Michał Łach, Agnieszka Przybek, Kinga Setlak, Patrycja Bazan, Maria Hebdowska-Krupa
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RESISTANCE OF LOW-EMISSION GEOPOLYMER BINDERS WITH FIBERS TO AGGRESSIVE EXTERNAL FACTORS

ODPORNOŚĆ NISKOEMISYJNYCH SPOIW GEOPOLIMEROWYCH Z WŁÓKNAMI NA DZIAŁANIE AGRESYWNYCH CZYNNIKÓW ZEWNĘTRZNYCH

Michał Łach*, Agnieszka Przybek, Kinga Setlak, Patrycja Bazan, Maria Hebdowska-Krupa
Cracow University of Technology, Poland

Abstract

Materials called geopolymers are considered an alternative to common hydraulic binders, but they have certain limitations in many applications due to their brittleness. The use of fibers to reinforce geopolymers can bring the expected results by increasing their compressive strength. This paper presents the results of accelerated durability tests of geopolymers based on coal shale and fly ash reinforced with natural fibers (1% by mass). The results of testing the resistance of such composites to UV radiation, variable temperature cycles and the results of the thermal conductivity coefficient are presented.

Keywords: geopolymer, natural fiber, composite, aging test; thermal resistance, UV resistance

Streszczenie

Materiały zwane geopolimerami uznawane są za alternatywę dla powszechnych spoiw hydraulicznych jednak posiadają one pewne ograniczenia w wielu zastosowaniach ze względu na ich kruchość. Zastosowanie włókien do zbrojenia geopolimerów może przynieść oczekiwane rezultaty zwiększając ich wytrzymałość na zginanie. W niniejszej pracy zaprezentowano wyniki przyspieszonych badań trwałości geopolimerów na bazie łupków węglowych i popiołu lotnego wzmocnionych włóknami naturalnymi (1% mas.). Przedstawiono wyniki badań odporności takich kompozytów na działanie promieniowania UV, zmiennych cykli temperaturowych oraz przedstawiono wyniki badań współczynnika przewodzenia ciepła.

Słowa kluczowe: geopolimery, włókna naturalne, kompozyty, badania starzeniowe, odporność termiczna, odporność UV

1. INTRODUDUCTION

Geopolymer composites have been known for at least several decades. One of the main motivations for researching and producing this type of material is that it is less harmful to the environment compared to materials based on hydraulic binders such as Portland cement [1, 2]. However, these materials are characterized by high brittleness, so it is necessary to reinforce them with fibers with different properties such as high corrosion resistance, resistance to alkaline environment or high mechanical strength. Different types of fibers such as steel, polymer, basalt, carbon and also natural fibers and increasingly so-called hybrid fibers are tested [3]. Most of the scientific work is concerned with the manufacture

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*Cracow University of Technology, Poland, e-mail: michal.lach@pk.edu.pl

of geopolymer composites reinforced with synthetic fibers [4, 5], but there is a growing interest in the use of natural fibers. Detailed studies are needed in this area to evaluate the durability of such solutions and the effect of the addition of natural fibers on physical and mechanical properties. Studies conducted to date [6-10] allow us to conclude that natural fibers can successfully replace synthetic fibers in geopolymer composites.

The use of natural fibers creates a number of advantages in terms of their availability, biodegradability and diversity, but can also bring a number of limitations related to the durability of these fibers. Studies have shown that geopolymer composites containing 1% ramie, hemp and bamboo fibers and 2% ramie fibers show higher compressive and tensile strengths and lower shrinkage than unreinforced geopolymers, with those containing 1% ramie fibers showing the highest strength and lowest shrinkage [11]. Banana-derived fibers are also used [12], which can realistically improve the aging resistance of geopolymers. Tests conducted under alternating freeze-thaw cycles showed that geopolymer composites containing 1.5 wt.% banana fibers had higher cohesion and less material damage compared to unreinforced samples. As studies have shown, natural fibers added to geopolymers can also help improve insulating properties and thermal energy storage capacity, as well as improve thermal stability [13, 14].

A very important issue related to the possibility of using natural fibers in concretes is to develop a method of manufacturing and introducing these fibers in such a way that the produced composites are resistant to high temperatures. This is particularly important in foamed insulating geopolymer materials, which are characterized by good thermal conductivity values and high thermal resistance up to temperatures of the order of 1000°C [15]. The scientific literature also gives many examples related to problems with the use of natural fibers. For example, the absorbency and moisture sensitivity of natural fibers such as jute, sisal and cotton can be a problem. The addition of such fibers can increase water absorption and chloride penetration through concrete (geopolymer) [16]. In order to increase the possibility of using environmentally beneficial biodegradable and renewable natural fibers, scientific research should focus on evaluating the durability of such fibers in a geopolymer matrix [17]. This is particularly important precisely in the case of alkali-activated composites or geopolymers, where the activator concentrations used are alkaline in nature and the use of such materials in humid environments constantly exposes the fibers present in the composite to harmful alkaline solutions. Alkalis present in geopolymers are capable of dissolving the main components of the fibers, especially lignin and hemicellulose. This can lead to a complete loss of fiber integrity especially with changes in humidity and temperature. In addition, the expansion and contraction of fibers associated with environmental changes results in the formation of tiny microcracks. This can result in complete loss of fiber strength properties and loss of long-term viability of the composites [18].

As presented in a number of scientific papers [19-25], the addition of fibers to binders may involve the need for appropriate treatment to increase their durability or improve the quality of the bond between the fibers and matrix. Cellulose is the compound responsible for the fiber's strength, while lignin and hemicellulose are responsible for its low durability, so it is often necessary to carry out treatments, most often with alkaline solutions, to improve the adhesion of the matrix to the fiber in the contact area. It has been confirmed, for example, that fibers such as açai, jute, sisal, bamboo and curaua can be used after appropriate treatment as a filler in cement matrices [26]. Kenaf fibers are also a promising addition to geopolymers due to their properties [27]. The authors of many works show that the properties of composites with fibers, in addition to their processing, can also be influenced by the way they are extracted from plants [28]. Some chemical modification of the fiber surface (e.g., the use of nano-additives) may also be recommended to improve contact at the fiber-matrix interface [29].

This paper presents the results of selected studies of geopolymer composites with natural fibers conducted to confirm the suitability of natural fibers as reinforcement of geopolymers based on specific precursors found locally in Poland. Rarely used calcined coal shale and ash from lignite combustion were used as precursors. Quantities of these precursors exist in Poland in the tens of millions of tons and their management is of great importance. The composites tested were produced by adding 1% by weight of fibers such as wood wool fibers, field grass fibers, and rafia fibers. The produced composites were subjected to flexural strength tests, accelerated aging tests in a UV chamber, tests of resistance to varying temperature cycles and determination of the thermal conductivity coefficient.

2. MATERIALS AND METHODS

Geopolymer composites were produced using precursors such as calcined coal slate from the heaps of the Janina Mine in Libiąż (TAURON) and lignite combustion ash from the Bełchatów Power Plant. The microstructure of the raw materials is shown in Figure 1 and the oxide composition is presented in Table 1. Microstructure observations showed that the lime fly ash is characterized by irregular particles different from conventional fly ash from pulverized coal boilers (probably the ash contains a small amount of amorphous phase). The microstructure of calcined coal shale is similar to that of metakaolin (calcined coal shale contains about 30-40% metakaolin).

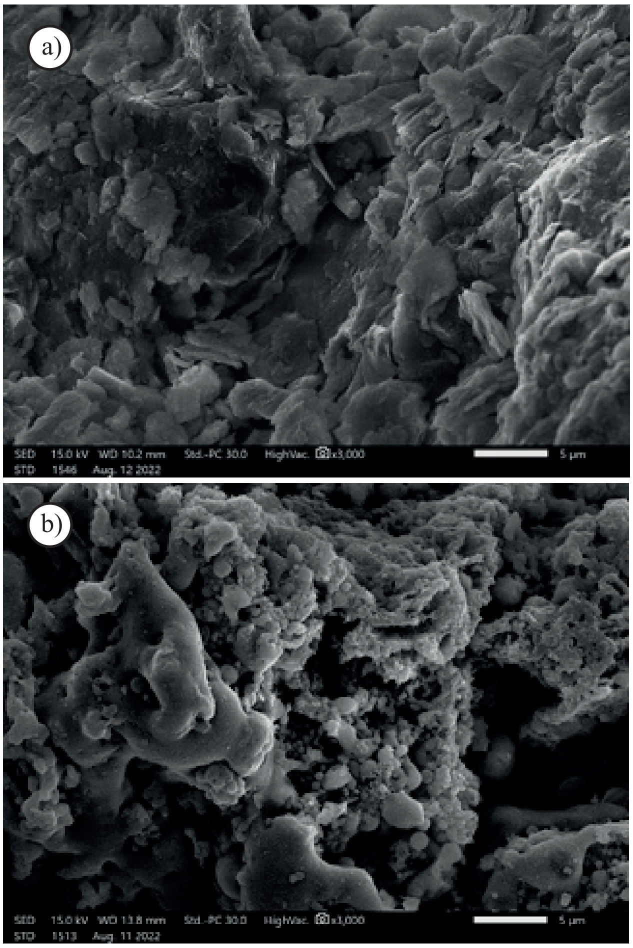


Fig. 1. Microstructure of raw materials used in the study as precursors of the geopolymerization process: a) calcined coal slate; b) lime fly ash

Table 1. Oxide composition of geopolymerization precursors used in the study

Oxide composition [wt.%]	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	Others
Lime fly ash	14.51	49.00	33.10	0.95	0.2	–	2.24
Calcined coal slate	55.84	36.91	0.94	1.26	3.51	0.34	1.20

The following 3 types of natural fibers were used as reinforcing fibers: rafia fibers (supplier NeoPak, Poland), fibers of field grasses found locally in Poland (supplier Dach-Wkręt, Poland) and wood fibers (produced in the form of wood wool (supplier Dach-Wkręt, Poland)). The appearance of the fibers is shown in Figure 2. Geopolymer composites were produced with the addition of 1% fibers (short- 1-1.5 cm; long- 5-7 cm). The fibers were added to the geopolymers already after the geopolymer mass was formed and mixed for about 15 min until the composition became homogeneous.

Fig. 2. Overview photo of the fibers used in the study to reinforce geopolymer materials: a) rafia; b) field grass fibers; c) wood wool fibers

Table 2. Designations of manufactured composite variants

Fibers / Geopolymer	1% rafia – long fiber	1% rafia – short fiber	1% grass fiber-long fiber	1% grass fiber-short fiber	1% wood wool – long fiber	Reference samples
Lime fly ash	RD.B	RK.B	TD.B	TK.B	DD.B	REF.B
Calcinated coal slate	RD.Ł	RK.Ł	TD.Ł	TK.Ł	DD.Ł	REF.Ł

Geopolymers were produced using a 10M sodium hydroxide solution, with R-145 sodium glass (aqueous sodium silicate solution). The ratio of sodium hydroxide solution to water glass was 1:2.5 by weight. Geopolymers were produced using 50 parts by weight of building sand, 50 parts by weight of ash or shale, 17.5 parts by weight of alkali solution (sodium hydroxide solution + water glass). Curing was carried out at 75°C for a period of 24 hours. Table 2 shows the determinations of the geopolymer composite variants produced.

Thermal conductivity tests were carried out using NETZSCH's HFM 446 Lambda Series. The measurements were carried out for the temperature range of 0-20°C. For the tests, 16×16×5 cm panels with dimensions of 16×16×2.5 cm were prepared by cutting from the manufactured samples.

Accelerated aging tests were carried out in a Q-UV SPRAY (Q-LAB) chamber by simulating sunlight, rain and dew using alternating cycles of UV light and moisture at controlled elevated temperature. Tests were performed in accordance with ASTM G-155 [30] Cycle 7 (UV intensity 1.55 W/m^2/nm; total time 500 h; spray time 0.15 h; condensation time 3.45 h, condensation temperature 50°C). Resistance tests to varying temperature cycles were carried out in a WEISS TECHNIK climate chamber for a temperature change cycle from -40°C to +40°C. The duration of one cycle was 2.5 h. The study was carried out by simulating the temperature change over 135 cycles. Microscopic images were taken on a JEOL IT200 scanning electron microscope. Flexural strength tests were performed using an MTS Criterion testing machine according to PN-EN 12390-5:2019-08 [31].

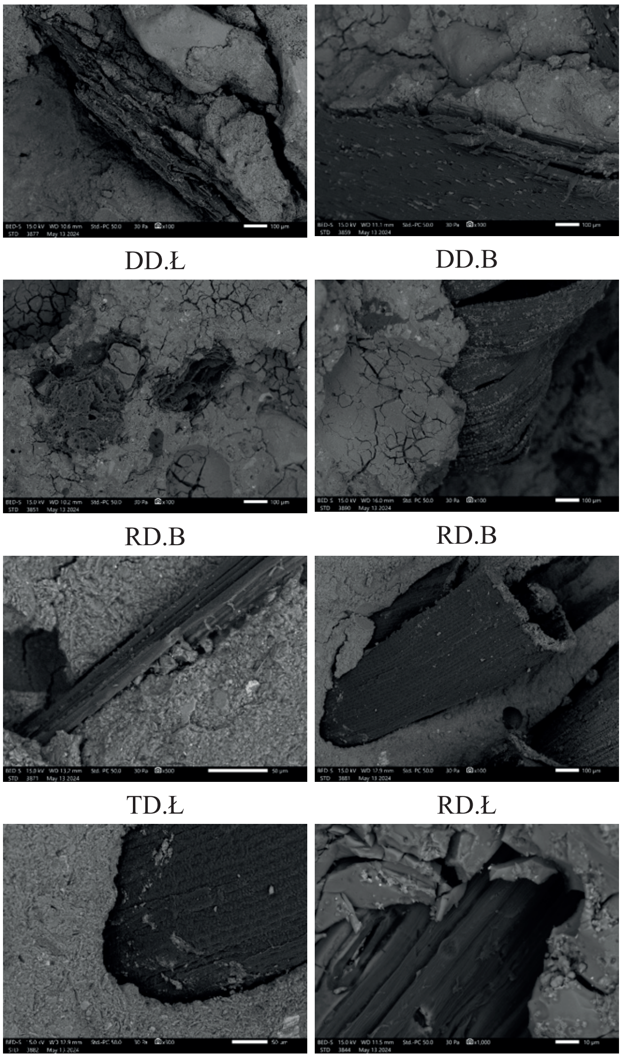
3. RESULTS

Table 3 shows the results of testing the thermal conductivity coefficient of geopolymer composites with fibers, as well as the results of testing the density, flexural strength and resistance to varying temperature cycles. In the case of fly ash-based composites, only rafia fiber has a positive effect on lowering the lambda coefficient, reducing the coefficient by 20%. The other fiber types negatively affect the thermal conductivity coefficient. In the case of composites based on calcined carbonaceous shale, both rafia and field grass fibers resulted in an improvement in insulating performance. In contrast, wood fibers had a negative effect. When analyzing the effect of fiber length on the thermal conductivity coefficient, it is difficult to determine the effect of fiber length and relationships. The length of rafia fibers had the greatest effect because, in the case of slate-based composites, long fibers were more favorable and in the case of ash-based matrix, short fibers were more favorable. The addition of fibers had no significant effect on the density changes of the composites. In the case of the coal shale-based matrix, the fibers caused a maximum density change of 3% by weight, while in the case of the Bełchatów ash-based matrix, the changes

Table 3. Results of density and thermal conductivity tests

	RD.B	RD.Ł	RK.B	RK.Ł	TD.B	TD.Ł	TK.B	TK.Ł	DD.B	DD.Ł	REF.B	REF.Ł
Conductivity coefficient [W/mK]	0.7292	0.6570	0.5516	0.8405	0.7314	0.7768	0.7365	0.8552	0.6949	0.9940	0.6881	0.9646
Density [kg/m^3]	1468.7	1641.7	1431.9	1618.4	1497.8	1655.4	1422.6	1566.1	1439.4	1595.7	1432.8	1606.4
Flexural strength [MPa]	1.69	5.75	1.81	4.65	2.22	5.57	1.83	4.95	2.01	5.02	1.98	5.70
Flexural strength after the climatic chamber [MPa]	1.39	4.23	1.70	4.76	1.93	5.94	1.21	4.94	1.33	4.13	1.66	4.02

amounted to a maximum of 4.5% by weight. Flexural strength tests showed that composites produced on the basis of calcined carbon slate showed higher strengths. All the fibers introduced into the composites due to the poor quality of the connection to the matrix did not have a positive effect on the flexural strength of the composites. The effect of varying temperature cycles reduced the flexural strength mainly for composites based on fly ash from the Bełchatów Power Plant.


DD.Ł DD.B

RD.B RD.B

TD.Ł RD.Ł

RD.Ł TD.B

Fig. 3. Selected SEM microphotographs showing the quality of the fiber-axial bond

Based on the observation of the microstructure (Fig. 3) of the fabricated composites and, in particular, the observation of the contact zone between the fiber and matrix, it can be concluded that all composites were characterized by a very weak bonding at the fiber-matrix interface. In each case, voids between the fiber and matrix and lack of cohesion are visible. This is a result of the lack of prior preparation of the fibers by chemical treatment. This type of treatment was not chosen due to the fact that geopolymerization is a process where alkalis are used and the curing process takes quite a long time. It was decided to see if the fibers without prior treatment would be "etched" just during the geopolymerization process. Based on SEM observations and on the results of flexural strength tests, it can be concluded that prior treatment is also necessary for the addition of fibers in geopolymerization processes.

Fig. 4. Appearance of samples after 500 cycles in UV chamber (accelerated aging tests) – RD.B. samples

Accelerated aging tests in a UV chamber showed no negative effect of UV radiation on the degradation rate of the composites. The evaluation was made by visually comparing the condition of the fibers and matrix surfaces. No surface loss or exfoliation was observed and fibers that were exposed and exposed to UV radiation did not degrade. Both natural fibers and matrix are resistant to the kind of factors used in this study (UV, rain, condensation). Examples of samples with visible reinforcement, after aging tests are shown in Figure 4.

4. CONCLUSIONS

The article presents the results of a study of geopolymer composites based on two waste materials found in Poland in significant quantities. The composites were reinforced by introducing 1% fibers such as wood fibers, field grass fibers, rafia fibers. The fibers were introduced in the form of long fibers with a length of 5-7 cm and chopped short fibers with a length of 1-1.5 cm. Studies were conducted on the effect of fiber addition on such properties of the composites as flexural strength, thermal conductivity coefficient, resistance to varying temperature cycles, density, resistance to UV radiation, rain and condensation. The results obtained allow the following conclusions:

1. Natural fibers introduced into geopolymer composites should be treated to etch and get rid of some organic matter. This conclusion is confirmed by an analysis of the literature [19-25]. The use of untreated fibers results in a very poor quality of the bond at the fiber-ossein interface. Even the strongly alkaline reaction of the activating solutions does not cause the fibers to bond well to the geopolymer matrix. This results in no effect of added fibers on the flexural strength of the composites. Untreated fibers even cause a reduction in strength properties due to a reduction in the active cross-sectional area of the matrix. The flexural strength values of RD.Ł, TD.Ł samples are comparable to those of the reference sample, while the ash samples showed a slight decrease or comparable values after the introduction of fibers. However, the intended effect resulting in improved properties was not achieved.
2. Rafia fiber has the most favorable effect on insulating properties and thermal conductivity coefficient. Field grass fibers and wood fibers can adversely affect the insulating properties by increasing the heat conduction coefficient. This is probably related to the absorbency of these fibers [32].
3. All the fibers used are resistant to UV radiation, rain simulation and condensation. Accelerated aging tests showed no degradation of the fibers in the geopolymer matrix. The geopolymer matrix itself also shows significant resistance to UV radiation, rain simulation and condensation.
4. Coal shale-based geopolymers have significantly better strength parameters compared to fly ash-based geopolymers. They also show greater resistance to variable temperature cycles.

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