

# Fly Ash Based Fiber-Reinforced Geopolymer Composites as the Environmental Friendly Alternative to Cementitious Materials

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**Abstract:** Currently geopolymer composites are the promising solution the problem of post processing waste such as fly ashes and slags from energy, mining and metallurgy industries. There are also the most promising alternative for the environment to cementitious materials. This technology is cost-effective, environmental friendly and needs moderate energy to produce. Geopolymers have good compressive strength, durability and thermal resistance. Weakness of this composites is primarily related to relatively low tensile and flexural strength, which limits their use in many areas. The paper describes the mechanical properties of fly ash based geopolymer reinforced with short polypropylene fibers (5–20 vol%). The article aims is to analyze the influence of addition polypropylene fibers on mechanical properties of geopolymers. The empirical part was based on the following tests: compressive strength, flexural strength and microstructure investigations. Results show that the appropriate addition of polypropylene fibers can improve the mechanical properties of geopolymer composites.

**Keywords:** fly ash, geopolymer composites, waste minimization

## 1. Introduction

The importance of energy, mining and metallurgy industries for the economy is evident. However, this industries produce huge amounts of post process waste, which deposit in the environment cause serious problems related to pollution of soil and water. It can be solved by used this waste for the production of new, environmentally safe materials. One of the possibilities is development of geopolymer cements on the base of fly ash and slag as a new construction materials [20, 22, 30].

Geopolymer composites could be used in industrial application, which are attractive for enterprises. Due to their properties such a chemical and thermal resistance and excellent mechanical properties [18, 28] the importance of these materials for the economy is constantly increased. Currently, there are i.e. used in [29]:

- Production of construction materials such as a material for the manufacture of bricks, the fire-resistant wood panels, sandwich panels and walls, energy efficient ceramic tile [22].
- In the industry as a material for various applications, especially in areas where the heat-and fire resistant products are required [17], and in particular, as a construction material used in power station, the heat shields for the space shuttle, as well as fire barriers in the construction industry.
- Protective coating material such as various types of steel.
- The foundries as a component molding, and the material for molds in aluminum foundries.
- Emergency repair runways.
- Material support to the stabilization of toxic waste, including radioactive substances [21].

In comparison to traditional building materials, such as concrete, geopolymers have number advantages. They can operate as well as under normal ambient conditions and in extreme conditions, in which traditional materials wear out quickly and cannot be used at all [10]. In addition, the production of composites, compared to

materials for special applications (working in difficult conditions) is economically more advantageous, including the low energy consumption of the process.

An important factor in the growth of interest of geopolymers is also an increasing public environmental awareness. Geopolymers during production emit much lower greenhouse gases comparing to traditional construction materials such as Portland cement. It is estimated that the production of geopolymers create 4-8 times less carbon dioxide than cement production [29, 31], the process needs twice less energy than the manufacture of Portland cement [20, 30, 34] and it is associated to low emissions of CO<sub>2</sub>, SO<sub>2</sub> and NO<sub>x</sub> [30]. At the same time, these materials obtain better properties than conventional materials, especially [9, 29, 40, 43]:

- High initial strength [24], wherein the standard Portland cement concretes exhibit a compressive strength of between 30 MPa - 60 MPa, and the strength of geopolymers reach up to 100 MPa.
- Reduced shrinkage and low thermal conductivity - high dimensional stability.
- A good fire resistance (up to 1000 °C) and no emission of toxic fumes when heated.
- High level of resistance to atmospheric conditions and a variety of acids and salts (also reinforcement geopolymers - with steel addition).
- Adherence to the new and old concrete, steel, glass, ceramics.
- A high capacity for mapping the surface patterns of amplified form.
- Availability of raw materials and lower costs, the possibility manufacturing from waste materials such as fly ash from power plants.
- Reduced energy consumption in the manufacturing process (environment friendly).
- Possibility of hazardous waste immobilization by shutting them in geopolymers composites [21].

The advantages of geopolymers are related to good mechanical properties such as compressive strength and thermal (high flame and heat resistance). The weaknesses are relatively low tensile and flexural strength, which limits their application in many areas. Currently, numbers of research on the feasibility of fiber as filler for geopolymer matrix are conducted. A literature review shows valuable results of experiments with addition to different fibers to geopolymers matrix. The most important research in this area included:

- metal fibers, especially steel - good consistency with the filler matrix and reducing corrosion [7, 36, 37].
- glass fibers, which improved the mechanical properties such as compressive strength composites [30].
- ceramic fibers (silica-alumina) [6], and basalt fibers [11, 26, 27, 39], which provide good mechanical properties also at the high temperatures.
- carbon fibers [16, 17, 25], including the examination of the influence of the carbon fiber composite matrix [24] and the test additive nano-carbon fiber [12].
- polymer additives, which caused a better elasticity and decrease weight of the material. In particular, the work related materials such as poly (vinyl alcohol) - PVA [31, 38], poly (vinyl chloride) - PVC [31, 39], polypropylene - PP [34, 43] and biodegradable polylactide - PLA [32].
- different types of additives "organic", both in the form of fibers, as well as in other forms such as epoxy resins, organic additive [13], cotton [2], cellulose fibers derived from plants (*Phormium tenax*) [3], the cellulose fibers of the paper waste [42] or palm oil additive [23], and the ash derived from the combustion of rice husk [18].

The addition of different fibers to geopolymer matrix generally improves their flexural properties and resistance (especially corrosion). The geopolymers matrix keep good mechanical properties such as compressive strength and thermal resistance. It gives new materials new fields of implementation.

## 2. Material and methods

### 2.1. Material

The fly ash from power station located in Skawina (Poland) was used to prepare geopolymeric matrix of composites. EDS analysis confirmed the possibility of using fly ash as a material to create a geopolymer matrix composites. Samples for initial tests were prepared using sodium and potassium promoter and fibres addition for

5%, 10%, 15% and 20% by volume of the composite. Characteristics of PP fibres (PM 12/32, acc. DIN EN 14889-2): density:  $\gamma = 0.91 \text{ tons/m}^3$ , module E:  $\sim 4000 \text{ N/mm}^2$ , tensile strength:  $\geq 300 \text{ N/mm}^2$ , flash point:  $> 320 \text{ }^\circ\text{C}$ , melting point:  $\sim 160 \text{ }^\circ\text{C}$ , length:  $\ell = 12 \text{ mm} \pm 10\%$ , diameter:  $d = 32 \text{ } \mu\text{m} \pm 50\%$ , cross section: round.

## 2.2. Preparation of geopolymer composites

19 different configurations of geopolymer matrix (sodium and potassium-based) were prepared on the basis of literature sources [1, 4, 5, 8, 9, 10, 14, 15, 19, 24, 32, 35, 41, 42], research conducted at the Institute of Materials Science and Engineering [30], and data on chemical composition of fly ash from a supplier (power station). The values for the different configurations are presented in Table I.

TABLE I: The Molar Ratios of the Prepared Compositions Geopolymer Matrix

The molar ratios of ash + Na <sub>2</sub> O + sodium water glass				
SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O/SiO <sub>2</sub>	Na <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O/Na <sub>2</sub> O	W/S
6.39 – 7.17	0.10 – 0.15	0.64 – 1.05	7.56 – 13.86	0.17 – 0.30
The molar ratios of ash + K <sub>2</sub> O + potassium water glass				
SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O/SiO <sub>2</sub>	K <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O/K <sub>2</sub> O	W/S
6.55 – 7.17	0.09 – 0.12	0.58 – 0.66	10.32 – 13.89	0.23 – 0.30

Selected composites were prepared in three versions: in normal conditions ie. at ambient temperature, stored in an open container in a desiccator (high humidity) and heat at a temperature of 65<sup>o</sup>C for a period of 7 days.

Geopolymers were visually assess - first after 24 hours, secondly after 7 days and finally 1 month after and compressive strength test was conducted. The few matrix for the further test (Table II) and the way of manufacturing (heating) were chosen. Selection was based on the following criteria:

- Results of initial compressive strength tests carried out after 1 month (for 2 - 3 samples).
- Surface appearance (cracked or "patches" on the surface).
- Miscibility of the components of a given composition.

TABLE II: Selected Compositions of Geopolymer Matrix

The molar ratios of ash + Na <sub>2</sub> O + sodium water glass					
No.	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O/SiO <sub>2</sub>	Na <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O/Na <sub>2</sub> O	W/S
01	6.86	0.12	0.82	11.34	0.27
04	6.55	0.12	0.80	10.40	0.27
12	6.55	0.11	0.69	13.86	0.32
13	6.55	0.11	0.75	9.51	0.23
The molar ratios of ash + K <sub>2</sub> O + potassium water glass					
No.	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O/SiO <sub>2</sub>	K <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O/K <sub>2</sub> O	W/S
10K	6.71	0.10	0.70	11.66	0.25

Next, initial samples were prepared with addition polypropylene fibres.

## 2.3. Measurements

The compressive strength test, due to the lack of standards for geopolymer materials was carried out according to the methodology described in the standard EN 12390 - Part 3:2001. Research - Concrete compressive strength. Samples used to the compressive strength test had cylindrical shape with dimensions:  $\varnothing = 36.5 \text{ mm}$   $h = 70 \text{ mm}$ . Similarly, the test of flexural strength, due to the lack of standards for geopolymer materials, was carried out according to the methodology described in the standard EN 12390 - Part 5:2009 Flexural strength test specimens. Tests were performed on an universal testing machine - single-point load (Instron type 4465). Samples used to the compressive flexural test had dimensions: 60 mm x 10 mm x 10 mm. The distance between the lower supports was 50 mm. The images of structure were taken from samples after compressive strength test using scanning electron microscope SEM with different magnification.

### 3. Results

Microstructure research has been performed by scanning electron microscopy (SEM) on samples previously broken while compressive strength test. It can be noticed that the geopolymerization process has successfully occurred and all researched samples proved a mainly amorphous structure.

Research on SEM have enabled the analysis of complex structures geopolymers and determine their elemental composition. Research gives a preliminary information on the consistency of fiber (filler) and the geopolymer matrix. In the matrix, there are zeolite structure type G - Na – chabazite (Fig. 1). Obtaining such structures significantly improves the mechanical properties of the material (compressive strength increases).

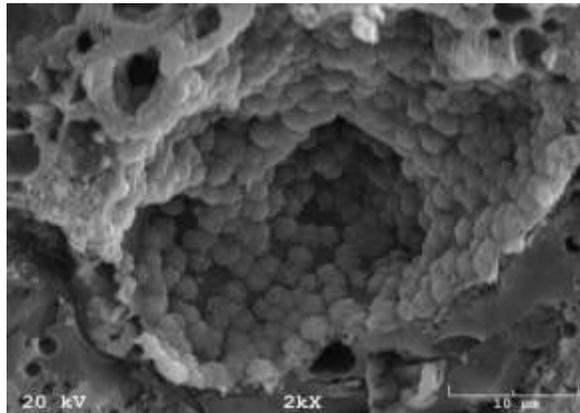


Fig. 1: SEM image obtained with zeolites type G - Na - chabazite, at a magnification of 2 000

SEM observations have also been used to examine the adhesion of the fibers geopolymer matrix (Fig. 2). Researched samples showed that polypropylene fibers are able to sufficiently adhere to the matrix.



Fig. 2: SEM image obtained with the addition of 5% PP fibers at a magnification of 500 x

Strength research allowed to determine the mechanical properties and practical applicability of tested materials. Tests were conducted on samples in the following time interval: after 1 week, 30 days (one month), 3 and 6 months. Samples after the preparation were stored in the oven for 1 week at 65<sup>0</sup>C. Each test was conducted on the 15 samples. Selected research results, after a period of 6 months, are presented in Table III.

TABLE III: Compressive Strength After 6 Months For Selected Samples

No.	Composite 1 (04)		Composite 2 (10K)		Composite 3 (12)		Composite 4 (13)	
	kN	Mpa	kN	Mpa	kN	Mpa	kN	Mpa
The best result	38.50	36.81	43.00	41.12	61.00	58.33	65.50	62.63
average	25.80	24.67	26.21	25.06	33.87	32.38	42.78	40.90

The strengthening of the geopolymer in the scope of time was also analyzed. The strengthening of the geopolymer under the influence of time were rising. It has 12 MPa after 1 week and about 25 MPa after 6 months.

The preparation of the samples, for flexural strength, without the addition of fiber was impossible, due to the limited flexural strength (they had broken before the tests started). For the samples with fibre, each test was carried out on 5 samples. The best result was observed on the samples with 15% of added PP fibers as filler (Table IV and V).

TABLE IV: The Results Of Flexural Strength Test Sample No. 01 On The Base With The Addition Of PP Fibers

	Flexural strength [MPa]	Young modulus [MPa]	Tensile strength at yield [MPa]	Strain [mm/mm]
5% PP fiber addition	2.14	896.99	1.04	0.03
10% PP fiber addition	3.30	966.94	2.00	0.03
15% PP fiber addition	7.66	1,584.59	7.09	0.03
20 % PP fiber addition	7.92	1,847.85	7.35	0.03

TABLE V: The Results Of Flexural Strength Test Sample No. 04 On The Base With The Addition Of PP Fibers

	Flexural strength [MPa]	Young modulus [MPa]	Tensile strength at yield [MPa]	Strain [mm/mm]
5% PP fiber addition	4.95	5,734.92	0.39	0.00
10% PP fiber addition	4.82	4,669.81	0.18	0.01
15% PP fiber addition	7.36	4,286.45	2.47	0.03
20 % PP fiber addition	5.62	5,154.00	1.00	0.01

#### 4. Discussion and Conclusions

New fiber-reinforced composite materials based on geopolymer matrix have been researched. The scanning electron microscopy observation proved that, in some samples there are the zeolite structure type G - Na - chabazite. This type of structures significantly improves the mechanical properties of the material (compressive strength increases), which can be seen in results of strengthening tests. The scanning electron microscopy observation also allowed to state that polypropylene fibers have good adhesion with the matrix. The strengthening of the geopolymer in the scope of time was also analyzed. Results show that geopolymers have high initial strength, which increase in time. The addition of fibers does not change it significantly.

The addition of polypropylene to geopolymer matrix improves the flexural strength of these materials. Moreover, obtained results showed that the composites with addition of 15 % vol. of reinforcing fibers have the best flexural strength among the analyzed compositions.

Currently geopolymer composites are the most promising alternative for the environment compering to the ordinary Portland cement and cementitious materials for construction purposes. Thanks to addition such as polypropylene fibres they can be applied to new area, especially this with required not only reasonable compressive properties, but also good elasticity.

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