Assessment of the applicability of a phase change material in horizontal building partitions

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Abstract. The contemporary building sector needs new technologies for reducing the energy consumption in heating and cooling processes used in building interiors. These solutions should be integral to ensuring the comfort of using such premises. Phase change materials (PCM) that absorb, accumulate and return large amounts of heat energy as part of their phase change temperature meet such needs. These materials can absorb excess heat in overheated premises at times when the temperatures are high, during long hours of sunshine in summer time, or from internal sources such as working machinery or office equipment. The stored heat energy, due to the postponement, will be transmitted to the room at a later time, usually in the evening. Thanks to these properties, it is possible to provide better utility conditions in building interiors. This article describes the test results of selected properties of PCM modified materials applied in floor systems that are used in premises where they are subject to significant overheating. The analysis took into account the impact of a phase change material on the mechanical and physical properties of the tested material solutions. This modification is intended to provide better thermal comfort in the rooms, while the use of PCM has a beneficial effect on reducing the temperature fluctuations in the premises where it is used.

1. Introduction

Currently, in the investment construction business phase change materials are used in various construction areas. Among other things they are incorporated into the traditional building materials used for the construction of various buildings. Phase change materials, in the form of microcapsules, are introduced as ingredients for construction components used for construction solutions and finishing of rooms in a building. This use of phase change materials is intended to absorb excess heat from the occupied premises during the day, and then return it in the evening and night. At the same time, the temperature fluctuations in the room are reduced, which results in higher comfort in the use of these rooms. Overheating can be caused by various appliances in the room that produce excess heat during operation, as well as by the sun's radiation through the glazing located in the outer walls. Studies that use phase change materials for building materials applications are being conducted in many research centres.

Article [1] presents the use of phase changemicroparticles in concrete floors. Experimental studies were carried out on four boxes of 1130 mm x 725 mm x 690 mm. PCM was introduced into two of them in a concrete floor. All of the boxes were located on a university campus in the Netherlands. Each of them had a window facing south. In the boxes containing PCM floor concrete, the maximum temperature was reduced by 16%, while the minimum was increased by 7%. Article [2] presents the development and characterization of a composite material, plastering mortar with microcapsule filler.
material. Quantitative analysis and experimental studies were conducted to compare the results obtained with the properties of commonly used mortars without phasechange materials. An improvement of the thermal performance of the modified mortars was achieved with a 25% proportion of phasechange material in the mortar mass. Article [3] describes a study of selected technical features of geopolymer mortars with varying content of phasechange material. In this work, the mechanical and thermal properties of geopolymer mortars synthesized from fly ash with low calcium content and different amounts of PCM were determined experimentally. A compressive strength of geopolymer mortar containing up to 20% PCM is sufficient for this material to be used in buildings. It has been shown that the use of PCM in these mortars increases the thermal inertia of buildings, while reducing energy consumption to provide internal thermal comfort. Article [4] presents considerations of the use of thermoregulatory material for gypsum. The thermoregulatory material is a composite PCM blend and a polymeric substance consisting of polystyrene-divinylbenzene. The influence of this material on the internal structure and the main physical, thermal and mechanical properties of gypsum were investigated. The results obtained were compared with other thermoregulatory materials. Article [5] discusses the use of microcapsule phase change material in cement mortar. Theoretical modelling and experimental research have been carried out. These considerations are important in the context of the general use of cement mortar in construction. Article [6] describes the use of microcapsule phase change material applied to a plastering mortar structure. An experimental study was conducted to determine the effects of PCM application on the plaster structure. The tests confirmed that the plaster with the phase change material showed higher insulation, and that the heat-storage effect of the analysed plaster component was significant. Plaster with PCM shows more absorption of solar radiation. Article [7] presents the operation of water-based floor heating technology using heat from solar radiation. Phase change material was used in the heating system, which enabled greater stabilization of the heating medium. Energy gains have been shown. Considerations were made theoretically with simultaneous experimental verification. Article [8] describes the use of phase change material in energy-efficient buildings. The possibility of incorporating PCM into building housing elements (walls, floor) is presented. This is aimed at absorbing solar energy and reducing daily temperature fluctuations in the premises of the building. Article [9] contains information on the issues of heat storage in phase change materials. It presents a set of phase change materials that are active at different temperature ranges, which enables them to be used in a number of technical solutions. It draws attention to the introduction of phase change material in the form of microcapsules, which eliminate many application problems in material and technological construction solutions. There are also studies connected with the location of phase change material in vertical building walls. The contribution of such material also increases the temperature stability in the utility room [10]. The literature compiled indicates the direction of current research in the field of new building materials and technologies. This article discusses the evaluation of selected technical features of horizontal construction partitions using phase change material.

2. Experimental program
To limit the number of experiments required, a statistical algorithm was used to significantly reduce the number of mortar specimens to be tested. The STATISTICA package was used to create the plan. A central compositional plan was selected with repetition of the central point experiment. The selection process of the experiment was similar to that described in [11,12]. The experiment plan took the form of a table (Table 1). Each of the ten lines of the table shows one point of the plan and describes the parameters of the experiments that were performed separately for cement and epoxy mortars. The selected experiment plan assumed repetition of the study at the central point, so the points marked as 2 and 10 and 3 and 9 for cement and epoxy mortars, respectively, do not differ in composition.
Table 1. Summary of parameters describing the composition of mortars for individual points of the experiment plan.

| No. point in plan | Cement mortar | Epoxy mortar |
|-------------------|---------------|--------------|
|                   | PCM content   | Water / cement ratio W/C | Ratio of aggregate to binder A/B | Ratio of binder to microfiber B/M |
|                   | $x_1$ % vol.  | $x_2$        | $x_1$ | $x_2$ |
| 1                 | 11.716        | 0.259        | 5.75  | 0.5   |
| 2                 | 11.716        | 0.4          | 5.04  | 0.64  |
| 3                 | 3.431         | 0.3          | 5.04  | 0.5   |
| 4                 | 11.716        | 0.541        | 5.54  | 0.6   |
| 5                 | 20.00         | 0.5          | 4.33  | 0.5   |
| 6                 | 23.432        | 0.4          | 4.54  | 0.4   |
| 7                 | 20.00         | 0.3          | 5.04  | 0.36  |
| 8                 | 0.00          | 0.4          | 5.54  | 0.4   |
| 9                 | 3.431         | 0.5          | 5.04  | 0.5   |
| 10                | 11.716        | 0.4          | 4.54  | 0.6   |

2.1. Materials

2.1.1. Cement mortars modified with PCM. As a cement binder in cement mortar, CEM I 42.5 R Portland cement was used. The aggregate was quartz sand with a grain size of 0-2 mm, compliant with the requirements of PN EN 196-1. The other ingredients were: tap water and modifier in the form of encapsulated phase change material Micronal DS 5038 X. The modifier was a partial substitute for sand, starting with the smallest fractions. Based on the available literature [13], it was established that the percentage of PCM relative to sand volume and water binder ratio would be the input values in the experimental plot (Table 1, Columns 2 and 3).

2.1.2. Epoxy resins modified with PCM. The binder in the epoxy mortar was epoxy resin Epidian 5. The hardener used was triethylenetetramine (Z-1) in an amount of 10% by weight relative to the weight of the resin. The aggregate was quartz sand with a grain size of 0-2 mm, compliant with the requirements of PN EN 196-1. As with cement mortars, Micronal DS 5038 X encapsulated phase change material was used as a modifier, but in resin mortars, it acted as a microfiller. After analysing the literature data [14-16] it was determined that the ratio of aggregate to binder A/B and the ratio of binder to microfiber B/M would be the input values in the experimental plan (Table 1, columns 4 and 5).

2.2. Methods

2.2.1. Method for strength testing. After mechanical mixing of the respective components of each type of mortar, samples of dimensions 40x40x160 mm and 200x200x50 mm or 200x200x10 mm were prepared for each point of the experiment plan, for strength and heat tests respectively. Cement mortars matured for 28 days under the conditions laid down in PN-EN 196-1: 2006. In order to cure the resin mortars, they were left for 7 days under laboratory conditions.

The flexural strength test and the compressive strength test were carried out on strength testing machines fitted with appropriate inserts in accordance with PN EN 196-1: 2006. For the testing of compressive strength, the beam halves formed after the flexural strength test were used.

2.2.2. Method for thermal testing. Determination of the possibility of using the latent heat of phase change material applied in a floor layer was carried out in a thermal box placed in a climate chamber. A box of 250x250x600 mm was made of extruded polystyrene, in which samples were placed.
A cement mortar sample from point No. 6 of the plan (table 1) with the highest content of 23% PCM and an epoxy mortar sample from point No. 4 of the plan (table 1) were chosen for the tests. The flexural and compressive strengths of these samples are satisfactory, despite the high content of the phase change material. For comparison purposes, a reference sample without PCM was also prepared (Figure 1).

Selected parameters characterizing the properties of PCM are given in Table 2.

**Table 2.** Selected properties of PCM.

| Material   | Density [kg/m³] | Melting temperature [°C] | Solid temperature [°C] | Specific heat [J/kg K] | Latent heat [kJ/kg] | Thermal conductivity [W/mK] |
|------------|-----------------|--------------------------|------------------------|------------------------|---------------------|----------------------------|
| Micronal DS5038X | 300-400 | 25                        | 24                     | ~ 2.0                  | 100                 | ~0.2                       |

Samples of 200 x 200 x (50 + 10) mm were placed in the bottom of the box. Figures 2 and 3 show the arrangement of the sample and temperature sensors in the box. The whole box was introduced into the climate chamber in order to perform the heating cycle of both samples.

Test samples in the first stage of the cycle were stabilized at 20°C for 8 hours. Then the temperature was gradually increased over a period of 6 hours. After the heating cycle, the temperature was lowered to 20°C for a period of 6 hours.
For another 28 hours, the temperature was stabilized at 20°C so that it was possible to determine the return time of the heated samples to the initial temperature before heating. The time of the entire cycle was assumed to be 48 hours.

3. Results and discussion
Analysis of the results of the strength tests began with the determination of mean values and standard deviations. These figures are presented in Tables 3 and 4.

Table 3. Summary of results of mean values and standard deviations of flexural and compressive strengths of samples of modified PCM mortars

| No. point in plan | PCM content | Water / cement ratio W/C | Flexural strength $f_{f}$ | Compressive strength $f_{c}$ |
|------------------|-------------|--------------------------|---------------------------|-----------------------------|
|                  | $x_{1}$ % vol. | $x_{2}$               | $z_{1}$ MPa | $z_{2}$ MPa |
| 1                | 11.716       | 0.259                   | 1.45 ± 0.29 | 2.70 ± 0.59 |
| 2                | 11.716       | 0.4                     | 9.52 ± 0.78 | 44.57 ± 0.94 |
| 3                | 3.431        | 0.3                     | 2.27 ± 0.35 | 2.42 ± 0.65 |
| 4                | 11.716       | 0.541                   | 8.46 ± 0.35 | 30.85 ± 2.65 |
| 5                | 20.00        | 0.5                     | 8.19 ± 0.81 | 28.03 ± 0.97 |
| 6                | 23.432       | 0.4                     | 9.00 ± 0.23 | 40.87 ± 1.16 |
| 7                | 20.00        | 0.3                     | 196 ± 0.05  | 2.28 ± 0.24  |
| 8                | 0.00         | 0.4                     | 9.24 ± 0.84 | 42.88 ± 3.23 |
| 9                | 3.431        | 0.5                     | 8.42 ± 0.31 | 30.55 ± 1.06 |
| 10               | 11.716       | 0.4                     | 8.51 ± 0.13 | 41.40 ± 2.65 |

Table 4. Summary of mean values and standard deviations of flexural and compressive strengths of epoxy mortar samples with PCM microfiber.

| No. point in plan | Ratio of aggregate to binder A/B | Ratio of binder to microfiber S/M | Flexural strength $f_{f}$ | Compressive strength $f_{c}$ |
|------------------|----------------------------------|----------------------------------|---------------------------|-----------------------------|
|                  | $x_{1}$                          | $x_{2}$                          | $z_{1}$ MPa | $z_{2}$ MPa |
| 1                | 5.75                             | 0.5                              | 23.47 ± 1.68 | 51.25 ± 1.15 |
| 2                | 5.04                             | 0.64                             | 15.67 ± 0.44 | 51.15 ± 3.25 |
| 3                | 5.04                             | 0.5                              | 13.58 ± 0.73 | 44.62 ± 1.07 |
| 4                | 5.54                             | 0.6                              | 23.92 ± 1.22 | 52.92 ± 2.70 |
| 5                | 4.33                             | 0.5                              | 19.74 ± 1.35 | 47.67 ± 1.32 |
| 6                | 4.54                             | 0.4                              | 16.98 ± 0.81 | 33.93 ± 1.25 |
| 7                | 5.04                             | 0.36                             | 14.73 ± 0.99 | 30.80 ± 1.08 |
| 8                | 5.54                             | 0.4                              | 15.96 ± 0.48 | 31.55 ± 0.79 |
| 9                | 5.04                             | 0.5                              | 21.37 ± 0.66 | 45.58 ± 3.46 |
| 10               | 4.54                             | 0.6                              | 21.67 ± 1.08 | 55.80 ± 1.02 |

On the basis of the Fisher-Snedecor F test it is possible to reject the statistical null hypothesis, which is equivalent to finding that the composition of the raw material mixture significantly differentiates the properties of the samples. Using the Brown-Forsythe test the hypothesis of homogeneity of variance was confirmed, which enabled the process of searching for a function describing the test object. This function roughly describes the relationship between output size and input quantities. It was assumed that the approximation function would take the form of a second-degree polynomial. This form of function is suggested by StatSoft experts for the adopted experiment plan [17] and the experience of the research centres indicates that the adopted regression function
adequately describes the relationship between the data characterizing the composition of the composite and the technical properties of the resin composites studied [18]:

\[ \hat{z} = A_0 + A_1 x_1 + A_2 x_1^2 + A_3 x_2 + A_4 x_2^2 + A_5 x_1 x_2, \]  

(1)

where:
\( \hat{z} \) – value of the test object function for the actual values of the variables,
\( x_1, x_2 \) – actual input variables accepted in the test plan:
\( A_i \) – coefficients of equation for actual variables.

This function can therefore take the form:

a) For cement mortar:
\[ \hat{z} = A_0 + A_1(\%PCM) + A_2(\%PCM)^2 + A_3(w/c) + A_4(w/c)^2 + A_5(\%PCM)(w/c) \]  

(2)

a) For epoxy mortars:
\[ \hat{z} = A_0 + A_1(\%K/S) + A_2(\%K/S)^2 + A_3(S/M) + A_4(S/M)^2 + A_5(\%K/S)(S/M) \]  

(3)

Table 5 summarizes the values of the approximation coefficients for each output variable for cement and resin mortar, respectively. Non-significant coefficients were discarded, and the tables were marked with a "-".

Table 5. Summary of polynomial coefficients and determinants for flexural compressive and strengths of cement mortar samples modified with PCM and epoxy mortar samples with PCM microfibers.

| Polynomial coefficient / Determination coefficient | Cement mortars | Epoxy mortars |
|---------------------------------------------------|----------------|---------------|
|                                                   | Flexural strength | Compressive strength | Flexural strength | Compressive strength |
| A0                                                 | -42.450          | -277.81        | 262.010          | 111.249             |
| A1                                                 | -              | 1.55           | -102.193         | -69.897             |
| A2                                                 | -0.006          | -0.06          | 9.491            | 7.053               |
| A3                                                 | 226.488         | 1443.96        | –                | 350.450             |
| A4                                                 | -248.646        | -1648.02       | –                | -247.835            |
| A5                                                 | –              | –              | –                | –                  |
| R²                                                 | 0.906           | 0.851          | 0.569            | 0.903               |

The tables also show the values of coefficients of determination determined for each function of the test object. These values of the coefficient, which are close to unity (0.851 – 0.906), show that the estimated model largely explains the variance of the dependent variable. This can be considered as one of the forms of verification of the adequacy of the function approximating the experimental data.

A summary of the results of the experiment based on the central composition plan can be presented as a spatial and contour plot of the response surface. For each of the output variables and for each type of mortar, a suitable response surface was set, which is shown in Figures 4-7.
Figure 4. Spatial and contour plot of response surface for flexural strength of epoxy mortar with PCM microfiber.

Figure 5. Spatial and contour plot of response surface for compressive strength of epoxy mortar with PCM microfiber.

Figure 6. Spatial and contour plot of response surface for flexural strength of modified cement mortar samples
Figure 7. Spatial and contour plot of response surface for compressive strength of samples of modified cement mortar samples.

The highest values of flexural strength for epoxy mortars were recorded in points 1 and 4 of the experiment plan, i.e. those with the highest aggregate content. On the basis of the data obtained, it can be concluded that the addition of phase change material as a microfiller only affects the flexural strength of B/M in the range of \(<0.5-0.6>\). PCM additive in cement mortars has a significantly greater influence on the parameter, but in order to obtain satisfactory results a suitable ratio of W/C greater than 0.4 should be used. The most favourable flexural strength results for cement mortars were noted for those points in the experiment plan where the PCM content was 11.716\% by vol. relative to the aggregate content.

Compressive strength values of epoxy mortar depend mainly on the ratio of binder to microfiller (B/M). From the graphs shown in Figure 2, it can be seen that the value of the measured parameter is improved as the B/M ratio increases. Therefore, to maintain high values of this strength, PCM microfibers should be added to the mortar at twice the amount of resin (B/M = 0.5). The spatial charts obtained for cement mortars after the flexural and compressive tests have a very similar shape (Figures 3 and 4). The highest values of compressive strength were obtained at point 2 of the experiment plan, i.e. at a W/C ratio of 0.4 and a PCM content of 11.716\% vol. The composition labelled in the experiment as 6 was also characterized by high compressive strength values while still containing the largest amount (23\%) of the phase change material. This fact influenced the selection of this composition for thermal research.

The temperature distribution of the analysed samples in the thermal test is shown in Figures 8-11. During the sample heating cycle, temperatures were recorded at five points. One of them registered the temperature in the climate chamber. Figure 8 shows the temperature distribution within a given time period inside the box for measuring point T_4, during the test of two the samples. At the maximum heating point, the sample with the phase change material was 1K lower than the sample without PCM. Approximately 12 hours after reaching the maximum temperature in both samples, a temperature drop occurred in the sample without PCM, while in the sample modified with the phase change material the temperature remained at 21°C until the end of the cycle.
On the surface of the epoxy layer (Figure 9), between the epoxy and the cement mortar (Figure 10) and the under the cement mortar (Figure 11) a similar temperature distribution was observed. The temperature difference in the above layers between the samples at the point of reaching the maximum temperature was 1K. During cooling and cycle stabilization, the maximum temperature difference between the samples was also approximately 1K.

Figure 8. Distribution of air temperature at the measuring point $T_4$ inside the box for both samples.

Figure 9. Distribution of temperature on the surface of the epoxy layer for both samples at measuring point $T_3$.

Figure 10. Distribution of temperature between the epoxy and cement layers for both samples at the measuring point $T_2$.

Figure 11. Distribution of temperature below the cement layer for both samples at the measurement point $T_1$.

4. Conclusions
This paper presents the effect of the addition of phase change material on selected properties of resin mortars, cement mortars and a composite composed of both types of mortars.

Based on the studies and statistical analyses of the results of the experiments, the following conclusions can be drawn:
- PCM additive has an influence on the strength properties of cement mortars, but the W/C ratio is predominant in this case. The best results were obtained with a PCM content of 12% and a W/C ratio of 0.45.
- In the case of epoxy mortar, PCM content was the main determinant of the attained strength values. Mortars were characterized by high flexural strength values from 13.58-23.92 MPa. The compressive strength results obtained were satisfactory (30.8-55.8 MPa), but significantly lower than for PCM-free mortar (97 MPa).
• When delivering heat to a sample containing PCM during laboratory testing, the ambient air temperature was lower compared to the air temperature surrounding the sample without PCM. This demonstrates the occurrence of the phenomenon of heat absorption by building material containing phase change material, which contributes to lower energy requirements for the cooling of utility interiors.

• The stored energy will be returned at a later time interval, as opposed to non-PCM materials, which at the same time have no capacity to return stored latent heat. This phenomenon can be used in times of increased demand for heat energy needed to heat utility interiors.

• The temperature difference at the measuring points $T_1$, $T_2$, $T_3$, $T_4$ of the tested samples was 1K. The obtained value indicates a low efficiency of thermal energy transfer between composite matrix and PCM. This fact can be explained by a comparable thermal conduction coefficient for PCM and resin composite at a level of 0.2 W/(mK). The research should be continued using modifier which improves heat transfer between the matrix and PCM.

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