Mechanical Properties of Gypsum-PCM Composite Refined with the Acrylic Copolymer

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Abstract
Nowadays, the construction sector is changing rapidly towards more energy-efficient solutions. Many companies strive to improve the properties of building materials by reducing the weight of materials, increasing mechanical properties, and improving insulation properties. Therefore, to bring closer the problems that need to be solved, it was proposed to develop a new gypsum composite that will be used in a drywall. In addition, phase change material (PCM) and copolymer were used to improve thermal properties and tighten the final product against paraffin leakage. The main goal of the study was to check the mechanical properties. The results of endurance tests were presented. Based on that, it was determined that PCM affects the strength properties of the gypsum. In analyzing the obtained results, it can be stated from a mechanical point of view that to a certain extent, it was possible to confirm the correctness of using PCM in gypsum with an acrylic copolymer. The other part of the article presents the course of research and the results confirming the presented hypothesis.

Keywords
paraffin, heat storage, mortar, compressive strength, bending strength

1 Introduction
Every year, the building materials market introduces newer and newer solutions aimed at improving insulation [1] and mechanical properties [2]. Based on treatments, new building materials are being reinforced with various admixtures [3]. PCM is often used in many types of building components. There are different types of applications of PCM in buildings. This can be done in several ways: separated layers, a mixture of different components, and materials such as cement and others [4–6]. Based on many years of analyses aimed at using PCM in construction, three methods have been identified that give the possibility of further research on these materials. One of them is the use of microencapsulated PCM coated with a polymer. This method prevents the PCM from coming into direct contact with concrete or plaster, which would lead to chemical degradation. In addition, there is no risk of PCM leakage when melted. The second way is to use a special polymer with high porosity of up to 80%. Thanks to this, the composites ensure stability even with changes in the state of aggregation, which are called "shape-stabilized PCM". The final method of using PCM is direct application. Unfortunately, this method is currently the least frequently used due to major flaws such as leakage of PCM or significant deterioration of strength. Many scientists are working on improving the properties of plaster. However, gypsum itself as a building material has been used for several hundred years. In the XIX century, the advantages of using plaster as decoration were noticed. It was the beginning of the production of plasterboard due to low production costs [7].

There are several ways to improve material for use in construction. One of them is microencapsulation [8–14]. Microencapsulation makes it possible to integrate PCM into conventional building materials with the advantages of easy application, good heat transfer and no need for protection against destruction [15]. Microencapsulation of the PCM prior to incorporation to the building material is favored in commercial applications. Microencapsulation of PCM prior to incorporation into a building material is preferred for commercial applications. Microencapsulation is a complex process that requires an appropriate method. Depending on which PCM is dealing, physical and chemical methods are used. Physical methods include spray
cooling, spray drying and fluidized bed processes. Physical methods are characterized by rather large capsules that are used on a larger scale. This is because the essence of micro-encapsulation is physical methods. On the other hand, chemical methods are suspension polymerization, dispersion, emulsion in-situ, polymerization, Interfacial polymerization, electroplating. These processes are more complicated, while their result are capsules of much smaller sizes, which makes chemical methods mainly nanoencapsulation [16–19].

It should be remembered that the introduction of phase-change material into the plasterboard can be done in several ways. One of them is microencapsulation, which involves enclosing paraffin in special capsules that prevent it from flowing to the surface. PCM particles enclosed in thin sealed polymer spherical-like capsules, which range in size from 1 μm to 300 μm, can maintain their shape and prevent leakage during the phase change process [8]. In contrast to microencapsulation techniques, the direct introduction of phase-change material has several disadvantages, primarily a deterioration in mechanical properties. As PCM has not binder properties, it is expected to decrease the mechanical properties of the final mortar [20]. The literature shows ways to prevent leakage. When porous materials are used, PCM is absorbed by capillary forces and surface tension, thus preventing leakage of functional PCM [21]. Another large disadvantage of direct application of PCM with mortar is corrosion. This mainly applies to reinforced cement, where the bars made of steel are subjected to corrosion, which causes faster cracking, chipping and reduces the overall mechanical strength of the surface. Such a situation was noticed when adding fatty acids to reinforced cement.

There are three groups on how PCM is defined in building material. Each of them is designed to improve the material properties, but each process gives different effects. Due to their origin, phase-changing materials can be divided into organic and inorganic. The advantage of these materials is the low thermal conductivity, which is advisable in the case of improving thermal properties. However, a serious disadvantage of such materials, mainly paraffin, is their flammability. As a result, the use of such material in the construction of external layers is unacceptable. In the case of inorganic materials, which are, for example, hydrated salts, they have high latent heat and have a greater heat capacity than organic materials. They are often used in installations for solar energy storage. Unfortunately, they cause corrosion and are thermally unstable (supercooling effect). Depending on the nature of the use of a given material, its advantages and disadvantages should be taken into account, but looking at the nature of the presented materials, it seems that organic materials are better in construction [22].

PCM can be divided according to the way of application. The division is as follows: PCM as components, PCM integrated with building materials and PCM in storage units. When the PCM element is one of the layers or parts of a structural section, it has been classified as a "component". On the other hand, when PCM is mixed, impregnated, or incorporated into building material, they are classified as "integrated". These two categories can be used in both passive and active applications. However, the third category, PCM in storage units [23], can only be applied to active systems and is essentially thermally insulated from the building by insulation [24]. This article explores the scenario of using PCM as an integrated material.

One of several challenges of creating a mixture of gypsum with the addition of PCM is mixing it. The method of mixing and adding the ingredients needed to obtain satisfactory results is not without significance. The simplest way is to directly add raw ingredients to get the mixture. Bajare et al. [25] faced such a problem, which added PCM with an addition of 5% and 10%. Then he mixed the contents for 2 minutes as the main reason why PCM cannot be connected directly to gypsum is poor mixing properties. A similar relationship was observed in previous studies [26]. When mixing gypsum with PCM, it occurred that PCM did not bind with gypsum while adding water to the mixture. However, the change in the order in which the ingredients were added caused the PCM to bond with the plaster without flowing to the surface of the mixture. Faced with such problems, many scientists use PCM resulting from microencapsulation, which is more stable for joining various materials as well as in subsequent applications used in construction. However, new solutions arise for PCM to be incorporated into the mortar. One of the more interesting ways to introduce PCM into the mortar, in this case for ceramic tiles, is to use a vacuum pump [27]. Initially, the PCM was dissolved, and after this treatment, the plate was inserted into a vacuum pump to remove air from the pores. To ensure the pores were completely impregnated, the samples were immersed in liquid PCM for 2 hours. The vacuum pump was then turned off to allow air to enter the furnace, and the fully impregnated ceramic tiles were removed from the liquid PCM. The PCM ceramic plates were then placed in an oven at 50 °C to remove excess PCM (present on the surface). The samples were finally...
epoxy coated to prevent the PCM from fusing during use. The process seems to be quite complex, but the application of resin to the surface of the plate ensures that the PCM does not leak. Another issue is the use of a porous material as the basis for direct application in the mortar.

The aim of this article has tested compressive strength and bending for the use of this mixture in construction in the form of plasterboard. In previous studies [26], a recipe for combining gypsum with paraffin and polymer was found, while in this study, an attempt was made to prove that the mixture prepared in this way can be used on a real scale, taking into account the further improvement of the composite.

The article presents the results of the compressive and bending strength tests of gypsum mortar containing paraffin and acrylic copolymer.

2 The effect of PCM application in the inner constructions
Thermal inertia of building affects energy demand [26] and the alignment of temperatures between the external and internal environments without the input of either heating or cooling sources [28]. A new approach to characterise the thermal inertia of the envelope was proposed by Corrado and Paduos [29]. Strzałkowski and Garbalińska [30] revealed that the use of lightweight cement-based materials improves the thermal insulation of the whole building and significantly increases the walls' thermal mass. These investigations led to optimizing building components' properties and developed a product with high thermal mass and good thermal insulation [31].

The use of PCM in walls and ceilings was already investigated in the early 1990s. One of the few studies that were done on a real scale was one that used PCM in the most important areas of a building. To test the validity of PCM operation on a real scale, heat was introduced in winter by fans. The research results were unexpected because it turned out that thermal energy was stored inside the building for about ten days.

In 2005, Zhang et al. [32] presented a wall model that was filled with PCM in the form of macro encapsulation. The authors also used a real-scale model, thanks to which it was possible to reduce the peak value of thermal energy by almost 40%, which shows a very large impact on the internal comfort in the building. Zhang also experimented with the ratio of PCM to the rest of the materials used. Therefore, the presented results are the reduction of the heat flux for 10% PCM by 9% and 20% PCM by 15%.

Oliver [5] came to some interesting conclusions. She noticed that at 30°C, a 1.5 cm thick gypsum board with 44.5% PCM stores as much energy as a brick wall 12 cm thick and five times more energy than an ordinary gypsum board, and at 35°C it 2.5 cm thick gypsum with 44.5% PCM stores as much energy as a 14 cm thick thermal brick wall. In addition, it was found that under the same test conditions, the new 45% PCM gypsum board stores five times more energy per unit mass than a thermal brick wall, 9.5 times more energy than a brick wall and almost three times more energy per unit mass than ordinary gypsum board.

In subsequent studies, many scientists have tried to place PCM in different parts of the building. A roof structure was used in which PCM was used in combination with other materials in the cooling space. PCM was combined with polyurethane foam, and PCM was soaked in special fabrics. Two types of PCM were also used with a melting point of 26°C and 32°C. Tests showed that when installing a metal roof using cool roof pigments, reflective insulation and ventilation ducts, the peak heat flow in summer passing through the roof deck was reduced by approximately 70% compared to a conventional shingle roof. Installing the PCM heatsink generated an additional 20% reduction in heat flow during peak hours for an overall reduction of up to 90%.

As can be seen, many scientists are trying to use PCM in various forms. Thanks to more and more modern technologies and mathematical modelling [34], it is possible to refine the influence of PCM and show the positives of its use in walls and ceilings or roof surfaces. It should be emphasized that a previous study of direct application of PCM was done for a relatively low amount of paraffin, e.g., 10% in weight [35]. Our study increased the PCM amount to 25%, which affected maintaining the material's structure and caused that gypsum composite is not a dry material. Although a decrease of mechanical properties is possible in that case [36], the more flexible material structure could be an advantage of such a product.
3 Sample's preparation and test methods

The purpose of the research was to check the compressive and bending strength of the mixture consisting of raw gypsum and polymer with paraffin. The mixture prepared in this way is intended to improve the thermal properties of gypsum by increasing the heat capacity. To determine whether a given material meets the strength standards and how the PCM and polymer additives affect the mechanical parameters by comparing the test results. Of course, it can be predicted that each additional admixture will change the consistency of the material, especially when paraffin is added, which changes its physical state depending on the temperature.

Instron equipment was used to conduct the following tests (Fig. 1). It is a device used both in laboratories as well as in industrial plants. The single-column construction allows checking the material with a strength of up to 5 kN. In addition, the device allows displacement testing according to Hook's theory.

The materials used to make the samples are synthetic gypsum from flue gas desulphurization and Rubitherm paraffin with a melting point of 21 ºC. The choice of temperature is important because it should be close to room temperature, and in the case of building users, it is justified to use paraffin with a melting point of 21 ºC. During the tests, the applied PCM was in a semi-liquid state, so the test results show the conditions where PCM begins to store thermal energy. When it is converted back to a solid phase, the heat will be released. The choice of temperature is important because it must be close to the room temperature, and in the case of building users, it is justified to use paraffin with a melting point of 21 ºC. The idea of such a mixture will allow it to be used in drywall.

The use of the acrylic copolymer in each mixing has one main purpose; namely, when the phase changes from solid to liquid paraffin, uncontrolled leakage may occur on the surface of the material. In this case, the task of the polymer is to retain the paraffin inside the material by sealing the surface. The choice of the polymer and the method of sample preparation were presented in the previous article, where the focus was on finding the best recipe [26].

Samples were made according to a strictly established recipe checked in previous studies:
1. Mixing water with an acrylic copolymer containing 0%, 0.1%, 0.5% and 1% by weight of the entire sample;
2. Mixing raw gypsum with water in the proportion of 0.35: 0.65;
3. Addition of paraffin to the entire mixture in a ratio of 0.2: 0.8;
4. Mixing for 15 seconds to achieve a homogeneous consistency.

After the samples were made in accordance with the points above, they were placed in a room with a temperature of 21ºC in moderately humid conditions. In order to maintain reproducibility, subsequent samples from days 3, 7, 14, 21, 28 were stored under the same conditions. The same was done for the bending samples.

3.1 Compressive strength test method

Compressive strength samples were made according to the above-mentioned recipe. In order for the measurement to give reliable results, it was decided to carry out more samples according to the list below:
• 4 samples with 0.0% acrylic copolymer and no paraffin as reference samples,
• 4 samples of 0.1% acrylic copolymer,
• 4 samples of 0.5% acrylic copolymer,
• 4 samples from 1.0% acrylic copolymer.

Samples were made in 6 series for the purpose of strength tests after 1, 3, 7, 14, 21 and 28 days after execution. In this way, 96 compression samples were made. The dimensions of the samples were 40 mm × 40 mm × 40 mm (Fig. 2). The diagram below and the photo illustrate how the compression tests were carried out. The test was carried out at a temperature of 20ºC. The compressive strength test was carried out based on the PN-EN 13279-2: 2014-02 standard [37].
3.2 Bending strength test method

As in the case of compressive strength, several samples were also made in the flexural strength test to confirm the validity of the thesis. The samples were also made according to the recipe given above, and so:

- 3 samples with 0.0% acrylic copolymer and no paraffin as reference samples,
- 3 samples with 0.1% acrylic copolymer,
- 3 samples with 0.5% acrylic copolymer,
- 3 samples with 1.0% acrylic copolymer.

Samples were made in 6 series for the purpose of strength tests after 1, 3, 7, 14, 21 and 28 days after execution. In this way, 72 bending samples were made. The dimensions of the samples were 160 mm × 40 mm × 40 mm. The bending strength test was carried out based on the PN-EN 13279-2: 2014-02 standard (Fig. 3). The test was carried out at a temperature of 20°C. The following diagram and photo illustrate how the tests were carried out.

4 Results and analyses

Based on the strength tests, results were obtained to determine whether the gypsum mixture can be used in building applications. Analyzing individual results, the samples in which the polymer was used have different mechanical properties. As expected, paraffin affected the strength of the samples; namely, it deteriorated depending on the polymer content. It can be seen that for seven days, the results differ quite significantly. In the first week, polymer samples had better results with more polymer for both samples intended for the compressive and bending strength tests. For 1% of polymer content, the compressive strength was higher and amounted to about 0.1 MPa in each of the three days. The situation changed after 14, 21 and 28 days. It is clearly seen that the strength along with the polymer decreased. It was respectively 0.3, 0.16 and 0.12 MPa for 14, 21 and 28 days for samples with 1% polymer.

However, the bending samples in the first seven days were comparable depending on the percentage of polymer. For a value of 1%, the strength for 1, 3 and 7 days was 0.24, 0.24 and 0.17 MPa, respectively. In the following days, samples with 0.1 % content were characterized by the highest bending strength.

An interesting relationship can be observed in the tests, which characterizes individual samples. Namely, during the compressive strength test, the samples behaved differently at the time of destruction. This relationship can be observed in Fig. 4, the very process of destruction.

By analyzing Fig. 4, it can be observed an interesting phenomenon that occurred during the measurement. As can be seen, the sample in Fig. 4(a) shows a clear destruction process in the diagram. The line in the diagram representing the compressive force is almost vertical
and ends with a sharp point. This means that the destruction occurred instantaneously with visible destruction. The conclusion is that the material is not flexible and can be damaged by small forces, especially when it is intended for plasterboards. However, in Fig. 4(b) it can be seen that the course of the destructive force is not clear. You can clearly see that the graph is rounded, and it is difficult to tell if the sample was completely damaged.

The surface is deformed, but the sample is not damaged by force, as was the case of pure gypsum. Based on this observation, it can be concluded that samples containing the polymer are more flexible, which may be advantageous in the case of plasterboards.

In Fig. 4, several relationships between the polymer and the paraffin content in the sample can be noticed. In the first seven days, the values are so small that the average is practically equal to the values obtained during the measurement. The results are different after 14 days from the measurement; namely, the maximum values vary between 0.5 MPa and 3.5 MPa. However, when analyzing the results in individual days, there is an upward trend in which, as expected, the highest value was achieved on day 28 for pure gypsum.

This means that the test itself was performed correctly, and the strength of the raw gypsum on day 28 was 3.38 MPa. As to be expected, samples with admixtures had lower strength. The 0.1% polymer samples showed slightly higher values on day 28 and were 1.63 MPa compared to 0.5%, where it was 1.53 MPa.

The comparison is made in the same way for the flexural strength test as is the compressive strength test; namely, the reference sample is the raw gypsum sample (Figs. 5 and 6). Although the result for the crude cast on day 28 was lower than on the previous days. It is noteworthy that the mean value for the sample on day 28 for raw gypsum is due to a fairly large range between 1 MPa and 3 MPa. Similarly, although not so large, ranges were found in other results after the 14th day of measurement. Comparing the results with samples with the participation of the polymer, the flexural strength increases with time. For comparison, this time, the best results were given by the sample with 1% polymer on day 28, where the value was 2.28 MPa.

![Fig. 5 Compressive strength as a function of time for (a) 0.0%, (b) 0.1%, (c) 0.5%, (d) 1.0% of acrylic copolymer content](image-url)
However, the results for the remaining shares are very similar; for 0.1% on day 28, it was 1.83 MPa, while for 0.5% on day 28, it was 1.78 MPa. There was no great reduction in the flexural strength of the polymer samples compared to pure gypsum.

5 Conclusions

Due to newer and newer construction trends, this article presents the strength tests of the gypsum mixture as a continuation of previous research, which aimed to find the best components, as well as mass ratios and the method of making samples. In referring to this article, it was possible to investigate the compressive and bending strength. Comparisons are presented in the form of diagrams of the measurements after 1, 3, 7, 14, 21 and 28 days from the time of performance. This allows us to compare which percentage of acrylic copolymer gives the best results in relation to a pure gypsum per unit time.

Based on the strength tests carried out, several promising conclusions can be formulated, which will allow the continuation of work on the proposed composite. One is the degree of flexibility of the material that can be applied to various structures. The main conclusion is that the addition of PCM and acrylic copolymer reduces strength, but not so much that the material becomes unsuitable for use in construction. Moreover, it is more flexible, which is a positive feature.

The most important conclusion that can be drawn from the strength tests is how much the compressive and bending strength has decreased compared to raw gypsum. In the flexural strength test, the best bending result is approx. 300 N of destructive force for the 1% acrylic copolymer sample on day 28 of the test and is close to the best result for raw gypsum on day 21. Moreover, the values for 0.1% are about 240 N of the destructive force, and for 0.5% are 237 N of the destructive force, so they do not differ from each other and allow for further testing of the given mixture. This means that despite the admixtures proposed in this paper, it is possible to increase the advantages of gypsum with heat accumulation properties without significantly deteriorating other parameters, in this case, bending and compressive strength. This allows for further research focusing on the determination of the heat capacity and the thermal conductivity coefficient.
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