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EXPERIMENTAL STUDY OF THE BASIC MECHANICAL PROPERTIES OF HARDENED GYPSUM PASTE MODIFIED WITH ADDITION OF POLYOXYMETHYLENE MICROGRAINS

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The development of the construction industry and the growing ecological awareness of society encourages us search for new solutions to improve building materials. Therefore, an attempt was made to improve building gypsum by modifying it with the addition of polyoxymethylene (POM). Polymer grains, with a particle size below and above 2 mm, were added to the samples in the amount of 1% and 2% relative to gypsum. The work contains the results of bending and compressive strength tests of prepared gypsum beams. It was shown that the compressive strength increased by 7% and the bending strength increased by 31% when compared to the reference test without the addition of polymer. All the obtained gypsum composites were characterized by a growth of strength. The best results were obtained for the sample containing gypsum composite modified with polymer in the amount of 1% and with a diameter of grains below 2 mm.

Keywords: polyoxymethylene (POM), building materials, modified gypsum, compressive strength, bending strength

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1. INTRODUCTION

Gypsum can be modified with the use of various chemical additives. Such additives can be, among others, accelerants, retarders, glass fibres, cellulose fibres, vermiculite, aerogels, microspheres and polymers, and copolymers [1,2,3,4,5,6,7]. All such modifications affect the properties of gypsum composite and its application.

Haratym and Klepka [8] made attempts to increase the strength of gypsum using polyethylene additives and rubber granulate additives. Depending on the percentage share of polymeric plastic in gypsum and polymeric mortar, the result was the change of bending and compressive strength and the speeding up the process of the reaction with water. Adding polymer granulate to the gypsum mixture influenced the growth of elasticity of mortar and enabled the option of carrying mechanical loads without damage to the structure of a sample.

Najim et al. [9] studied the influence of adding poly(vinyl acetate) PVAc on the properties of gypsum composite. The PVAc content in gypsum was varied from 2.5 to 10%. The physical and mechanical properties of the produced composite were evaluated. Water absorption by the composite was reduced with an increase of the percentage of PVAc. The prepared composites showed a clear improvement in terms of impact, compressive strength and modulus of elasticity when the PVAc proportion in gypsum was increased from 2.5 to 10%. Bending strength, however, was reduced.

Apart from gypsums, concrete is also modified with the use of plastics. Management of plastic waste in the modification of concrete and mortars is an object of research in many research centres. In the subject literature, we can find articles about the modification of concrete with the use of, among others, ABS (acrylonitrile-butadiene-styrene), and the waste of polyurethane foam, expanded polystyrene (EPS), polystyrene (PS), fitted carpet waste containing polyamide (PA) and polypropylene (PP), polyethylene waste of high and low density (LDPE, HDPE), poly(vinyl chloride) (PVC), tyre waste, tyre fibres, grinded electric cables, powdered rubber, melamine-formaldehyde resins and poly(ethylene terephthalate) (PET) [2,5,10,11,12,13,14,15,16]. The use of such materials, which are considered as an environmental problem, may considerably reduce the costs of producing concrete modified with polymers. The researchers examining the use of plastic waste, due to the positive results they have obtained, see a huge potential in continuing such research.

Acetal polymers, also known as polyoxymethylene or polyacetal, are formaldehyde-based thermoplastics that have been commercially available since the 1960s. Polyformaldehyde is thermally unstable. It decomposes on heating to yield formaldehyde gas. Two methods of stabilizing

polyformaldehyde for use as an engineering polymer were developed and introduced by DuPont in 1959 and Celanese in 1962 [17]. DuPont's method for making polyacetal yields a homopolymer through the condensation reaction of polyformaldehyde and acetic acid (or acetic anhydride). The acetic acid puts acetate groups ($CH_3COO–$), which provide thermal protection against decomposition to formaldehyde, at the ends of the polymer, as shown in Fig. 1 [17].

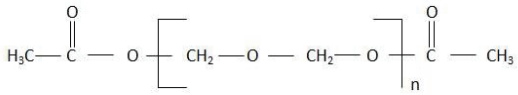

Fig. 1. Chemical structure of acetal homopolymer [17]

The Celanese route for the production of polyacetal yields a more stable copolymer product via the reaction of trioxane, a cyclic trimer of formaldehyde, and a cyclic ether, such as ethylene oxide or 1,3 dioxolane. The polymer structure is given in Fig. 2 [18].

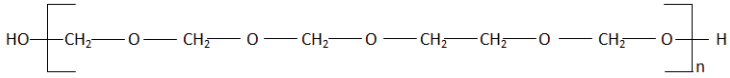

Fig. 2. Chemical structure of acetal copolymer [18]

The improved thermal and chemical stability of the copolymer versus the homopolymer is a result of randomly distributed oxyethylene groups. These groups offer stability to oxidative, thermal, acidic, and alkaline attack. The raw copolymer is hydrolysed to an oxyethylene end cap to provide a thermally stable polyacetal copolymer. The copolymer is also more stable than the homopolymer in an alkaline environment [18].

A review of the literature showed only a few publications related to the using of POM for the modification of building materials. Most works are related to the modification of concrete. Therefore, only the results of measurements of concrete were quoted. The authors did not find any articles related to the use of polyoxymethylene in gypsum materials, and therefore the interest of using this polymer in gypsum composites is a new area of research.

In the United States Patent [19], we can find a work related to the use of polyoxymethylene fibres in concrete. Bassetti et al. [19] showed that POM copolymers can be utilized to form fibrous additives for concrete, i.e., microfibers and/or macrofibers. For instance, concrete can include microfibers in an amount up to about 1.78 $kg \cdot m^{-3}$, and include macrofibers in an amount up to about 4.75 $kg \cdot m^{-3}$. The authors showed that the addition of polyoxymethylene to concrete may improve its properties. Fibers formed of POM copolymers can exhibit increased hydrophilicity when compared to fibers formed of more traditional polymers, such as polypropylene. The increased hydrophilicity of the

POM copolymer can improve mixing between POM polymeric fibers and the concrete mix, and also prevent blooming of the fibers to the surface of the concrete during the curing process [8]. In works [20,21], the researchers studied the flexural performance and the splitting tensile strength of polyoxymethylene fiber with different contents and different lengths reinforced concrete. The results showed that the flexural performance of 6 mm POM fiber reinforced concrete at a 0.6 $kg \cdot m^{-3}$ content was the best. POM fibers at the length of 6 mm, and with 12 mm reinforced concrete, had good flexural properties at a content of 0.9 $kg \cdot m^{-3}$. In addition, concrete that is rainforced with the 3 mm and 6 mm long POM fibres has the best flexural performance [20]. The results show that the splitting tensile properties of 6 mm POM fiber reinforced concrete at a 0.9 $kg \cdot m^{-3}$ content is the best [21].

The main aim of this work was to check the possibility of using polyoxymethylene waste to increase the bending and compressive strength of hardened gypsum paste. The authors assumed that polyoxymethylene will significantly improve the mechanical properties of modified gypsum. In addition, it was assumed in the study that the size of polymer grains may have an effect on the mechanical properties of gypsum. There are only a few studies in the literature related to the modification of building materials with the addition of polyoxymethylene. However, most of them concern concrete. Therefore, there is a need to study the effect of polyoxymethylene on the properties of gypsum modified with this polymer.

2. EXPERIMENTAL PROCEDURES

2.1. MATERIALS

The material applied during the research was ordinary β building gypsum (Dolina Nidy, Pinczow). As a starting material, the natural gypsum powder widely available on the market and that meets the standard requirements was selected. The amount calcium sulphate ($CaSO_4$) in the gypsum was 90.98%, whereas other components were as follows: $CaCO_3$—2.79%, SiO_2—1.62%, montmorillonite—3.07%, clays—0.79%, chlorite—0.16%. The basic physical properties of the building materials used in the research are presented in Table 1. These parameters were obtained from safety data sheets made available by the producer [22].

Table 1. Physical properties applied in research of β building gypsum [22]

Building material	Relative density d_R [kg·m^{-3}]	Bulk density d_B [kg·m^{-3}]	*pH* in aqueous solution	Colour	Appearance	Setting time [min]
Building gypsum	2300	900	7÷8	Grey	grey-yellow powder	3

In this research, the copolymer waste of polyoxymethylene (POM) Hostaform C9021 (Celanese, Sulzbach, Germany) was used. POM possesses a linear structure with a highly crystalline quality that provides a variety of characteristics: outstanding wear, long-term fatigue, toughness, and creep resistance, as well as excellent resistance to moisture, solvents and strong alkalis [18,23].

POM products are very versatile and as a result they are used in a wide array of applications across many industries. POM may be processed by injection molding, extrusion, compression molding, rotational casting, or blow molding [18,23]. Applications and uses include the following: metal and glass replacement in automotive and industrial equipment, consumer goods, appliances, and electrical and office automation [24]. Table 2 shows the basic physical properties of the polyoxymethylene used in the study.

Table 2. Physical properties of polyoxymethylene applied in research [23]

Parameter	Value	Unit
Density	1410	[kg·m^{-3}]
Water absorption (23°C)	0.65	[%]
Humidity absorption (23°C/50%RH)	0.2	[%]
Melting temperature (10°C/min)	166	[°C]
Tensile modulus (1mm/min)	2850	[MPa]
Tensile stress at yield (50 mm/min)	64	[MPa]
Tensile strain at yield (50 mm/min)	9	[%]
Nominal strain at break (50 mm/min)	30	[%]
Tensile creep modulus (1h)	2500	[MPa]
Tensile creep modulus (1000h)	1300	[MPa]
Charpy impact strength at 23°C	220	[kJ·m^{-2}]
Charpy impact strength at -30°C	220	[kJ·m^{-2}]
Charpy notched impact strength at 23°C	6.5	[kJ·m^{-2}]
Charpy notched impact strength at -30°C	6	[kJ·m^{-2}]

The grain size distribution of the gypsum binder and of the addition of polyoxymethylene was determined using an Analysette 22 MicroTec laser grain size analyser [25]. Three repetitions of particle size distributions measurements were made during the experiment for both gypsum (Fig. 3) and POM (Fig. 4). These graphs show particle size distribution (PSD) and frequency distribution (FD) dependencies for studied building gypsum and polymer.

In articles [25,26], the authors analysed the granulation of building materials used during tests according to Fraunhofer theory. The accurate methodology of measurement with the use of laser diffraction method was presented in the above mentioned works. The results obtained from the three

measurements are similar, which explains the fact that the lines mostly overlap in the graph.

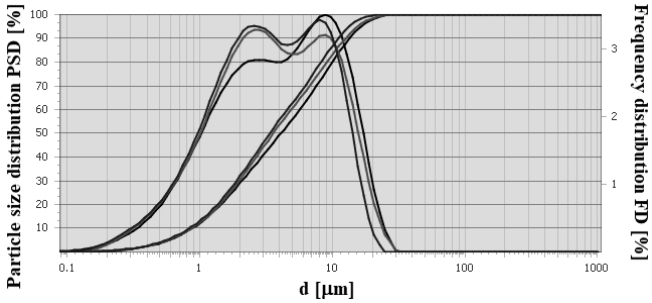

Fig. 3. Grain size distribution and frequency distribution of gypsum binder

Polyoxymethylene granulate had a quite diversified granulation, and therefore it was sifted and divided into two fractions. The first fraction had granulation with a diameter of up to 2 mm, and the second one of more than 2 mm. The fraction with a diameter of up to 2 mm was analysed in the granulometric analysis with the use of laser diffraction. The results were presented in article [26].

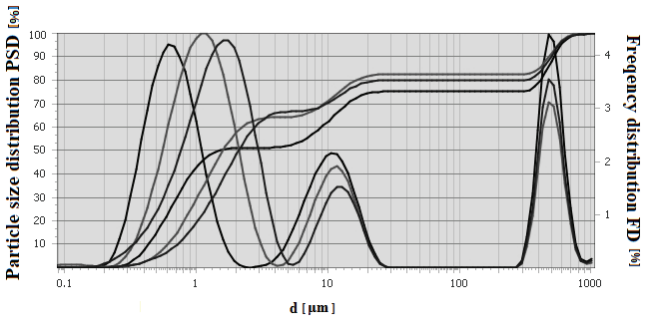

Fig. 4. Grain size distribution and frequency distribution of POM addition [26]

In order to present the structure of gypsum and polymer, images of the used substances were made by a laboratory stereoscopic microscope with the use of 100x magnification and the Moticam camera, which allowed for observation of details with a size of up to 20 µm (Figs. 5a-5c). Due to the visible differences in the structure of the micro additive, it was assumed that the polymer would change the strength of gypsum composites to a different extent. Because the polymer had very large particles (Fig. 5b) not used to form gypsum composites, they had to be sieved and separated. The polymer used

in this experiment was waste obtained from a factory producing precision plastic parts.

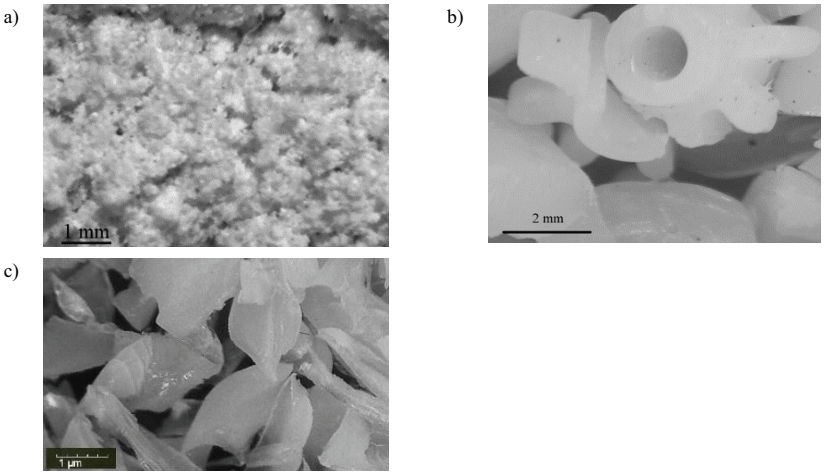

Fig. 5. Microscopic images: a) gypsum, b) polyoxymethylene constituents prior to the sieving process, c) polyoxymethylene grains obtained after the sieving process

2.2. EXPERIMENTAL SETUP

Gypsum beams with dimensions of 160x40x40 mm were prepared in accordance with the PN-EN 13279-2:2014-02 norm [27]. In all the tests, the constant ratio of water to gypsum (w/g=0.6) was applied, which was recommended by the producer of the product.

For the purpose of the experiment, 15 samples were prepared that were demolded after 24 hours and then conditioned in room temperature between 26°C and 28°C at a relative humidity of 53±2%. Then, according to the norm [27], after 7 days the specimens were used during bending and compressive strength tests. In order to conduct the strength tests, standard gypsum beams and beams containing gypsum with an additive of polyoxymethylene were prepared. The prepared samples are presented in Table 3. The sample markings included the numbers between 0 and 4 and defined the amount and size of the grains of the applied polyoxymethylene in the gypsum mixture. The letter markings (A, B, C) described the three repetitions that were made during the experiment. Two fractions were used to prepare the gypsum specimens with a polyoxymethylene additive in order to check the influence of the diameter of the added polymer on the strength of these beams.

Table 3. The specimens used in bending and compressive strength tests

Sample marking	The composition of tested sample
0 - A, B, C	Gypsum
1 - A, B, C	Gypsum with the addition of POM in the amount of 1 % and diameter of grains up to 2 mm
2 - A, B, C	Gypsum with the addition of POM in the amount of 2 % and diameter of grains up to 2 mm
3 - A, B, C	Gypsum with the addition of POM in the amount of 1% and diameter of grains over 2 mm
4 - A, B, C	Gypsum with the addition of POM in the amount of 2% and diameter of grains over 2 mm

The bending strength tests included the determination of the force necessary to break the sample made of gypsum mortar with the dimensions of 160 x 40 x 40 mm. This force works centrally on the sample supported on the supports distant from each other by l = 100 mm. The test was carried out with the use of the testing machine ZD 10/90 for static tests in an air temperature of 25.1°C and humidity of 42.8%. The compressive strength tests included the compression of a sample until the moment of its destruction with the use of the same machine as in the previous strength tests.

3. RESULTS AND DISCUSSION

3.1. BENDING AND COMPRESSIVE STRENGTH TESTS

According to the PN-EN 13279-2:2014-02 norm [27], the bending strength for all the samples was calculated. The obtained results of the bending strength f_{zg} and compressive strength f_{sc} of the tested samples are presented in Table 4. The practical effect of the addition of POM grains on bending tensile strength is negligible.

Table 4. The average values of bending strength f_{zg} and compressive strength f_{sc} of modified gypsum composites

Sample tested	f_{zg} [N·mm^{-2}]	f_{sc} [N·mm^{-2}]
0	4.88	10.55
1	4.82	11.99
2	5.22	12.00
3	4.95	13.80
4	4.33	11.06

Compressive strength in accordance with the PN-EN 13279-2:2014-02 norm is calculated according to the following formula (3.1), where f_{sc} is compressive strength [N·mm^{-2}], F_{sc} is maximum breaking force [N], and the number *1600* is a compressed surface with dimensions of 40mm x 40mm [mm^2].

(3.1) $$f_{sc} = F_{sc}/1600$$

It was found that all the applied modifications caused the growth of compressive strength. The highest strength f_{sc} was obtained for sample 3, which is the gypsum composite modified with polyoxymethylene in the amount of 1% in comparison with the gypsum with a diameter of grains below 2 mm. The average value of strength f_{sc} for this mixture is 13.80 N·mm^{-2}. It is higher by almost 31% in comparison with the sample without polymer. In the case of samples 1 and 2, which were the composites containing polyoxymethylene in the amount of 1% and 2% with a diameter of grains of up to 2 mm, the average values are very similar and cause the growth of strength of the beams by about 1.45 N·mm^{-2}. This value increased by 13.7% in comparison with the gypsum. The lowest value of strength was obtained for sample 4 containing modified POM in the amount of 2% with a diameter of grains of up to 2 mm. In this case, the compressive strength increased by 0.51 N·mm^{-2}. There are few studies in the literature related to the modification of building materials with the addition of polyoxymethylene, with most of them concerning concrete. Therefore, there is a need to further study the effect of polyoxymethylene on the properties of gypsum modified with this polymer.

4. CONCLUSIONS

The growth of compressive strength and the lack of growth of strength during bending is caused by reduction of porosity of gypsum samples. The addition of polyoxymethylene that is built in composite structure results in its modification, causing visible changes in compressive strength of gypsum samples. The less porous gypsum, the higher compressive strength. Whereas, structure of gypsum is disturbed during bending, causing its quick destruction.

Adding microfilaments to gypsum mixtures can bring many benefits. The filaments distributed in gypsum mortar create three-dimensional network. In this way, they bind substances in mortar and thicken the whole structure. It constitutes some sort of micro-reinforcement of mortar. Such polymers contribute to increased mechanical strength of gypsum composite. They have higher mechanical parameters, therefore, they are applied to reinforce construction products of high strength, especially within the scope of bending strength.

Therefore, it may be assumed that the strength of gypsum can have the form of applied micro-additions. Bending strength improves if the addition is in the shape of filaments and gest tangled in the whole structure of a sample. However, polyoxymethylene has no such structure, causing tearing of homogenous form of gypsum and unnoticeable impact on changes of bending strength. The plastic

used, which is POM, has different particle sizes, irregular shape and rough surface, which can affect the way polymer connect with gypsum and thus its strength.

The analysis of the obtained results showed that the use of polyoxymethylene in order to modify gypsum is an issue with promising prospects and one which requires further research. Adding polyoxymethylene to gypsum composites in the amount of 2% and with a diameter of grains below 2 mm caused the growth of bending strength by 7% in comparison with the standard sample. The strength values of composites containing polymer decreased in the remaining cases. The results of the compressive strength tests of modified gypsums are very interesting and promising. All the obtained composites were characterized by a growth of strength. The best results were obtained for the sample containing gypsum composite modified with polymer in the amount of 1% and with a diameter of grains below 2 mm. The value f_{sc} in the discussed case increased by 30% in comparison with the standard sample. The lowest value of strength was obtained for the sample containing polymer in the amount of 2% and with a diameter of grains of more than 2 mm. In this case, the compressive strength was higher by only 5% in comparison with the gypsum sample without polymer.

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Tab. 4.Wartości średnie wytrzymałości na zginanie f_{zg} oraz na ściskanie f_{sc} modyfikowanego gipsu

BADANIE EKSPERYMENTALNE PODSTAWOWYCH WŁAŚCIWOŚCI MECHANICZNYCH UTWARDZONEGO ZACZYNU GIPSOWEGO MODYFIKOWANEGO DODATKIEM MIKROZIAREN POLIOKSYMETYLENU

Słowa kluczowe: polioksymetylen (POM), materiały budowlane, gips modyfikowany, wytrzymałość na ściskanie, wytrzymałość na zginanie

STRESZCZENIE:

WPROWADZENIE

Głównym celem pracy było sprawdzenie możliwości wykorzystania odpadów polioksymetylenowych (POM) w celu zwiększenia wytrzymałości na zginanie i ściskanie zaprawy gipsowej. Autorzy założyli, że polioksymetylen znacznie poprawi właściwości mechaniczne modyfikowanego gipsu. Ponadto przyjęto w badaniach, że wielkość ziaren polimeru może mieć wpływ na właściwości mechaniczne gipsu. W literaturze jest niewiele badań dotyczących modyfikacji materiałów budowlanych z dodatkiem polioksymetylenu. Większość z nich dotyczy jednak betonu. W związku z tym istnieje potrzeba zbadania wpływu POM na właściwości gipsu.

MATERIAŁY I METODY

Modyfikowane próbki wykonano przy użyciu gipsu budowlanego zmieszanego z granulatem polioksymetylenu o dwóch frakcjach uziarnienia do 2 mm i powyżej 2 mm. Wykonano również próbki czystego gipsu, będące próbkami referencyjnymi. Stosunek wody do gipsu był stały i wynosił w/g = 0,6, zgodnie z zaleceniem producenta. Na potrzeby eksperymentu wykonano 15 próbek. Zawierały one polimer w ilości 1% oraz 2% i uziarnieniu do 2 mm i powyżej 2 mm. Badanie wytrzymałości na zginanie i ściskanie zostało wykonane na maszynie wytrzymałościowej model ZD 10/90 do prób statycznych.

WYNIKI

Zgodnie z normą PN-EN 13279-2:2014-02 oraz uwzględnieniem poprawek wymiarowych dla belek dla wszystkich próbek obliczono wytrzymałość na zginanie ze wzoru:

$$f_{zgp} = \frac{1.5 \cdot l \cdot F_{zg}}{b \cdot h^2}$$

gdzie: l - rozkład podpór przy zginaniu [mm], b i h – wymiary belki [mm], F_{zg} – siła niezbędna do złamania [N]

Średnie wartości wytrzymałości na zginanie dla prób z domieszką POM w ilości 1%, o średnicy ziaren poniżej 2 mm oraz z domieszką POM w ilości 2% i średnicy ziaren powyżej 2mm wynosiły odpowiednio 4,82 N·mm^{-2} oraz 4,33 N·mm^{-2}. Wartości te są niższe niż wartość średnia dla belek wykonanych w czystego gipsu. Dla próby numer 4 wytrzymałość na zginanie zmniejszyła się o ponad 11 % w stosunku do próby wzorcowej. Najwyższe średnie wartości uzyskano dla

kompozytu zawierającego 2% polioksymetylenu o średnicy do 2 mm. W tym przypadku wartość wytrzymałości wzrosła o około 7% w porównaniu do czystego gipsu.

Wartość wytrzymałości na ściskanie została obliczona zgodnie ze wzorem:

$$f_{sc} = F_{sc}/1600$$

gdzie: F_{sc} – maksymalna siła niszcząca [N].

Stwierdzono, że wszystkie zastosowane modyfikacje spowodowały wzrost wytrzymałości na ściskanie. Największą wytrzymałość f_{sc} wykazuje próba 3 będąca kompozytem gipsowym modyfikowanym polioksymetylenem w ilości 1% o średnicy ziaren poniżej 2 mm. Średnia wartość wytrzymałości f_{sc} dla tego kompozytu wyniosła 13,80 N/mm^2, i była większa o prawie 31% w stosunku do próby z czystego gipsu. Dla prób 1 i 2 będących kompozytami polioksymetylenu w ilości 1% i 2%, o średnicy ziaren do 2 mm wartości średnie były bardzo zbliżone i powodowały zwiększenie wytrzymałości próbek o około 1,45 N/mm^2, czyli 13,7% w porównaniu do próby referencyjnej. Najmniejszą wytrzymałością wykazała się próba 4 zawierająca polimer w ilości 2% o średnicy ziaren powyżej 2 mm. Wytrzymałość na ściskanie w tym przypadku wzrosła o 0,51 N/mm^2, czyli o prawie 5%.

WNIOSKI

Analiza uzyskanych wyników wykazała, że zastosowanie polioksymetylenu do modyfikacji gipsu ma obiecującą perspektywę i wymaga dalszych badań oraz analiz. Dodanie polioksymetylenu do kompozytów gipsowych w ilości 2% o średnicy ziaren poniżej 2 mm spowodowało wzrost wytrzymałości na zginanie o 7% w porównaniu z próbką wzorcową. Wyniki badań wytrzymałości na ściskanie wszystkich zmodyfikowanych gipsów charakteryzowały się wzrostem wytrzymałości. Najlepsze wyniki uzyskano dla próbek kompozytu modyfikowanego polimerem w ilości 1% o średnicy ziaren poniżej 2 mm. Wartości f_{sc} wzrosła wówczas o 30% w porównaniu z próbą wzorcową. Najniższą wartość wytrzymałości uzyskano dla próbki zawierającej POM w ilości 2% i średnicy ziaren powyżej 2 mm. W tym przypadku wartość wytrzymałości na ściskanie była większa jedynie o 5% w porównaniu z próbką referencyjną wykonaną z czystego gipsu.

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