

## Sustainable Gypsum Composites with a Complex of Porous Modifiers

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The study is devoted to one of the acute problems of construction materials science - the creation of mineral lightweight composites based on low-carbon binders with high-performance properties and efficiency. Gypsum stable composites modified with a complex of porous technogenic fillers were studied using thermogravimetry and mercury porosimetry. Perlite in combination with thermally activated technogenic carbon was used as a filler in the composition of gypsum stable composites. Carbon was separated from ash and slag mixtures of hydraulic removal by the flotation method. The aim of this work was to study the structure and properties of gypsum stable composites, which can provide unique characteristics for a number of materials based on them. The most significant effect from the standpoint of the formation of structural characteristics of stable composites was the effect of nano- and microreinforcement with a porous technogenic filler. Modification with technogenic carbon powder made it possible to improve the properties of the stable composite by creating a porous structure at different scale levels. It was found that in the process of structure formation of stable composites in the presence of a carbon filler with a mesh structure, hydration occurs, and subsequently, the formation and development of crystallization contacts of nano- and microporous gypsum stone with a complete absence of macropores. Gypsum composites with a complex of porous modifiers are characterized by sufficient strength and high insulating properties, as well as low water absorption. This opens up great prospects in the field of their application.

### 1. Introduction

The United Nations (UN) requirements for achieving zero carbon emissions limit the ability of cement industries to increase the production of traditional types of Portland cement. Since they account for about 7-8 % of carbon emissions. Under the UN Paris Agreement, countries must find ways to radically limit carbon emissions and improve carbon recovery in a relatively short time (Amran et al., 2022). Research efforts are aimed at developing alternative materials. They should replace Portland cement, which has been considered the most versatile binder in construction technologies for two centuries. The mass volumes of its production are not yet covered by the production of geopolymers or other alternative binders based on industrial by-products. Despite all the efforts of researchers to study the impact of the cement industry on the environment and ways to reduce it, the community recognizes that it is impossible to achieve "net zero" by 2050 (Ma et al., 2016) without a transition to completely new generations of building materials (Zhu et al., 2019). Portland cement is undoubtedly the leader among binders. It has high-performance properties, high strength, and resistance to physical and chemical corrosion. However, currently, binders and materials based on them are subject to increased requirements for comfort, environmental friendliness, thermal insulation (Gaião et al., 2011), and other insulating characteristics (Petropavlovskii et al., 2021). Gypsum meets all modern requirements in the field of sustainable development and green building (Wakili et al., 2009), does not emit greenhouse gases when obtained, is fire-resistant (Brunello et al., 2020), energy efficient (Petropavlovskaya et al., 2022), and a technological binder (Petropavlovskaya et al., 2021). Reducing energy consumption in buildings using gypsum materials (Zhu et al., 2019) attracts more and more researchers (Karua et al., 2024). The main disadvantages of gypsum materials and structures are their brittleness, creep, relatively low flexural strength, and high density (Jia et al., 2021). To

increase strength properties (Yildizel, 2018) and reduce creep, natural and man-made fibers of organic or mineral nature are used (Sair et al., 2019). Carbon nanostructures can be used as additives, and in addition to the high-strength properties of the gypsum matrix, they can additionally provide it with electrically conductive properties (Yakovlev et al., 2022). At the same time, it is necessary to take into account the aspects of using complex technology and an increase in cost for such materials.

The production of gypsum energy-efficient composites with high-performance characteristics has been studied (Domanskaya et al., 2025) based on a high-strength matrix and fillers in the form of microfoam spheres, cenospheres, microgranules, micro- and nanofibers, fulleroids (Sair et al., 2019). For this purpose, porous natural or technogenic filler additives (Petropavlovskaya et al., 2021) or phase transition substances (Zhu et al., 2019) are used in the gypsum matrix. The use of technogenic carbon (underburnt, isolated from the ash and slag mixture) in the raw material mixture of gypsum stable composites requires additional research.

The aim of the work was to obtain an energy-efficient lightweight gypsum composite based on a complex of porous modifiers with reduced resource consumption.

## 2. Materials and methods

### 2.1 Materials

The work used gypsum binders of  $\alpha$ -modification of the G-16 grade of the Samara Gypsum Plant. Additives of technogenic carbon and perlite were used as porous modifiers. Carbon was obtained as a result of foam flotation and magnetic separation of the ash and slag mixture from the combustion of solid fuel at a thermal power station in the Moscow region. Enrichment of mixtures and carbon extraction were carried out in semi-factory conditions of production of the Caroline company (Moscow region). The flotation time for the extraction of technogenic carbon was 6 min. A concentrate containing 52.0 % C was obtained, with a carbon extraction of 87.6 %. The specific surface area of technogenic carbon was 400 m<sup>2</sup>/kg. Thermal treatment of carbon during its stay in the furnace of the power unit affected its structure (Figure 1). An additive of expanded perlite was additionally introduced. The chemical additive DKG-602 (Dongke, China) was used to reduce water in the mixtures.

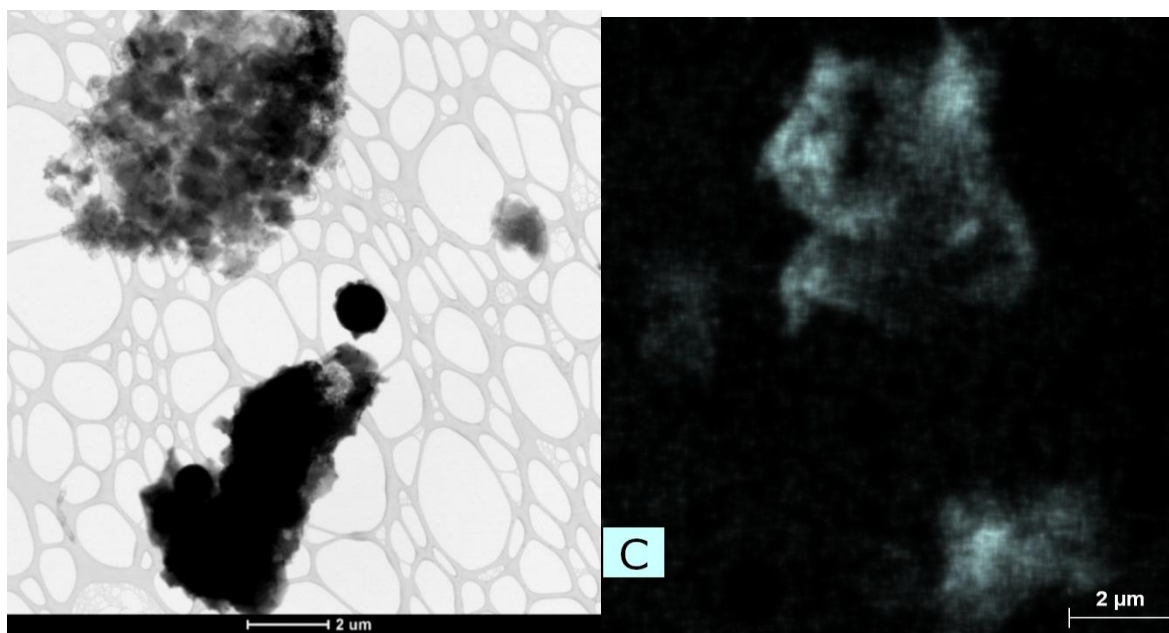


Figure 1: Bright-field and dark-field imaging of man-made carbon particles (with registration of electrons scattered at large angles - HAADF)

The studies of structural changes in gypsum composites with porous fillers were conducted using four gypsum-based compositions (Table 1). They were obtained by hydration hardening of the original gypsum and modified binder.

Table 1: Material composition of mixtures

Composition	Gypsum	Plasticizer	Carbon	Perlite
1	+	-	-	-
2	+	+	-	-
3	+	+	+	-
4	+	+	+	+

## 2.2 Methods

Samples of mineral binders based on gypsum were studied by thermogravimetry methods. The study was conducted using a thermobalance device (Netzsch, Germany) with an argon purge of 40 mL/min. The temperature program included two sections: the first - heating from 40 to 600 °C at a rate of 10 K/min, the second - isothermal for 30 min. The pore structure of the obtained gypsum composite was studied by mercury porosimetry. The work used a mercury porosimeter PASCAL 140 and a dilatometer CD6. The additional measuring instruments used were the SARTOGOSM CE224-C scale with a measurement accuracy of 0.0001 g, a thermometer, and a VIT-2 psychrometric hygrometer. The pore measurement range was 3.8–110  $\mu\text{m}$ . The results were calculated taking into account the cylindrical pore model. The samples were made in the form of cubes with a side length of 20 mm. The total porosity was determined by taking into account the true density of the samples, obtained by testing the samples using a standard method using a Le Chatelier flask.

## 3. Results and Discussion

The results of the studies of the structure of gypsum composites are presented in Figures 2 – 6. The control composition (composition 1) is characterized by a crystalline structure with a predominance of well-formed prisms of calcium sulfate dihydrate. When studying it using thermogravimetric methods, two peaks of mass loss were observed, corresponding to the loss of crystallization water (Figure 2). The first peak with a maximum of 150 °C is associated with the loss of 1.5 mol H<sub>2</sub>O and the formation of calcium sulfate hemihydrate, and the second peak (190 °C) corresponds to the formation of anhydrous calcium sulfate (anhydrite). At a higher temperature, the sample was thermally stable.

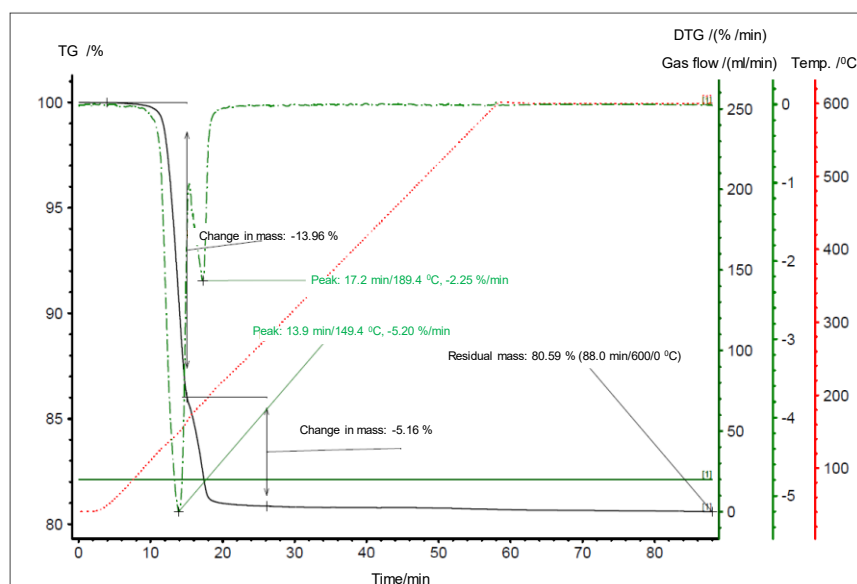


Figure 2: Mass loss curve of sample composition 1

To confirm the role of technogenic carbon as a modifier in the gypsum composite, compositions 2, 3 and 4 were studied. They included porous fillers and a water-reducing chemical additive (Table 1). When using a plasticizer in the mineral binder, a similar mass loss process is observed (Figure 3).

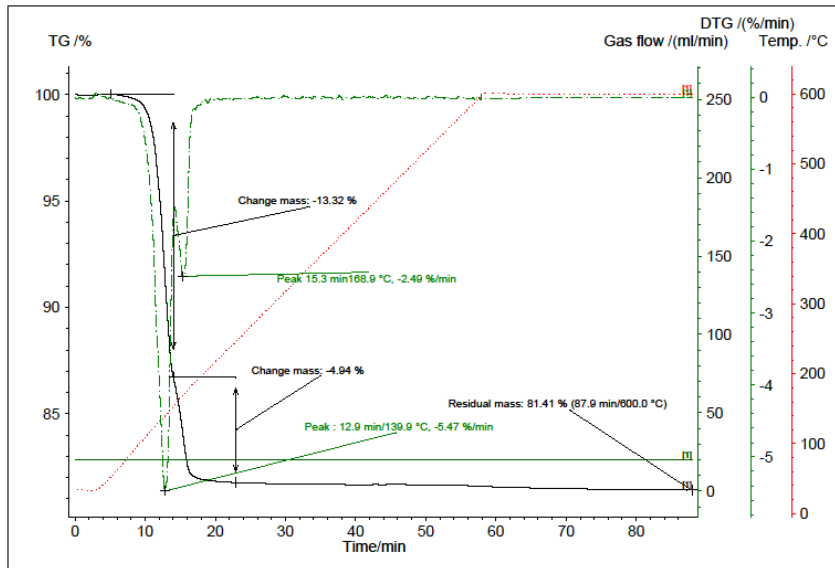


Figure 3: Mass loss curve of sample composition 2

The plasticizer used was a water-reducing modifier DKG on a polycarboxylate base, which demonstrated heat resistance under the conditions of the study. The introduction of a plasticizer into the system changes the structure of the gypsum stone. This is confirmed by the data of thermogravimetric studies, the effect of increasing the mass shifts towards lower temperatures.

In the case of samples with the addition of carbon and perlite (samples of compositions 3 and 4), in addition to the peaks of loss of crystallization water, an increase in the mass of the samples at temperatures above 300 °C, which is not typical for gypsum, was observed (Figure 4).

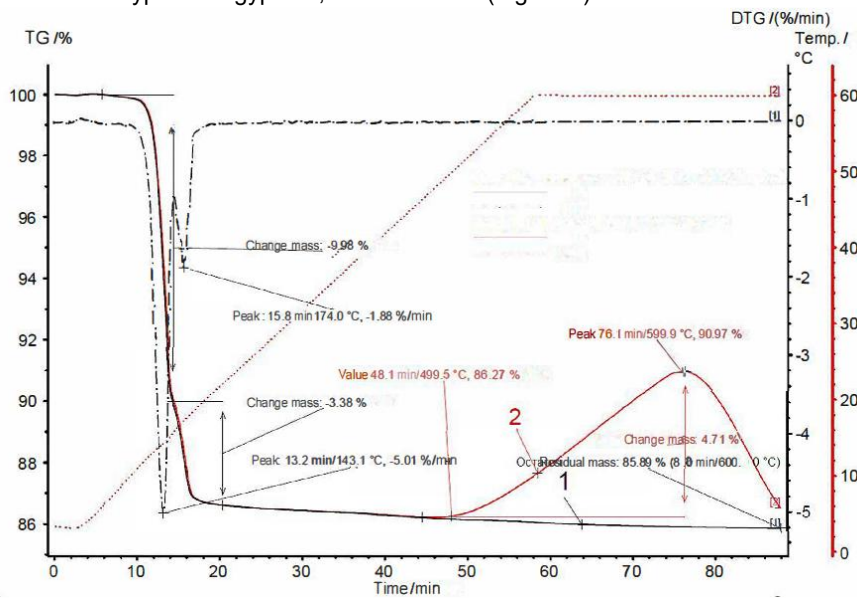


Figure 4: Mass loss curve of sample composition 3 (1 - crushed sample; 2 - lump sample)

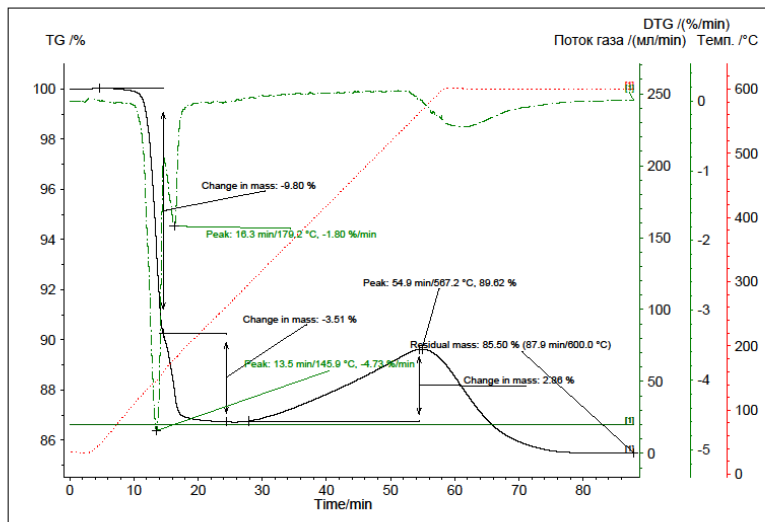


Figure 5: Mass loss curve of sample composition 4

When kept for 30 min at a temperature of 600 °C, the mass of the sample decreased again. The system came to hydrodynamic equilibrium. This effect may be associated with the destruction of the closed porous structure and filling the free volume with argon. This is confirmed by the fact that this effect was not observed when studying the crushed sample (Figure 4, curve 2). In general, the studies confirm that porous fillers are active participants in the hardening processes of the modified gypsum composite.

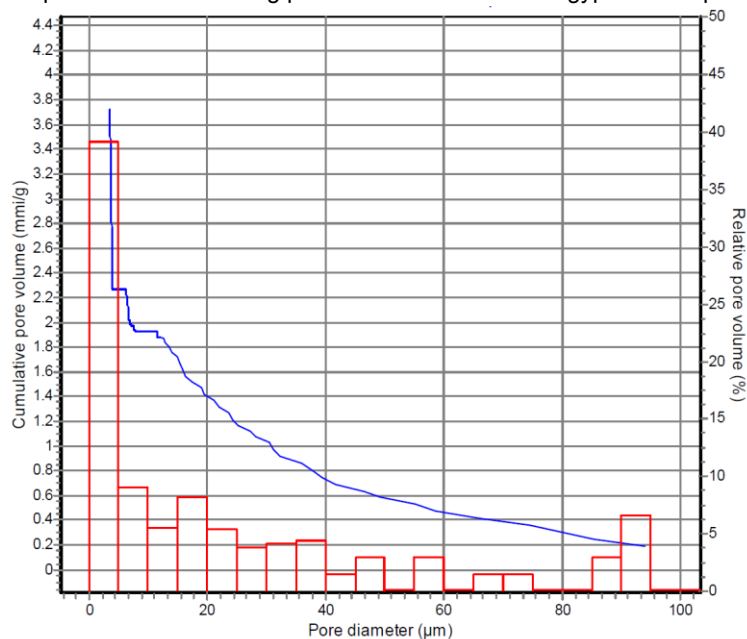


Figure 6: Pore size distribution composition 4

Studies of the pore space of gypsum composites have confirmed the positive role of porous fillers in its modification, and primarily, technogenic carbon. Studies and analyses of the pore size distribution in the gypsum structure with the addition of porous fillers, depending on the total and differential pore volume, have shown that most pores are in the range of less than 10 µm. In the original (control) composition, the samples have a wide pore distribution in the range from 10 to 90 µm. This is due to the formation of new formations of matrix gypsum stone in the contact zone. The structure of technogenic carbon is similar to the structure of fulleroids. However,

it additionally includes silicate particles. This contributes to active nucleation and further growth of tree-like structures based on a modified mixture of gypsum with additives. Intertwining in the volume of the composite, such structures cause microreinforcement of gypsum.

#### 4. Conclusions

The possibility of synthesizing a modified gypsum composite based on a gypsum binder and porous fillers has been theoretically substantiated and experimentally proven. It has been shown that modification of the structure with man-made carbon from ZShS and perlite affects the structure and properties of the resulting composite. Changing the structural porosity of the modified gypsum helps to improve its insulation properties without losing quality. The micro-reinforced structure of lightweight stone reduces water absorption and creep of materials based on it and creates prerequisites for their wider application. The conducted research and determination of physical and mechanical properties at different curing times, as well as the durability of the material in the future, will allow us to expand the scope of application of the developed lightweight composites.

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