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The production of gypsum materials with recycled gypsum-bearing components using semi-dry pressing technology

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ABSTRACT

Issues of industrial waste recycling are very relevant for the entire global scientific community. The search for technological solutions that would allow the production of high-quality materials using industrial waste will not only reduce the environmental load, but also expand the raw material base for the production of gypsum materials. The study examined the possibility of improving the surface quality of molded gypsum samples by replacing the metal mold with a plastic one and introducing a surfactant into the raw mixture. As a result of the research, it was found that the use of a surfactant and a plastic mold allows to avoid defects on the surface of the samples. At the same time, the use of a plastic mold, which has low adhesion to the citrogypsum-based binder, helps to reduce the amount of adhesion friction and optimize the raw mixture compaction process. This makes it possible to obtain samples with improved physical and mechanical characteristics (compressive strength increases by 30–85.5%, average density - by 1.7–2.7% and water absorption decreased by 1.7–16%) or lower consumption of binder up to 20% compared to samples prepared in metal mold.

1. Introduction

Finding ways to recycle industrial waste to obtain final high-quality commercial products is a priority for most countries in the world (Cárcamo & Peñabaena-Niebles, 2022; Gao et al., 2023; Bhairappanavar et al., 2021; de Abreu & Ceglia, 2018). The “recycling” principle greatly reduces the carbon footprint of industry-driven construction materials; releases useful areas occupied by waste, as well as restores the ecosystems of the areas where this waste is located (Yang et al., 2023; Carrasco-Amador et al., 2022; Moreno et al., 2024; Poranek et al., 2023). Most of the solutions proposed nowadays are aimed at finding the possibility of replacing natural raw materials with waste, taking into account their disadvantages. One of these wastes that requires the search and implementation of recycling routes is a by-product of the biochemical synthesis of citric acid – citrogypsum (CG). CG, in general, consists of $CaSO_4 \cdot 2H_2O$ and is often used as an analogue of natural gypsum when the production of gypsum binders and building materials (Sverguzova et al., 2010; Kozhukhova et al., 2023). However, the instability of the chemical composition, the presence of various impurities, high dispersion and humidity of citrogypsum make the use of standard technologies and equipment ineffective, and the resulting gypsum products are of low quality (Alfimova et al., 2020). Therefore, this approach, in general, deliberately limits the scale of the achieved result.

A more promising approach seems to be the perception of industrial waste as a separate type of raw material, which has no analogues in nature and was formed as a result of large energy consumption, which influenced its phase and material composition, and structure, as well. This concept reveals the higher energy potential of technogenic raw materials, which, with the right approach, should be used as efficiently as possible in the technological process. One of the features of citrogypsum is its high dispersion, which for a natural gypsum analogue is achieved due to significant energy consumption.

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On the one hand, high dispersion is the reason for the low quality of gypsum binder produced using the classical direct firing technology. But, on the other hand, high dispersion of precursors is a key factor for producing artificial gypsum stone using the powder-reaction mechanism, which has proven its effectiveness (Petropavlovskii et al., 2021; Petropavlovskaya et al., 2015).. When highly dispersed gypsum particles come together during the compaction process and low water consumption, a large number of contacts are formed per unit volume. As a result, systems are formed that allow achieving high strengths. Another important feature of the powder-reaction mechanism is the high degree of saturation of the gypsum paste with inert products, which is aimed to reducing the consumption of the binder component, reducing porosity and autogenous deformations. One way to implement powder-reaction mechanism is to use semi-dry pressing technology, which involves the use of a mixture of gypsum binder and original citrogypsum, vs. casting technology, where only gypsum binder is used (Alfimova & Pirieva, 2023; Alfimova et al., 2023).

It was previously found that pressing a semi-dry mixture consisting of citrogypsum binder (CGB) and fractionated citrogypsum (FCG) makes it possible to obtain products with good physical characteristics such as green strength: 0.99–1.99 MPa; compressive strength at the age of 2 days: 6.2–29.9 MPa (Alfimova & Pirieva, 2023); water absorption: 11–18% (Alfimova et al., 2023a). However, the surface of the resulting samples in some cases was distinguished by a significant number of defects in the form of cracks and delaminations. It was also found that the determining negative factor affecting the quality of the surface, as well as the uniformity of the compaction, is the increased adhesion of the binder component to the metal surfaces of the mold, which causes a high level of wall friction (Alfimova & Pirieva, 2023). Since the standard (Russian Standard GOST 6428-2018, EN 12859-2011) for gypsum wall products implies a limitation on the number of defects on the surface (the presence of no more than three separate shells with a maximum diameter of 10 mm per one front surface of the slab), studies were carried out, aimed to improve the quality of the front surface (Alfimova et al., 2023b). In particular, the possibility of introducing surfactants into the mixture was considered, as well as replacing the metal material of molding by plastic one, made on a 3-D printer. It was also found in that introducing a surfactant separately into the raw mixture and replacing the metal mold with a plastic one makes it possible to increase the average density and compressive strength of the samples. The authors found that only the combination of a surfactant and a plastic mold makes it possible to completely get rid of defects (Alfimova et al., 2023b).

This study is a continuation of the previously conducted research (Alfimova et al., 2023a) and aimed at studying the influence of recipe (ratio of molding sand components) and technological parameters (molding pressure) for the manufacture of pressed products from citrogypsum on their physical characteristics, such as «green» average density and «green» strength; average density and compressive strength of gypsum paste at the age of 2 days; water absorption.

2. Materials and Methods

2.1. Materials

The object of study was citrogypsum (Russia, Belgorod) is a by-product from the biochemical synthesis of citric acid. In the study, citrogypsum was used as a gypsum-containing raw material for the production of citrogypsum binder (CGB), and also as a filler of a certain fraction, i.e. fractionated citrogypsum (FCG). Full information on the characteristics for CGB and FCG are given in (Alfimova et al., 2023a). The additive “Penostrom” (LLC «Fire extinguishing equipment plant», Russia) was used as a surfactant.

2.2. Methods

For the experiment, a non-compositional plan for three factors described by the Box-Benkin Planning Matrix (**Table 1**) was used (**Table 2**).

Table 1. Experiment planning conditions

Parameters		**Variation Levels of Studied Parameters**			**Variability**
Original Form	**Coded Form**	**-1**	**0**	**+1**	**Interval**
FCG content, wt.%	X_1	10	30	50	20
Molding pressure, MPa	X_2	2	3.5	5	1.5
Surfactant content, wt. %	X_3	0	0.095	0.19	0.095

The preparation of experimental samples was realized at a constant mixing water content (W/S ratio) – 22.5%, selected in (Alfimova et al., 2023a,b). The detailed sample preparation process is given in previously published works (Alfimova et al., 2023a,b). The pressing procedure was carried out at a molding pressure of 2–5 MPa, which is due to preliminary data on a significant increase in compressive strength (by 60.6%) of samples prepared using a plastic mold and the introduction of a surfactant into the raw mixture (Alfimova et al., 2023b). A reduction in molding pressure will have a positive impact on the technical and economic indicators of the entire production process, and, in particular, will reduce energy consumption, metal consumption and equipment wear. The amount of surfactant required to facilitate the molding process was determined earlier in (Alfimova et al., 2023b), where exceeding the dosage by more than 0.19% contributed to the formation of air cavities on the surface of the samples, which negatively affected both the appearance of the samples and their physical and mechanical characteristics.

Table 2. Experiment design matrix

No	Input parameters					
	Coded Form			Original Form		
	X_1	X_2	X_3	FCG Content, wt.%	Molding Pressure, MPa	Surfactant content, wt. %
1.	+1	+1	+1	50	5	0.19
2.	+1	+1	-1	50	5	0
3.	+1	-1	+1	50	2	0.19
4.	+1	-1	-1	50	2	0
5.	-1	+1	+1	10	5	0.19
6.	-1	+1	-1	10	5	0
7.	-1	-1	+1	10	2	0.19
8.	-1	-1	-1	10	2	0
9.	+1	0	0	50	3.5	0.095
10.	-1	0	0	10	3.5	0.095
11.	0	+1	0	30	5	0.095
12.	0	-1	0	30	2	0.095
13.	0	0	+1	30	3.5	0.19
14.	0	0	-1	30	3.5	0
15.	0	0	0	30	3.5	0.095
Reference mixes						
16.	–	-1	-1	–	2	0
17.	–	0	0	–	3.5	0.095
18.	–	+1	+1	–	5	0.19

The controlled output parameters were the average density and compressive strength for the «green» samples, i.e. immediately after the molding procedure; average density and compressive strength for consolidated samples, i.e. dried under ambient conditions for 2 days until constant weight is achieved; and water absorption. Additionally, the appearance of the samples was assessed; and the presence of defects on the surface was recorded. The reference samples without FCG was molded as well (**Table 2**, Mixes16–18). The strength test was carried out using a laboratory press with a force of 10 tons.

Regression equations were obtained by statistical computer processing of experimental data using Excel. To plot the dependences of output parameters on varying factors, SigmaPlot software was used, designed for the analysis and visualization of scientific and statistical data.

The microstructure and morphology of new formations in CGB samples at the age of 2 days were studied using a scanning electron microscope Mira 3 FesSem (Tescan, Czech Republic) operating in a high vacuum mode (InBeam) with a high brightness Schottky cathode. Before measurements, the samples were preliminarily covered with chromium (Cr). Compressive strength characteristics were determined on cylinder specimens in accordance with standard test methods. Pressing equipment was used: a 10-ton laboratory press with a measurement range of 0–100 kN (graduation 1 kN). The average loading rate during the test was (1.0 ± 0.5) MPa/s. The compressive strength of an individual specimen was calculated as the quotation of the breaking load divided by the specimen area. The compressive strength for the mixes was calculated as the arithmetic mean of the test results of the samples.

3. Results

3.1. Appearance of Experimental Mixes

The appearance of the experimental mixes at two days after demolding is presented in **Fig. 1**; and for reference Mixes – in **Fig. 2**.

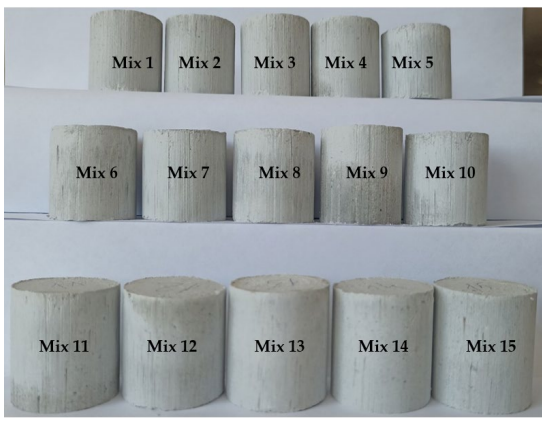
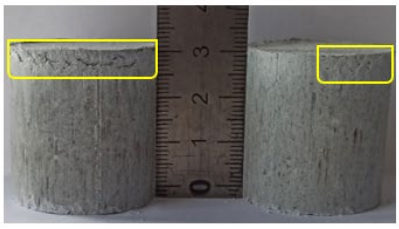
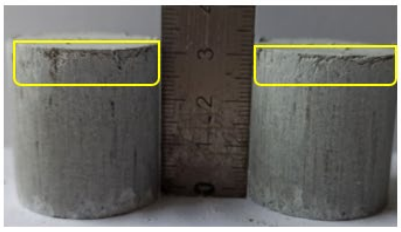


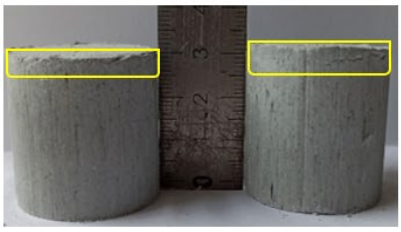
Fig. 1. Appearance of experimental Mixes



Mix 16
There is a 3–7 mm high detachment in the upper part of the sample.



Mix 17
There is a 1–6 mm high detachment in the upper part of the sample



Mix 18
A smooth surface is observed, with minor delamination in the form of a surface crack at a distance of 3–5 mm from the top of the sample

Fig. 2. Appearance of reference Mix

2.2. Properties of experimental mixes

The physical and mechanical characteristics of the experimental Mixes are given in Table 3.

Table 3. Physical and mechanical characteristics of experimental Mixes

Mix ID	Average values of controlled output parameters				
	Green fverage density, kg/m^3	Green compressive strength, MPa	Average density of consolidated mixes, kg/m3	Compressive strength of consolidated mixes, MPa	Water absorption, wt. %
Mix 1	1746±1.31	1.22±2.01	1624±1.37	11.07±1.98	18.7±1.23
Mix 2	1737±1.00	1.26±1.87	1564±1.12	11.01±1.77	16.6±1.22
Mix 3	1766±0.98	1.40±2.22	1579±1.07	8.98±1.45	16.6±1.17
Mix 4	1708±0.61	0.95±2.41	1570±0.99	10.65±1.85	16.6±1.23
Mix 5	1858±1.22	1.36±2.02	1783±1.41	28.81±0.45	11.7±1.31
Mix 6	1917±1.01	1.26±2.00	1760±1.22	28.36±0.67	11.6±1.15
Mix 7	1899±0.63	1.33±1.88	1770±0.97	27.91±1.00	12.9±1.12
Mix 8	1821±0.50	1.19±1.77	1753±1.15	25.31±1.23	12.3±1.25
Mix 9	1735±1.02	0.95±2.05	1538±1.44	9.50±1.77	16.5±1.34
Mix 10	1814±1.10	1.19±1.82	1760±1.57	28.72±0.98	10.3±1.56
Mix 11	1857±1.23	1.19±2.41	1703±1.77	21.43±1.34	12.7±1.65
Mix 12	1803±1.07	1.19±1.99	1643±1.37	19.27±2.01	16.1±1.45
Mix 13	1798±0.77	1.19±2.41	1685±1.67	21.79±2.00	14.1±1.77
Mix 14	1793±0.68	0.95±2.07	1666±1.25	21.17±1.99	12.4±1.34
Mix 15	1799±1.57	1.19±2.22	1683±1.85	24.09±1.83	15.6±1.62
Reference mixes					
Mix 16	1790±1.98	0.75±2.34	1710±2.01	19.58±1.77	12.1±1.98
Mix 17	1837±1.87	0.99±2.17	1720±2.00	21.42±2.03	11.8±1.44
Mix 18	1891±1.30	1.12±2.21	1738±1.89	28.78±2.11	11.5±1.41

4. Discussion

4.1. Influence of formulation and technological parameters on the appearance of samples

A visual assessment of the experimental samples (see **Fig. 1**) showed that replacing the metal mold with plastic one and introducing a surfactant into the mixture had a positive effect on the appearance and made it possible to completely get rid of defects on the surface of almost the entire series of samples. Minor defects in the form of small-sized peelings on the surface were observed on samples of Mix 6 and Mix 8. These mixes were prepared from a raw material mixture with a minimum content of fractionated citrogypsum – 10% and without surfactants (see **Table 1**).

Also, it should be noted that in the case of the reference mixes (**Fig. 2**), defects in the form of a slight «wedge» (up to 1 mm) and delamination are observed on the surface of the samples. The samples of Mix 16, made from surfactant-free mixture, have the maximum number of defects. It should be noted that with an increase in the proportion of surfactants in the mixture, the number of defects decreases. For example, the sample of Mix 18, prepared with the maximum surfactant dosage, is characterized by a smooth surface. However, there is a minor defect in the form of a surface crack at a distance of 3–5 mm from the top of the sample (**Fig. 2**). The results obtained from a visual assessment of the reference samples indicate that both the amount of surfactant and the amount of FCG, as well, make a significant impact on improving the surface. The presence of FCG in the mixture helps to reduce the adhesion of the molding mass to the surface of the mold.

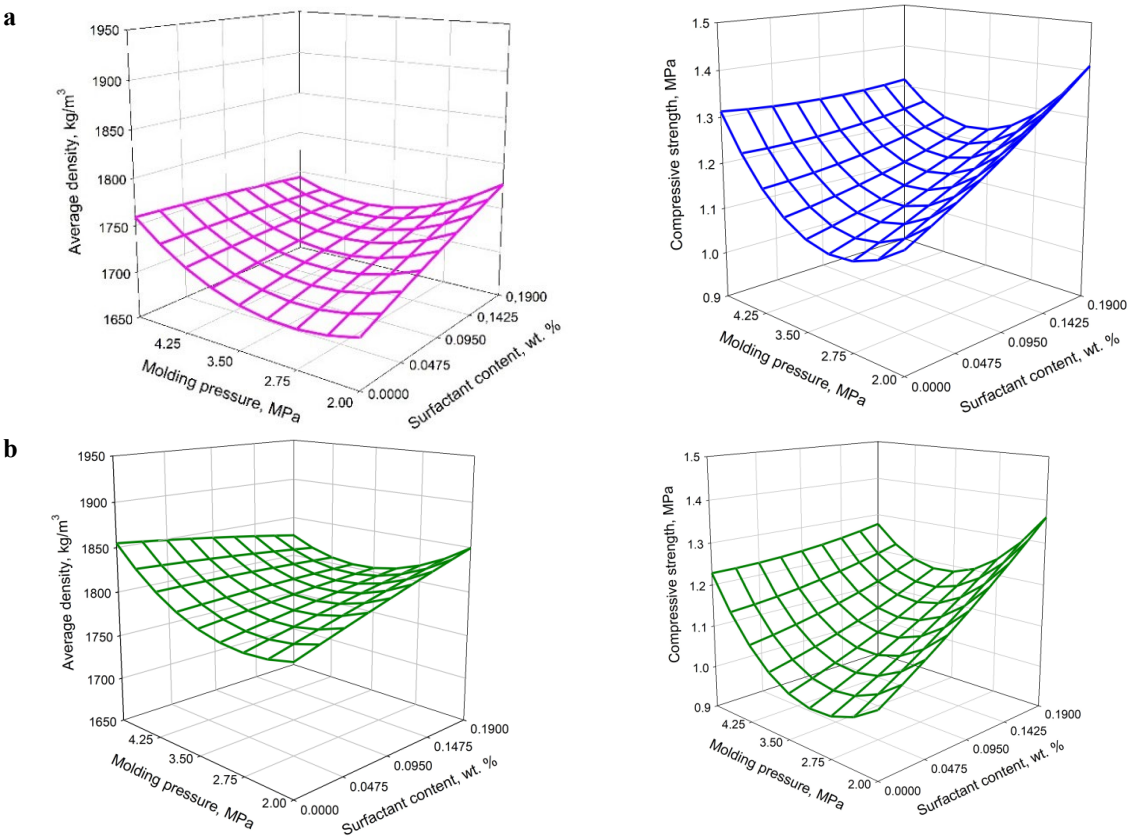
3.2. Influence of formulation and molding pressure on the physical and mechanical characteristics of samples

By analogy with the previous study (Alfimova & Pirieva, 2023), the influence of variable factors on the «green» average density and «green» strength, i.e. average density and strength of the samples immediately after molding was analyzed. After

statistical computer processing of the experimental data, nomograms of the dependence of these parameters (Figure 3) on varying factors: molding prwssure and surfactant content (**Table 1**) were plotted. Based on the data in Figure 3, the following dependencies were identified. At a molding pressure of 2 MPa, regardless of the FCG content in the mixture, with an increase in the proportion of surfactants in the mixture, an increase in the average density of the samples is observed, which may indicate a positive effect of the surfactant on compaction process. However, at a molding pressure of 5 MPa, with an increase in the proportion of surfactants in the mixture, a decrease in the average density is observed. The «green» compressive strength of the samples increases with the increase in the proportion of surfactants in the mixture, the more noticeably the higher the content of FCG in the mixture and the lower the molding pressure. The maximum strength values of raw materials will be achieved at a molding pressure of 2 MPa, a 10%- content of FCG and a surfactant dosage of 0.19% (**Fig. 3, a**). At a molding pressure of 5 MPa, for samples with 30 and 50% of FCG content, with an increase in the proportion of surfactants in the mixture, an increase in compressive strength is observed (**Fig. 3, b, c**). At the same time, for samples with a 10% of FCG content in the mixture, the «green» compressive strength remains unchanged (**Fig. 3, a**).

It has been established that with an increase in molding pressure from 2.25 to 3 MPa, a decrease in «green» average density and «green» compressive strength is observed; and as the pressure increases from 3 to 5 MPa, the average density of the «green» samples begins to increase. This is most likely due to the fact that at low molding pressures the structure is quite gas-permeable and, after removing the external influence, the air trapped in the volume of the samples during the molding process freely escapes without violating the integrity. As the molding pressure increases, the gas permeability of the mixture decreases due to its compaction, and the air pressure inside the bubbles increases. When the external molding pressure is released, the air inside the bubbles begins to expand, destroying the structure and reducing the average density of the «green» sample. This is observed until the critical value of the molding pressure is overcome: 3–4.3 MPa, above which, most likely, the area and strength of contacts between particles becomes enough to resist the air pressure in the bubbles. This, in turn, leads to a decrease in the degree of decompaction of the mixture and, as a consequence, an increase in the average density and compressive strength of the «green» sample. At the same time, the higher the content of a surfactant in the mixture, which forms gas-tight shells on the surface of the bubbles, the more pronounced is the process of reducing the average density of the «green» sample at a molding pressure below the critical level (**Fig. 3, b**).

Most likely, decompaction is also the reason that samples prepared at a molding pressure of 5 MPa have lower average density and compressive strength of «green» samples than samples prepared at a molding pressure of 2 MPa. Analysis of the effect of the forming surface material on the average density of «green» samples showed that with equal parameters of W/S ratio = 0.225, and without surfactant content, the average density of «green» samples prepared using a plastic mold is 1–6% higher, than for samples prepared in a metal mold (Alfimova, 2023a).



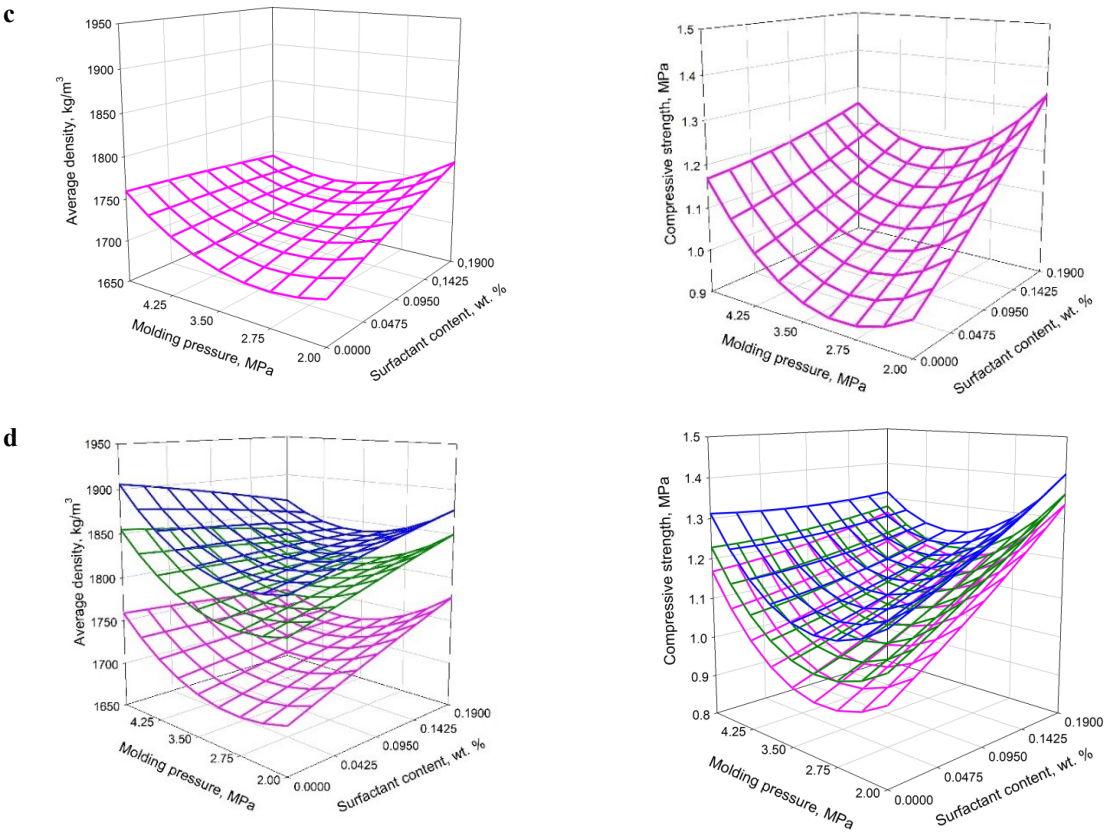


Fig. 3. The effect of molding pressure and surfactant content on the average density and compressive strength of sample immediately after demolding at the different FCG content* in the mixture:
***a** – 10%; **b** – 30%; **c** – 50%; **d** – summary nomogram

A comparison of the «green» strength values of samples prepared in metal and plastic molds (Alfimova et al. 2023a); showed that in the first case, an increase in the proportion of FCG in the mixture increases the compressive strength. Specimens with a 50% of FCG content in the mixture are characterized by maximum strength. At the same time, when using a plastic mold, an inverse relationship was observed: maximum strength was achieved with a minimum content of FCG in the mixture – 10%. This is most likely explained by the fact that in the case of a metal mold, the major factor contributing to a decrease in the adhesion of the molding mixture to the molding surface was an increase in fractionated citrogypsum in the mixture, which had a positive effect on the molding process and, as a result, on the «green» strength of samples. The use of a plastic mold in combination with the introduction of a surfactant, which ensures a reduction in interparticle and adhesion friction, significantly reduced the negative impact of CGB on the molding processes, ensuring the formation of the most compact and strong structure of «green» samples at lower values of CGB in the mixture.

In general, the value of the «green» strength of a sample prepared in a plastic mold is in the range of 0.8–1.4 MPa, which is quite enough to ensure the integrity of the products after demolding and further transportation. Analysis of the effect of recipe and technological parameters on the average density and compressive strength of the reference samples (**Table 3**) showed a direct dependence of the average density and strength on the molding pressure and surfactant dosage in the mixture. The maximum values of average density and compressive strength were observed for samples manufactured at a molding pressure of 5 MPa and containing 0.19% of surfactant (**Table 3**, Mix18). The «green» strength for most of the samples are comparable or exceed the values for the reference mixes (**Table 3**).

3.3 The influence of recipe and technological parameters on the physical and mechanical characteristics of consolidated sample

After statistical computer processing of the experimental data, nomograms of dependences of the average density and compressive strength of the consolidated samples on molding pressure and surfactant content were plotted (**Fig. 4**).

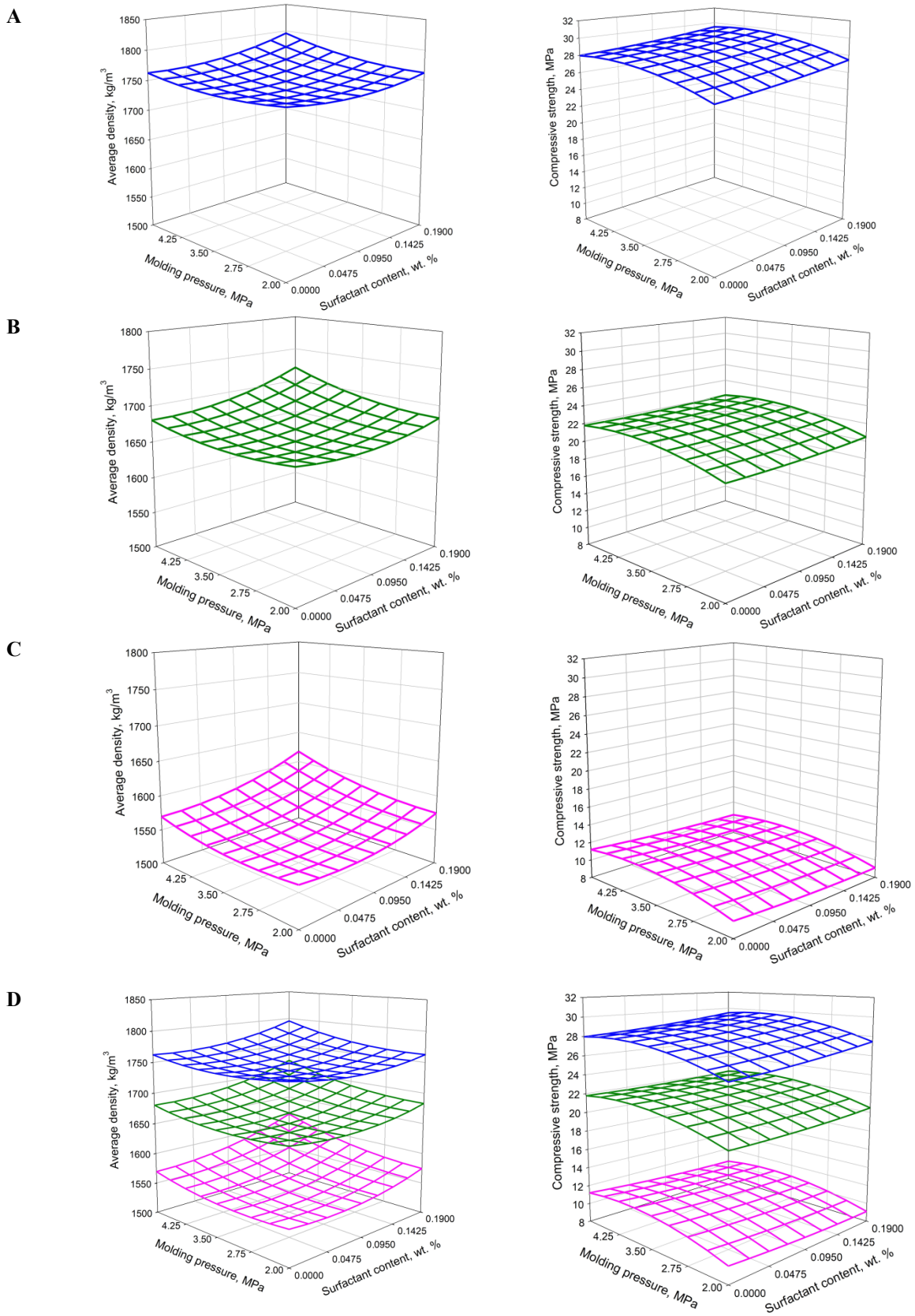


Fig. 4. The effect of molding pressure and surfactant content on the average density and compressive strength for samples at two days after demolding at the different FCG content* in the raw mixture:
***a** – 10%; **b** – 30%; **c** – 50%; **d** – summary nomogram

An analysis of the influence of recipe and technological parameters on the average density of the consolidated samples showed (**Fig. 4**) that, regardless of the content of FCG in the mixture, the nomograms are of a similar nature. In particular, in the nomograms in all cases, there is an area in which, with an increase in the molding pressure and the proportion of surfactants in the mixture, the average density remains almost unchanged. The average density begins to grow at a molding pressure of

≈ 4 MPa and a surfactant content of ≈ 0.14% by weight. Maximum values of average density are achieved at maximum values of molding pressure – 5 MPa; anfsurfactant content – 0.19% by wt.) (**Fig. 4**).

Analysis of the results obtained (**Fig. 4**) showed that at 10% and 30% of FCG content in the mixture, the dependences of compressive strength on molding pressure and surfactant content also have a similar character: regardless of the amount of surfactant in the mixture, with increasing molding pressure the compressive strength of the samples increases (**Fig. 4, a, b**). It should also be noted that the maximum increase in strength is observed at molding pressures from 2 to 3.5 MPa: with a 10% of FCG content in the mixture, the increase in strength is 7.7% (**Fig. 4, a**); at 30% of FCG content in strength increasing is 10% (**Fig. 4, b**). With a further increase in the molding pressure from 3.5 to 5 MPa, the increase in the compressive strength in both cases is about 1.8%. The identified dependencies confirm the previously made conclusion about the presence of a molding pressure limit, exceeding which provides enough contacts between particles to resist decompaction processes due to the expansion of trapped air in the molding mixture, which ensures a significant increase in the physical and mechanical characteristics of the final products. The maximum value of compressive strength (29.7 MPa) in this case is achieved with a surfactant content in the mixture of 0.019% and a molding pressure of 4.25 MPa (**Fig. 4, a**). With a 30% of FCG content in the mixture, regardless of the molding pressure, an increase in the proportion of surfactants has almost no effect on the strength. The maximum value of the compressive strength (22.4 MPa) in this case is also achieved with surfactant content in the mixture of 0.019% and a molding pressure of ≈ 4.25 MPa (**Fig. 4, b**).

At 50% of FCG content in the mixture, an increase in the surfactant, regardless of the molding pressure, leads to a decrease in the average density and asrength (**Fig. 4, c**). The maximum value of compressive strength (11.5 MPa), in this case, is demonstrated by samples prepared without surfactants and at a molding pressure of about 4 MPa. The observations obtained allow concluding that when the FCG content increases above 30%, the surfactant begins to have a negative effect on the compressive strength of the samples. This is most likely due to an increase in the negative effect of surfactants on the hydration processes of the citrogypsum binder with a decrease in its concentration in the total volume of the mixture. An increase in the proportion of FCG in the mixture from 10 to 30%, on average, leads to a 25% decrease in the compressive strength (Figure 4, a, b); and with an increase in the proportion of FCG from 30 to 50%, the strength loss is significantly higher: amounts up to 48%, on average, (**Fig. 4, b, c**).

Comparison of the results obtained from the reference mixes (Mix 16 – Mix 18, **Table 3**) showed that the strengths of samples with 10 and 30% of FCG content are either comparable (Mix 11–Mix 14) or exceed (Mix 5–Mix 8, Mix 10, Mix 15) compressive strength for reference samples prepared without FCG (**Table 2**). A comparison of nomograms of average density and compressive strength demonstrated that at 10 and 30% of FCG content (**Fig. 4, a, b**) a proportional dependence of the parameters is observed in the mixture, i.e. with increasing average density, the compressive strength increases, and maximum values are achieved at maximum values of molding pressure – 5 MPa and the surfactant content is 0.19%. At the same time, at a 50% of FCG content in the mixture (**Fig. 4, c**), with an increase in the surfactant content, despite the increase in average density, a decrease in the compressive strength is observed, which confirms the previous assumption about the negative effect of surfactants on the process of structure formation of samples with a low content of CGB in the mixture. To determine the influence of the mold material on the physical and mechanical characteristics of the samples, a comparison was made of the average density and compressive strength of samples prepared without surfactants at the same values of molding pressure – 5 MPa and W/S ratio – 0.225 (Alfimova et al., 2023a). A comparative analysis showed that, regardless of the content of citrogypsum in the mixture, samples prepared in plastic mold showed higher average density: with a 50% of FCG content in the mixture, the average density increased by 2.6–2.7%, with a 30% FCG content in the mixture - by 2–2.1%; at 10% of FCG content in the mixture – by 1.7–1.8%.

Based on (Alfimova et al., 2023a), it is interesting that when using a metal mold, the average density of only Mix 5, containing 10% of FGC, is comparable to the average density of the reference Mix 18. At the same time, when using a plastic mold and surfactant, the average density of all samples with 10% of FGC content (**Table 3**, Mix 5–Mix 8, Mix 10) exceeds similar values for the reference Mix 18 (**Table 3**). This indicates that the introduction of fractionated citrogypsum into the mixture, as well as the use of a plastic mold, has a positive effect on the compaction process, ensuring the formation of a more compact structure of the samples. Replacing the mold material with other equal parameters (PCG content in the mixture, W/S ratio and molding pressure) contributes to a significant increase in compressive strength. The higher the FCG content in the raw mixture, the higher the increase in strength. Thus, for samples containing 10% of FCG, prepared in a plastic mold, the increase in strength relative to samples prepared in a metal mold was 30.4–35.1%; for samples with 30% of FCG content – 37.8–44.1%; and for samples with 50% of FCG content – 67.9–85.5%. The obtained dependencies can be explained by the fact that the more fractionated citrogypsum in the raw material mixture, the lower its bulk density and, therefore, the greater the potential for increasing mechanical characteristics due to a more homogeneous compaction of the molding mixture. The compressive strength of samples prepared in a plastic mold with 30% of FCG content is 0.3–1.23 MPa higher than that of samples with 10% of FCG content prepared in a metal mold.

Thus, the results obtained demonstrate that replacing the mold material in combination with the introduction of a surfactant significantly increases the efficiency of the molding process for products based on citrogypsum binder using the semi-dry pressing method, and provides significant potential for saving material and energy resources. In particular, when using a metal

mold, the strength of samples with a 10% of FCG content (26 MPa) was achieved at a molding pressure of 7 MPa (Alfimova et al., 2023a), while when using a plastic mold, such strength can be achieved at a molding pressure of 2 MPa (**Fig. 4, a**).

3.4. The influence of recipe and technological parameters on water absorbtion

After statistical computer processing of experimental data, the dependences of the average density and compressive strength of consolidated samples on molding pressure and surfactant content were plotted (**Fig. 5**).

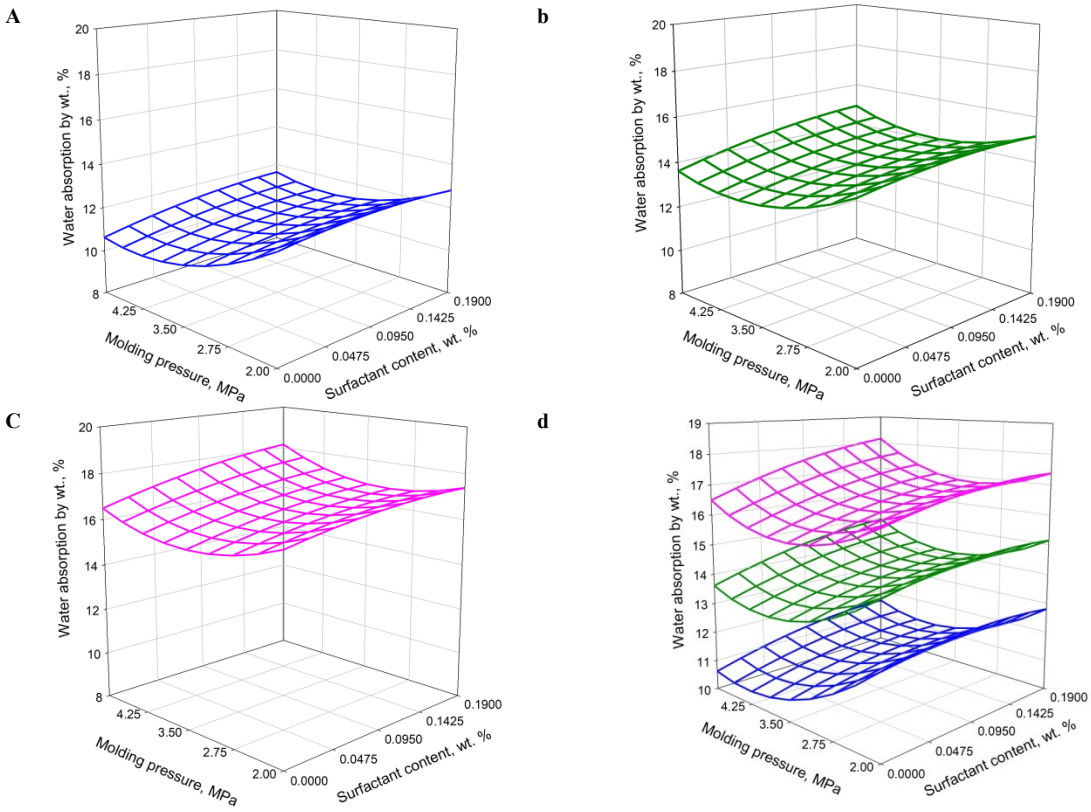


Fig. 5. The effect of molding pressure and surfactant content on the water absorption for samples at two days after demolding at the different FCG content* in the raw mixture:
***a** – 10%; **b** – 30%; **c** – 50%; **d** – summary nomogram

Analysis of the obtained nomograms demonstrated some dependencies

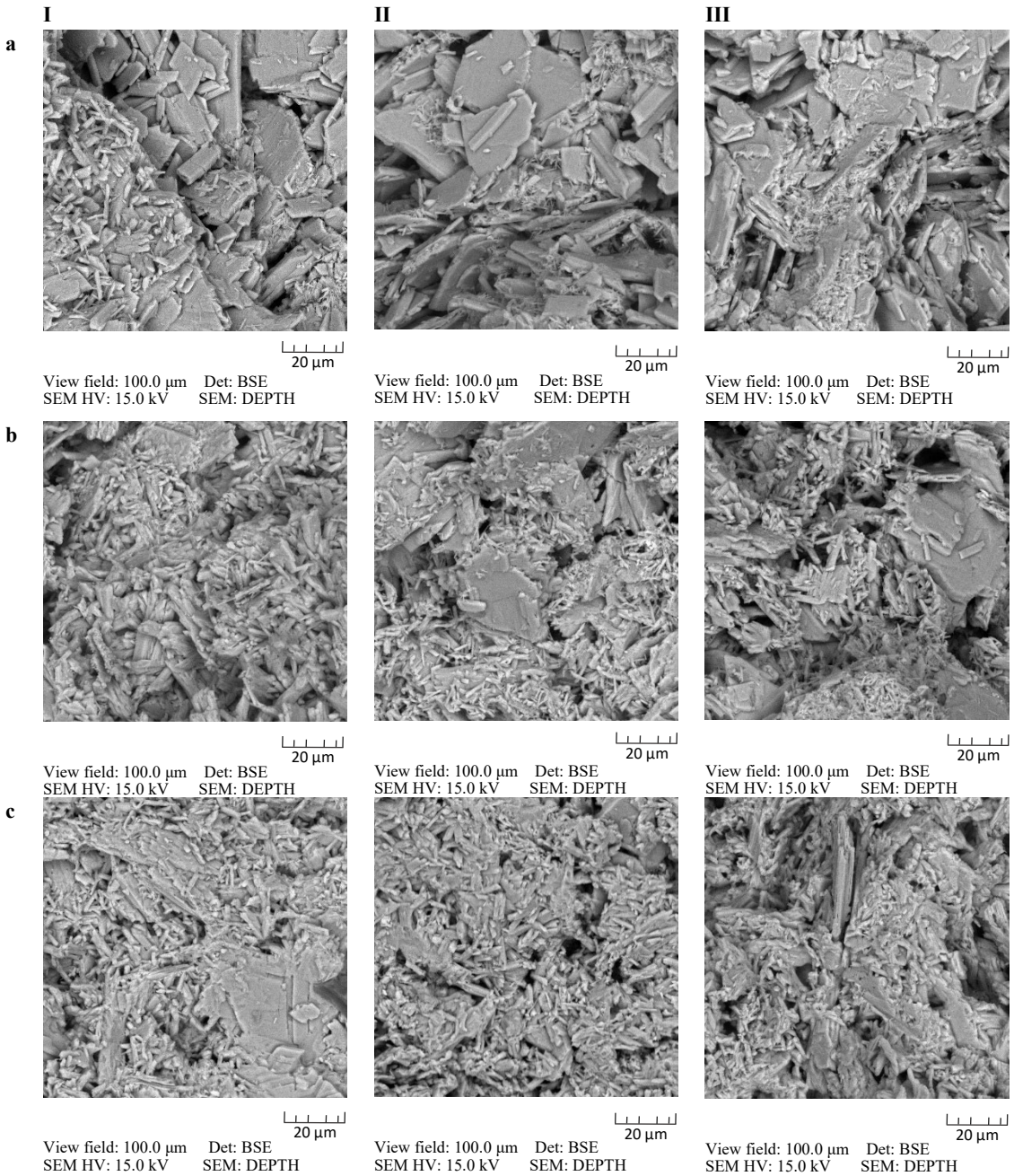
Regardless of the molding pressure and the FCG content in the mixture, an increase in the surfactant content in the mixture promotes an increase in water absorption, which is due to the natural increase in the proportion of capillary pores. With a 10% of FCG content, regardless of the surfactant content in the mixture, an increase in molding pressure leads to a decrease in water absorption. Minimum water absorption values are achieved at a molding pressure of ≈ 4 MPa; a further increase in molding pressure to 5 MPa does not contribute to a decrease in water absorption – a flattening of the nomogram is observed (**Fig. 5, a**).

With an increase in the FCG content in the mixture to 50%, minimal water absorption is achieved at a molding pressure of ≈ 3.5–4 MPa; a further increase in the FCG content leads to an increase in water absorption by 0.8–1%. The additional increase in water absorption takes place with the increasing proportion of surfsctant in the mixture (**Fig. 5, c**). The revealed phenomenon can be explained by the fact that water absorption largely depends on the amount and type of pores. In particular, it increases with the number of small pores and capillaries. As noted earlier, in the specified range of molding pressures of 3.5–4 MPa, there is a slight decrease in the average density of «green» samples (**Fig. 3**), which is associated with the decompaction of the molding mixture under the excess pressure of trapped air. At the same time, a fairly large number of large pores are formed in the mixture, disrupting the capillary system, which in turn leads to a decrease in water absorption rates. With an increase in the content of FCG in the mixture, this effect is manifested to a greater extent. The water absorption of the reference samples without FCG (**Table 3**, Mixes 16–18) are 11.5; 11.8; 12.1%, respectively, which is more consistent with water absorption for samples with a 10% of FCG content (10.6–12.8%) (**Fig. 5, a**). A further increase in the FCG content in the mixture from 10 to 50% leads to an increase in water absorption rates, which is associated with a decrease in the average density and an increase in the porosity of the samples (**Fig. 5, d**). Samples with a 10% of FCG content prepared in a metal mold, despite higher molding pressures (3–7 MPa), are distinguished by higher water absorption rates: 11–17.5% (Alfimova

& Pirieva, 2023) in relation to samples made in plastic mold: 10.5–13% (**Fig. 5, a**). The water absorption values for samples with a 30% of FCG content are almost comparable to each other and range from 13.5–17.5% for a metal mold (Alfimova & Pirieva, 2023) and 13.2–15% for a plastic mold (**Fig. 5, b**). At the same time, at a 50% FCG content, samples prepared in a plastic mold are characterized by slightly higher water absorption (15.7–18%) (**Fig. 5, c**) in comparison with samples prepared in a metal mold (14–17, 5%) (Alfimova & Pirieva, 2023). Thus, the use of a plastic mold in combination with a surfactant, even when using lower molding pressures, contributes to a more uniform compaction of the molding mixture, which ensures an increase in the average density and helps reduce the water absorption rates of the samples.

3.5. Effect of formulation and molding pressure on the microstructure of the samples at 2 days after demolding

Analysis of the microstructure of the samples (**Fig. 6**), containing FCG, prepared in a plastic mold, showed that, other things being equal, the number of new formations, their density and size increases with a decrease in the FCG content in the mixture. Samples prepared from a mixture with 10% of FCG content (**Fig. 6, .c**) are characterized by the maximum density and the largest number of new formations; and samples with 50% of FCG content are characterized by the minimum density and number of new formations (**Fig. 6, a**).



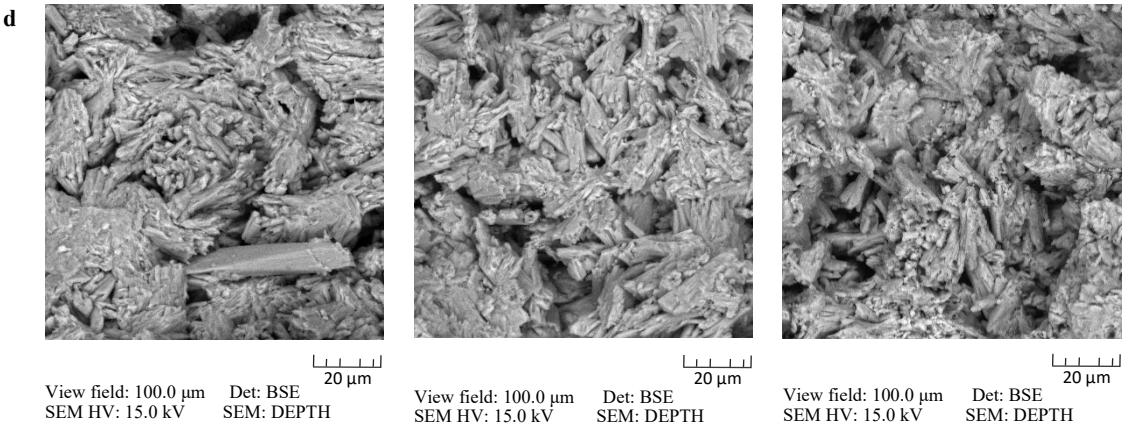


Fig. 6. Microstructure of the samples at 2 days after demolding with different mix design and molding pressure:
a – FCG content is 50%, by wt. (X1 = +1); **b** – FCG content is 30%, by wt. (X1 = 0); **c** – FCG content is 10%, by wt. (X1 = -1);
d – reference – FCG content is 0%
I – molding pressure is 5 MIIa (X2 =+1), surfactant content is 0.19%(X3 =+1); **II** – molding pressure is 3.5 MIIa (X2 = 0) surfactant content is 0.095% (X3 = 0); **III** – molding pressure is 2 MIIa (X2 = -1) surfactant content is 0% (X3 = -1)

Regardless of the FCG content in the mixture, increasing the surfactant content and molding pressure increases the density of the structure. This dependence is most clearly visible when 0.095% ofsurfactant is introduced into the raw mixture and the molding pressure is increased from 2 to 3.5 MPa. A further increase in the surfactant dosage in the mixture to 0.19% and the molding pressure to 5 MPa has a lesser effect on the porosity of the structure (**Fig. 6, I**). The more dosage of binding component there is in the mixture, the less the increase in surfactant content and molding pressure affects the density of the structure.

Analysis of the microstructure of reference samples prepared from a CGB (**Fig. 5, d**) demonstrated that Mix 18 has the most compact structure with fewer pores (**Table 3**), manufactured at a molding pressure of 5 MPa and containing 0.19% of surfactant (**Fig. 5, d** I). At the same time, Mix 16 (**Table 3**), prepared at a molding pressure of 2 MPa and without a surfactant, is characterized by a structure with a maximum pore content (**Fig. 5, d** III). Mix 17 occupies an intermediate position between Mix 18 and Mix 16 (**Fig. 5, d** II). This is due to the fact that the raw mixture without FCG, which is the main factor in reducing the value of wall friction of the molding mass against the walls of the mold, is the presence of a surfactant in the mixture. Therefore, with increasing surfactants and increasing molding pressure, the density of samples prepared from CGB increases. In the case of preparing samples in a metal mold, the reference samples were distinguished by a more compact structure with fewer pores compared to samples prepared from a mixture containing FCG (Alfimova et al., 2023a). When replacing a metal mold with a plastic one, dependence is visible: the most compact structure with a minimum number of pores is observed for samples prepared with a 10% of FCG content (**Fig. 5, c**). This is probably because the replacement of the mold material in combination with the presence of certain amounts of FCG in the mixture, all other things being equal, minimizes the wall friction effect, which contributes to the formation of the most compact structure.

5. Summary

The influence of the raw mixture formulation and molding pressure of samples from CGB and FCG using a surfactant and a plastic mold on the quality of the surface of the samples and their physical and mechanical characteristics such as: average density and strength of «green» samples, average density and compressive strength of aged samples 2 days, and water absorption was studied. It has been established that the application of a semi-dry pressing method for a raw mixture consisting of CGB and FCG using a plastic mold and the introduction of a surfactant into the mixture allows avoiding defects on the surface of products. The use of plastic molds, characterized by low adhesion to the binding mixture, helps reduce wall friction and optimize the compaction process of the raw mixture. This makes it possible to obtain samples with higher physical and mechanical characteristics (compressive strength increases by 30–85.5%, average density by 1.7–2.7% and water absorption decreased by 1.7–16 %) or lower consumption of binder up to 20% compared to samples prepared in metal mold.

When using a metal mold for samples with a 10% of FCG content, prepared at a molding pressure of 7 MPa, strength up to 26 MPa is achieved. When using a plastic mold, a similar strength value is achieved at a molding pressure of 2 MPa. In general, replacing the mold from metallic to plastic one, together with the introduction of a surfactant, significantly increases the efficiency of molding processes for CGB samples using the semi-dry pressing method, and provides significant potential for saving material and energy resources, as well.

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