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LABORATORY ASSESSMENT OF MICRO-ENCAPSULATED PHASE-CHANGE MATERIALS

LABORATORIJSKA OCENA MIKROENKAPSULIRANIH MATERIALOV S FAZNO PREMENO

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The operation of low-energy-consumption and passive houses can be based on the passive or active utilization of renewable energy sources. Thermal energy storage plays a key role in the application of renewable energy sources and it thus contributes to the reduction of global CO_2 emissions. Thermal energy storage is commonly based on the sensible- or latent-heat-storage techniques. Latent-heat thermal storage is based on the absorption or release of heat when a storage material is changing phase. The thermal-storage materials suitable for latent-heat storage are called Phase-Change Materials (PCMs). PCMs have considerably higher thermal-energy-storage densities than the sensible-heat-storage materials and they are able to absorb large quantities of energy in a small range of temperatures during the phase change. Nowadays, micro-encapsulation of phase-change materials is one of the promising approaches in the integration of latent-heat storage in various applications. The most important properties of a latent-heat-storage medium are the heat of the fusion and the temperature range of the phase change. The correct determination of the physical and chemical properties is essential for a practical use of phase-change materials. The paper deals with the results of a laboratory assessment of the selected micro-encapsulated PCMs and shows a practical example of a possible integration in the building structures.

Keywords: phase-change materials, latent heat, differential scanning calorimetry, micro-encapsulation

Delovanje nizkoenergijskih pasivnih hiš lahko temelji na uporabi aktivne ali pasivne uporabe obnovljivih energijskih virov. Shranjevanje toplotne energije igra ključno vlogo pri uporabi obnovljivih virov energije in zato vpliva na zmanjšanje globalne emisije CO_2. Shranjevanje toplotne energije navadno temelji na smiselnih tehnikah ali tehnikah shranjevanja latentne toplote. Shranjevanje latentne toplote temelji na absorpciji ali sproščanju toplote, ko material za shranjevanje spreminja fazo. Materiale, ki so primerni za shranjevanje latentne toplote, imenujemo Materiali s fazno premeno (PCM). PCM imajo občutno višjo gostoto shranjenje toplotne energije v primerjavi z občutljivimi materiali za shranjevanje toplote in so sposobni absorbirati med fazno premeno večjo količino energije v manjšem temperaturnem intervalu. Dandanes je vgradnja mikroenkapsulacijskih materialov s fazno premeno obetajoča za številne možnosti uporabe. Najpomembnejši lastnosti medija za shranjevanje latentne toplote sta talilna toplota in temperaturno področje fazne premene. Pravilno določanje fizikalnih in kemijskih lastnosti je bistveno za praktično uporabo materialov s fazno premeno. Ta članek obravnava laboratorijsko oceno izbranih PCM in kaže praktičen primer možnosti njihove uporabe v gradbeništvu.

Ključne besede: materiali s fazno premeno, latentna toplota, diferenčna dinamična kalorimetrija, mikroenkapsulacija

1 INTRODUCTION

Sensible-heat storage utilizes the heat capacity and the change in the temperature of a thermal-storage material during the process of charging and discharging the heat[1]. The amount of stored heat depends on the specific heat of the storage material, the temperature difference and the amount (mass) of the material.

Any building material can generally be used for sensible-heat thermal storage but the materials with high specific heat and high density usually perform the best. The typical representatives of sensible-heat-storage materials are common building materials such as ceramic bricks or blocs, concrete, lime-cement bricks and stone. The indoor environments with the envelopes made of such materials exhibit a much higher degree of thermal stability than the light-weight envelopes (e.g., timber-frame walls). The thermal storage in common building structures has its limits. The first limit is the use of heavy-weight structures. This is a very important constraint, especially in modern buildings. For example, glass-building envelopes would need to be supplemented with heavy-weight indoor structures and that is not always possible or desirable. This is where latent-heat storage can be employed.

1.1 Latent-heat storage

Latent-heat storage is based on the absorption or release of heat when a storage material undergoes a phase change from solid to liquid[1]. Such thermal-storage materials are called Phase-Change Materials (PCMs). They use chemical bonds to store and release heat[2]. PCMs have a high ability to store thermal energy. PCMs are able to absorb large quantities of heat in a small range of temperatures during a phase change. Latent-heat storage is one of the most efficient ways of storing thermal energy[3]. The selection of a PCM is mainly based on its melting temperature. A PCM's melting temperature should be within the operating temperature range of the thermal system. With respect to building use, it means within the thermal-comfort temperature range of an occupied space.

1.2 Selection of phase-change materials

Phase-change materials can be chosen from both organic and inorganic materials. The organic phase-change materials melt and freeze repeatedly without a phase-change segregation and crystallize with little or no supercooling. The organic phase-change materials, e.g., the paraffins, are compatible with metals without any risk of corrosion. The paraffins have a rather poor thermal conductivity and they are flammable. The melting point of the alkanes increases with an increased number of carbon atoms[1].

The inorganic PCMs are compatible with plastics and their storage capacity is higher than the capacity of the organic PCMs due to their higher density. The inorganic PCMs, e.g., salt hydrates, are incompatible with uncoated metals. The salt hydrates are important PCMs because of the high heat of fusion and a small volume change during the process of melting and solidification. The main disadvantages of salt hydrates are their poor nucleating properties that result in supercooling.

Suitable PCMs from both organic and inorganic groups are available for applications in the latent-heat-storage technology. Many phase-change materials cannot be used as latent-heat-storage mediums because of the problems with their chemical stability, toxicity, corrosion, volume change and price. The phase-change materials should meet the following thermodynamic, kinetic, chemical and economic criteria[4].

Thermodynamic criteria:
- high heat of fusion;
- melting range in the desired operating-temperature range;
- high specific heat;
- high thermal conductivity;
- high density and low volume change;
- congruent melting.

Kinetic criteria:
- little or no supercooling during the solidification process;
- sufficient crystallization rate.

Chemical criteria:
- compatibility with the container;
- long-term chemical stability;
- no toxicity;
- no flammability.

Economic criteria:
- availability in the required quantities;
- low cost.

2 MATERIALS AND METHODS

The research and development at the Brno University of Technology is focused on the utilization of the latent-heat storage in passive and active solar-heating and cooling technologies. The development of the advanced latent-heat-storage technologies is strongly dependent on the possibility to find suitable phase-change materials that fulfill the above-mentioned requirements. The second problem lies in finding a suitable technology for an integration of latent-heat-storage media in building structures.

Micro-encapsulation is one of the possible approaches to the PCM integration in building structures[5].

2.1 Micro-encapsulated PCMs

Micro-encapsulation is based on enclosing a PCM in a very small capsule. The micro-capsules can be included in the common building materials and structures. Special attention has to be paid to the choice of the material of the capsule to avoid a chemical reaction between the capsules and the building material[5]. Micro-capsules can be added to the composition of lime or gypsum plaster, concrete, fibrous wooden slabs and gypsum wall boards.

A special composition of gypsum plaster and micro-encapsulated PCMs was developed for the application in building structures. The gypsum plaster contains 30 % of micro-capsules Micronal DS 5008 X. The plaster is the final layer of the walls and ceilings in the buildings with a low thermal mass.

2.2 Differential scanning calorimetry

Differential Scanning Calorimetry (DSC) is a thermo-analytical technique where a temperature range is scanned. The difference between the amount of the heat required for changing the temperature of a sample and its reference is measured as a function of temperature. Both the sample and the reference are maintained at nearly the same temperature throughout the experiment. The reference is used to determine the heat stored in the sample by considering the difference between the signal of the sample and the reference[6].

The DSC heat flux has got a Siamese structure[5]. The sample and the reference are connected to the same metal disc. The behavior difference between the sample and the reference submitted to the same temperature excitation leads to a voltage difference between the sample and the reference. The absorbed heat in the PCM sample is deduced from the voltage[5]. The weight of the sample is only a few grams. A calorimeter PYRIS1 Perkin Elmer was used in the tests.

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3 RESULTS AND DISCUSSION

The experiments focused on determining the thermal properties of a micro-encapsulated PCM and the plaster containing 30 % of a PCM.

Figure 1 shows the results after two heating/cooling cycles for the micro-capsules with a PCM. The results were obtained with the continuous scanning mode at the rate of 1 °C/min. This rate is rather quick compared to common conditions in rooms. The black and blue curves represent heating, while the red and green curves show the results of the cooling mode. There is some supercooling in the cooling phase that is not really problematic for a practical application in building structures. The presence of supercooling plays a role in discharging the heat stored in the PCM.

As can be seen, the PCM displays two heat-flow peaks. The first peak is well below the comfortable indoor air temperature, thus, it has no implications for the practical use. **Table 1** shows the peak temperatures for both cycles. The difference between the peaks of the cooling and heating phases is about 2 °C. **Table 2** shows the heat of fusions for the heating and cooling phases.

Table 1: Peak temperatures for the PCM
Tabela 1: Maksimalne temperature za PCM

Cycle	Peak temperature T/°C	
	cooling	heating
I	22.32	24.32
II	22.40	24.20

Table 2: Heat of fusions for the PCM
Tabela 2: Talilna toplota za PCM

Cycle	Heat of fusion in J/g	
	cooling	heating
I	–84.16	86.83
II	–81.29	86.76

Thermal stability of PCMs was determined with a thermogravimetric apparatus (TGA) Q500 TA Instruments. The results from TGA are shown in **Figure 2**. Significant weight losses start at 140 °C, which is above the commonly used temperature range. The temperature range of the indoor climate between 15 °C and 35 °C can be assumed for building applications. **Figure 3** shows DSC results for a gypsum plaster with 30 % of Micronal D5008 X. The chart shows a significant reduction of the latent heat during the heating and cooling processes. Three temperature rates of (1, 10 and 20) °C/min were tested. As can be seen, the onset and the peak temperatures during the heating and cooling strongly depend on the temperature ramp. Melting temperatures rise with an increased heating rate. A shift of the solidification-temperature range follows an increased rate of cooling. The risk of supercooling increases with a faster cooling rate.

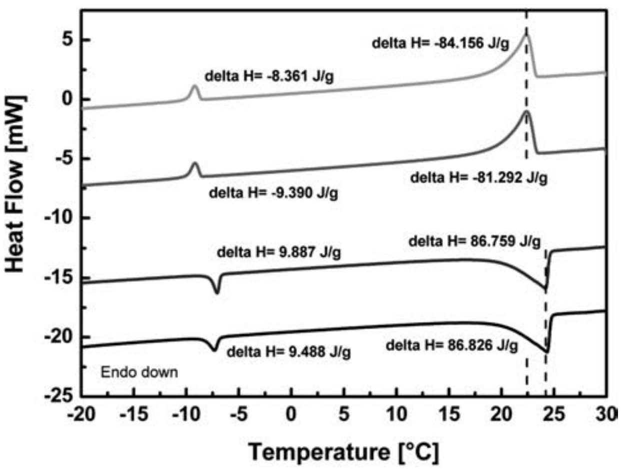

Figure 1: DSC results for the PCM
Slika 1: Rezultati DSC za PCM

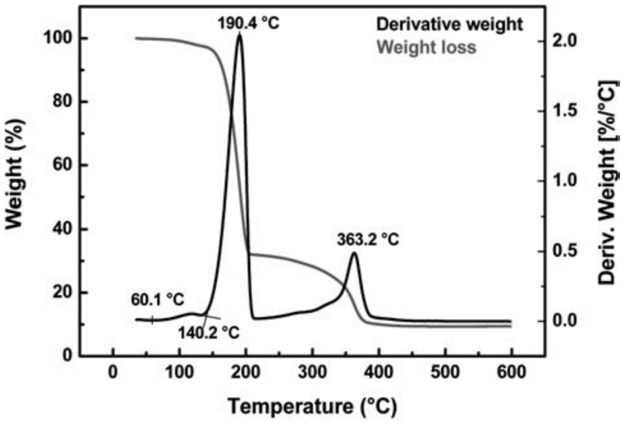

Figure 2: Thermal stability of the PCM
Slika 2: Toplotna stabilnost PCM

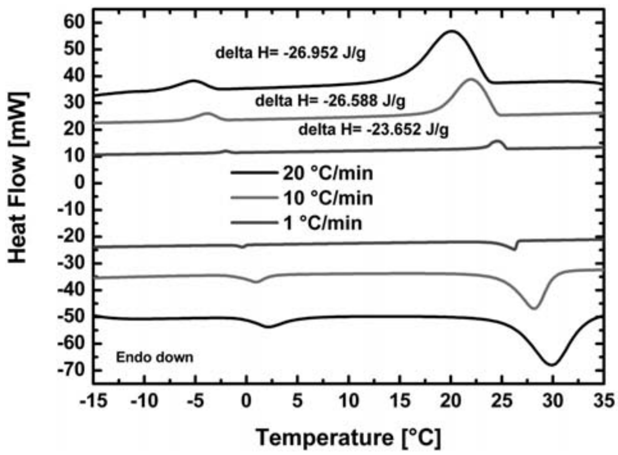

Figure 3: DSC results for gypsum plaster
Slika 3: Rezultati DSC za mavčni omet

4 CONCLUSION

The results obtained with DSC confirm suitability of the tested PCMs and the plaster for their integration in building structures. The peak temperature during the heating is about 24 °C. The micro-encapsulated PCMs have a required melting range for the thermal-energy storage during the summer season. The difference between the peak-melting and solidification temperatures of a PCM is about 2 °C allowing a proper, natural or driven, regeneration of the heat-storage medium at night.

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