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MECHANICAL AND PHYSICAL PROPERTIES OF CEMENT MIXTURES FOR 3D PROCESSING

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Abstract. In this paper, information about cementitious composite materials for further 3D processing is discussed and supplemented. Many of the research in this area focuses primarily on cement composites suitable for 3D printing. Nevertheless, 3D printing is not the only robotic processing technique. Another such a technology is modelling with the help of a robotic arm, which can be used to create various elements that fulfil their original but also aesthetic function. The robotic arm creates, using a variety of sculptural or hand tools, a final unique relief of a given element. Three different cement composite mixtures are discussed and their mechanical, physical and thermophysical properties are evaluated. The research aims to investigate and optimise these composites for robotic sculpturing and 3D printing.

Keywords: 3D processing, robotic sculpturing, cementitious composite, printing technology.

1. Introduction

Currently, there is a lot of research in the world focused on optimising the composition of mixtures in 3D processing. Especially 3D printing has received a considerable development and investment in recent years. In general, technologies based on 3D processing are on the rise. The surface processing of cement composites using a robotic arm has been overshadowed by 3D printing technology, although this technology has great potential. The research that led to this paper focuses primarily on the development of a special mixture for this purpose and its comparison with cementitious composite mixtures used for 3D printing. The use of cement composites modified with the help of robots is a new, innovative, and partly automated technology, which has recently been significantly promoted in construction. This production method has started to develop very fast, which brings the need to design a suitable composition of mixture for this technology. It is important to clearly define the required properties of the material in the fresh state, as well as during the processes, solidification and hardening.

Such a technology brings the potential for an interesting architectural concept, either of the whole building or its parts and elements. An important and interesting characteristic of modern structural design is increasing the fire and dynamic resistance of buildings. However, this effort also increases the financial impact on the resulting architectural work. Therefore, the principle of prefabrication is used in civil engineering, which has the main advantage especially in the speed of construction and the possibility of fast delivery of the prepared precast directly to the construction site. A significant advantage of using precast parts is the possibility of control and guarantee of the mechanical properties of these elements. However, for precast elements, it is not advantageous to create original shapes due to the difficulty of formwork of irregular shapes and the impossibility of reusing such a formwork. This has a very negative impact, especially on the financial aspect of the whole process and the final product. This situation can be conveniently solved by the mentioned 3D processing using a robotic hand.

With the mentioned robotic sculpture, the robotic hand quickly and efficiently creates the original shape on the surface of the mixture in a simple formwork with the help of programmed techniques and appropriate tools. Therefore, the need to create a special formwork for each element, which would be used only once, is essentially eliminated.

A more popular 3D technique using cement composite is 3D printing. The robotic processing, mentioned above, shapes the element only on the surface directly into the fresh cement mixture in the formwork and the elements are mostly of a non-load-bearing character. The 3D printing can afterwards be used mainly for creating load-bearing structures. Both these 3D cement composite processing technologies are suitable for producing original construction shapes and eliminate the use of formwork. 3D printing of buildings and other structures are used mainly for vertical load-bearing structures, various home furnishings, or for interesting design works. These 3D technologies have started to develop rapidly, but there are several conditions associated with them.

During the use of a robotic hand, plasticity and controllable setting time are required from the mixture, while for 3D printing technology, mainly good extrudability and hardening of the layers immediately after application is preferred. Another important factor is to ensure a suitable consistency, sufficient processing time, and maintaining the shape of the already extruded structure after the application. When shaping the surface of a cement composite, it is crucial to achieve good workability and consistency of the mixture. The primary object is to create a mixture that will be able to remain in a plastic state for a time sufficient for the robot or artist to create the desired work of art. The plastic state is understood in 3D processing mixtures as a mixture that has the consistency of a stiffer paste, but the onset of solidification is significantly delayed. Currently, there is a lot of extensive research being carried out worldwide to design suitable cementitious mixtures for 3D printing technology. Most of these studies, such as Nerella et al. [1] were focused mainly on extrudability, buildability and processing time. Another team of Kazemian et al. [2] at the University of Southern California have shown that the addition of micro-silica and nanofibers leads to a significant improvement in shape stability, and these fibres have better properties in such a material than the addition of polypropylene fibres. Rahul et al. [3] focused on optimum buildability and extrudability, the optimal value of extrudability was achieved when the yield strength of the mixture was in the range of 1.5-2.5 kPa. After the addition of the additives, the yield strength and also the processing time of the mixture increased significantly. After 30 minutes, the increase was almost double as compared to the reference mixture. Unfortunately, there is currently not many studies focusing on the area of robotic hand sculpturing, so no significant progress was observed here.

Another important and also necessary advancement in the field of civil engineering is the improvement of energy efficiency in buildings. This topic is tightly related to the thermo-physical properties of materials. The thermo-physical properties of the material subsequently affect the heating demand and the associated greenhouse gas emissions. The knowledge of thermo-physical properties can be applied to cementitious composites used in 3D technologies such as robotic sculpturing, specifically in the design and implementation of cladding elements created by this method. It is generally known that the requirements for additional insulation are lower if the material has better thermal properties. This finding is particularly important for 3D printed buildings where the potential implementation of thermal insulation could cause difficulties during installation.

Last but not least, the rheology plays very important role during the design process of any concrete or concrete-similar materials. In the literature, we can find, for instance, Feys [4] and Paul [5], who were dealing with this phenomenon.

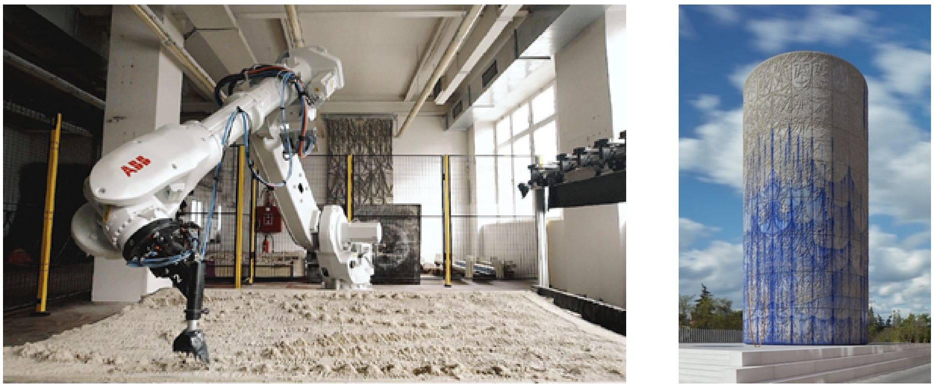
2. Mixtures

As part of the experimental programme, three mixtures were selected and subjected to detailed testing. The first reference mixture marked as number 1 is a commercially available dry mixture MasterFlow 3D 100 from BASF intended for 3D printing. This mixture is based on the main component – Portland cement. The manufacturer describes this dry bagged mixture as a special non-shrinking mixture with a grain size of up to 0.5 mm. As an additional characteristic of the mixture, the manufacturer also mentions a good workability, suitable for 3D printing, of 1 h/+ 20 °C at 0.165 l·kg^{-1} of water, which means high strength, zero segregation, and also fast hardening. The two newly designed mixtures number 2 and 3 are composite mixtures resulting from a suitable combination of cement and commercially available additives. These mixtures in the specific mixing ratios lead to the desired effect. Thanks to a controlled and continuous hydration process, such mixtures retain a sufficient workability time and also have a rapid increase in initial, especially mechanical, properties. These mixtures are subsequently able to achieve a reliable stability after curing. All of these properties make the mixture easy to handle and suitable for a wide range of practical use.

The first mixture (number 1) was mixed exactly according to the procedure specified by the manufacturer. The water-cement ratio for this reference mixture is prescribed to be 0.156. This value of the water-cement ratio is generally relatively low and so it can be assumed that the mixture will contain admixtures or additives which will affect the workability of the fresh mixture. The procedure was, therefore, as follows – Dry mixes 2 and 3 were firstly weighed with a small amount of water and mixed thoroughly. Then, more water was gradually added until the desired consistency of the mixture was reached. The appropriate water-cement ratio was considered to be 0.183 for mixture 2 and 0.163 for mixture 3, and these water-cement ratio values are very close to those of the reference mixture. The mixtures exhibited optimum consistency during casting of the mixtures and forming in the formwork – the consistency of a stiffer paste. Earlier, during the collaboration with Federico Díaz, 3D forming technology was used. Figure 1a shows the forming of sand by a robotic hand, which is then used as part of the formwork for the individual cladding pieces. Figure 1b shows the cladding created by 3D sculpture – Federico Díaz (TUNELBLANKA-INFO) in Prague [6].

3. Experimental programme

The experimental programme included testing of a series of selected mechanical, physical, and thermo-physical properties of mixtures. Test specimens in the



(a). The formwork surface formation process by the robotic hand, see [6].

(b). Artistic application of tiling elements created by a robotic hand in Prague 6 [6].

Figure 1. Two examples of robotic sculpturing, manufacturing process and the final application.

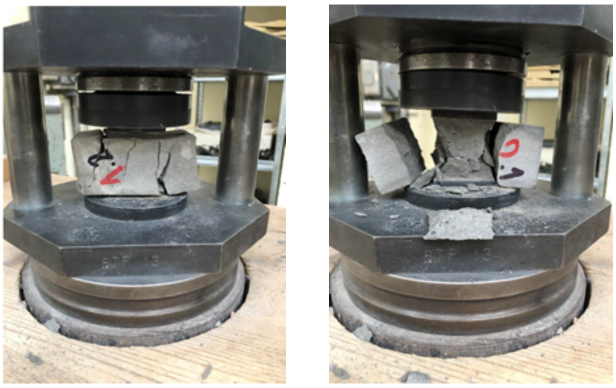
form of cubes with edge lengths of 70 mm and 50 mm and beams with dimensions of $160 \times 40 \times 40$ mm were used to test these properties. The beams were used for mechanical properties tests and cube-shaped specimens for thermal and moisture experiments. Most of the samples, namely two thirds, were stored in an environment with elevated humidity. The remaining samples intended for 7-day physical properties were placed in a hot-air oven where drying was carried out until the weight settled. Physical and mechanical properties of the samples were tested, as usual, after 7, 14, and 28 days. Due to the continuous procedure of measurements carried out at different stages of concrete hardening, we are able to obtain a more comprehensive overview of the investigated cementitious composites and the evolution of their characteristics.

3.1. Mechanical properties

The whole experimental programme started with the determination of the mechanical properties on five samples for each mixture. Flexural tensile strength test was performed on $160 \times 40 \times 40$ mm specimens with a support distance of 100 mm using a three-point bending test. After the bending test, a compression test according to EN 12390-3 was performed on both fragments, with a contact area of 40×40 mm. Figure 2a and Figure 2b show a compression test in which a clear difference in failure can be observed between the sample of mixture 1 and 2. This mode of failure indicates that mixture 1 contains an admixture of a specific type of microfibre, which here acts as a dispersed reinforcement.

3.2. Physical and thermo-physical properties

Basic physical parameters were determined using standard test methods, namely gravimetry and pycnometry, such as bulk density and matrix density. In this research, the thermo-physical parameter tests were



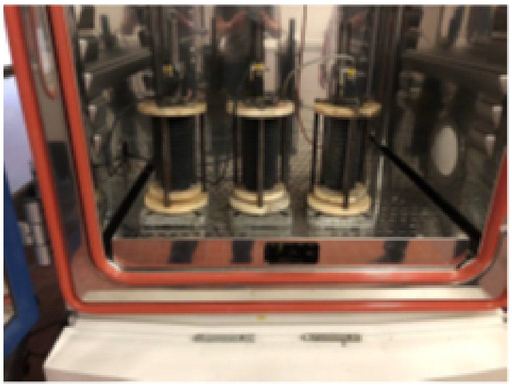
(a). Pressure test of the sample 1 with fibres and its failure pattern.

(b). Pressure test of the sample 2 without fibres and its failure pattern.

Figure 2. Testing of the mechanical properties by hydraulic jack ISOMET 2114.

carried out using the stationary hot disk method. This method is based on the transfer of heat to the material and subsequently, depending on the response, the coefficient of thermal diffusivity, the coefficient of thermal conductivity and also the coefficient of specific heat capacity were evaluated as well. An ISOMET 2114 instrument was used for these measurements, which is equipped with interchangeable probes. These measurements were performed on cubes with an edge length of 70 mm, one measurement for each sample. After the drying process, the samples were placed in a desiccator until the weight was stabilised, see Figure 3a. A silica gel was applied to the bottom of the desiccator to provide a stable and especially non-moist environment. Subsequently, after the connection, the entire instrument was set up and a hot disk was attached to the top wall of the sample according to the expected measurement range. Finally, the required number of measurements was set.

(a). Samples placement of tested mixtures in a desiccator.



(b). The measurement of changes using a rubber wavy-line mould.

Figure 3. Testing of the physical and thermo-physical properties.

As a part of the experimental program, measurements of volume changes in the solidification and hardening phases were also performed. It is generally known that the formation and subsequent development of cracks in the hydration phase is the result of volume changes and these significantly affect the durability and service life of the entire structure. In the case of artistic processing, crack management is even more important. The aim, is therefore, to restrict, as much as possible, the volumetric changes that play an important role in the artistic processing of cementitious composites. Most often, these volume changes are examined during the hydration phase, when the fresh mixture changes from a so-called quasi-fluid to a solid phase of the mass. The volume changes of the mixtures were investigated using the rubber wavy-line false mould method on the rubber, see Figure 3b. A mould of this shape is placed in a gripping chair, which primarily prevents horizontal deflection of the mould. This wavy-line shape has the required properties, such as high vertical flexibility, while at the same time sufficiently resisting horizontal length changes.

In the first step, the moulds were filled with the prepared mixtures and then compacted by hand. Immediately afterwards, the filled and compacted moulds with the fresh mixture were covered with a foil on the upper surface. This step should eliminate the exchange of moisture parameters with external conditions. A reflective surface was then placed on top of it for measurements by laser sensors. To ensure a constant ambient temperature, the whole assembly was placed in a thermostatic chamber. Thereafter, in order to achieve a non-contact method of optical distance measurement, the device is equipped with a sensor support structure for a simple displacement measurement in the vertical direction. Scanning was performed by using laser reflective sensors. There is a reflective surface on the top of the specimens which allows the reflection of the transmitted and received light beam by the laser sensor. The reflection is recorded by the measuring station during the measurement, from where it is exported as a text file and then evaluated. With this method, we can continuously measure length changes with an accuracy of $0.6\,\mu$m.

4. Results and Discussion

4.1. Mechanical properties

The graphs below show the behaviour of the tested cementitious composites in flexural tensile strength (Figure 4) and the evolution of compressive strengths (Figure 5) over time.

If we take a look at the flexural tensile strength, it can be seen that mixes 1 and 2 showed a rapid increase at 7 days and a further step increase at 28 days compared to mix 3 where the strength increased continuously in a linear trend. The presence of fibres in mix 1 was already visible in the flexural tensile strength tests. These fibres are activated as the load develops and increase the flexural tensile strength. It is ensured by preventing brittle fractures. The values of mixes 1 and 3 were comparable for the 28-day tensile strengths. Thus, it can be concluded that the results obtained from the compressive and tensile strength tests indicate that the tested mixes 1 and 3 are close to the values of high strength concrete (60 MPa in compression), see [7].

Regarding the development of the compressive strength values of the individual mixtures 2 and 3, we can observe a standard trend with almost the same values after 7 and 14 days. After 28 days, however, we can notice a significant difference between the mixtures, especially for mixture 3, where we can observe a rapid increase in compressive strength from 20 MPa to 71.3 MPa. For both new mixtures 2 and 3, we can see a faster increase in strength in comparison to the reference mixture 1. However, compared to commercially available concretes, our reference mixture 1 still achieved relatively high strength characteristics.

The Table 1 summarises samples 1, 2 and 3, their densities, and measured mechanical properties. According to Pytlík [8], these composites can be classified

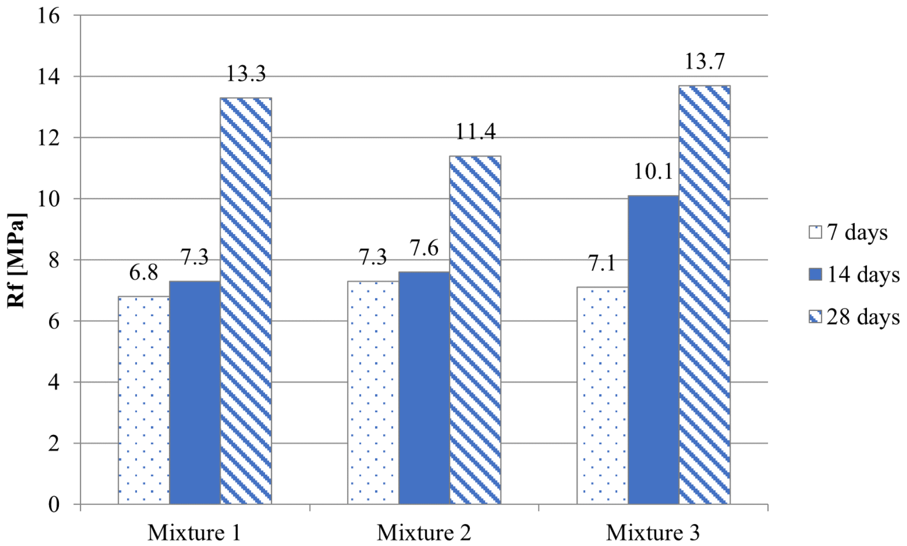


Figure 4. Graphical representation of the flexural tensile strength after 7, 14, and 28 days.

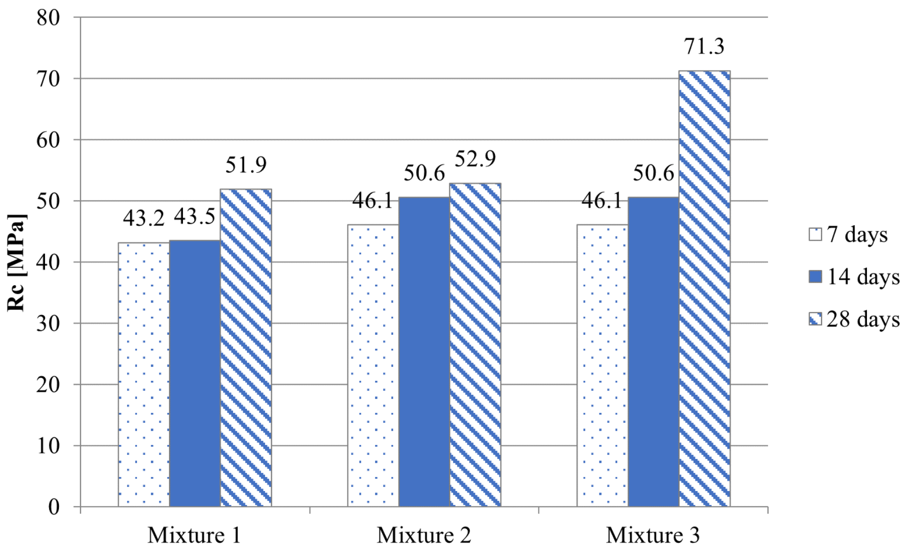


Figure 5. Graphical representation of the compressive strength after 7, 14, and 28 days.

Mixture	Density ϱ [kg·m^{-3}]	Compressive strength $\mathbf{R_c}$ [MPa]	Tensile bending strength $\mathbf{R_f}$ [MPa]
1	1990	51.9	13.3
2	2040	52.9	11.4
3	2100	71.3	13.7

Table 1. The density and mechanical properties of the cement composites for 3D printing technology.

Mixture	Age [days]	λ [$\mathrm{Wm^{-1}K^{-1}}$]	$\mathbf{c}_{\varrho}$ [$\mathrm{Jkg^{-1}K^{-1}}$]	**A** [$\times\mathbf{10^{-6}}$] [$\mathrm{m^2s^{-1}}$]
1	7	1.17	881.01	0.68
	14	1.19	922.43	0.68
	28	1.28	896.85	0.73
2	7	1.24	869.60	0.76
	14	1.28	948.13	0.72
	28	1.35	951.95	0.75
3	7	1.36	876.09	0.79
	14	1.45	914.09	0.81
	28	1.48	870.86	0.87

Table 2. Thermophysical properties of measured mixtures.

from lightweight concrete LC (800–2000 $\mathrm{kg \cdot m^{-3}}$) to ordinary concrete C (2000–2600 $\mathrm{kg \cdot m^{-3}}$), see [8].

The reference mixture 1 had the lowest bulk density, while mixture 3 reached the highest values out of the three mixtures, as shown in Table 1. These bulk densities also correspond to the measured compressive strengths. From the bulk density evaluations, it is apparent that mixture No. 1 is the most suitable for 3D printed structures. Mixtures 2 and 3 have higher bulk densities and can, therefore, be considered less suitable for non-load bearing structures. Although they have a higher compressive strength, which might seem to be an advantage, their relatively high bulk density increases the self-loading of the structure in a negative way. For the purposes of lightweight cladding materials, such as tiles or pavers, a high bulk density is disadvantageous.

4.2. Physical and thermo-physical properties

This section summarises the results and evaluation of the physical and thermo-physical properties of the tested cementitious composite mixtures. The data in Table 2 presents the material results, specifically the thermo-physical characteristics, as they relate to research in the development of mixtures for 3D robotic processing. The table shows the measurements of these properties over a range of 7/14/28 days. The time setting is determined by the simultaneous measurement of the mechanical properties. Only a slight increase in the measured thermo-physical values can be observed, while the strength characteristics increase significantly with increasing concrete age.

As can be seen from the measured values of the thermal conductivity coefficient, all investigated composites can be classified according to their thermophysical properties and compared to the standard concrete with a standard density of 2100–2300 $\mathrm{kg \cdot m^{-3}}$, which has a thermal conductivity coefficient of approximately 1.23–1.36 $\mathrm{Wm^{-1}K^{-1}}$, mix number 3 reached values of 1.481 $\mathrm{Wm^{-1}K^{-1}}$, which is the same value of thermal coefficient as for reinforced concrete according to Ražnjević, see [9]. The authors here presented measured values in the range of 1.43–1.74 $\mathrm{Wm^{-1}K^{-1}}$.

When considering the design of elements that will be exposed to different temperature and humidity conditions, we must take into account that these values are given for dry material. Therefore, we must also consider the practical humidity value for the cladding element. For materials with a porous structure, the higher temperature is directly proportional to the heating of the material in the pores, which can cause an increase in the values of the thermo-technical and thermo-physical properties. Therefore, a material with a lower thermal conductivity coefficient value works better as an insulator. The values shown in Figures 6 and 7 correspond to the strength values of the individual mixtures. This indicates that the higher the strength of the material, the worse the thermal insulation properties of the material.

The mixture 1 shows the best results in terms of conductivity due to the lowest value of thermal conductivity coefficient, next is mixture number 2, and the last mixture 3 is the least suitable from the thermal conductivity point of view, as shown in Figures 6. However, this comparison has no real meaning with respect to the settled moisture and the sample (drying) parameters. In real conditions, moisture would play an important role in increasing thermal conductivity. Figures 7 shows the evolution of specific heat capacity, where mixture number 2 demonstrates the best values and its values improve further with increasing age, for example, after 28 days. On the contrary, the mixtures 1 and 3 report a decrease in their heat capacities after 28 days.

However, all mixtures displayed different values during the solidification phase in volume changes, as shown in Figures 8. Mixture 3 showed the most significant volume changes, while the other mixtures 1 and 2 had almost identical values in this aspect. Interestingly, it also has the best strength properties. The importance of each these properties depends on the purpose of application and use. It is a matter of

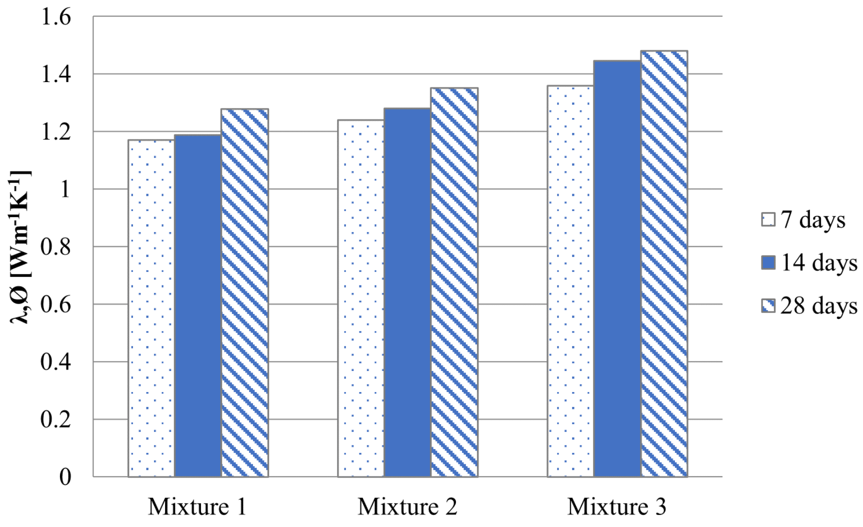


Figure 6. Graphical representation of the thermal conductivity coefficient after 7, 14, and 28 days.

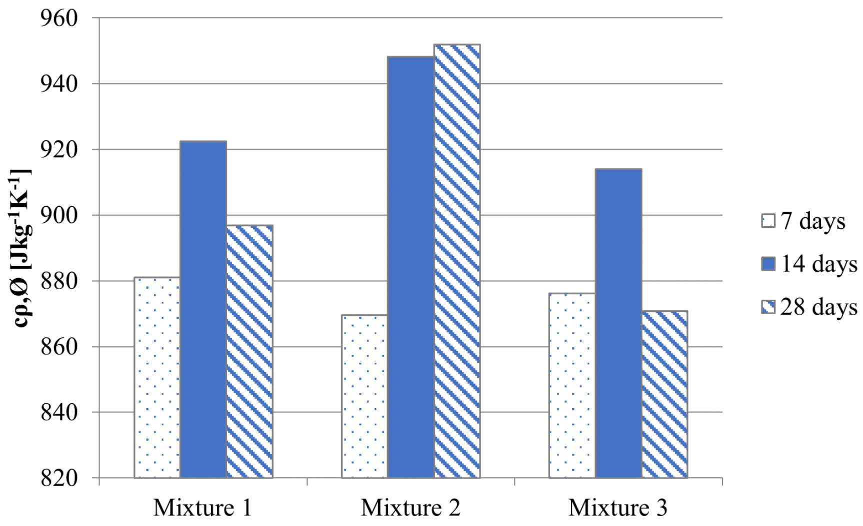


Figure 7. Graphical representation of development of specific heat capacity at 7, 14, and 28 days.

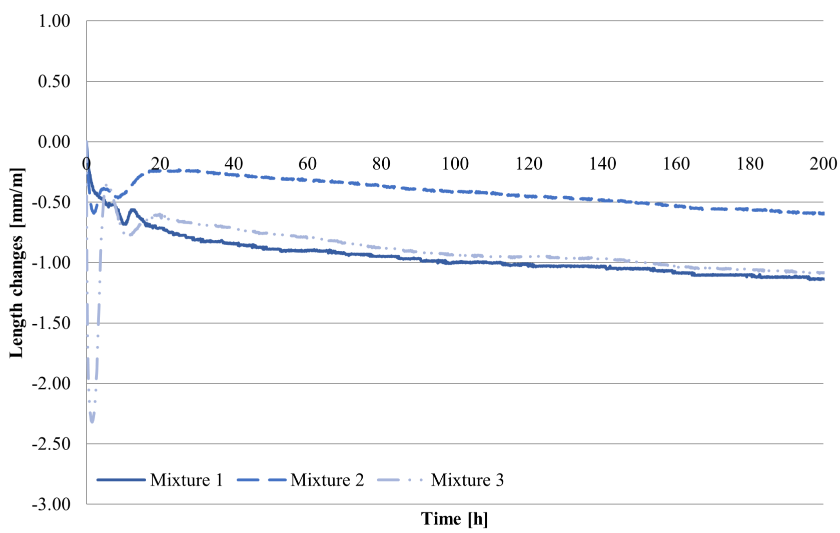


Figure 8. Volume changes overview of measured mixtures.

preference whether high strength or durability is more important for the resulting material.

5. Discussion

As other authors who also devoted their efforts to the development of materials for 3D robotic printing, we too arrived to results that showed good and similar values of compressive strength as, for example, mixtures from S. J. Woo et al. [10], who determined the appropriate compressive strength after 28 days of such materials to be at least 50 MPa, our reference mixture 1 and mixture 2 slightly exceeded these values, with mixture 3 reaching a level of compressive strength exceeding 70 MPa after 28 days. This is almost similar value to the mixture tested by A. P. Rubin et al. [11]. In terms of tensile strength during bending, our samples showed almost two-times higher values after 28 days than the mixture from S. J. Woo et al. [10], on the contrary, they were only slightly lower than the measured mixture values from A. P. Rubin et al. test study [11]. According to the volume weights of our reference mixture and our mixtures 2 and 3, we classify these materials in the category of light concrete according to Pytlík [8]. Our mixtures showed slightly lower bulk weights than the material from S. J. Woo et al. [10]. According to the study of E. Lublasser et al. [12], we know that the lower the volumetric weight, the more suitable the material is for use in such a technology. Therefore, we can state that our achieved results reached very similar measured values to similar studies by other authors who dealt with the development and measurement of mechanical and thermophysical properties of such mixtures of materials for 3D printing.

6. Conclusion

The results of the experimental program show that all tested mixtures are suitable for robotic processing. It can be stated that mixture 1 is more suitable for 3D printing and mixtures 2 and 3 are more suitable for robotic sculpturing, mainly due to their rheology.

According to the mechanical property measurements, the proposed mixtures show better compressive strength values than the reference mixture. However, their bulk density and thermal insulation values are higher, and therefore worse than in the case of the reference mixture. The intended application of these materials is very important. Unless it is necessary to use a high-strength composite, it is not recommended to use mixtures 2 and 3, which would significantly increase the self-weight of the structure. Considering the physical properties of the tested composites, density may be the most important factor when the mixture is used for cladding material with artistic elements. On the contrary, strength and thermal insulation properties will be crucial if the material is used for load-bearing, solid or other external structure.

The obtained knowledge and measured values will be used in a future investigation within the project, which will be focused on a better balance of cement mixtures with focus on adjustability of processing time and other physical-mechanical properties, whose optimal configuration is very important for 3D processing. The main goal of the project is to create a mixture that is suitable for robotic processing and at the same time commercially available as a dry bagged mixture.

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References

[1] V. N. Nerella, S. Hempel, V. Mechtcherine. Effects of layer-interface properties on mechanical performance of concrete elements produced by extrusion-based 3D-printing. *Construction and Building Materials* **205**:586–601, 2019. https://doi.org/10.1016/j.conbuildmat.2019.01.235

[2] A. Kazemian, X. Yuan, E. Cochran, B. Khoshnevis. Cementitious materials for construction-scale 3D printing: Laboratory testing of fresh printing mixture. *Construction and Building Materials* **145**:639–647, 2017. https://doi.org/10.1016/j.conbuildmat.2017.04.015

[3] A. Rahul, M. Santhanam, H. Meena, Z. Ghani. 3D printable concrete: Mixture design and test methods. *Cement and Concrete Composites* **97**:13–23, 2019. https://doi.org/10.1016/j.cemconcomp.2018.12.014

[4] D. Feys, K. H. Khayat, R. Khatib. How do concrete rheology, tribology, flow rate and pipe radius influence pumping pressure? *Cement and Concrete Composites* **66**:38–46, 2016. https://doi.org/10.1016/j.cemconcomp.2015.11.002

[5] S. C. Paul, Y. W. D. Tay, B. Panda, M. J. Tan. Fresh and hardened properties of 3D printable cementitious materials for building and construction. *Archives of Civil and Mechanical Engineering* **18**(1):311–319, 2018. https://doi.org/10.1016/j.acme.2017.02.008

[6] TunelBlanka-Info. Výtvarník a robot proměnili výdech v umělecké dílo. 2017, [2022-11-30], https://www.tunelblanka.info/vytvarnik-a-robot-promenili-vydech-v-umelecke-dilo/.

[7] ČSN EN 12390-3 Část 3. Pevnost v tlaku zkušebních těles. 2017, Úřad pro technickou normalizaci, metrologii a státní zkušebnictví.

[8] P. Pytlík. *Technologie betonu*. 2. vydání. VUTIUM, Brno, Czech Republic, 2000.

[9] K. Ražnjevič. *Thermodynamic tables*. 1. vyd. Bratislava: Alfa, 2 sv. Edícia energetickej literatúry, 1984.

[10] S.-J. Woo, J.-M. Yang, H. Lee, H.-K. Kwon. Comparison of properties of 3D-printed mortar in air vs. underwater. *Materials* **14**(19):5888, 2021. https://doi.org/10.3390/ma14195888

[11] A. P. Rubin, L. C. Quintanilha, W. L. Repette. Influence of structuration rate, with hydration accelerating admixture, on the physical and mechanical properties of concrete for 3D printing. *Construction and Building Materials* **363**:129826, 2023. https://doi.org/10.1016/j.conbuildmat.2022.129826

[12] E. Lublasser, T. Adams, A. Vollpracht, S. Brell-Cokcan. Robotic application of foam concrete onto bare wall elements – Analysis, concept and robotic experiments. *Automation in Construction* **89**:299–306, 2018. https://doi.org/10.1016/j.autcon.2018.02.005