Leveraging 3D Printing Capability for Geopolymer Composites Based on Fly Ash with Cotton Fibers Addition

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Abstract

The study explores the use of fly ash as a base material for extrusion-based 3D printing and the impact of incorporating 1% cotton fibers on print properties. Characterization of the base material involves X-ray techniques, particle size distribution analysis, and microscopy. Mechanical properties are tested via bending and compressive strength. Meanwhile, thermal conductivity is also tested. Cotton fibers reduce print strength for loads applied perpendicularly and parallel to the printed sample layers by about 20-23% for compressive strength and 14-24% for flexural strength, possibly due to fiber agglomeration. Thermal conductivity decreases by approximately 12.17% compared to the base material. The results indicate the importance of the current study, i.e., assessing the different types of additives to enhance the mechanical and thermal properties of printed materials. Such ongoing research will facilitate the utilization of 3D printing in creating geopolymer composites.

Keywords: composite, geopolymer, 3D-printing, fiber

1. Introduction

Materials engineering makes a substantial contribution to the design of modern production methods, with particular emphasis on the development of materials with advanced properties and complex structures, such as geopolymers, which can potentially be used in the construction sector. The use of geopolymer materials in 3D printing technology, offering an enticing and sustainable alternative to traditional concrete, is gradually drawing interest and prospects scientifically and architecturally.

1.1. Background and motivation

The constant pursuit of innovative production methods is the primary objective of modern materials engineering, aiming to facilitate the development of materials with advanced properties and intricate structures. 3D printing typifies the rapidly evolving technology, enabling precise object creation in three dimensions using various materials [1]. The construction sector's escalating productivity in recent years has significantly contributed to the advancement of 3D printing technology [2-3]. One of the key advantages of this technology lies in its resource efficiency and energy-saving attributes compared to subtractive methods [4-5].

Moreover, factors bolstering 3D printing technology encompass waste reduction in construction by 30 to 60%, labor cost reduction by 50 to 80%, production cost reduction by 50 to 70%, streamlining supply chains, and enhancing flexibility in shaping components, enabling the fabrication of intricately designed parts unattainable conventionally [6-7]. Given the

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aforementioned advantages, 3D printing emerges as a sustainable technology, exerting minimal impact environmentally and atmospherically. Currently, furthermore, 3D printing facilitates the utilization of diverse material groups during production, including concrete [8], clay [9], ceramics [5], metals [10], cermets [11], biomaterials [12], and polymers [13]. Moreover, numerous benefits result from the adoption and utilization of waste materials from construction in 3D printing processes. Specifically, some of these advantages include reducing environmental impact, increasing sustainability, and improving cost-effectiveness in construction projects through the integration of waste materials into 3D printing [14-15].

To increase the strength of materials produced using 3D printing technology, the feasibility of using various additives, such as fibers, is increasingly examined. This research mainly concerns the production of printing materials based on plastics, whereas practical research focusing on the possibility of fiber reinforcement of building materials, such as cement or geopolymers, produced using 3D printing technology, is currently absent.

1.2. Literature survey

Initially, the application of 3D printing was mainly in the automotive and aviation industries, where part prototyping was especially crucial. Meanwhile, in architecture, 3D printing has been widely used to produce structural models. Over the years and the development of 3D printing, the construction industry has witnessed the potential of this technology [4]. Therefore, predictably, the development of 3D printing in this industry has evolved expeditiously from prototyping to the construction of full-size buildings [16].

In the construction industry, the initial solution for additive manufacturing, i.e., concrete impression, was pioneered in the 1990s [17]. This technique entails the gradual application of molten material to form the desired contours [16]. Meanwhile, another innovative approach, inspired by binder jetting [3], is particle printing. This method involves the selective deposition of a liquid binder onto a powder bed to fuse its particles [16].

The rapid development of 3D printing in the construction industry was possible owing to the constant emergence of innovative solutions covering materials and printers. Currently, giant printers on the market enable the printing of parts or entire buildings in various forms and dimensions [18].

The most prevalent additive manufacturing technique in the construction sector is extrusion-based 3D printing. This process involves the gradual construction of an object by depositing successive layers of material extruded from a nozzle [3]. This manufacturing method facilitates the creation of intricate three-dimensional structures and promotes efficient material usage, aligning with the principles of sustainable development and environmental conservation [7].

The utilization of geopolymers as base materials is emerging as a prominent trend in 3D printing, offering an alluring and more sustainable alternative to traditional concrete. Geopolymers are inorganic materials with a three-dimensional structure, synthesized through a polymerization process resulting from the interaction of an alkaline solution with raw materials, which can include natural and waste materials such as fly ash, metakaolin, red mud, slag, rice husks, glass waste, etc [19-20]. The term “geopolymer” commonly refers to systems whose binding phase primarily consists of aluminosilicates and is highly coordinated [7]. Besides, geopolymers exhibit favorable mechanical properties, chemical resistance, and adaptability to various environmental conditions [5]. However, given that geopolymer blends are complicated and inconsistent, it is hard to determine the perfect composition commensurate with the strict needs of the additive manufacturing process [7, 17].

For the development of 3D printing technology based on geopolymers, appropriately selected settings and curing times for extruded layers are crucial to ensure high adhesion. Additionally, the geopolymer paste prepared for printing must possess thixotropic properties – the viscosity of such paste decreases when subjected to shearing forces (the material “flows”). After the cessation of these forces, the material returns to a higher viscosity state [21]. This course is of great importance due to the superior precedence of pumping and screw mixing operations to the extrusion of the paste through the nozzle in the 3D printing process by the extrusion method. Additionally, the geopolymer paste requires good fluidity, while the printed layer of geopolymer paste must maintain its structure, ensuring the stability of the construction. Another crucial aspect is ensuring the high quality and uniformity of the material used for printing, as it significantly influences the final properties of the structure [22].

To strengthen materials produced using 3D printing technology, scientists are increasingly introducing fibers as additives to materials used in 3D printing. The research conducted so far highlights the potential of using fibers to strengthen 3D printed materials to produce composites with specific geometric features, characterized by low carbon dioxide emissions, reduced density, and high strength, simultaneously [23]. These studies mainly concern the production of printing materials based on plastics, whereas the articles focusing on research showing the impact of adding fibers to 3D printing of building materials such as cement or geopolymers are insufficient. Nematollahi et al. [24] conducted research on introducing polypropylene fibers into geopolymer mixtures for 3D printing, showing an improvement in the properties of printed materials. In turn, research conducted by Perrot et al. [25] focused on the possibility of 3D printing using the extrusion method using earth materials.

However, these studies were conducted on a laboratory scale and did not consider the introduction of additives in the form of fibers. Korniejenko et al. [26] attempted to develop materials based on ceramics for applications in civil engineering, which is possible to use in 3D printing technology. Specifically, a 3D printing simulation (injection molding) of fiber (green linen and carbon fibers) reinforced geopolymer composites based on fly ash was performed. The research results obtained are promising concerning bending strength and material properties - the mechanical properties of the new composites produced using injection methods were comparable to the properties of samples made using the traditional casting method.

1.3. The question of the study and the novelty

The research question to be investigated herein is: "How does the use of fly ash as a base material in extrusion 3D printing influence the mechanical strength properties and thermal conductivity of the printed samples, and what are the effects when 1% by weight of cotton flock is added?". The novelty of this study lies in its focused exploration of the feasibility of utilizing fly ash-based materials for 3D printing, particularly in the context of geopolymer materials. In addition, the possibilities of using natural fibers as a material for 3D printing technology in the construction sector were explored. The influence of the addition of 1% cotton on the strength properties and thermal conductivity of printed samples was examined. This research would contribute to bio-based composites in the construction industry by adopting natural fibers in 3D printing processes, which is one of the sustainable manufacturing methods addressing environmental issues currently with value-added benefits.

1.4. Purpose of the study

In the context of the dynamic development of 3D printing using geopolymer materials, a focused study was conducted to explore the feasibility of extrusion-based 3D printing using materials based on fly ash from the combustion of hard coal in a heat and power plant in Skawina, Poland. Furthermore, the impact of adding 1% by weight of cotton flock on the mechanical strength properties and thermal conductivity of the printed samples was investigated. The objective of this research is to delve into the capabilities and potential applications of 3D printing technology for geopolymer materials.

2. Material

The following section presents the characteristics of fly ash, which was used as the base material in the research. The research carried out on fly ash including oxide analysis using X-ray fluorescence (XRF) analysis, phase analysis using X-ray diffraction (XRD), and observation using a scanning electron microscope (SEM). The results of examining the particle size distribution of the base material were also presented. The section below also contains a description of the used additives and the alkaline solution used in the geopolymerization process.

2.1. Base material

For the tests, fly ash class F from a heat and power plant in Skawina, Poland was used as a base material. The microstructure of fly ash particles was imaged using an SEM in Fig. 1. The oxide composition is shown in Table 1. Analysis of the oxide composition using the XRF method was performed using an EDX-7200 spectrometer (Shimadzu, Kyoto, Japan).

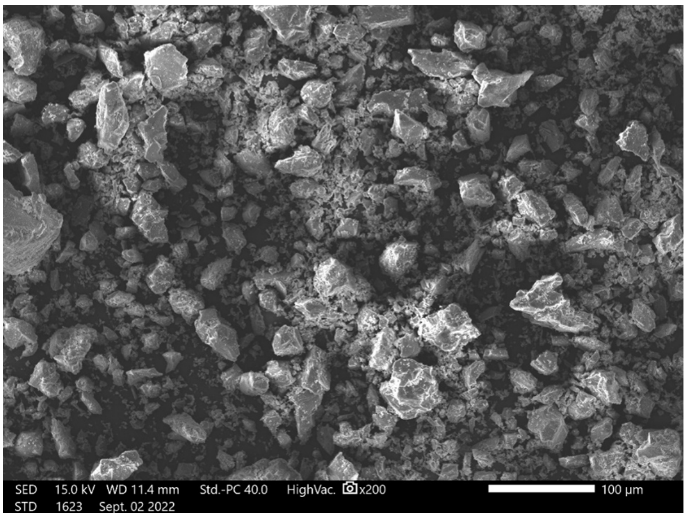


Fig. 1 SEM microphotographs of fly ash particles

Table 1 Oxide composition of fly ash

Fly ash											
Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	TiO_2	SrO	ZrO_2	Ag_20	MnO	Ball
Concentration [%]	44.140	22.629	20.226	3.758	3.533	1.652	0.984	0.592	0.287	0.241	2.245
3-sigma	0.158	0.205	0.025	0.009	0.014	0.008	0.004	0.007	0.037	0.003	-

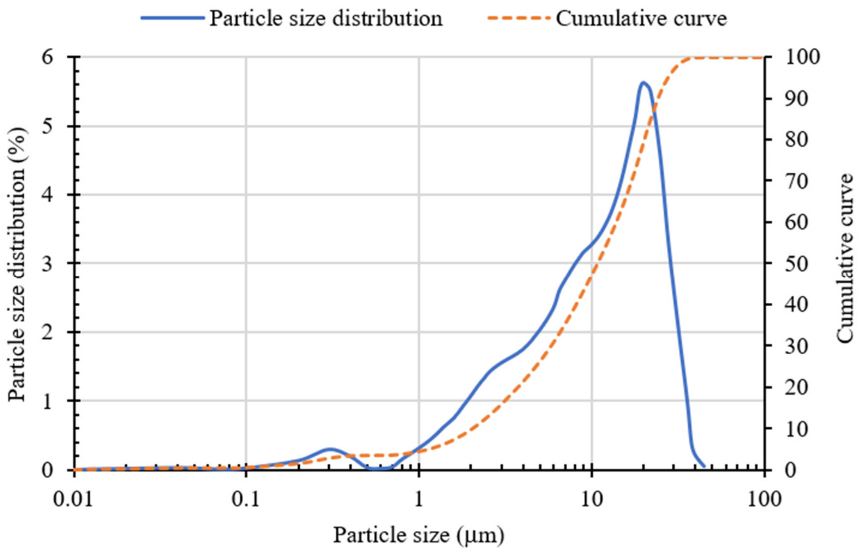


Fig. 2 Particle size distribution and cumulative curves for fly ash

The particle size distribution and cumulative curves of fly ash are shown in Fig. 2. Presented data comes from own study conducted using a particle size analyzer from Anton Paar GmbH in Graz, Austria, on which tests are carried out using laser diffraction technology.

Table 2 provides fly ash particle size distribution data, including key parameters such as D_{10}, D_{50}, and D_{90} values, mean size, and span parameters. The analysis exhibited that the mean particle size was 13.21 µm with a standard deviation of 0.345 µm, suggesting a uniform particle size distribution. The D_{10}, D_{50}, and D_{90} values were 2.08 µm, 10.98 µm, and 25.33 µm, respectively, while the span value of 2.12 indicates a moderate width of the particle size distribution of the tested material.

Table 2 Statistical grain size values for fly ash

	Mean value	Standard deviation
D_{10} (μm)	2.08	0.082
D_{50} (μm)	10.98	0.233
D_{90} (μm)	25.33	0.850
Mean size (μm)	13.21	0.345
Span	2.12	0.021

The XRD analysis was carried out on a Panalytical Aeris X-ray diffractometer (Malvern Panalytical, Almelo, the Netherlands), using Cu-Kα radiation in the scanning range from 10° to 100° 2θ, with a step of 0.003° (2θ). The obtained results of the tests are presented in the form of a diffractogram in Fig. 3. Phase identification was carried out using the International Centre for Diffraction Data (ICDD) PDF4+ database, and qualitative analysis was performed using the Rietveld method. The XRD analysis, both qualitative and quantitative, enabled the identification of phases such as quartz (46.2%), mullite (47.1%), hematite (1.9%), and orthoclase (4.8%).

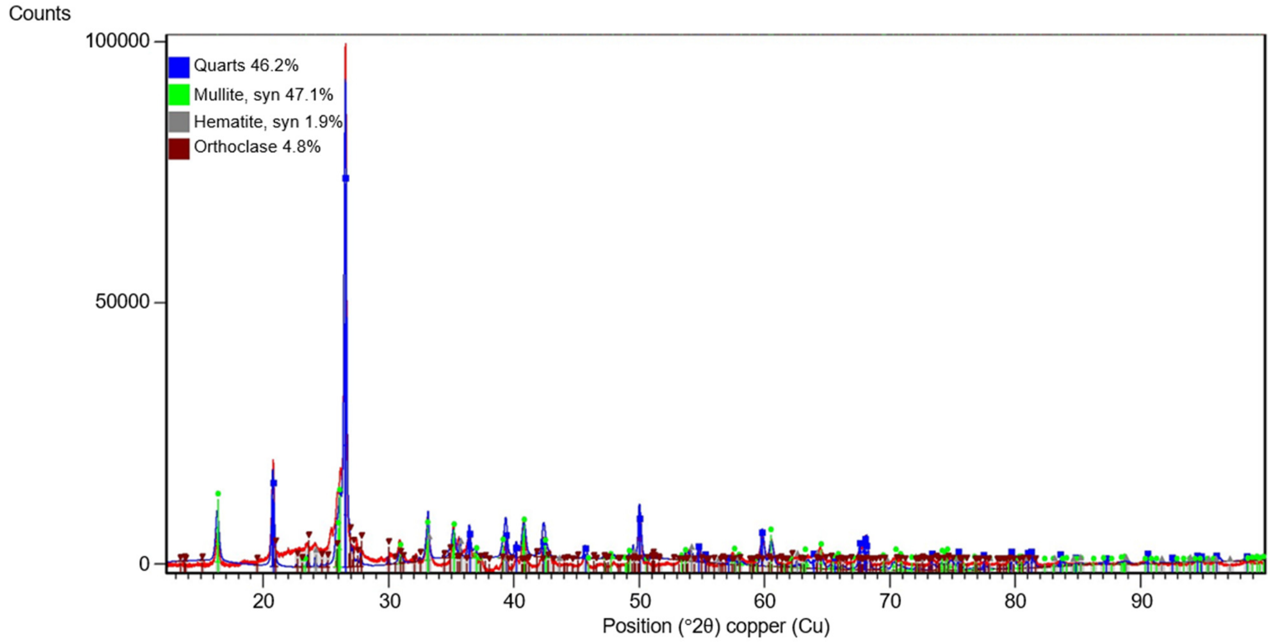


Fig. 3 X-ray diffraction analysis of fly ash

2.2. Additives and alkali solution

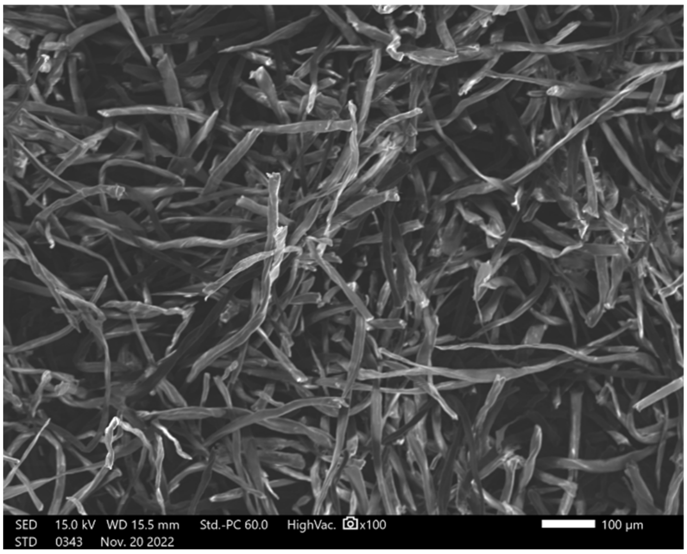


Fig. 4 SEM microphotographs of cotton flock

The cotton flock was employed as an additive, introduced in an amount of 1% by weight concerning the mass of fly ash (Fig. 4), which is classified as a biodegradable material. Cotton flock is characterized by low density (0.22 g/cm^3), with an average fiber length of 350 μm (usually 150-500 μm) and a width ranging from 10 μm to 25 μm.

As an additive improving printability and averting the geopolymer paste from spreading during printing (giving thixotropic properties), 1% by weight, concerning the weight of fly ash, of sodium alginate C6H9NaO7 from Pol-Aura Sp. z o.o. (Zabrze, Poland). In the tests, an alkaline activator was comprised of a 10-molar solution of sodium hydroxide mixed with an aqueous solution of sodium silicate R-145 in a 1:2.5 volume ratio.

3. Methodology

This section presents the organization and scope of the research conducted. Additionally, the chapter contains a description of the methodology used in the work. This research methodology consists of the preparation of a mixture based on fly ash for printing using the extrusion method, a description of the method and parameters of 3D printing of plates from which samples were subsequently cut out for testing, and a description of the tests on the prints including the measurements of density, thermal conductivity, bending strength, and compressive strength.

3.1. Organization of the research work

The procedure and scope of the research included:

(1) Analysis of the physical and chemical properties of the base material, including:

 (i) analysis of the oxide composition using the XRF method,

 (ii) phase composition analysis using the XRD method,

 (iii) particle size distribution analysis and microscopic observation of fly ash particles.

(2) Formulate a suitable mix design of geopolymer paste for 3D printing.

(3) Printing of plates with dimensions of 460 × 460 × 40 mm from geopolymer paste based on fly ash without and with the addition of cotton fibers.

(4) Cutting samples for testing.

(5) Carrying out tests on printouts, including:

 (i) density measurement,

 (ii) measurement of bending strength,

 (iii) measurement of compressive strength,

 (iv) measurement of the thermal conductivity coefficient for three temperature ranges: 0-20 °C, 20-40 °C, and 30-50 °C.

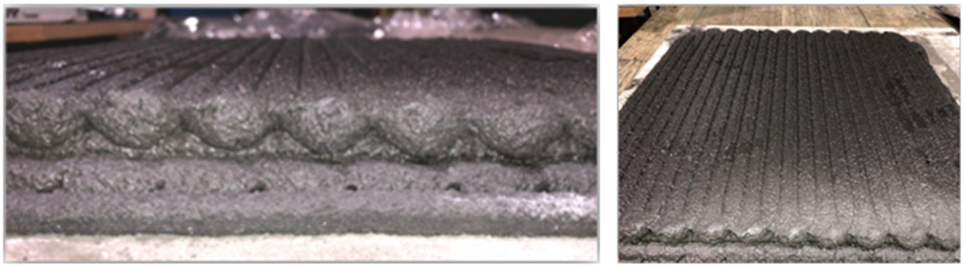
(6) Analysis and evaluation of the obtained research results.

3.2. 3D printing

The fly ash-based geopolymer paste used for 3D printing was prepared by mechanically mixing dry ingredients with an alkaline solution. Then, the prepared paste was transferred to the stainless steel tank of the IMER Small 50 pump (IMER Group, Kraków, Poland), which fed the material directly to the printer nozzle via a transport hose. The pump used is equipped with an inverter, which enables smooth adjustment of the material flow rate during printing, with the material feeding speed for printing the tested samples being approximately 1.6 dm^3/min. A printer from ATMAT (Kraków), specially adapted to printing from concrete/geopolymer materials, was used to print the plates. This device enables printing to deploy fused deposition modeling (FDM) and fused filament fabrication (FFF) technology. The dimensions of the printer's worktable are 460 × 460 × 40 mm. When printing from the tested material, a nozzle with a diameter of Ø20 mm was used.

Before printing, a g-code was prepared that precisely defined the print parameters. The printing process itself consisted of applying 10 mm thick layers (4 layers) at a speed of 250 mm/s onto a rough ceramic plate. The movement of the printer nozzle took place in the XY plane, and the movement in the Z axis was achieved by changing the height of the table. After the printing process was completed, the printed ceramic plate was removed from the working field. The prints were left to harden under foil at ambient temperature for 28 days. After the seasoning time, the boards were cut into smaller samples for testing. Fig. 5 depicts photos taken during printing, the printed plate, and samples cut for testing.

(a) Printing process with parameters



(b) 3D-printed plate

(c) Dry-cut test samples from the plate

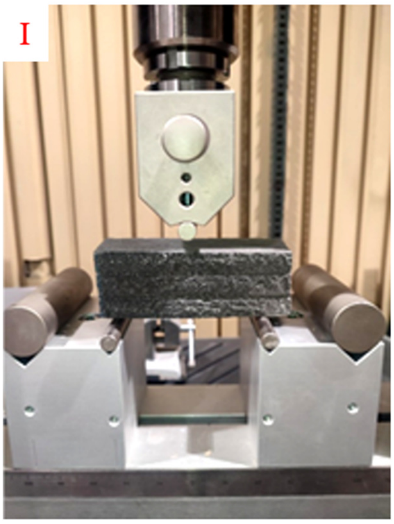
Fig. 5 Photo taken during printing, the printed plate and samples cut for testing

3.3. Testing methods

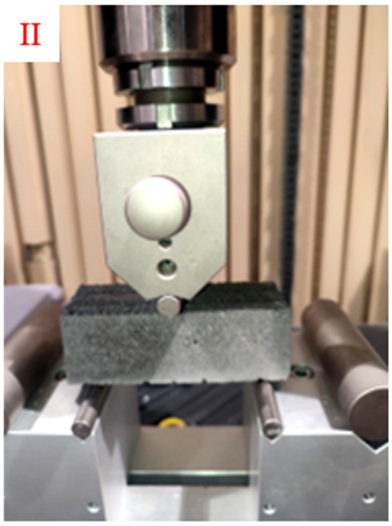
The density of the printed samples was determined geometrically by measuring the dimensions and weight of the samples prepared for thermal conductivity measurements. Thermal conductivity measurements were carried out on samples with dimensions of 140 × 140 × 40 mm, according to the ASTM C518-21, JIS A1412-1, ISO 8301:1991, and DIN EN 12667 standards on the HFM 446 Lambda Series device from NETZSCH (Netzsch GmbH & Co., Selb, Germany). Measurements were performed for three temperature ranges: 0-20 °C, 20-40 °C, and 30-50 °C.

Bending strength and compressive strength measurements were performed on the Autograph AGS-X universal testing machine (Shimadzu, Kyoto, Japan). Tests for each analyzed print variant were sequentially carried out on six samples. The dimensions of the samples prepared for bending strength and compressive strength tests were 120 × 40 × 40 mm and 40 × 40

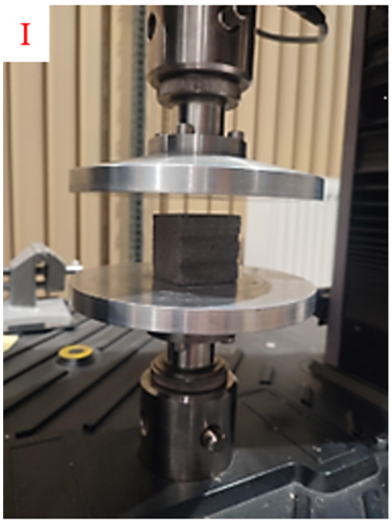
× 40 mm, respectively. The test speed was 5 mm/min, and regarding three-point bending tests, the distance between the supports was 100 mm. The tests were carried out based on the PN-EN 12390-5:2019-08 and PN-EN12390-3:2019-07 standards. Both compressive and bending strength were tested in two directions: I-load normal to the layers and II-load parallel to the layers (Fig. 6).



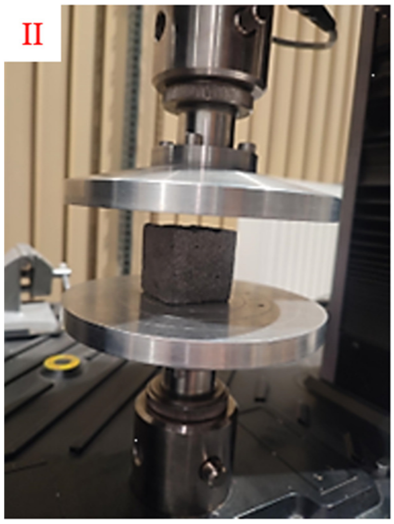
(a) The bending load normal to the layers



(b) The bending load parallel to the layers



(c) The compressive load normal to the layers



(d) The compressive load parallel to the layers

Fig. 6 Arrangement of samples during bending and compressive strength tests

4. Results

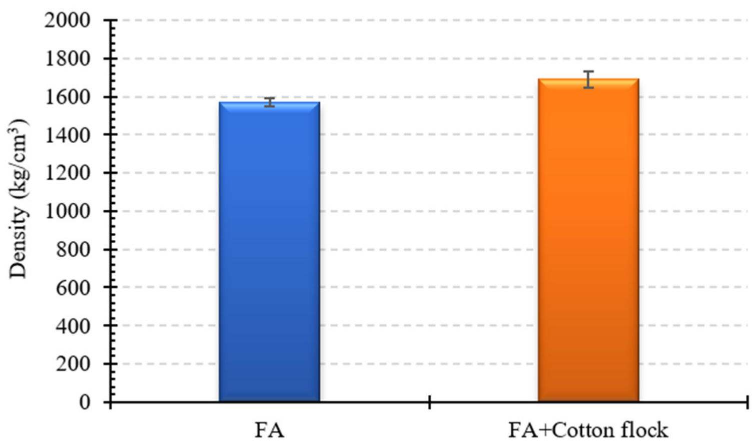


Fig. 7 Density of printed geopolymers based on fly ash

Fig. 7 demonstrates the results of density measurements. The use of the addition of cotton flock resulted in a slight increase in the density of printed geopolymers, amounting to approximately 8% compared to reference samples, the density of which was 1568.3 kg/cm^3.

The obtained results of the thermal conductivity coefficient for the tested prints are presented in the chart shown in Fig. 8. For both types of samples, the reference one and the one with the addition of cotton fibers, the values of thermal conductivity coefficients were determined in three temperature ranges: 0-20 °C, 20-40 °C, and 30-50 °C. Cotton fibers, which are natural materials with insulating properties, had a significant impact on these results. The addition of cotton fibers resulted in a decrease in the thermal conductivity coefficient in all tested temperature ranges. The greatest decrease in thermal conductivity, approximately 12.17%, was obtained in the most frequently studied temperature range: 0-20 °C. Concerning the temperature ranges 20-40 °C and 30-50 °C, the decreases were 2.63% and 5.13%, respectively, which are predictable given that cotton fibers act as an insulator, reducing the thermal conductivity of the material.

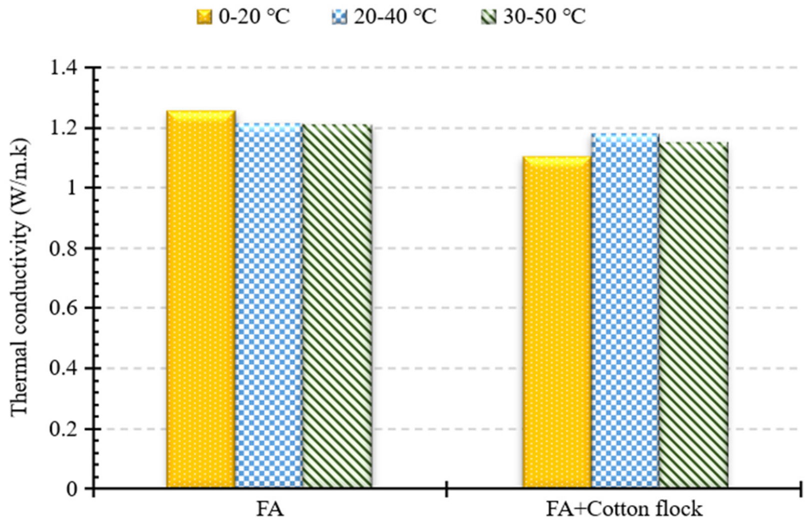


Fig. 8 Thermal conductivity of printed geopolymers based on fly ash across temperature ranges

Figs. 9 and 10 present the results of compressive and bending strength measurements. Tests were carried out by subjecting samples to loads in two directions: perpendicular and parallel to the layers of printed materials. It was obtained that the introduction of cotton fibers did not result in the expected increase in strength properties. For both compressive and flexural strengths, a decrease in values was observed in both loading directions. The reduction in compressive strength was approximately 20% and 23% for the load applied perpendicular and parallel to the layers of the printed samples, respectively. However, for bending strength, these decreases were approximately 14% and 24%, respectively.

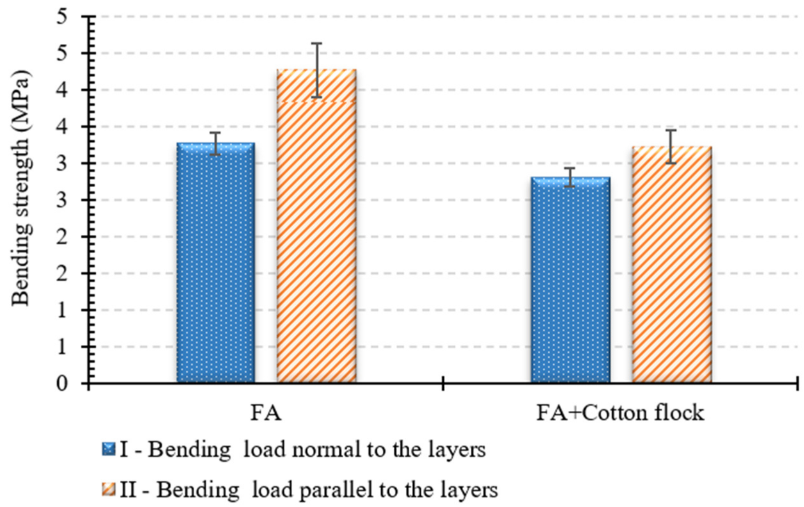


Fig. 9 Compressive strength of printed geopolymers based on fly ash

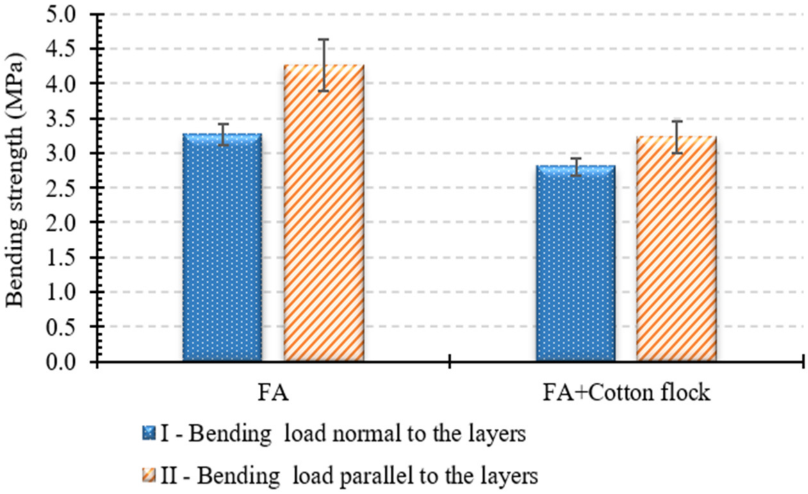


Fig. 10 Bending strength of printed geopolymers based on fly ash

It is also noteworthy that the samples possessed higher values of strength properties when loaded parallel to the layers. Based on the observations of the printing process and the results obtained, it can be interpreted that the decrease in strength properties for samples with the addition of cotton fibers may result from the deterioration in the printability of the geopolymer paste observed during the printing process and, consequently, from the increased risk of the formation of fiber agglomerates in the structure of the printed materials and geopolymer materials. Moreover, the sample hardening process is carried out in open air at ambient temperature may induce a significant impact.

Al-Qutaifi et al. [27] attempted to investigate the possibility of building structural layered objects based on fly ash by conducting printing simulations. The influence of the addition of steel fibers in the amount of 1% and polypropylene fibers in the amount of 0.5% on the bending strength of the produced geopolymer composites was examined. The obtained results of bending strength measurements signified that the layering process incurs a negative impact on the mechanical strength of the surface materials. The obtained bending values for reference samples without reinforcement, for samples with the addition of steel fibers, and for samples with the addition of polypropylene fibers were 4.99 MPa, 6.31 MPa, and 5.13 MPa, respectively.

In turn, the research conducted by Nematollahi et al. [28] was to investigate the effect of fiber type on the interlayer bond strength and bending strength of a 3D extrusion-printed geopolymer. The extrudable geopolymer paste based on fly ash developed by the authors was reinforced with three types of polyvinyl alcohol (PVA), polypropylene (PP), and p-phenylene benzobisoxazole (PBO) fibers. The results indicated a reduction in the bond strength of the interlayer geopolymer printed in 3D as a result of the addition of fibers, regardless of their type. Nevertheless, the obtained results for the flexural strength of geopolymers reinforced with fibers ranged from 9.0 MPa to 10.3 MPa and were higher compared to the reference material without the addition of fibers, for which it amounted to 7.7 MPa.

Lim et al. [29] in work focused on solving the problem of low tensile strength and poor ductility due to the lack of reinforcement of 3D concrete, by designing reinforced concrete. The tests used a special nozzle that enabled direct insertion of a steel cable to be installed into the extruded mortar. The obtained test results indicated that the use of steel ropes as reinforcement can improve the bending strength of 3D-printed concrete by 290%. Similar research was carried out by Ma et al. [30], who focused on the micro-reinforcement of geopolymer material with a steel cable. In the research, three different printing path configurations were considered (oblique, orthogonal, and rectangular). The test results indicated that the most favorable configuration, characterized by the highest bending strength of approximately 48.9 MPa, 600% higher compared to samples without reinforcement, was a sample with an oblique cross-section as reinforcement.

Korniejenko et al. [26] compared the compressive and bending strength of designed compositions and geopolymers reinforced with short fibers using 3D printing injection technology. During the tests, the green tow flax fibers and carbon fibers were used, introduced in an amount of 1% by weight. The obtained test results indicated a subtle increase in compressive strength in the case of samples with the addition of fibers, but only in a short time after production. After 28 days, a significant decrease was observed. Regarding the bending strength, a significant increase in the value was observed for samples reinforced with flax fibers, for which, after 28 days of seasoning, the strength was approximately 38% higher than that obtained for samples made using the traditional casting method.

In this work, bending strength at a similar level was unattainable as in the discussed literature. It may be attributed to the printed composites, which were not hardened at elevated temperatures which distinguishes the research presented in this paper from those presented in the literature. According to literature reports, the introduction of fibers into the mixture should result in an improvement in the bending strength of prints, whereas the improvement didn't emerge as expected in the case of the tests performed. It is most likely related to the formation of fiber agglomerates, and another method of introducing fiber agglomerates should be considered. Therefore, this study requires further research, including the alternative method of fiber introduction, using a variable amount of fibers, or examining the impact of adding other natural fibers.

5. Conclusions

The article presents research aimed at examining the feasibility of 3D printing using geopolymer materials based on fly ash. Additionally, the influence of the addition of cotton flock in the amount of 1% by weight on the mechanical properties and thermal conductivity of the printed samples was examined. The results of the tests showed that:

(1) The addition of cotton fibers resulted in a slight increase in density, approximately 8%, and a decrease in the thermal conductivity coefficient, approximately 12.17%, for a 0-20 ℃ temperature range for the printed geopolymers. It is noteworthy that cotton fibers, which are natural materials with insulating properties, yielded a significant impact on these results by acting as an insulator and reducing the thermal conductivity of the material.

(2) Contrary to expectations, the addition of cotton fibers did not improve strength properties. Both compressive and flexural strength values decreased when loads were applied perpendicular or parallel to the printed layers. The reduction in compressive strength was approximately 20% and 23% for the load applied perpendicular and parallel to the layers of the printed samples, respectively; regarding bending strength, these decreases were approximately 14% and 24%, respectively.

(3) Observations during printing and test results suggest that the reduced strength in samples with cotton flock may be due to compromised printability of the geopolymer paste, probably caused by leading to fiber agglomeration. Moreover, curing samples in open air at the ambient temperature had a substantial impact.

The findings highlighted above underscore the significance of the present work. Given these findings, further research is warranted to optimize the 3D printing process using geopolymers. Additionally, assessing various types of additives is essential for enhancing the mechanical and thermal properties of printed materials. This ongoing research will contribute to advancing the application of 3D printing technology in the development of geopolymer composites.

Acknowledgments

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Conflicts of Interest

The authors declare no conflict of interest.

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