

Izabela Marchewka^{1*}, Ewa Downar-Zapolska¹, Waldemar Pichór²

¹Lakma SAT, Cieszyn, Poland

²AGH – University of Science and Technology, Faculty of Materials Science and Ceramics, al. A. Mickiewicza 30, Krakow, Poland

*Corresponding author. E-mail: izabela.marchewka@lakma.com

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EFFECT OF PREPARATION METHOD AND POLYMER IMPREGNATION ON FORM STABILITY OF EXPANDED PERLITE/PARAFFIN COMPOSITE

Interest in the use of phase change materials in construction materials is constantly increasing. However, the problem is to develop an effective method to introduce them. The challenge is to obtain shape-stabilized composites with a high heat storage capacity. In this paper, expanded perlite was proposed as a potential carrier of phase change material (paraffin). The effectiveness of two impregnation techniques – vacuum and immersion – was compared. Composites with various amounts of paraffin were prepared, and then their properties were characterized. It has been shown that the vacuum impregnation method can be used to obtain composites with better shape stability. Despite this, leakage was observed in the materials with high proportions of paraffin. It was been proven that, regardless of the impregnation method, the application of a thin polymer dispersion coating can effectively protect the composite against leakage of the phase change material. Thanks to the use of epoxy resin, a stable composite was obtained with a paraffin content of over 80% by weight. The high PCM content and no leakage effect during the phase transition make the presented composite show great potential for many different applications.

Keywords: phase change materials, expanded perlite, perlite/paraffin composite, leakage prevention, polymer resin impregnation

INTRODUCTION

The building sector is responsible for the largest consumption of energy worldwide. According to data provided by the International Energy Agency, the amount of energy consumed by residential and commercial buildings is constantly increasing, and this trend is expected to continue [1]. A significant share is the energy used for heating and cooling [2]. In the search for new solutions to reduce energy consumption, many scientists have become interested in phase change materials. The use of the latent heat of phase change may be a form of thermal energy storage. Therefore, phase change materials are used in all situations that require maintaining a stable temperature. This also applies to maintaining temperature in a closed room [3].

Numerous research works are being carried out on the possibility of using PCM in building materials. The analyses conducted in 2012 by Kosny et al. showed that placing PCM panels in the attic reduces the energy used to heat by 30% and the energy used to cool by 50% [4]. Xin, Medina, and Zang performed simulations on the placement of panels made of phase change material in different places in the building envelope [5]. Attempts are also made to directly create PCM composites with building materials such as cement, concrete, and mortars. The main problem concerns the selection of an effective method of introducing PCM into the material

and ensuring shape stability during phase transformation [6]. PCM in liquid form tends to bleed out of the material and form greasy stains. In most cases, the application of phase-change material by direct introduction or immersion is not able to limit this phenomenon. Therefore, it is necessary to look for effective methods of encapsulation and stabilization of the shape of phase change materials [7].

To date, attempts have been made to limit this phenomenon by means of coating materials [8], the use of polymerization techniques [9] or by depositing the material in a porous carrier. Research is being conducted on PCM stabilization by depositing them in expanded graphite [10, 11], activated carbon [12], diatomite [13, 14] expanded perlite [15, 16] and polymer carriers [17]. Other attempts to solve this problem have been to cover the composite with a hydrophobic coating prior to incorporation into cement [18] or applying protective coatings, for example, epoxy, to the composite [19].

In this work, an attempt was made to create a tight composite containing PCM for use in building materials. Two techniques for introducing PCM into expanded perlite were compared: immersion and vacuum impregnation. The selected samples were coated with the selected polymer dispersions to reduce leakage of the heat storage material. The aim of the work is to determine

how to obtain a stable composite with the highest possible content of phase change material.

Among the diverse group of phase change materials, paraffin was selected, which is characterized by an appropriate melting point, relatively high phase change heat, good stability, and low price [20].

MATERIALS

Tables 1 and 2 present the basic characteristics of expanded perlite and paraffin used in this work for preparation of the composites.

TABLE 1. Basic characteristics of expanded perlite

Parameter	Value
bulk density	48 kg/m ³
water absorption	450%
granulation	1.4-2 mm

TABLE 2. Basic characteristics of paraffin

Parameter	Value
density	880 kg/m ³
melting point	45°C
flash-point	> 180°C

To reduce the leakage of paraffin from the composite, three different polymer dispersions were attempted. Selected dispersions differ in their chemical nature and performance parameters. Table 3 presents a comparison of the most important parameters of the selected binders.

TABLE 3. Basic characteristics of selected polymer dispersions

	A	B	C
Solid content	50%	53%	45%
Chemical characteristics	Styrene-acrylic copolymer	2 component epoxy system	cationic epoxy-resin-ester (prepolymers)
Density [g/cm ³]	1.04	1.05	1.07
Tg [°C]	5	nd.	nd.
Dynamic viscosity [mPa·s]	< 3000	1000-6000	25-200

nd. – no data available

PREPARATION OF COMPOSITES

The perlite-paraffin composites were prepared using two different techniques – immersion impregnation and vacuum impregnation.

Immersion impregnation was performed at an elevated temperature. The paraffin was dissolved at 100°C, and then the appropriate amount of expanded perlite was poured into it and stirred. The container with paraffin and perlite was placed back into the heating chamber. The process was carried out for 8 h, then the con-

tent of the tank was poured through a sieve. The perlite was placed in a separate container where it was stirred until the paraffin solidified. Because of the inability to precisely control the amount of paraffin absorbed by the perlite, it was only possible to determine the maximum saturation. Tests with other perlite:paraffin ratios were not performed.

Vacuum impregnation was performed in a specialized vacuum mixer. Perlite was weighed in the vessel and then the appropriate amount of previously dissolved paraffin was poured into it. The whole was placed in the heating chamber until the temperature reached 100°C. The whole was then placed under a vacuum stirrer for 10 minutes. Tests were carried out on composites with the following perlite:paraffin weight ratios: 1:2, 1:3, 1:4, 1:5.

The selected samples were coated with different polymer binders. The application process was performed by measuring a specific volume of the composite, and then pouring the measured volume of dispersion into the container. The amount of polymer was different for each sample. The whole mixture was stirred until the resin solidified on the surface of the composite. Coating tests were carried out by introducing the following binder volumes into 100 ml of the composite: 5, 10, 15 and 20 ml. The application of 20 ml of resin had to be carried out in two stages, 10 ml each due to excessive sticking of the composite particles.

RESULTS

Maximum paraffin absorption

The total paraffin content in the composite was determined by burning the samples with the largest percentage of PCM at 450°C. As a result, there was complete decomposition of the organic content. Table 4 presents the obtained results.

TABLE 4. Maximum paraffin absorption capacity

Method of impregnation	Maximum paraffin content [%mas.]
Immersion	83%
Vacuum	69%

The sample impregnated by immersion by the action of capillary forces absorbed 83% of the mass of paraffin. This result could not be replicated in the case of vacuum impregnation. When paraffin was added in amounts above the ratio of 1:3, unabsorbed material remained visible at the bottom of the vacuum container. After burning a sample of the drained composite prepared in a 1:5 ratio, the result was 69% by mass of paraffin.

Nonetheless, it should be considered that the presented results may be subject to errors resulting from the presence of paraffin also on the surface of the composites. There were noticeable visual differences be-

tween the individual composites. The samples prepared by immersion and vacuum impregnation in a ratio higher than 1:3 were characterized by a reflective light coating, which indicates the remains of paraffin on the surface. The comparison is presented in Figure 1.



Fig. 1. Perlite:paraffin composites, from left: vacuum impregnated 1:5, immersion impregnated, vacuum impregnated 1:3

Leakage test

Evaluation of leakage resistance was carried out by measuring the same volume of individual composites and placing them on white paper. The samples prepared this way were placed in a heating chamber at 50°C for 4 hours. After the designated time, the composite was removed and the size of the greasy trace left on the paper was compared. ImageJ software was employed to assess the leakage area of the samples. Figure 1 presents the results for selected samples before impregnation with polymer dispersion.



Fig. 2. Leakage of composite paraffin, from left: vacuum impregnated 1:5, immersion impregnated, vacuum impregnated 1:3

The composites prepared by immersion impregnation were characterized by the greatest tendency to leak PCM. The vacuum impregnated samples left a smaller mark on the paper, even in the case of the composites with previously observed paraffin residues on the surface. The most promising results were obtained for the vacuum impregnated composite in the ratio of 1:3.

The immersion impregnated and vacuum impregnated perlite in the ratio of 1:3 were further modified with polymer dispersions. Figures 3 and 4 present the results of the surface of the observed leak.

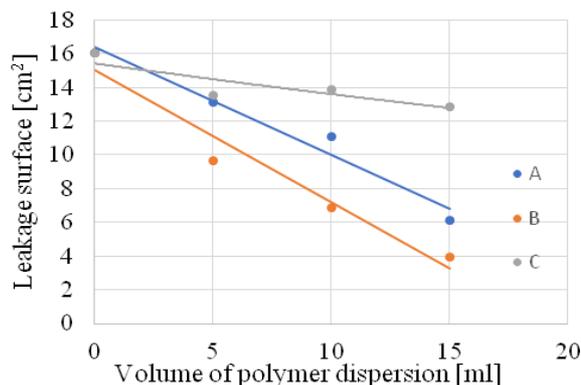


Fig. 3. Leakage surfaces for immersion impregnated samples

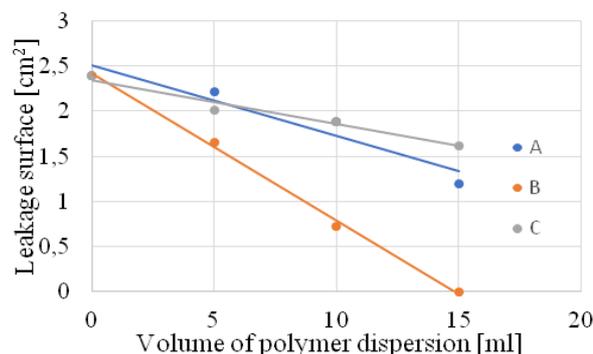


Fig. 4. Leakage surfaces for vacuum impregnated samples (perlite: paraffin ratio 1:3)

The effectiveness of protection against paraffin leakage from the composite was different for both selected composites. Significant differences were also observed between the individual binders. In the case of acrylic dispersion, the effectiveness of protection was the lowest and the leakage observed for the samples with different polymer additions remained at a similar level.

The most effective protection for both composites turned out to be the dispersion of epoxy prepolymers. The difference was in the amount of resin needed to completely stop the PCM leak. For the vacuum-prepared composite, no paraffin stains were observed on the sheet at 15 ml. The composite from the immersion process required the application of 2 layers of 10 ml. The observed difference may result from the presence of paraffin on the surface of the immersion impregnated sample, which makes it difficult for the polymer dispersion to wet the surface and surround the composite with an uninterrupted coating.

Bulk density

The bulk density of individual composites was characterized by weighing the composite poured into a metal container with a volume of 100 ml. Figure 5 shows the obtained results.

The density of paraffin is higher than the density of the air contained in the pores of the expanded perlite.

For this reason, the saturation of pores with paraffin changes the bulk density of this material. The density of the composite with the maximum paraffin filling was almost six times higher than the density of the expanded perlite alone.



Fig. 5. Bulk density of composites

Influence on thermal conductivity

The thermal conductivity coefficient was determined by pouring the material into a previously prepared plastic frame and then placing the sample prepared in this way in the measuring apparatus. Thermal conductivity measurements were carried out using the stationary method with a TA Instruments FOX 50 apparatus. Measurements were conducted in the temperature range of 5 to 15°C. The values obtained from the thermal conductivity coefficient measurements are listed in Table 5.

TABLE 5. Thermal conductivity coefficient of perlite and composite perlite/paraffin

Sample	λ [W/mK]
Expanded perlite	0.041
Perlite/paraffin composite (immersion)	0.068

The impregnation of perlite with paraffin resulted in an increase in thermal conductivity. The value obtained for the composite was over 60% higher than that of the expanded perlite alone. The obtained results are the effect of filling the empty pores with paraffin with a higher thermal conductivity than air.

CONCLUSIONS

The studies showed that expanded perlite can be an effective carrier for phase change material. Both vacuum and immersion impregnation can be a successful technique for incorporating selected PCM into expanded perlite. The experiment showed that the impact of capillary forces during immersion is large enough for the investigated carrier to absorb up to 83% of the paraffin. Only 69% was achieved using vacuum. The difference in the results may be due to air trapped in the

perlite pores during introduction of the paraffin by the vacuum technique.

The vacuum impregnated perlite exhibited a lower tendency to leak compared to the immersion impregnated sample. The molten paraffin trace was the smallest for this sample. This was justified by the lack of paraffin residue on the surface of the composite. The entire PCM was effectively trapped under the influence of vacuum in the pores of the material. However, the characteristics of the immersion impregnated samples are different. The observed leakage is very large, and even on visual inspection there is a layer of paraffin on the surface of the perlite.

Attempts to protect the composite against PCM leakage demonstrated that both the method of impregnation and the characteristics of the selected coating material are important. Basic acrylic dispersions commonly used in polymer coatings turned out to be too soft for this type of application. The most effective was epoxy prepolymers, which were good at wetting even the surface with paraffin residues.

Filling the voids in the pores of expanded perlite with paraffin significantly affected the density of this material. Also, as a result of the higher heat conductivity coefficient of paraffin than that of air, the thermal conductivity coefficient of the composite increased. Nonetheless, it remains low enough for the presented composite to be used in materials with increased thermal insulation.

The research results presented in this article are the beginning of a project related to PCM-modified coating materials. Further stages of work will be an attempt to use the prepared composite in wall coatings such as polymer plasters. The research will also cover insulating coatings.

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