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# A Comparative Study of Linen (Flax) Fibers as Reinforcement of Fly Ash and Clay Brick Powder Based Geopolymers

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**Abstract.** Geopolymers have great potential for the development of sustainable building materials due the utilization of industrial by-products and waste materials for their production. The introduction of natural fibers to geopolymer matrices has shown interesting results for producing alternative building materials with improved mechanical properties. This research explores the use of linen (flax) fibers as reinforcement of clay brick powder based-geopolymers and fly-ash based geopolymers. Linen fibers were obtained from the Institute of Natural Fibres and Medicinal Plants while clay brick powder were obtained from construction waste in Lima (Peru) and fly ash were extracted from Skawina (Poland). The influence of the addition of linen fibers on the mechanical properties of geopolymer matrix was studied by unconfined compression tests and three-point bending tests. The results show that addition of flax fibers significantly enhanced both compressive and flexural strengths of fly ash and clay brick powder based geopolymers.

## 1. Introduction

The search for eco-friendly building materials to replace traditional ones is a global concern due to all the environmental issues and unsustainability regarding construction industry. Recycling Construction & Demolition Wastes (C&D) for the production of alternative building materials will have significant benefits, from the reduction of energy and raw materials usage, to the decrease of use of available land for extracting resources or for landfills [1]. Clay brick powder, a C&D product, has shown great potential as raw material for geopolymer production. Baronio and Binda [2] claimed that clay brick powder has pozzolanic activity potential due to the destruction of the crystalline network when structural hydroxyl groups of clay minerals are lost by high temperatures during production. On the other hand, as global electricity production shows an important dependency on coal sources, enormous quantities of fly ash are produced around the world, e.g. only US produced 51 million tons in 2014 [3]. The percentage of fly ash used in other applications is low, so the major part of these ashes are stored in landfills and storage lagoons resulting in potential land, environmental and human health issues [4]. The chemical composition of fly ash varies depending of the coal source and the combustion process but is mainly



composed by  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  [4]. Therefore, fly ash is also suitable for geopolymer production since it contains high amounts of silica and alumina.

Unfortunately, unreinforced geopolymers exhibit a brittle behavior, similar to Portland cement pastes, which makes geopolymers inadequate for many applications. However, addition of synthetic and natural fibers to the geopolymer matrix has shown promising results as it enhances mechanical properties e.g. increases fracture toughness, improves flexural and impact strength, provides ductility and suppresses brittle behavior [5,6]. Natural fibers are eco-friendly and have lower density, higher specific strength and lower cost than synthetic fibers, and do not require a high energy consuming process [7]. Since a composite material based on recycled clay brick powder or an industrial by-product as fly ash reinforced with natural fibers would be a good option for the development of an eco-friendly building material, the objective of this article is to explore the use of untreated linen fibers as a reinforcement of fly ash and clay brick powder based geopolymers.

## 2. Experimental plan

### 2.1. Materials

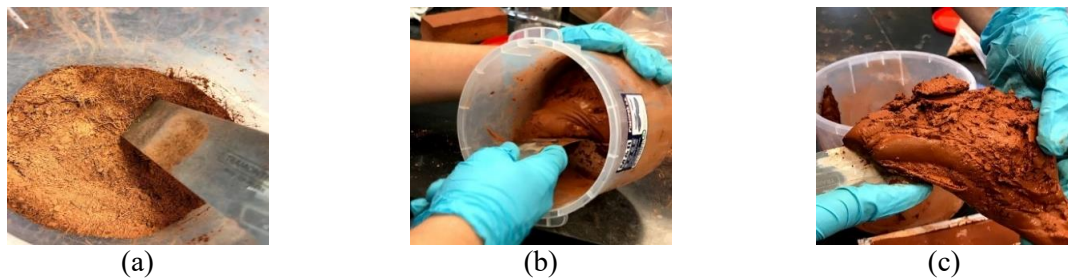
Fired clay bricks from construction wastes in Lima (Peru) were subjected to a milling process consisting of four phases with different equipment. First, the entire brick was placed in an impact crusher to obtain pieces smaller than 25 mm. These pieces were then placed in a jaw crusher to reduce their size. After that, a rolling mill was used to produce brick particles smaller than 0.5 mm, which were finally placed in a ball mill for 3 hours. Resulting brick powder was passed through ASTM sieve No. 200 and particles smaller than 0.075 mm were used as a raw material in the production of clay brick powder-based geopolymer. Analysis of elemental composition of brick powder was performed by energy dispersive spectroscopy (EDS) with an Ametek EDAX TEAM system coupled to a FEI Quanta 650 scanning electron microscope. The brick powder showed high content of O (~35%), Fe (~29%), Si (~17%) and Al (~11%).

Geopolymer matrices were also made from fly ash from the CHP plant in Skawina (Poland) and sand in ratio 1:1. This fly ash from CHP plant in Skawina (Poland) is rich in oxides such as  $\text{SiO}_2$  (~56%),  $\text{Al}_2\text{O}_3$  (~23%) and  $\text{Fe}_2\text{O}_3$  (~6%) [8].

The flax fibers were after retting process. The fibers were cut to a length of 5 mm approximately and were added to the geopolymer matrix in the production process. The addition of fibers in geopolymer mixture was 1% by weight of both clay brick powder as well as fly ash based matrix.

### 2.2. Preparation of samples

Unreinforced and fiber reinforced geopolymer samples were prepared to study the effect of linen fiber reinforcement on the mechanical properties of clay brick powder-based geopolymers. In both cases, geopolymer matrices with an alkaline solution to clay brick powder ratio of 0.4 were considered. Alkaline solution consisted of a mixture of 10 M sodium hydroxide (NaOH) and commercial sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) in a NaOH: $\text{Na}_2\text{SiO}_3$  ratio of 1:2.5 by volume. First, clay brick powder and linen fibers were dry-mixed to ensure homogenous distribution (see Figure 1a). Then, alkaline solution was gradually added and manually mixed until a homogenous paste was obtained (see Figure 1b and c). The geopolymer paste was hand-formed into prismatic silicon moulds and subject to vibration in order to remove trapped air bubbles. Specimens were kept in an oven at approximately 70°C for 24 hours, after which specimens were left at ambient temperature until the mechanical tests. All specimens were tested after 1, 7, 14 and 28 days of age.



**Figure 1.** Clay brick powder-based geopolymer production: (a) Dry-mixing of clay brick powder and linen fibers; (b) Hand-mixing after solution addition; (c) Geopolymer paste.

In case of fly-ash based samples the manufacturing process was very similar. The process of activation has been made by 8 M sodium hydroxide solution combined with the sodium silicate solution (liquid glass at a ratio of 1:2.5). Technical sodium hydroxide was used and an aqueous solution of 2.5 M sodium silicate (R-145) with density about 1.45 g/cm<sup>3</sup>. Tap water was used instead of the distilled one. The alkaline solution was prepared by means of pouring the aqueous solution of sodium silicate over the solid sodium hydroxide. The solution was mixed and left until its temperature became stable and the concentrations equalized about 2 hours. The fly ash and sand, alkaline solution and fibers were mixed about 20 minutes by using low speed mixing machine (to form a homogeneous paste). Next, samples were hand-formed into two sets of plastic moulds and then subjected to vibratory removal of air bubbles. Tightly closed moulds were heated in the laboratory dryer for 24h at 75 °C. Then, the samples were demoulded and investigated after 28 days.

### 2.3. Research procedures

Scanning electron microscopy (SEM) type JEOL JSM 820 with EDS has been performed for microstructure research. The research has been made for fibers and the samples made from fly ash-based geopolymer previously broken from compressive or flexural strength tests. Samples were covered with a thin layer of gold with JEOL JEE-4X vacuum sputter. The investigations were made at various magnifications (between 20 – 100x).

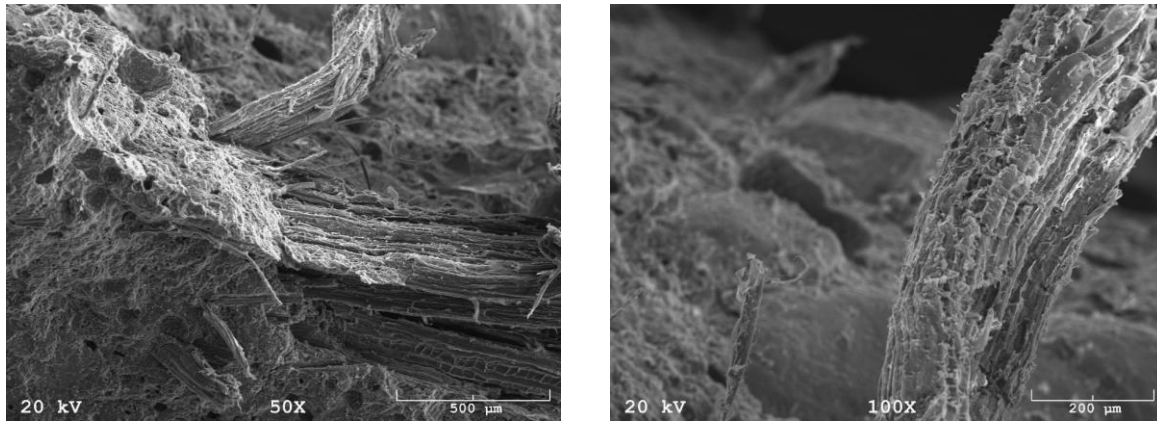
To evaluate the influence of linen fibers as a reinforcement of clay brick powder-based geopolymers, three-point bending tests and compression tests were performed according to ASTM C348 [9] and ASTM C349 [10], respectively. Three-point bending tests involved at least testing 3 prismatic samples with approximate dimensions of 40 mm x 40 mm (cross section) x 160 mm (length). Then, the two resultant broken parts from each bending test (cubic specimens with approximate dimension of 40 mm x 40 mm x 40 mm) were tested in compression. An electromechanical testing machine MTS Exceed 45.105 [11] was used for all mechanical testing. The displacement rate of the load frame was set at 0.3 mm/min for three-point bending tests and 0.5 mm/min for compression tests. Displacements of the load frame were recorded in all tests and were taken as global deformations. Furthermore, a digital image correlation technique was also used to measure localized deformations in the middle third of geopolymer specimens during mechanical tests by means of an ARAMIS 5M system with a strain precision of 0.005% [12].

The fly ash-based geopolymers were tested according to EU standards. Compressive strength tests were carried out according to the standard method EN 12390-3 ('Testing hardened concrete. Compressive strength of test specimens') [13], because of the lack of separate standards for geopolymer materials. The tests involved at least 6 samples and were performed on a universal testing machine - single-point load (Instron type 4465). Samples used for compressive strength test had cubic shape and dimensions (approx.): 50 mm × 50 mm × 50 mm. Flexural strength tests were carried out according to the standard EN 12390-5 ('Testing hardened concrete. Flexural strength of test specimens') [14]. Samples used to the flexural strength test had cuboid shape and dimensions (approx.): 200 mm × 50 mm × 50 mm. Tests were performed with a universal testing machine - single-point load (Instron type 4465).

### 3. Results

#### 3.1. Microstructure investigation

SEM observation of microstructure for composites gives a preliminary information about the coherency of flax fibers with the geopolymer matrix. Figure 2 shows exemplary images that presented the composite morphology.



**Figure 2.** SEM image morphology for geopolymer matrix reinforced flax fibers

SEM images also showed that fiber distribution is regular. Fiber distribution in the matrix influences the properties of the specific composite, especially aggregation of fibers can decrease its mechanical properties.

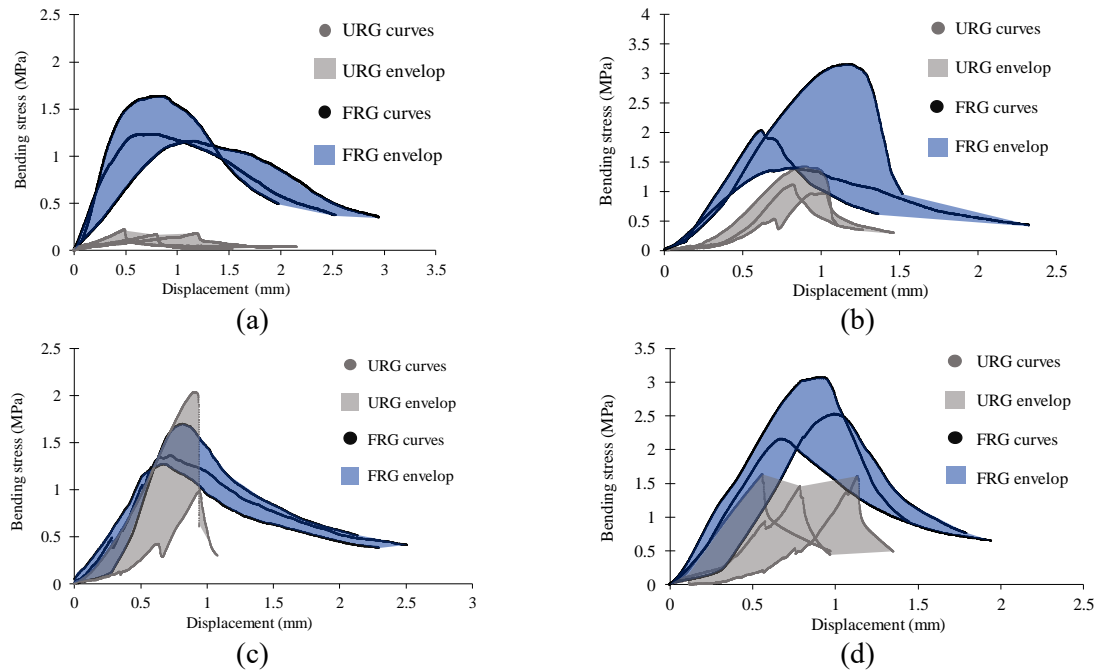
#### 3.2. Three-point bending tests

Three-point bending tests were used to study the flexural behavior of unreinforced geopolymer (URG) and fiber reinforced geopolymer (FRG) by means of applying a concentrated load in the middle of a simply supported prismatic sample. The history of applied force and mid-span vertical displacements of the load cell during three-point bending tests were used to generate the curves that relate bending stress and displacement. The bending stresses ( $\sigma_f$ ) were calculated using equation 1.

$$\sigma_f = \frac{3FL}{2BH^2} \quad (1)$$

where F is the applied force while L, H and B denote the space between supporting points (for conducted tests: 120 mm), height and width of the prismatic samples, respectively.

Figures 3a, b, c and d show the curves and envelopes resulting from the three-point bending tests for URG and FRG samples at ages of 1, 7, 14 and 28 days, respectively. A notorious difference in flexural response between UGR and FGR samples due to the reinforcement of linen fibers is evidenced in Figure 3. FGR samples could withstand higher bending stresses as a result of the linen fiber's presence. Likewise, the abrupt reduction of resistance after reaching the maximum stress observed in URG samples is not seen in the FRG curves, which indicates that the linen fibers help to avoid a fragile failure.



**Figure 3.** Bending stress vs. mid-span vertical displacements curves and envelopes for clay brick powder-based geopolymer specimens tested at different ages: (a) 1 day age test; (b) 7 days age test; (c) 14 days age test; (d) 28 days age test.

The research for samples based on fly ash was performed after the 28 days. For the calculation of flexural strength of fly ash-based geopolymer ( $f_{cf}$ ) has been applied following equation 2:

$$f_{cf} = \frac{Fl}{d_1 d_2^2} \quad (2)$$

where  $F$  is maximal load while  $l$ ,  $d_1$  and  $d_2$  denote the space between supporting points (for conducted tests: 140 mm), height and width of the prismatic samples, respectively. The results of flexural strength test are presented in table 1.

**Table 1.** Flexural strength of fly ash-based geopolymer after 28 days.

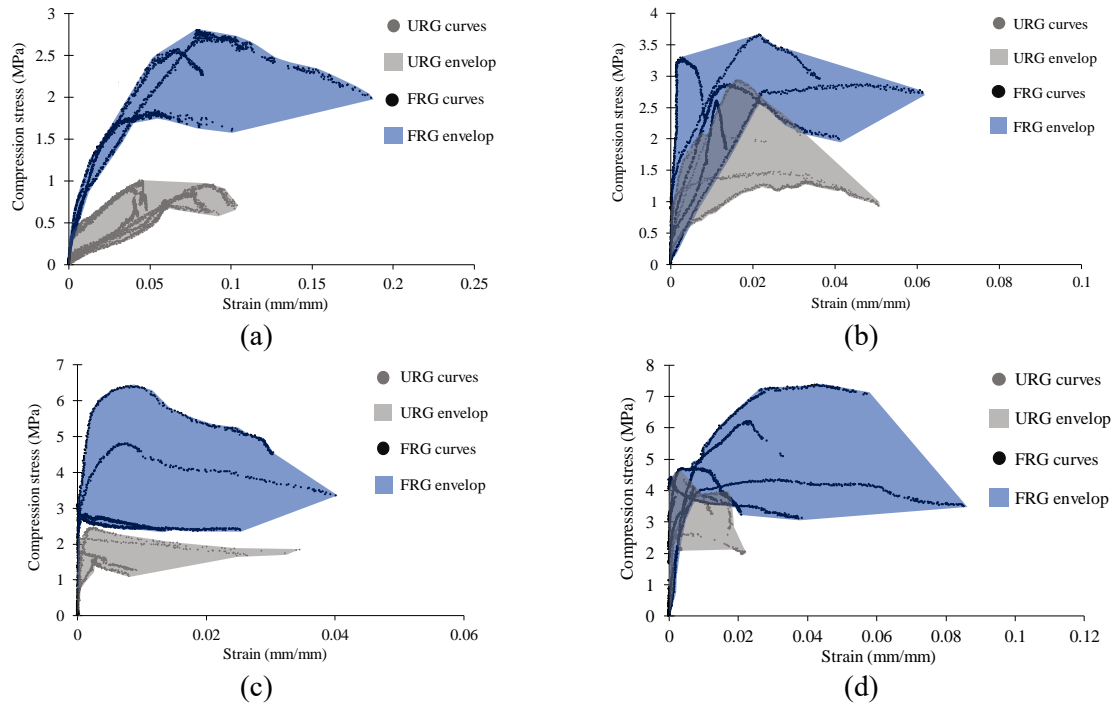
Sample	MPa	StD
Geopolymer without reinforcement	5.22	0.251
Geopolymer with flax fibers (1%)	8.39	0.098

The results for samples with reinforcement are significantly better than for geopolymer matrix. Additionally, similar as in case of clay brick powder-based geopolymers, the fiber addition helps avoid the brittle failure.

### 3.3. Compression tests

Compression stress-strain curves were obtained by means of uniaxial compression tests on the two resultant portions from each three-point bending test. Compression stresses were calculated as the ratio of the applied load recorded during the test and the transversal area of the 40 mm x 40 mm metal plate. Local deformations measured in the middle third of the lateral face in URG and FRG samples were considered as the strains to calculate compression stress-strain curves. Figures 4a, b, c and d show the stress-strain curves and envelopes resulting from the uniaxial compression tests for URG and FRG samples at ages of 1, 7, 14 and 28 days, respectively. Again, FRG specimens exhibited higher

compressive strength than URG samples at all studied aging times. Also, FRG samples evidenced higher capacity of deformation than URG presenting higher level of ultimate strains. This behavior is associated with an increment in ductility capacity of the FRG specimens.



**Figure 4.** Compression stress vs. strain curves and envelopes for clay brick powder-based geopolymer specimens tested at different ages: (a) 1 day age test; (b) 7 days age test; (c) 14 days age test; (d) 28 days age test.

The research for samples based on fly ash was performed after the 28 days. The results of compressive strength tests are presented in table 2.

**Table 2.** Compressive strength of fly ash-based geopolymer after 28 days.

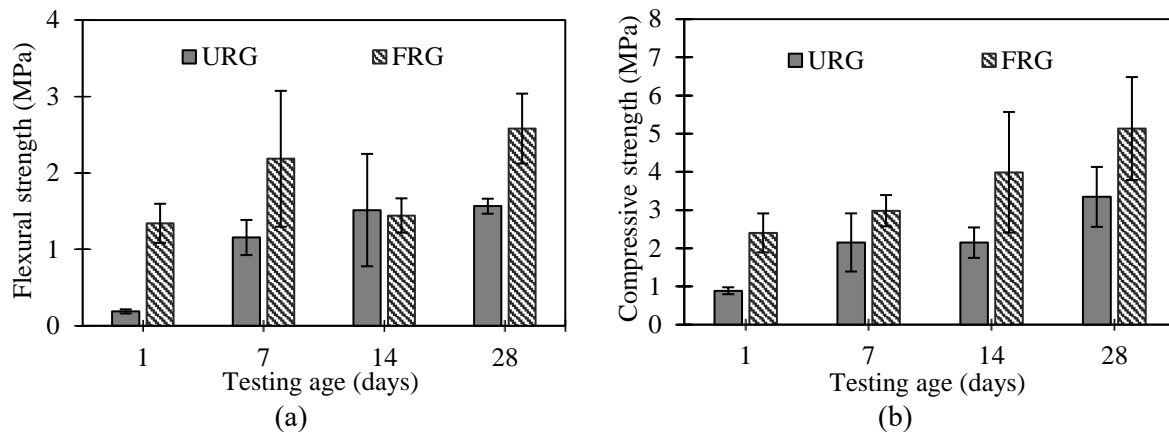
Sample	MPa	StD
Geopolymer without reinforcement	42.70	3.381
Geopolymer with flax fibers (1%)	44.64	4.047

The results for samples with reinforcement and without are very similar. Even though results for samples with reinforcement are slightly better, there are no statistically significant differences.

#### 4. Discussion of results

In order to illustrate the influence of flax fiber reinforcement regarding flexural and compressive strengths, two column graphs with error bars are given in Figure 5a and b. Values of flexural and compressive strengths presented in Figure 5 are the arithmetic mean of peak values for each bending stress vs. displacement and compression stress vs. strain curves shown in Figure 3 and 4, respectively. For data processing, the Interquartile Range (IQR) criterion was used to discard outliers and work with more representative values. Even though data were filter with IQR criterion, error bars display high values of standard deviation. High variability of data could be explained by differences during manual production i.e. hand-mixing and hand-operated vibration of geopolymer paste. Both column graphs also show the development of flexural and compressive strengths over time, reaching their maximum values at 28 day. However, FRG samples at 14 days exhibited a flexural strength much lower than 7-day-age

FRG, thus, it did not follow the marked trend (see Figure 5a) so 14-day-age FRG flexural strength were taken as an inconsistent value. Column graphs in Figure 5 reveal the large influence of the linen fiber reinforcement on the mechanical properties of geopolymer matrices. Addition of linen fibers to produce FRG composites caused an increase in flexural strength respect to URG of 605%, 89% and 64% at 1, 7 and 28 days, respectively. This improvement in flexural strength due to natural fiber reinforcement has been reported by other researches [5,7]. Despite the fact that the main objective of fiber reinforcement is to provide post-cracking ductility [15], compressive strength also increased significantly by the presence of linen fibers. FRG composites evidenced an increase in compressive strength of 170%, 39%, 86% and 53% in comparison to samples with no fibers at 1, 7, 14 and 28 days, respectively.



**Figure 5.** Summary of mechanical test results for clay brick powder-based geopolymer specimens tested at different ages: (a) Flexural Strength; (b) Compressive Strength.

In case of composites based on fly ash it is possible also observe the positive influence of flax fibers. The composites with fibers have better mechanical properties flexural strength (table 1) as well as compressive strength (table 2). Additionally the fragile is more ductile. The SEM observation confirms the good coherence linen fibers to matrix.

A comparative study of flax fibers as reinforcement of fly ash and clay brick powder based geopolymers shows that in both cases linen fibers have positive influence for composite properties. The differences of final properties of the composites, in this case, are depending on the matrix properties.

## 5. Conclusions

This work focused on the study of the reinforcement of clay brick powder-based geopolymers and fly ash geopolymers with the addition of natural linen fibers. Mechanical characterization consisted on three-point bending tests and compression tests showed that their addition to the mixture significantly enhanced flexural and compressive strengths when compared with the unreinforced matrices. Fiber reinforcement increased flexural strength by 605%, 89% and 64% at 1, 7 and 28 days, respectively. While compressive strength was upgraded by 170%, 39%, 86% and 53% at 1, 7, 14 and 28 days, respectively. The increasing the mechanical properties for fly ash-based geopolymers was: 161% in case of flexural strength and 104% in case of compressive strength.

Besides strength improvements, bending stress vs. displacement and compression stress vs. strain envelopes evidenced that fiber reinforced geopolymer samples did not exhibit an abrupt decrease of resistance after peak stress was reached, and samples had the capacity to withstand larger deformations and presented a more ductile failure mode than unreinforced matrices. Therefore, the use of natural linen fibers to improve the mechanical properties of geopolymer matrix based on waste materials as clay brick powder or fly ash allows the development of an eco-friendly building composite material with relatively good mechanical properties.

The research results confirms the possibility of modification of geopolymers and alkali activated materials by short natural fibers addition. It is supposed this kind of additive will be suitable also for geopolymers based on, others materials, exemplary slags or natural ashes, because there is only minor difference between the chemical reactions of these process.

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