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A Comparative Study of Mechanical Properties of Fly Ash-Based Geopolymer Made by Casted and 3D Printing Methods

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Abstract. Currently additive manufacturing (AM) is a rapidly developing industrial sector and a disruptive technology. It is an answer for new challenges such as resources saving and energy effectiveness as well as response for circular economy needs. Unfortunately, the full exploitation of 3D printing technology for ceramic is currently limited due to the in-process and in-service performance of the available materials' sets, especially in application in construction industry. The main aim of the article is comparison mechanical properties such as compressive and flexural strength between casted and injected samples (simulation for 3D printing process). The same geopolymer mix, plain as well as with flax fibres, based of fly ash class F was casted and cured in the same temperature prior to its mechanical test with 'printed' samples.

1. Introduction

3D printing is a rapidly developing industrial sector and, potentially, a disruptive technology that have a lot of advantages such as resources saving and energy efficiency compared to subtractive technologies [1]. Unfortunately, the full exploitation of 3D printing technology for ceramic is currently limited due to the in-process and in-service performance of the available materials' sets, especially in application in construction industry: 'While 3D printing techniques have been successfully applied in a wide range of industries such as aerospace and automotive, its application in concrete construction industry is still in its infancy' [2]. These achievements attract the attention of different industries and science fields about 3D printing technology. 3D printing technology used in the concrete is currently being studied in the construction industry applications and academia, such as military constructions or rapid rehabilitations [1].

With respect to the construction materials, additive technologies for concrete have been found to be the most viable option of widespread use in automated construction processes in the near future. Cementitious materials, such as concrete or mortar, are well understood and have unique fresh and hardened properties as well as an extensive variety of readily available admixtures to customize its performances [3, 4]. In conclusion, the requirements and properties of materials which are being used with a layer based automated construction approach. However, in most cases only a limited amount of powder has been used to fabricate structures. Shakor was developed for Z-Corporation's threedimensional printing process with a water-based binder using a unique mix of cements [5]. Khalil was indicated that a 3D printable mix made out of Calcium Sulfo-aluminate cement and ordinary Portland cement was developed and the extrudability and buildability was done and tested with a compressive strength of 79 MPa for printed specimens [6]. Ma was applied to accommodate the rapid development of 3D printing using geopolymer. It was proved the well bonding and coordination of the micro-cable and geopolymer [7]. In addition, use of 3D printing could reduce 35–60% of the overall costs of concrete construction due to the removal of formwork [8].

The further development of this technology required improvements to design new materials, to assess materials performance and to improve processing strategies. The research in the area in case of concreates have been done, but the investigation connected with other ceramic materials such as geopolymers are still new area [9, 10]. Contemporary only some prototype solution in the area of geopolymers materials were designed in Australia, China and Russia [11]. The current state-of-art in this area is presented in table 1.

Table 1. The state-of-art for AM technologies [12].

Country	Existing technology	Main limitation	Source of information
Russia	AM technology - 3 D printing for geopolymers is developed by RENCA company. The company has working prototype. It was tested in relevant environment.	The technology is presented as working solutions, but according to the information presented by the RENCA company on the conference in France (2018). The technology is not stable and required mixing manually. Recipes are individually created for each customer.	https://youtu.be/pKekH5beIZU https://youtu.be/hXdE8ozDfhg www.geobeton.ru, www.apis-cor.com, http://renca.org/ Information presented by RENCA on GEOCAMP 2018
China	- Printing mainly from powder. - Printing based on long steel fiber. - Addition short fiber glass.	Some information about theoretical concept and information about laboratory prototype.	[13, 14]
Italy	The project WASP declared development 3D technology form different materials.	There is lack of detailed information about geopolymers.	www.wasproject.it
Australia	Swinburne University in Melbourne work on the technology. Laboratory prototype have been done.	Lack of information about possible applications.	https://www.youtube.com/watch?v=6v8sYuJdRqw
Singapore	Cooperation in consortium. Laboratory prototype have been done .	Lack of information about possible applications.	https://www.youtube.com/watch?v=1ZyMd8CaONQ
Belgium	KULeuven created the group called SREMat. The prototype has not been created yet.	The group starts his work in 2018.	https://www.mtm.kuleuven.be/Onderzoek/sremat
Finland	Project financed by UIA.	Project started in November	https://www.uia-

	The prototype has not been created yet.	2017.	initiative.eu/en/city/lappeenranta

Some researchers have conducted to include short fibers in cementitious materials to achieve desired toughness and ductility enhancement for 3D printing [15, 16]. It also indicated the freeform additive manufacturing processes was useful to acquire sufficient mechanical capacities as the rebar reinforced structures.

Previous studies [17] have demonstrated that the performance of cement-based mixtures in construction-scale 3D printing mixtures is characterized in terms of print quality, shape stability, and printability. The main aim of the article is comparison mechanical properties between mechanical properties such as compressive and flexural strength for casted and injected samples (simulation for 3D printing process).

2. Materials and methods

2.1. Materials

Geopolymers were made from fly ash from the CHP plant in Skawina (Poland) and sand in ratio 1:1. Additionally half of the samples with 1% by mass of green tow flax fibers.

The fly ash is rich in oxides such as SiO_2 and Al_2O_3. The detailed contains of oxides is presented in table 2. The morphology of the particles of fly ash was typical of such by-products of coal combustion and suitable for the process of alkali-activation [18].

Table 2. Chemical composition of the fly ash [18].

	SiO_2	**Al_2O_3**	**Na_2O**	**CaO**	**Fe_2O_3**	**MgO**	**K_2O**	**SO_3**	**TiO_2**	**P_2O_5**	**BaO**
[% mass]	55.89	23.49	0.59	2.72	5.92	2.61	3.55	0.16	1.09	0.82	0.2

The fibres have been purchased from the Institute of Natural Fibres and Medicinal Plants in Poland. Tow is a coarse, broken fiber, removed during flax processing. They are usually shorter than 30 cm and are semi-products for textile fibers production [18]. Green tow flax fibres (figure 1 and 2) – the fibre is similar to tow flax fibre after dew retting process, but it is more stiff. The fibre for samples were 3-5 cm long.

Figure 1. Green tow flax fibre [19].

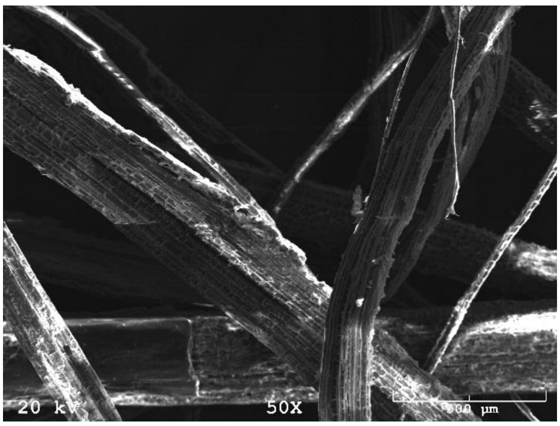

Figure 2. Green tow flax fibre – SEM picture.

The process of activation has been made by 10M sodium hydroxide solution combined with the sodium silicate solution. The ratio of liquid parts to dry parts was 0.4 by mass.

2.2. Samples preparation

The all samples were prepared using sodium promoter, fly ash, sand and half of them with using flax fibers (1% by mass of the composite). The main goal of fibers additive was reinforcement, especially increasing the flexural strength [20]. The process of activation has been made by 10M sodium hydroxide solution combined with the sodium silicate solution (the ratio of liquid glass - 1:2.5). To produce geopolymers, flakes of technical sodium hydroxide were used and an aqueous solution of sodium silicate (R-145) which molar module was 2.5 and density was about 1.45 g/cm^3 and the tap water. The alkaline solution was prepared by means of pouring the aqueous solution of sodium silicate over the solid sodium hydroxide. The solution was mixed and left until its temperature became stable and the concentrations equalized, which took, about 2 hours. The fly ash, sand, alkaline solution and fibers were mixed about 10 minutes by using low speed mixing machine (to receive the homogeneous paste).

Next, the two methods of production were implemented: injection molding made manually and traditional method (figure 3 and 4), were the material was put into two sets of plastic moulds (for compressive strength and flexural strength tests). The injection molding was made by using a syringe.

Figure 3. Plain sample (without fibres) made by injection method.

Figure 4. Sample with 1 % of green tow flax fibres, made by traditional casted method.

The following samples has been performed:

- Plain samples made by injected process (simulation for 3D printing process) PS3D (figure 5),
- Plain samples made by traditional casted technology – PSC (figure 6),
- Samples with 1% of green tow flax short fibres made by traditional casted technology - FSC,
- Samples with 1% of green tow flax short fibres made by injected process (simulation for 3D printing process) – FS3D.

Figure 5. Plain sample made by injected process (simulation for 3D printing process).

Figure 6. Sample made by traditional casted technology.

The samples were NOT subjected to vibratory. Tightly closed molds, by using foil, were heated in the laboratory dryer for 24h at 75 °C. Then, the samples were unmolded. They were investigated after 7 days.

2.3. Methods
Compressive strength tests were carried out according to the methodology described in the standard EN 12390-3 ('Testing hardened concrete. Compressive strength of test specimens'), because of the lack of separate standards for geopolymer materials. The tests involved 6 cubic samples: 50 x 50 x 50 mm. Tests were performed on an concreate press - MATEST 3000kN with speed 0,5 [MPa/s].

Flexural strength tests were carried out according to the methodology described in the standard EN 12390-5 ('Testing hardened concrete. Flexural strength of test specimens'), because of the lack of separate standards for geopolymer materials. The tests involved 3 prismatic samples: 50 x 50 x 200 mm (space between supporting points 150mm). Tests were performed on an universal testing machine - MATEST 3000kN with speed 0,05 MPa/s. The calculations were based on following equation:

$$f_{cf} = \frac{Fl}{d_1 d_2^3} \quad (1)$$

where:
f_{cf} – compressive strength, MPa
F – maximal load, N
l – space between supporting points, mm (for conducted tests:140 mm)
d_1, d_2 – sample dimensions, mm

3. Results and discussion

3.1. Compressive strength test
The results of the compressive strength test are shown in table 3. The results of both methods and samples with fiber reinforced and plain (without fibers) specimens are presented.

Table 3. Compressive strength test after 7 days.

Sample	MPa	standard deviation
PSC	44.9	5.1
FSC	34.7	5.3
PS3D	47.1	5.2
FS3D	39.6	5.6

Better results were attained for plain samples than ones with fibers, regardless of the technology of samples' manufacturing. The technology of injection gives slightly better results than traditional casting method. However, the difference between mechanical properties of the samples produced by different methods could be caused by lack of vibrating during the traditional method and removing the air bubbles in injection process. The best results have been achieved for plain samples made by injected process (simulation for 3D printing process).

3.2. Flexural strength test

The results from the flexural strength tests are shown in table 4. The results of both methods and samples with fiber reinforced and plain (without fibers) specimens are presented.

Table 4. Flexural strength test after 7 days.

Sample	MPa	standard deviation
PSC	6.1	0.9
FSC	6.0	0.5
PS3D	7.4	0.3
FS3D	7.1	0.7

Similarly to the compressive strength tests, better results were attained for plain samples than ones with fibers, regardless of the technology of samples' manufacturing. However, in case of flexural strength this differences are statistically not important. The best results give the injection process (simulation for 3D printing process). It could be caused by lack of vibrating during the traditional casting method.

4. Conclusions

The development of the AM technologies requires: improvements to design new materials, to assess materials performance and to improve processing strategies. The achieved results show that mechanical properties of the new composites made by injection methods (simulation of 3D technology) are comparable with the results achieved by traditional casting process. It could by caused by lack of vibrating during the traditional casting method and because of that some air-bubbles may remain, however macro observations do not shows any voids in material.

The results for compressive and flexural strength are better for plain samples than ones with fibers, regardless of the technology of samples' manufacturing. It shows that short fibers addition could influence similarly on both of the technologies. However, this topic required future research, including fiber orientation and distribution change depending on 3D printing parameters (flow seed, nozzle diameter, fiber length, trajectory etc.) and how it influence the flexural, tensile, compressive strength and fracture energy.

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