days at 80 °C. Another approach that has been

Table 4
Reported mixtures for CWP-based geopolymer pastes and mortars.

SiO_2/Al_2O_3 molar ratio	Alkaline solution	Liquid/binder (weight ratio)	Compressive Strength (MPa) [age]	Curing conditions	Reference
8.95	Na_2SiO_3 + NaOH (Na_2O = 6%, SiO_2/Al_2O_3 = 8)	0.22	46.4 [28d] (with metakaolin replacement of 10%)	24 h at 25 °C + curing chamber (covered with polythene sheet)	[101]
6.63	Na_2SiO_3 + 8 M NaOH (Na_2SiO_3 = 6%, H_2O = 17%)	0.48	13 [7d]	7 d at 90 °C (covered with plastic bags)	[44]
3.56	Na_2SiO_3 + NaOH (Na_2SiO_3/NaOH mass ratio = 2)	0.29[a]	35 [7d] (with fly ash replacement of 50%)	At room temperature (covered with plastic bag)	[1,2]
8.97	Na_2SiO_3 + NaOH (Ms = 0.6, Na_2O = 8% by weight)	0.26[a]	16.5 [28d] (with clay brick replacement of 40%)	At room temperature (humid bath)	[3]

[a] Water to dry binder weight ratio.

Table 5
Reported mixtures for MT-based geopolymer pastes and mortars.

(SiO_2/Al_2O_3 molar ratio)	Alkaline solution	Liquid/binder (weight ratio)	Compressive strength (MPa) [age]	Curing conditions	Reference
Tungsten MT (5.46)	Na_2SiO_3 + NaOH (Ms = 1.34)	1	45.5 [56 d]	At room temperature	[63]
Tungsten MT (5.46)	Na_2SiO_3 + NaOH (Na_2SiO_3/NaOH mass ratio = 2.5)	1	40 [28 d]	At room temperature	[65]
Tungsten MT (6.34)	Na_2SiO_3 + NaOH (Na_2SiO_3/NaOH mass ratio = 4)	0.25	24 [63 d]	7 days at 20 °C + 56 days at 80 °C	[91]
Cooper MT (15.53)	$NaAlO_2$ + 10 M NaOH ($NaAlO_2$/ NaOH = 1.25)	0.27[a]	17 [7 d]	7 days at 90 °C (capped mold)	[1,2]
Cooper MT (15.53)	NaOH 15 M	0.27[a]	3 [7 d]	7 days at 60 °C (Plexiglas cap)	[115]
Iron MT (10.15)	Na_2SiO_3 (8.9% Na_2O, 28.7% SiO_2 and 62.4% H_2O by weight)	0.31	50 [7 d]	1 day at room temperature and 6 days at 80 °C	[50]
Gold MT (8.14)	NaOH 10 M	0.27	25 [28 d] (with GGBFS replacement of 25%)	At room temperature	[43]
MA Vanadium MT (14.39)	Na_2SiO_3	0.36	25 [14 d] (with MK replacement of 50%)	At room temperature	[102]

[a] Water to total dry binder weight ratio.

gaining attention is mixing MT with other alumino-silicate materials to obtain a blended raw material with enhanced reactivity. In this line, Zhang et al. [115] produced hybrid geopolymers made from mixtures of fly ash and cooper MT, which resulted in compressive strengths in the range of 3 MPa (pure copper MT-based geopolymer) to 14 MPa (75% of flay ash replacement) at 7 days of age. Kiventerä et al. [43] investigated the alkaline activation of a mix of gold MT and GGBFS. They found that GGBFS has great potential as a co-binder of gold MT since a replacement of this material by 25% (wt%) of GGBFS increased the compressive strength from 3 MPa (pure gold MT-based geopolymer) to 25 MPa. Another example of hybrid MT geopolymer is the one reported by Wei et al. [102]. The main objective of their work was to explore the mechanical activation (MA) of vanadium MT. However, they used a constant metakaolin replacement of 50% (wt%) during all the investigation to provide additional reactive Al to the mix. They pointed out that milling raw MT modify the physicochemical characteristics of the vanadium MT as particle size and amorphous content leading to a better reactivity of the raw material [102]. The alkaline activation of the mix of the MA vanadium MT and metakaolin in a weight ratio of 1:1 produced a geopolymer matrix with 14-day compressive strength of 25 MPa, 190% higher than the strength obtained by geopolymer produced with raw vanadium MT.

3. Natural fibers used as reinforcement for geopolymers

3.1. Plant fibers

The synthetic or man-made fibers such as carbon, glass, aramid, and polypropylene as a reinforcement of polymer matrices producing fiber-reinforced composites with improved mechanical properties are widely applied to the automotive, aerospace as well as construction industries [51]. The application of fiber reinforcement to construction materials can modify the tensile and flexural strength, and fracture energy of cementitious matrices [108]. In the fiber-reinforced composites, fibers function not only as a reinforcement but also as the main source of strength while matrix glues all the fibers together in shape and transfers stresses between the reinforcing fibers. At the same time, fibers carry loads along with their longitudinal directions [19].

Due to an increasing environmental concern for developing environmentally-friendly and energy-efficient materials, natural fibers are more preferred than synthetic fibers as reinforcements within cementitious and polymeric composites for decades [39,90,108]. Many authors carried out the studies about the natural reinforcements considering only plant fibers although there are also animal and mineral natural fibers. Animal fibers are less favorable in comparison to plant fibers considering that their collection from animals is more difficult to implement on a large scale. The most of mineral fibers are undergone several processing before applications and the unique mineral fiber obtained without such processes is an asbestos, classified as carcinogenic material [94] and not suitable for eco-friendly composite preparation. Therefore, plant fibers are the better option for the production of natural fiber-reinforced geopolymer composites. Plants fibers have several attractive advantages that surpass synthetic fibers: i) low weight, ii) low cost, iii) widely available, iv) biodegradable, v) renewable and non-hazardous sources, vi) desirable aspect ratio, and vii) good relative tensile and flexural strength [105,109,110]. In the use of plant fibers, there are several important factors might be considered, i.e. fiber selection (including type, harvest time, extraction method, aspect ratio, treatment and fiber content), matrix selection, interfacial strength, fiber dispersion, fiber orientation, composite manufacturing process, and porosity [70].

Plant fibers have been used as reinforcing materials for geopolymer matrices for a long time. They are very suitable since the geopolymerisation occurs at high alkaline environments and lignocellulose fibers have a strong resistance to these conditions. All plant fibers are mainly composed of cellulose and lignin (lignocellulose fibers) and the cellulose content varies according to the species and age of the plant. Cellulose is a hydrophilic glucan polymer formed by a linear chain of glucose units linked together through $\beta(1\rightarrow4)$ bonds, while lignin is a biochemical material that works for structural support in plants [56]. The polymerization degree of cellulose is responsible for the mechanical properties of the fiber and varies depending on the species of the plant [56]. The mechanical properties of plant fibers depend also on physical properties of the fiber such as diameter, length, moisture gain, microfibril angle, etc. The tensile strength of cellulose fibers decreases with an increase of fiber length since longer fibers have possibly more defects and thus could fail prematurely compared to shorter fibers [110] and can vary significantly depending on the maturity level [112]. Cellulosic plant fibers have high moisture absorption capacity and poor dimensional stability because they usually swell in contact with water. When natural fibers are used as reinforcement, adhesion between fibers and matrix can be affected by both the hydrophobic/hydrophilicity characteristics of the fibers and its interaction with the matrix. The presence of pendant hydroxyl and polar groups in the components can lead to high moisture uptake, poor fiber and matrix adhesion causing a low mechanical performance of the composite [110]. There are several efforts to improve the fiber and matrix adhesion to obtain better durability of components: i) Hornification [13]: fibers undergo drying and rewetting cycles; ii) Mercerization [40,107]: alkaline pretreatment of fiber; and iii) Silane treatment [71]: improvement of water resistance.

3.2. Plant fibers reinforcement for geopolymer matrices

The number of published papers focusing on the use of natural plant fibers to enhance mechanical properties of geopolymer matrices has increased in recent years [110], however, it is still limited compared to the number of studies conducted in conventional cementitious materials. Review articles elaborated by Pacheco-Torgal and Jalali [64] and Hejazi et al. [38] have shown that natural fiber reinforcement of conventional building materials such as soil and OPC-based products is a topic that has gained great attention. There are a few papers that present the state of the art of fiber-reinforced geopolymer composites [82,88], but, they only focus on man-made or synthetic fibers and fabrics such as geopolymer matrices reinforced with steel fibers [117], carbon fibers, glass fibers [58], polypropylene fibers [73], polyvinyl alcohol fibers [95,111], basalt fibers [29,52] as well as carbon fabrics [53], basalt fabrics [118] and glass fabrics [69]. Therefore, this section will focus on presenting researches done with plant natural fibers as reinforcement in two forms: randomly oriented short natural fibers and non-woven and woven fibers (layers).

3.2.1. Applications of short plant fibers randomly oriented

Short random fiber reinforcement of geopolymer matrices is of special interest for large-scale applications as building materials since they do not require advanced processing techniques; traditional mixing machines can be used. As shown in Table 6, several works are available in the literature regarding the analysis of mechanical properties of geopolymer composites using short natural fiber reinforcement. Correia et al. [25] studied the reinforcement of metakaolin-based geopolymers with sisal fibers and pineapple leaf fiber (PALF). Sisal fibers were extracted from the leaves of the *Agave sisalana*, while PALF was obtained from *Ananas conosus* plant. 25 mm-long fibers were used in 3% ratio (vol%) with the geopolymer matrix. Even though the compressive strength of MT-based geopolymers reinforced with sisal fibers and PALF decreased compared to unreinforced geopolymers, the tensile and impact performance of the resulting composites significantly improved for both fibers. For PALF reinforced geopolymers, the flexural, tensile and impact strengths increased in respect to the unreinforced matrix by 100%, 111%, and 200%, respectively while for the sisal fiber, the reinforced MT-based geopolymer showed an increase of 43%, 100%, and 113% in its flexural, tensile and impact strengths, respectively. Similar behavior was reported by Chen et al. [20,21] using alkali-treated sweet sorghum fiber to reinforce fly ash-based geopolymers activated with 10 M sodium hydroxide with a liquid/solid ratio of 0.36. These researchers performed the alkaline pretreatment of the fibers to improve adhesion and cohesion between matrix and fibers ((([4,20,21]. Chen et al. [20,21] performed unconfined compression, splitting tensile, and flexural tests to determine the optimum content of fibers and found it to be 2% (wt%) of fiber with respect to fly ash. Correia et al. [25], also observed that the presence of fibers induced a little loss of compression strength in all specimens. Nevertheless, the main function of fiber reinforcement is to provide ductility and control of cracking and not to enhance compressive strength [17]. Sá et al. [81] explored the use of micro and short bamboo fibers to reinforce metakaolin-based geopolymers. They also evaluated the influence of water and alkali pretreatment of the fibers in the mechanical properties of the resulting geopolymer composites. Compression tests results indicated again that the addition of water and alkali-treated bamboo fibers caused an important reduction of compressive strength. However, the flexural strength significantly increased by 450% when 5% (wt%) alkali bamboo microfibers and strips were used to reinforce the geopolymer matrix. Korniejenko et al. [46] have also reported a wide variety of plant fibers to reinforce fly-ash based geopolymers. They have worked with 1% (wt%) of cotton fibers (30 mm length), sisal fibers (3 mm length), raffia fibers (3 mm length), and coir fibers (3 mm length). Unconfined compressive test and three-point bending tests showed that the addition of cotton, sisal, and coir caused a slight improvement of the mechanical properties after 28 days of curing. However, the addition of raffia fibers proved not to be compatible with fly ash-based geopolymers and resulted in a decrease of both compressive and flexural strengths. Similar to this, Alomayri et al. [4,5] evaluated different cotton fiber contents to reinforce fly-ash based geopolymers. They used an alkaline solution to solid ratio of 0.35 and found that a cotton fiber content of 0.5% (wt%) produced the highest flexural strength, flexural modulus, and fracture toughness. They observed an adequate fiber dispersion and good interaction between the matrix and the fibers with this fiber content. More fiber, as also mentioned by Chen et al. [20,21], resulted in lower mechanical properties due to the formation of voids and fiber agglomeration. Silva et al. [86] investigated the use of linen (flax) fibers as reinforcement of fired-clay brick powder and fly ash-based geopolymers by performing compression and three-point bending tests. The inclusion of linen fibers caused a significant increase of 60% in both fired clay brick powder and fly ash-based geopolymers. However, there are differences regarding the effect of linen fibers addition in the compressive strength of geopolymer composites: the resistance under compression loads of the fiber-reinforced fly ash-based geopolymers is almost the same than exhibited by the unreinforced matrix, while linen fibers caused an increment of 53% in the compressive strength with respect to the fired clay brick powder-based geopolymer matrix.

It is known that, in general, fiber length plays an important role in the final strength achieved by composites. In the specific case of geopolymer composites, Lin et al. [53] have reported that 7 mm carbon fibers gave the highest flexural strength in comparison to the resulting matrices containing fiber lengths of 2 and 12 mm.

Table 6
Reported studies of mechanical properties of geopolymer composites reinforced with short plant fibers.

Natural fiber	Geopolymer type	Fiber content (wt%)	Compressive strength (MPa)		Flexural strength (MPa)		Tensile strength (MPa)		Reference
			Without fibers	With fibers	Without fibers	With fibers	Without fibers	With fibers	
Sisal	Metakaolin	3[a]	6.9	6	1.4	2.8	0.45	0.95	[25]
Pineapple leaf	Metakaolin	3[a]	6.9	3.3	1.4	2.0	0.45	0.90	[25]
Alkali-treated sweet sorghum	Fly ash (Class F)	2	27.7	22.9	3.6	5	2.5	3.4	[20,21]
Bamboo fibers and strips	Metakaolin	5	55.7	29.7	4.50	24.95	N/A	N/A	[81]
Cotton	Fly ash (Class F)	1	24.78	28.42	5.55	5.85	N/A	N/A	[46]
Sisal	Fly ash (Class F)	1	24.78	25.16	5.55	5.90	N/A	N/A	[46]
Raffia	Fly ash (Class F)	1	24.78	13.66	5.55	3.05	N/A	N/A	[46]
Coir	Fly ash (Class F)	1	24.78	31.36	5.55	5.25	N/A	N/A	[46]
Cotton	Fly ash (Class F)	0.5	N/A	N/A	10.4	11.7	N/A	N/A	[4]
Linen	Fly ash (Class F)	1	42.70	44.64	5.22	8.39	N/A	N/A	[86]
Linen	Fired clay brick powder	1	3.35	5.14	1.57	2.5	N/A	N/A	[86]

[a] Fiber content by volume of the composite.

Table 7
Reported studies of mechanical properties of geopolymer composites reinforced with plant fabrics.

Natural fiber	Geopolymer type	Fiber content (wt%)	Compressive strength (MPa)		Flexural strength (MPa)		Impact strength (kj/m^2)		Reference
			Without fibers	With fibers	Without fibers	With fibers	Without fibers	With fibers	
Flax bundles	Dehydroxylated halloysite	10	N/A	N/A	5.8	70	N/A	N/A	[9]
Cornhusk bundles	Metakaolin	5	N/A	N/A	14.1	8.8	N/A	N/A	[49]
Luffa Cylindrical Fiber	Metakaolin	10[a]	13.0	31.0	3.4	14.2	N/A	N/A	[8]
Cotton fabric	Fly ash (Class F)	2.1	N/A	N/A	8.2	~12.5	2.1	~6.8	[5]
Cotton fabric (perpendicular to fabrics)	Fly ash (Class F)	8.3	21.0	~90.0	8.2	31.7	2.1	15.6	[6,7]
Cotton fabric (parallel to fabrics)	Fly ash (Class F)	8.3	21.0	~60.0	8.2	~26.0	N/A	N/A	[6]
Flax fabric	Fly ash (Class F)	4.1	19.4	91	4.5	23	N/A	N/A	[14]

[a] Fiber content by volume of the composite.

3.2.2. Applications of plant fiber layers

Plant fibers reinforcement with non-woven and woven layers with alternated fiber orientation have been studied to produce fiber-reinforced geopolymer panels. As shown in Table 7, the use of non-woven plant fibers layers to reinforce the geopolymer matrix has been reported by Alzeer & MacKenzie [9], who reinforced geopolymers based on halloysite with flax fibers. Their fabrication process consisted of alternating layers of geopolymer resin and unidirectional stripped flax fiber bundles. The best reinforcement was achieved with the highest content of fibers in the composite (10%, wt%) showing an impressive increase in flexural strength from 5.8 to 70 MPa when the composite was subjected to three-point bending tests. This remarkable increase of strength was resulted by transferring the stress between the fibers and the matrix. Alshaaer et al. [8] also investigated non-woven fibers as reinforcement in the development of laminate metakaolin-based geopolymer composites. They employed a unidirectional and randomly reinforcement of Luffa cylindrical fibers with a content of 10% (vol%). The laminate reinforced composite reached a compressive and flexural strength 140% and 320% higher than the strengths developed by the unreinforced geopolymer matrix, respectively. On the other hand, Kriven et al. [49] employed quasi-aligned and random corn husk fiber bundles to produce composite panels. Their results showed that, although both flexural and impact strengths decreased in the reinforced panels, a significant improvement in deformation resistance was gained with the addition of fiber bundles. There are also reports regarding woven plant fibers, best known as fabric-reinforced composites. For instance, Alomayri et al. [5] produced a reinforced class F fly ash geopolymer composite with cotton fabrics. In this study, the authors evaluated the flexural and impact strengths and fracture toughness with two (1.4 wt%) up to six (4.1 wt%) layers of pre-dried fabric. The results showed that all three mechanical properties increased with the presence of the fabric, with a 2.1% (wt%) fiber content the optimum, which corresponds to 3 layers. The loss of mechanical performance with more layers was suggested to be caused by a lower fiber-matrix interfacial bonding. However, in a more recent study, Alomayri et al. [7] solved this limitation by wetting the fabric with the geopolymer paste and applied a 25 kg load for 3 h on the composite. This fabrication method produced geopolymer composites with up to 40 (with an 8.3 wt%) fabric layers that showed significantly better mechanical properties (31.7 MPa flexural strength and 15.6 MPa impact strength). The same procedure was used by Assaedi et al. [14] with flax fabrics and fly ash based-geopolymers obtaining a compressive strength of 91 MPa and flexural strength of 23 MPa. It is important to highlight that mechanical performance of these fabric-reinforced geopolymer composites depend on the orientation of the fabrics with respect to the applied load, as it was demonstrated by Alomayri et al. [6]. They reported that the mechanical properties in the parallel direction of the fabrics are lower than those in the perpendicular direction. They suggested that loads applied perpendicularly to the cotton fabrics they used resulted in detachment and delamination of the composite.

3.3. Other natural fibers

Table 8 summarizes published research focusing on the usage of protein-based fibers as reinforcement of geopolymer composites. Alzeer and MacKenzie [10] studied metakaolin-based geopolymers activated with NaOH and sodium silicate and reinforced with two types of wool fibers at 5 wt% of the content. They used merino and carpet wool fibers in three different conditions: without any cleaning and treatment, cleaned and treated. They reported that the addition of bundles of wool fibers could increase the flexural strength of geopolymer matrices and that cleaning and treatment of fibers had an effect on the mechanical properties of the reinforced composite.

4. Conclusion

This paper has presented a review of a wide range of research studies that involve different geopolymer pastes and mortars, and also their reinforcement with natural fibers. All reported materials have the potential to be developed into eco-friendly construction materials since their silico-aluminate raw materials are industrial by-products or wastes, and the fibers for reinforcement are renewable and easily available since they are obtained mainly from plants. Application of fiber-reinforced geopolymer composites in the construction industry has great potential to be used in the production of non-structural and structural pre-fabricate elements since the literature review evidenced that this type of materials required special curing conditions. It has been reported that geopolymers produced from raw materials like fly ash, GGFBS, clay brick powder, concrete demolition waste, and mine tailings can show good mechanical properties that are comparable to OPC products. Morphology, size, and the molar ratio of SiO_2/Al_2O_3 in the raw material, together with the alkaline solution/solid ratio, NaOH concentration, SiO_2/M_2O molar ratio in the total alkaline solution and the

Table 8
Reported studies of mechanical properties of geopolymer composites reinforced with other natural fibers.

Natural fiber	Raw material	Content (%)	Compressive strength (MPa)		Flexural strength (MPa)		Tensile strength (MPa)		Reference
			Without fibers	With fibers	Without fibers	With fibers	Without fibers	With fibers	
Cleaned merino wool	Metakaolin	5	N/A	N/A	5.8	9.1	N/A	N/A	[10]
Treated carpet wool	Metakaolin	5	N/A	N/A	5.8	8.7	N/A	N/A	[10]

curing conditions have shown to be key parameters in the formulation of geopolymers to produce good mechanical properties. Available data show that these parameters need to be adapted to the raw material used. As it composites materials in general, the review of the scientific literature shows that fiber type, content (usually between 1 and 5 wt%), dimensions and strength affect its capacity to reinforce different types of geopolymers. Geopolymer matrices reinforced with natural fibers such as sweet sorghum, wool, cotton, sisal, and coir result in materials with increased compressive, flexural and tensile strengths (up to 53%, 454% and 111% compared to the unreinforced matrix, respectively) and form a material with improved ductile behavior. Moreover, a more efficient reinforcement of geopolymer matrices can be achieved with a layered approach by adding natural fiber bundles and fabrics to allow the development of a reinforced layered composite material with improved mechanical properties as a compressive, flexural and impact strengths of up to 369%, 1106% and 642% higher in comparison to the unreinforced matrix, respectively. The effectiveness of this type of fiber reinforcement depends mainly on the number of woven or non-woven layers related to the fiber content (normally between 2.1 and 10 wt%) and the fabrication technique. In conclusion, the key parameters that need to be considered in the formulation of geopolymers were compiled and compared according to their composition, fabrication and resulting mechanical properties. Based on the reviewed information, it is evident that more research needs to be performed to help optimize formulations for the production of fiber-reinforced geopolymers with improved properties.

Declaration of Competing Interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

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