

Vegetable origin-natural fibres for reinforcement of geopolymer composite

Fibre naturale de origine vegetală pentru armarea compozitului geopolimeric

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Abstract. *The current trend to decrease greenhouse gas emissions through increasing the efficiency of fossil fuel consumption implies the use of lighter materials in building construction, with a lower carbon footprint compared to materials commonly applied in the present. Composites prepared from vegetable fibres constitute a viable solution to reach this objective. The precursors adopted for the production of the geopolymer composite were fly ash, granulated blast furnace slag, and rice husk ash, as alumina-silicate materials with cementitious properties, to which hemp fibres were added with a major role in reinforcing the composite. Results showed increasing the compression and flexure strength.*

Key words: *vegetable fibre, geopolymer composite, reinforcement, strength, hemp fibre.*

Rezumat. *Tendința actuală de a reduce emisiile de gaz cu efect de seră prin creșterea eficienței consumului de combustibil fosil implică utilizarea materialelor mai ușoare în construcția clădirilor, cu amprenta de carbon mai redusă, comparabil cu materialele aplicate în prezent în mod obișnuit. Compozitele preparate din fibre vegetale constituie o*

soluție viabilă pentru atingerea acestui obiectiv. Precursorii adoptați pentru producerea compozitului geopolimeric au fost cenușa zburătoare, zgura de furnal granulată și cenușa cojilor de orez, ca materiale aluminosilicaticice cu proprietăți de cimentare, la care au fost adăugate fibre de cânepă cu rolul major de armare a compozitului. Rezultatele au arătat creșterea rezistenței la compresiune și încovoiere.

Cuvinte cheie: fibră vegetală, compozit geopolimeric, armătură, rezistență, fibră de cânepă.

1. Introducere

The current trend to reduce greenhouse gas emissions by increasing the efficiency of fossil fuel consumption implies the use of lighter materials in the construction sector, with more reduced carbon footprint compared to the materials commonly used today. Composites made from natural plant-based fibres represent an interesting solution to achieve this objective. According to [1], this fibre type comes from renewable resources such as flax, hemp, jute, cotton, coconut, ramie, sisal, kenaf, etc. placed in a matrix derived from biomass.

The fibres contain variable proportions of cellulose, hemicellulose, and lignin. Cellulose and hemicellulose are polymers containing glucose and polysaccharides, respectively. Unlike these, lignin is an amorphous mixture of aromatic polymers and phenylpropane monomers [2].

Due to their excellent chemical and thermal resistance, high adhesion and mechanical strength properties, thermosets are widely used as matrices in bio-composites. Although it seems that thermoset matrices are more difficult to recycle at the end of their life, a more environmentally friendly matrix can be obtained by adding biodegradable fillers [3].

The prices of the main natural fibres available in the world vary according to [4] within quite wide limits between 200-4200 USD/ton. Among the cheapest, coir, kenaf, and pineapple are mentioned (between 200-550 USD/ton), followed by sisal and bamboo (600-900 USD/ton). Hemp fibre has values considered average (between 1000-2100 USD/ton), while cotton and flax reach the highest price ranges (between 1500-4200 USD/ton).

According to the literature [5], natural fibres exhibit high tensile strength, but their modulus of elasticity is slightly deficient. Several advantages of using natural fibres have been experimentally identified by researchers: the setting time of the composite cement is low as a result of releasing acidic components of fibres, the cement hydration is avoided due to the presence of hemicellulose and lignin, most natural fibres naturally decompose at end-of-life, the high availability and durability of resources from which natural fibres come, the low denseness of fibres favouring preparing lightweight materials, and the price of natural fibres mentioned above is much lower than that of glass, carbon or polymer fibres, the price of carbon fibres exceeding 12,000 USD/ton.

Traditionally, the techniques applied until recently were those based on reinforcing fibers from steel and synthetic materials. Currently, economic reasons make these fibre types no longer acceptable. Also, their manufacture is based on the consumption of non-renewable resources and emits CO₂ into the atmosphere [6].

Under the conditions in which of composite materials progress is in continuously growing, developing the new construction materials could not be left behind. In principle, the addition of fibres aims to prevent tensile cracking of materials, and improve other characteristics such as impact resistance and thermal insulation. Vegetable fibres whose structures include both crystalline and amorphous areas create problems related to porosity and water absorption, generating weak bonding between fibres and matrix [6].

A new challenge in the construction field in the last decade has emerged in the form of using vegetable materials as bio-aggregates in ecological concrete. As a result of recent research, innovative applications of natural fibres in construction have been promoted. Among the natural fibres mentioned above, hemp has been adopted for the manufacture of the new hemp concrete, considering its special peculiarities. Thus, fibres constitute about 33 % from the entire stem material, the remainder being the woody core. These components are processed into pieces with lengths between 2-25 mm called "hurds". Hemp does not need special protection against parasites because it does not contain nutrients. According to [7], the new hemp-based concrete is fireproof.

Results of research undertaken in the last two decades on the use of vegetable fibres as reinforcement in cement-matrix composites were presented in [8]. Applications of composites with reinforced vegetable fibre in structural and non-structural constructions to improve their mechanical properties were highlighted in this paper. It has been found that short vegetable fibres used as internal reinforcement increase the flexural and tensile strength as well as the hardness of cement-based composites. Long vegetable fibres are mainly used as external reinforcement, increasing the strength and ductility of composites. In accordance with authors, the information regarding the durability of fibres incorporated into cement matrices is still insufficient, leading to the deferral of their application in structural building components requiring high resistance to mechanical stress.

Reinforcing with hemp fibres (in weight ratios of 3 and 9 %, respectively) of a geopolymer material based on fly ash and red mud was experimentally tried [9]. The geopolymer matrix was made of 70 % fly ash with a grain size under 21 µm and 30 % red mud with a grain size under 75 µm. The aqueous solution including sodium silicate, sodium hydroxide in the form of pellets dissolved in deionized water, and hemp fibres was poured over the solid mixture. It has been observed that a hemp fiber content higher than 9 % adversely affects the homogeneity of the fibre distribution in the matrix, disturbing the mechanical properties of the geopolymer. Also, increasing the fibre content causes a decrease in workability, allowing air bubbles to enter and to be trapped in the geopolymer mass during the pouring of the paste into the mould. On the other hand, loading hemp fibres in the mentioned range of 3-9 % does not allow the formation of voids and uniform dispersion of fibres can be obtained. In general, it

was found that by increasing the proportion of added fibres, the compressive strength of the geopolymer composite tends to slightly decrease.

In another work [10], the effect of the binder type used to produce hemp-lime concrete on its mechanical strength and durability was investigated. The following types of binder were tested: hydrated lime, hydraulic lime, and Portland cement. Metakaolin and granulated blast furnace slag as pozzolanic materials were added to the manufacturing mixture due to their rapid setting and high reactivity. Due to its role on improving mechanical properties, hydroxymethyl cellulose has been used in optimal ratio of 0.8 % [11] as well as hemp fibres. The mixing procedure consisted in creating a slurry with binder and water before adding the fibres. The fresh concrete was poured into a mould and lightly pressed. Then, the material removed from the mould was placed in a curing chamber at about 16 °C and relative humidity between 50-60 %. The compression strength of the product had values in the range of 0.02-0.04 MPa after 5 days and 0.29-0.39 MPa after 28 days.

In a recent work [12], the influence of length and amount of hemp fibres on the properties of composite based on blast furnace slag reinforced with fibres was investigated. Thus, fibres with length of 10 and 20 mm, in proportions of 0.5; 1; 2; and 3 % from the cement weight were tested. Determining the compression and flexure strengths were carried out after 7 and 28 days. Values of the two resistance types after 28 days varies between 1.28-2.73 MPa (in the case of compression) and between 0.48-1.65 MPa (in the case of flexure), Determining the sample resistance to temperatures within the limits of 250-750 °C showed that the use of 20 mm hemp fibres led to increasing the compression and flexure strength, but also contributed to increase the heat conductivity.

An authors' team of the current paper recently investigated [13] a technique for manufacturing a reinforced concrete composite with hemp fibres using as a binder a mixture composed of alumina-silicate industrial by-products (fly ash and granulated blast furnace slag) as well as residual building concrete from demolition. Hemp fibres were added to the starting mixture, their weight proportion varying within the limits of 2.44-8.67 %. By mixing, a slurry resulted, being poured into a mould. Introduced into an laboratory electric oven, the mould containing the paste was heated with hot air at 80 °C for 4 hours. The hemp concrete specimen removed from the mould was subjected to the curing process at ambient temperature for 28 days. The experimental results indicated the following values: density between 303-350 kg·m⁻³, porosity in the range of 83.5-85.8 %, heat conductivity in the range of 0.084-0.102 W·m⁻¹·K⁻¹, compressive strength between 7.6-8.5 MPa, and water uptake in the range of 2.2-2.9 vol. %. The optimal version was identified as the one in which the hemp fibre amount was 5.66 g per 100 g solid mixture. The product corresponding to the optimal version had the density of 327 kg·m⁻³, heat conductivity of 0.094 W·m⁻¹·K⁻¹, and compression strength of 8.1 MPa.

The current work presented below brought several changes in the composition of the starting materials and the peculiarities of preparing and hardening process of the geopolymer composite reinforced with vegetable fibres, aiming to improve its mechanical properties.

2. Materials and methods

Materials adopted for manufacturing geopolymer composite reinforced with vegetable fibres were coal fly ash, ground granulated blast furnace slag, and rice husk ash, all being cementitious materials with pozzolanic properties, suitable for producing geopolymers. The combination of fly ash and granulated blast furnace slag in this process type is quite frequently used, according to the literature, while the addition of rice husk ash to this mixture is much less applied.

Coal fly ash was the main material used in the solid mixture of the geopolymer composite, coming as a by-product of the energy producing industry. Fly ash is the result of burning the coal in boilers of the Paroseni thermal power plant, being captured in its electrostatic precipitators. The grain size of this by-product is below 250 μm . The ash was purchased by authors about 10 years ago, at a period when anthracite was the used coal type. For this reason, the fly ash is classified as class F, characterized by low proportion of calcium oxide. The waste purchased from Paroseni was subjected to mechanical processing for reducing its grain size under 80 μm . The chemical composition of the ash was determined through analysis in the AXIOS type X-ray fluorescence spectrometer from the Romanian Metallurgical Research Institute. The oxide composition of fly ash included the following components: 50.2 % SiO_2 , 25.6 % Al_2O_3 , 3.5 % CaO , 3.3 % MgO , 5.9 % Na_2O , 4.0 K_2O , 6.9 % Fe_2O_3 .

Granulated blast furnace slag procured from ArcelorMittal Galati (Romania) 12 years ago is a by-product of the metallurgical industry. Granulated slag is obtained by pouring hot slag into a water bath, resulting in the form of spheres with diameter between 2-6 mm. For use in the manufacture of the geopolymer composite, the granulated slag was ground in a ball mill to particle size below 70 μm . The chemical composition of the slag includes 36.5 % SiO_2 , 11.7 % Al_2O_3 , 41.5 % CaO , 5.7 % MgO , 0.5 % MnO , 0.9 % Fe_2O_3 , 0.3 % Na_2O , 0.4 % K_2O .

Rice husk ash is a by-product of the rice milling industry, resulting from the burning or incineration of rice husks at high temperature. The process of controlled burning in a furnace or reactor reduces the rate of silica crystallization, simultaneously increasing the amorphous silica content. Burning at temperatures between 550-800 $^\circ\text{C}$ favours the formation of amorphous silica, while higher temperatures lead to the formation of crystalline silica. The particle size of rice husk ash is within the limits of 5-10 μm and the silica content of the ash varies between 84.4-93.1 % [14].

The alkaline activation of the above-mentioned alumina-silicate materials involved the formation of a liquid mixture including NaOH in the form of pellets dissolved in distilled water (the molarity of the solution being 12M) and a sodium silicate solution (38 % concentration). Also, short hemp fibres were introduced and mixed into the alkaline liquid solution.

NaOH pellets were purchased from the market. The basic properties of NaOH pellets are high solubility, high alkalinity, non-volatility, and hygroscopic nature [15]. Also, sodium silicate solution with 38 % concentration was procured from the market. Sodium silicate or water glass is produced by melting quartz sand together sodium carbonate. Most commonly, water glass is a silicate binder dissolved in water. The

main suppliers of water glass are in Germany (Bockling GmbH, Caldic Deutschland Chemie B.V.), Poland (Chematex Sp.z.o.o.), Austria (Deuring GmbH), China (MC Glass). Short hemp fibres originating from the United States were also commercially purchased. Hemp production has significantly increased in the last decades due to the intense demand for environmentally friendly and sustainable products. Hemp fibres are concentrated in the stem of the plant, so harvesting the stems and separating the fibres are the main fibre recovery operations.

The method of turning alumina-silicate waste as inert materials into geopolymer composites was based on their alkaline activation with a liquid mixture of NaOH and water glass. Through alkaline activation, the geopolymerization reaction is initiated and developed leading to the formation of a geopolymer, according to the discovery of the French researcher J. Davidovits [16].

Peculiarities of the geopolymerization process of alumina-silicate minerals are presented in [17]. Geopolymers having an amorphous microstructure are formed through co-polymerization of individual aluminum and silicates species that come from the dissolution of precursor materials containing silicon and aluminum in the presence of soluble alkali-metal silicates. Alumina-silicate materials are largely soluble in the alkaline activator solution, the dissolution degree being higher in NaOH solution compared to KOH solution. It was found that materials with a higher dissolution degree facilitate to obtain higher compression strength after geopolymerization. On the other hand, the use of KOH solution instead of NaOH solution favours the development of the geopolymerization process.

Geopolymerization involves a non-homogeneous chemical reaction between the alkaline solution and alumina-silicate oxides. Thus, conditions are created for the formation of semi-crystalline polymer systems containing Si-O-Si and Si-O-Al bonds. Poly(sialates) are known as ring polymers and chain with Al^{3+} and Si^{4+} [18]. J. Davidovits, the inventor of geopolymer, has found that the by-product of the reaction of alkaline activator with aluminum and silicon could be used for producing geopolymers.

The methods used for investigating the composite specimen features were the following. The geopolymer composite density was determined by Archimedes' method through the water intrusion technique (ASTM D792-20). Porosity was calculated as a percentage of the difference between the estimated density of vegetable fibre composite without voids and the measured density including the void volume related to the composite density without voids, in accordance with NE 012-99 (Code 1999). The compression strength was measured with a 2000 kN-compression fixture Wyoming Test Fixture [19] and the flexural strength was determined by carrying out the three-point bend test on the specimen (SR EN ISO 14125:2000). Heat conductivity at room temperature was determined with HFM 446 Lambda apparatus using the heat-flow method (SR EN 1946-3:2004). Water uptake was determined by the immersion method of the sample under water for 24 hours (ASTM C948) at the end of the curing process (28 days) and the specimen mass was identified through weighing. The microstructural appearance of geopolymer composite specimens was investigated with the Biological Microscope model MT5000.

3. Results and discussion

Four experimental versions were chosen for making the hemp fibre reinforced geopolymer composite. The quantity and composition of materials that composed up the starting mixture are shown in Table 1.

Table 1

| Composition | Experimental version (g) | | | |
|-------------------------------|--------------------------|-------|-------|-------|
| | 1 | 2 | 3 | 4 |
| Coal fly ash | 45.2 | 44.0 | 42.8 | 41.6 |
| Granulated blast furnace slag | 29.5 | 29.0 | 28.5 | 28.0 |
| Rice husk ash | 22.7 | 22.3 | 21.9 | 21.5 |
| Hemp fibre | 2.6 | 4.7 | 6.8 | 8.9 |
| Total solid | 100.0 | 100.0 | 100.0 | 100.0 |
| Water glass solution | 30.5 | 30.5 | 30.5 | 30.5 |
| 12M NaOH solution | 12.0 | 12.0 | 12.0 | 12.0 |
| Water addition | 7.5 | 7.5 | 7.5 | 7.5 |
| Total liquid | 50.0 | 50.0 | 50.0 | 50.0 |
| Total slurry | 150.0 | 150.0 | 150.0 | 150.0 |

In accordance with the data in Table 1, the amount of coal fly ash varied between 41.6-45.2 g, granulated blast furnace slag had values between 28.0-29.5 g, and rice husk ash within the limits of 21.5-22.7 g, all being decreasing, while hemp fiber had increasing values between 2.6-8.9 g. The total solids of the mix in all four versions were set to be 100 g. Liquid components were kept at constant values: Water glass solution (30.5 g), 12M NaOH solution (12 g), and water addition (7.5 g), and the total of their value was 50 g. The water glass/NaOH ratio was also constant for all versions, i.e. 2.54.

Applying the requirements of the adopted production method, including also the parameters of the curing process of the fresh materials (at 28 days), four cured specimens of hemp fiber geopolymer composite were obtained, shown in Fig. 1.





Fig. 1. Images of the surface of geopolymer composite specimens reinforced with hemp fibre
a – version 1; b – version 2; c – version 3; d – version 4.

Physio-mechanical and heat characteristics of composite specimens are presented in Table 2.

Table 2

Physio-mechanical and heat characteristics of the hemp fibre geopolymer composite specimens

| Characteristic | Version 1 | Version 2 | Version 3 | Version 4 |
|--|-----------|-----------|-----------|-----------|
| Density ($\text{kg}\cdot\text{m}^{-3}$) | 352 | 335 | 317 | 300 |
| Porosity (%) | 83.0 | 83.9 | 84.8 | 85.9 |
| Heat conductivity ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) | 0.105 | 0.098 | 0.091 | 0.083 |
| Compression strength (MPa) | | | | |
| - after 7 days | 2.5 | 2.6 | 2.1 | 1.5 |
| - after 28 days | 6.3 | 6.8 | 5.5 | 4.7 |
| Flexural strength (MPa) | | | | |
| - after 7 days | 1.2 | 1.0 | 0.9 | 0.6 |
| - after 28 days | 1.7 | 1.6 | 1.2 | 0.8 |
| Water uptake (vol. %) | 3.0 | 2.8 | 2.7 | 2.5 |

Table 2 presents the evolution of geopolymer composite features between the four experimental versions made by slight decrease in the proportion of the main components of the mix (fly ash, slag, and rice husk ash) and the quite pronounced increase in the amount of hemp fibre. A significant decrease in the density of specimens (from 352 to 300 $\text{kg}\cdot\text{m}^{-3}$) was observed simultaneously with an increase in their porosity (between 83.0-85.9 %). Interesting effects on the compressive strength were felt after 28 days of curing. Thus, first, the strength value slightly increased, then in the case of the last two versions it had a clear decreasing trend, still remaining in remarkably high value range (up to 4.7 MPa). The improvement in compressive strength at early age (after 7 days) was achieved slowly in all tested versions with values of 2.5-2.6 MPa in the case of the first two versions and 1.5-2.1 MPa in versions

3-4. On the other hand, the flexure strength value registered a decrease from 1.7 to 0.8 MPa after 28 days in the ascending order of experimental versions. The measurement results carried out at early age showed that the flexure strength values reached high levels compared to the values at the end of the curing process (between 62.5-75 %). Water uptake measured in the case of geopolymer composite reinforced with hemp fibres was in the range of 2.5-3.0 vol. %, being considered at a normal level for this material type.

Microstructural peculiarities of geopolymer composite specimens reinforced with hemp fibre are shown in Fig. 2.

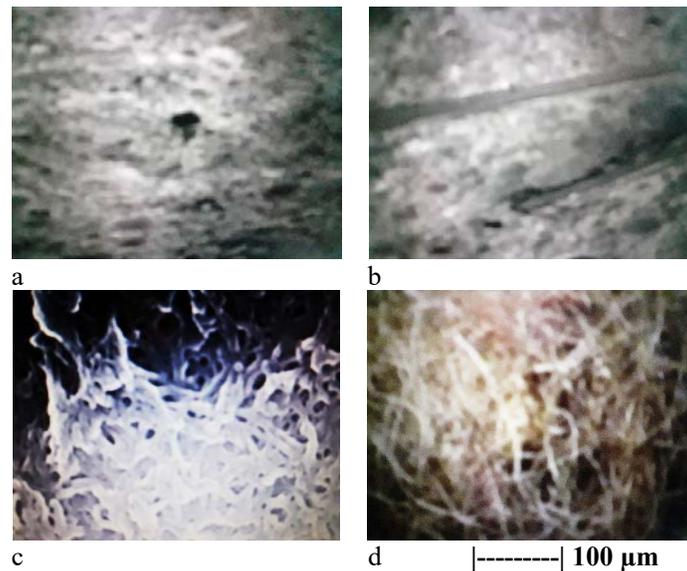


Fig. 2. Microstructural peculiarities of geopolymer composite specimens reinforced with hemp fibre

a – version 1; b – version 2; c – version 3; d – version 4.

The length of fibres used in this experiment was within the limits of 13-19 mm. The pictures in Fig. 2 show the increase conditions of hemp fibres ratio between versions 1-4.

The environmentally friendly and economic role of using various alumina-silicate industrial by-products in the production process of geopolymer composites as substitutes for traditional cementitious and pozzolanic materials (Portland cement) has already been evaluated in recent decades. These wastes, as well as other natural alumina-silicate materials nominated and claimed by the inventor of geopolymers J. Davidovits, have allowed the manufacture, through various combinations in the starting mixture, of new construction materials with excellent physical, mechanical, chemical, and thermal features.

Vegetable fibres have already confirmed their effect on increasing the strength of these composites. In the current work, coal fly ash, granulated blast furnace slag, and rice husk ash were adopted as precursor materials and hemp fibres as reinforcing

vegetable fibres. Regarding hemp fibre, this type of vegetable fibre is probably one of the most appreciated materials for reinforcing geopolymer composites.

Of course, it is well known that vegetable fibres do not have a reinforcing effect comparable to that of steel, glass, or carbon fibres, which were used intensively until recently, but the price of these fibres is very high, incomparable to that of vegetable fibres. This is the main reason why there is a clear trend in the world to adopt vegetable fibres in manufacturing processes of composites.

According to [5], natural fibres show high tensile strength, although their modulus of elasticity is deficient. Based on experimental determinations, several advantages of using natural fibres should be mentioned: availability and durability of resources containing natural fibres, setting time of the composite cement is low, cement hydration is avoided due to hemicellulose and lignin presence, most natural fibres decompose at end-of-life, contributing to environmental protection.

4. Conclusions

The paper aimed at the production of a geopolymer composite based on alumina-silicate precursor materials (fly ash, granulated blast furnace slag, and rice husk ash) from industrial by-products. The composite was reinforced with vegetable fibres (hemp fibres). The inert material mixture was activated with a liquid alkaline solution containing NaOH dissolved in deionized water and water glass (sodium silicate). Therefore, the work fell within the modern trend of using cheap residual materials, including also vegetable reinforcement fibres, incomparably less expensive than steel, glass or carbon fibres intensively utilized until now. Among the many types of vegetable fibre available in the world, short hemp fibres with a length between 19-29 mm were chosen. The advantages of their use include some peculiarities such as durability, versatility, and availability. Lately, the interest in this plant and especially, the recovery of its fibres is growing. The method for producing the hemp fibre-geopolymer composite tried in this experiment generally adopted the principles expounded and claimed by the inventor of geopolymers the French researcher J. Davidovits at the end of the 20th century and the beginning of the new millennium. The coal fly ash as well as the metallurgical slag are industrial by-products suitable for the manufacture of geopolymers, being recommended by the French inventor. The rice husk ash used by authors in the experiment described in this paper constitutes another adequate, but less applied, waste type. Apart from the particular combination of material precursors, another element of originality of the work is represented by the adopted ranges of their quantity values. The test results showed the possibility of obtaining relatively high values of compressive strength after the 28-day curing process (6.8 MPa in the case of experimental version 2). The peculiarity of the flexural strength was the high level of the values of this resistance at early age (after only 7 days) compared to its final values after 28 days.

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