Energy Conversion and Management 64 (2012) 516–521

Contents lists available at SciVerse ScienceDirect

Energy Conversion and Management

journal homepage: www.elsevier.com/locate/enconman

Performance evaluation of the microencapsulated PCM for wood-based flooring application

Su-Gwang Jeong, Jisoo Jeon, Jungki Seo, Jeong-Hun Lee, Sumin Kim *

Building Environment & Materials Lab, School of Architecture, Soongsil University, Seoul 156-743, Republic of Korea

ARTICLE INFO

Article history:
Received 5 January 2012
Received in revised form 14 March 2012
Accepted 16 March 2012
Available online 20 April 2012

Keywords:
MPCM
Adhesive
Energy saving
Heat storage
Wood-based flooring

ABSTRACT

Thermal energy storage (TES) systems using microencapsulated phase change material (MPCM) have been recognized as one of the most advanced energy technologies in enhancing the energy efficiency and sustainability of buildings. In this research, we examined a way to incorporate MPCMs with building materials through application for wood-based flooring. Wood-based flooring is commonly used for floor finish materials of residential buildings in Korea. There are three types of wood flooring: laminate flooring, engineered flooring and solid wood flooring. However, wood-based flooring has not performed the characteristic of heat storage. This study is aimed at manufacturing high thermal efficiency wood flooring by increasing its heat storage using MPCM. To increase the heat storage of wood-based flooring, MPCM was used with adhesive for surface bonding of wood-based flooring. As a result, this study confirmed that MPCM is dispersed well in adhesive through the scanning electron microscopy (SEM) analysis. From the differential scanning calorimetry (DSC) analysis, it can be confirmed that this composite has the characteristic of a thermal energy storage material. Also, we analyzed how this composition was formed by physical combination through the Fourier transform infrared (FT-IR) analysis. Also, we confirmed the bonding strength of the material by using the universal testing machine (UTM).

1. Introduction

Recently, the reduction of energy consumption is an important factor for residential buildings around the world [1]. Furthermore, thermal energy storage systems are essential for reducing the dependency on fossil fuels and then contributing to a more efficient environmentally friendly energy use [2]. As the demand for thermal comfort of buildings increasingly rises, the energy consumption is also increasing correspondingly in both domestic and residential buildings. Currently, the number of residential buildings using a radiant flooring heating system is increasing globally due to its thermal comfort. Comfort heating depends on the mean effective temperature, which is a function of the ambient air and the radiant surface temperatures. Therefore, it is possible to achieve comfortable conditions with a cooler air temperature by a large floor-heating surface, which results in reduced infiltration heat losses [3]. Differences between head and feet temperatures are small. Because the air temperature is not greater than that of the walls, no condensation takes places on the walls and there is no need to humidify the air in the winter. Differences in air temperatures are minimal and air convection appears to be negligible and the movement of dust is therefore diminished. Therefore, radiant floor heating systems are cleaner than more conventional heating systems [4]. The radiant floor heating system is created by radiant heat which is created by hot water or steam flowing in the buried pipe under the floor. The increasing use of radiant floor heating systems is due to various peculiar advantages offered by these systems, such as the possibility of employing low temperature water and the absence of terminal heaters in the heated environment such as fan-coils that could cause particulate movement and reduce the air quality, usually required in some important environments such as hospitals [5]. Thermal mass integrated into a floor heating system can be used for off-peak storage of thermal energy. Thus, peak loads may be reduced and shifted to nighttime when electricity costs are lower [6]. The combination of radiant floor heating and thermal storage offers the possibility of significantly improving building energy efficiency without compromising thermal comfort [7]. Wood flooring is mainly used in the radiant floor heating system because it is harder, more durable and has more fire stability than general flooring. Also, a wood floor is appealing as it has an aesthetically good appearance for satisfying the needs of the consumer. There are three types of wood floor including solid wood floor, laminate floor and plywood floor. Epoxy adhesives are used when these wood floors are installed in a building. These wood floors are applied to the radiant floor heating system such as the ondol system, and many studies have been carried out globally on this. Also, these studies have confirmed that radiant floor heating systems have high efficiency. However, the thermal

* Corresponding author. Tel.: +82 2 820 0665; fax: +82 2 816 3354.
E-mail address: skim@ssu.ac.kr (S. Kim).

0196-8904/$ - see front matter © 2012 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.enconman.2012.03.007

performance of these wood-based floor heating systems needs to improve because they consist of a wood floor and epoxy adhesive which have no characteristic of thermal storage.

Phase change materials (PCMs) can provide large latent heat storage over the narrow range of temperature typically encountered in buildings; thus they can improve the degree of thermal comfort. PCM was applied in many fields such as radiant floor heating systems, solar energy systems, central airconditioning systems, energy efficient buildings and industrial waste heat recovery, due to their high energy storage density and small temperature variation from storage to retrieval. In a latent heat storage system, energy is stored during melting and recovered during freezing of PCM. It performs an important role in solving the energy imbalance, improving the energy efficiency and protecting the environment [8–10]. PCM have been considered for thermal energy storage in buildings since 1980. By implementing the PCM in gypsum boards, plaster, concrete or other wall covering materials, thermal energy storage becomes part of the building structure, useful even for light-weight buildings. In the literature, development and testing were conducted for prototypes of PCM wallboard and PCM concrete systems, with particular interest in peak load shifting and solar energy utilization. During the last 20 years, new PCM products to be used in buildings appeared on the market [1]. However, the application of PCM as itself for building is difficult due to its phase instability of liquid state. Therefore, PCMs usually need to be encapsulated in shell materials such as melamine and SiO_2 In recent years, microencapsulated phase change materials [11,12] have been studied to solve the liquid leakage problems in the solid–liquid phase change process. Various methods have been developed for the encapsulation of PCMs, such as complex coacervation [13], interfacial polycondensation [14] and in situ polymerization [15]. A typical microencapsulation process uses formaldehyde resins for the protection of PCMs, such as melamine–formaldehyde resins [16–18] and urea–formaldehyde resins [19–21]. Microencapsulated PCM provides some important advantages such as the ability of microcapsules to handle phase change materials as a core, as they tolerate volume changes; there is a reduction of the reactivity of the paraffin wax with the outside environment; it increases the heat-transfer area and the handling and manipulation of paraffins becomes easier. In this paper, a performance evaluation was carried out of the composite epoxy adhesive with MPCM for application of a wood-based floor heating system in buildings. Also, this paper confirmed that the MPCM/wood-based flooring construction adhesive composite (MWAC) can improve the thermal performance in its application fields.

Therefore this paper handles that enhancing thermal properties of the construction adhesive without heat storage properties. Also we evaluated the applicability of MWAC for applying wood-based flooring using MPCM through measurement of bonding strength from universal testing machine (UTM) analysis. And we confirmed that MWAC has enough bonding strength to apply wood-based flooring as adhesive with heat storage performance. Also, although MWAC has poor heat storage capacity, it could be a good material for applying building material sector.

2. Experimental

2.1. Materials

The MPCM with a melting point of 27–31 °C and latent heat capacity of 66.61 kJ/kg was obtained from the Celsius Korea company in South Korea. Also, the MPCM was prepared through the Celsius Korea (http://www.celsius.co.kr) by using RT31 as the core material from Rubitherm corporation (http://www.rubitherm.de) and using melamine as the shell material. The epoxy resin was purchased from SamChang Tech Co. Ltd. (http://www.samchang-tech.co.kr/) in South Korea. The epoxy resin consists of a base with a hardener, and the mixture ratio is 1:1.

2.2. Preparation of MWAC

The preparation process of MPCM/wood-based flooring construction adhesive composite (MWAC) is as follows: First, the MPCM, which was dried for 5 h, is prepared for the beaker. The epoxy resin and hardener are then mixed into another beaker, and the mixture ratio is 1:1. Then, 3 wt%, 5 wt%, 7 wt% and 10 wt% of the MPCM is added to the mixture. This mixture was continuously stirred at a rate of 1000 rpm for 10 min with a shearing stirrer at room temperature. Table 1 shows the contents of the mass ratio of MWAC. This MWAC is then cured in the mold for 2 days. Fig. 1 shows the schematic of the preparation process of the MWAC.

2.3. Methods

To confirm the thermal and mechanical properties of MWAC, we have been carried out scanning electron microscopy (SEM, JEOL JSM-6360A), Fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC) and UTM measurements. The morphology and composition of the MPCM and MWAC at different loadings were observed by means of scanning electron microscopy at room temperature. An SEM with an accelerating voltage of 12 kV and working distance of 12 mm was used to collect the SEM images. The samples were coated with a gold coating of a few nanometers in thickness [23]. And FTIR spectroscopy (300E Jasco) was also utilized to monitor the change of chemical groups upon curing. Clear potassium bromide (KBr) disks were molded from the powder and a pure KBr disk was used as the background. The samples were analyzed over the range of 525–4000 cm^{-1} with a spectrum resolution of 4 cm^{-1}. All spectra were averaged over 32 scans. This analysis of the composites was performed at point-to-point contact with a pressure device [22]. Thermal properties such as the melting temperature and latent heat capacity of MWAC were measured using a DSC instrument (DSC Q 1000). DSC measurements were performed at a 5 °C/min heating and cooling rate and a temperature range of 10–50 °C and 50–10 °C. The latent heat capacity is not depend on a heating and a cooling rate. And a heating and a cooling rate has an effect on the absolute value of *x*-axis. In this paper, we focused to find the latent heat capacity of MPCM. So we had chosen a heating and a cooling rate of 5 °C/min in the measurements with the DSC. The melting temperature was measured by drawing a line at the point of maximum slope of the leading edge of the peak and extrapolating to the base line. The latent heat of the MWAC was determined as total by numerical integration of the area under the peaks that represent the solid–solid and solid–liquid phase transition. Also the bonding strength of MWAC was examined by means of the universal testing machine (Zwick Co.) according to ASTM standards D638 and D790. The crosshead speed of tensile testing was 5 mm min^{-1} with the gage length of 63 mm of a dumbbell shape. All tests were performed

Table 1
Contents mass ratio of epoxy resin, hardener and MPCM for preparation of MWAC.

wt% of MPCM	Epoxy resin (g)	Hardener (g)	MPCM (g)
0	50	50	0
3	48.5	48.5	3
5	47.5	47.5	5
7	46.5	46.5	7
10	45	45	10

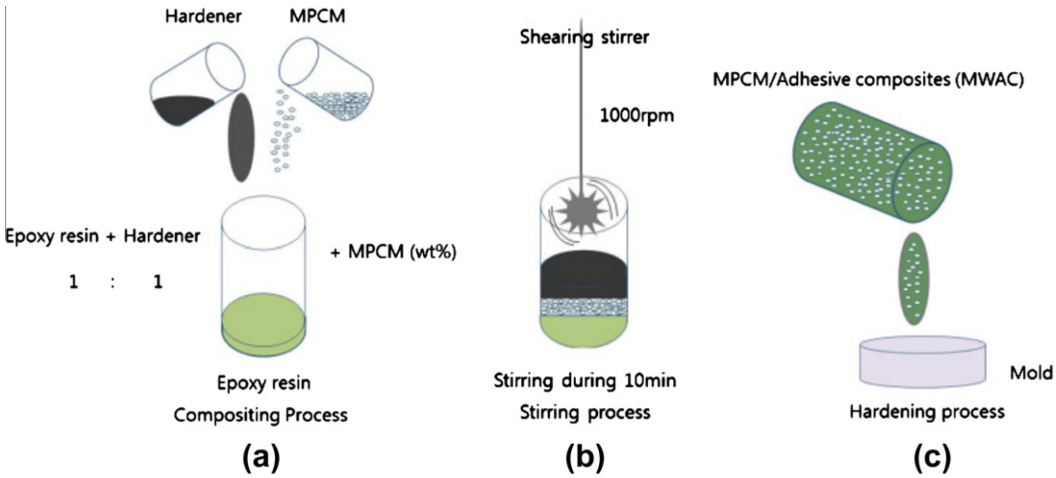

Fig. 1. A schematic of preparation process for MWAC: (a) compositing process, (b) stirring process, (c) hardening process.

for at least five specimens and the data was taken into account of the average values.

3. Results and discussion

3.1. Morphology analysis

Fig. 2 shows the morphology of the MWAC after hardening. The MWAC, of which from 0 wt% to 5 wt% MPCM is added, shows a spherical form similar to the shape of the mold, but MWAC exceeding 7 wt% of MPCM does not show a form similar to the shape of the mold, because the viscosity of the MWAC increases as the wt% of the powder form of MPCM increases. Also, this caused a reduction of the bonding strength of the MWAC. This can adversely affect the workability for the installation of the wood floor and it can also reduce the life of the radiant floor heating system and reduce the life of the wood floor. Finally, it negatively affects the reduction of the building energy. Therefore, an additional study on the reduction of workability as wt% of the MPCM increases is needed for high thermal efficiency of the radiant floor heating system.

3.2. Analysis on microstructure of MWAC

The cryogenically fractured surface of the MWAC was studied by SEM. Fig. 3 shows the SEM photographs of the MWAC of 3%, 7% and 10% MPCM loading contents. Fig. 3a shows the micro structure of the adhesive but it does not show the spherical micro particles of MPCM. However, Fig. 3b–d shows that the dispersions of the MPCM in the adhesive are uniform. The spherical micro particle of MPCM was well-dispersed in the adhesive [22]. Also, this study confirmed that the spherical micro particle of MPCM was not shattered during the stirring process, and is composed of a physical combination rather than a chemical combination. In this SEM analysis, we checked existence of the spherical micro particles and it seems to be MPCM particles. So we considered that the particles were not shattered because of conservation of its shape. Of course, maybe some particles are supposed to be shattered, however we confirmed that the most of all particles are not shattered because latent heat peaks were increased as increasing MPCM loading contents from DSC analysis. And the numbers of spherical micro particles of MPCM increased as the MPCM loading contents increased due to good dispersion. In this respect, the thermal performance of MWAC in which MPCM was well dispersed in the adhesive structure is revealed for the application of a radiant floor heating system.

3.3. FT-IR analysis

FTIR results on the MWAC are shown in Fig. 4. As the MPCM uses melamine as the shell material, we checked the melamine peaks from the FTIR spectra. Melamine has a molecular formula of $C_6H_2N_6$, and it is relevant to molecular structural 1,3,5-triazine-2,4,6-triamine, and it is also used widely due to the superior thermal resistance, water proofing and mechanical strength. The 3369 cm^{-1} and 3298 cm^{-1} of the peak appeared on the FTIR spectra due to the NH_2 group of melamine. Also, this peak increased as the MPCM load contents increased. Also, the N–CH_3 combination peak was shown on the FTIR spectra, due to the combination of the CH_3 group of the core material which consists of paraffin. The N–CH_3 peak is shown as 2775–2765 cm^{-1}. These numerical peaks are shown in Table 2. And we checked that FTIR peaks of the adhesive are maintained after mixing with MPCM. Also increasing peaks in the FTIR graph show the N–CH_3 and NH_2 group

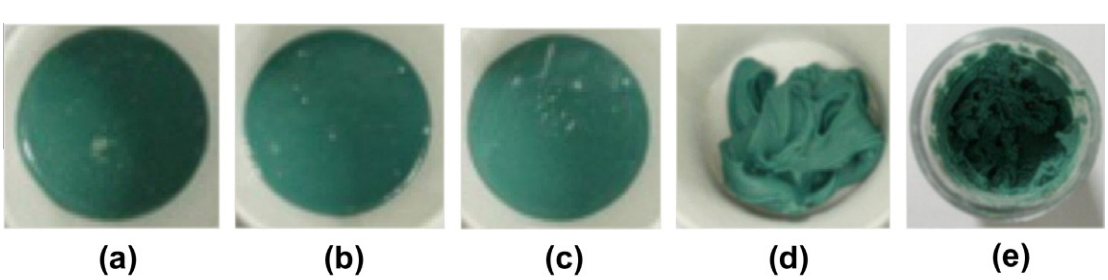

Fig. 2. The morphologies of MWAC after hardening: (a) adhesive, and the MWAC with (b) 3 wt%, (c) 5 wt%, (d) 7 wt%, (e) 10 wt% of loading contents of MPCM.

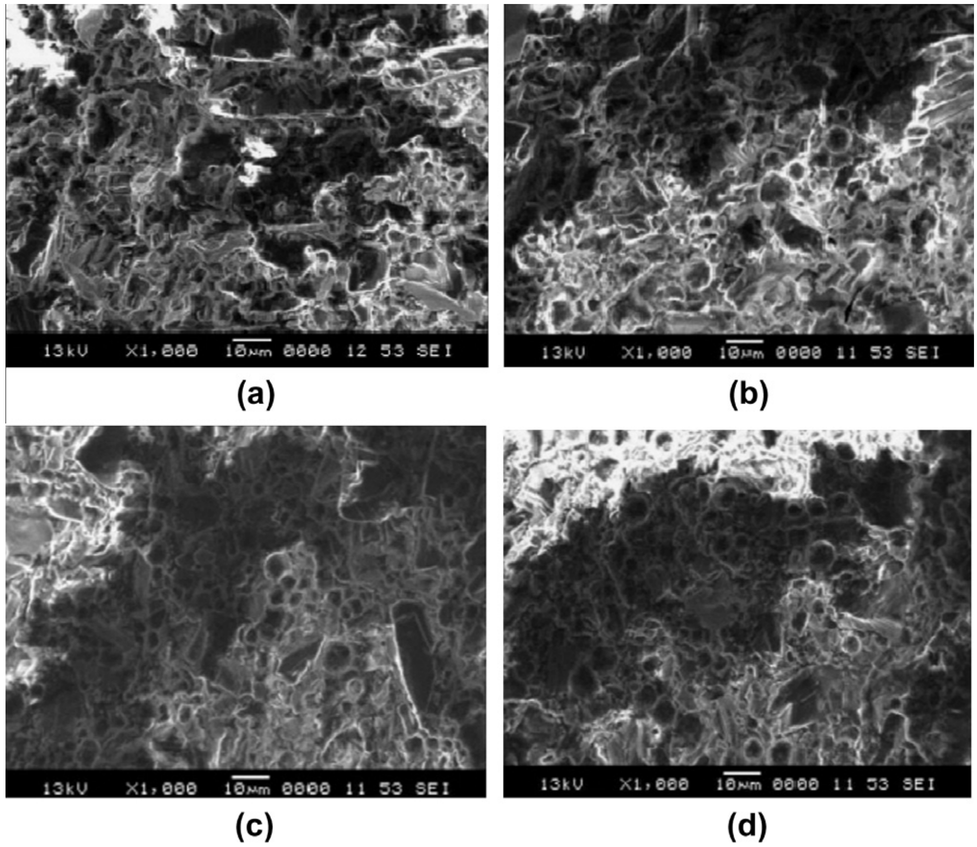

Fig. 3. Microstructure of the adhesive and MWAC: (a) adhesive, and MWAC with (b) 3 wt%, (c) 7 wt%, (d) 10 wt% of loading contents of MPCM.

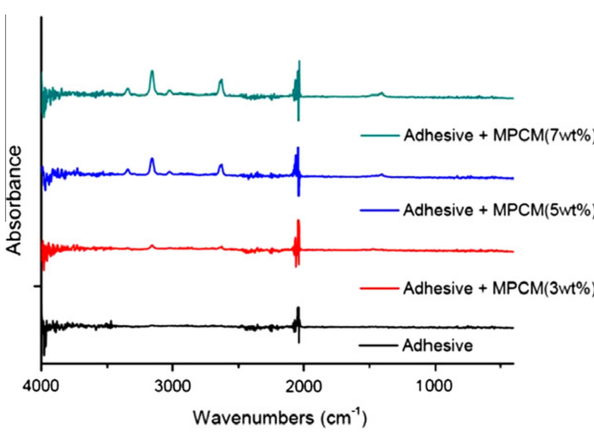

Fig. 4. FT-IR spectra of adhesive and MWAC.

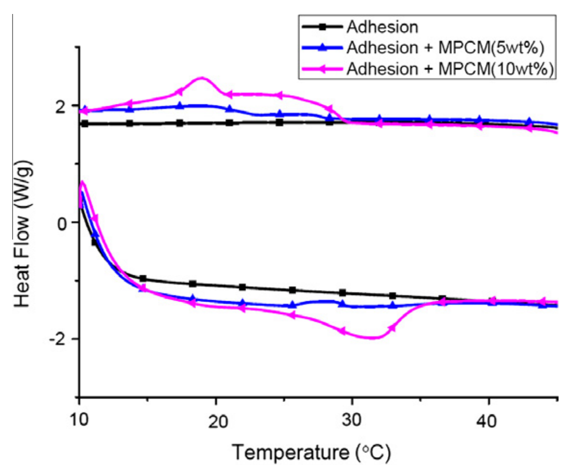

Fig. 5. DSC graph of the adhesion and MWAC with 5 wt%, 10 wt% of MPCM.

Table 2
The FTIR spectra of the amin group and N–CH_3 bonding.

Vibration	Wavenumber range (cm^{-1})
Asymmetric NH_2 stretch	3369
Symmetric NH_2 stretch	3298
N–$(CH_3)_2$ bonding	2775–2765

and it is MPCM peaks which were consist of melamine as shell material. In this results show the peaks of shell material and adhesive are not changed. So I judged that MWAC was composed of a physical combination rather than a chemical combination.

3.4. Analysis of thermal characteristic of MWAC

In the case of the combination of epoxy resin and hardener, MPCM has no properties of heat storage. Therefore, the composition of MPCM and adhesion was carried out in this paper to provide the characteristic of heat storage. The MPCM used in the experiment has 66.61 kJ/kg of heat storage capacity and the phase change temperature range is 27–31 °C. The heat performance of the adhesive and 5 wt% and 10 wt% of MPCM are analyzed by DSC measurement to carry out the heating and cooling process. At this point, we found the differences between the melting temperature and the solidification temperature. It is caused by supercooling during

Table 3
Heat storage capacity of the adhesive and MWAC with 5 wt%, 10 wt% of MPCM.

wt% of MPCM	Heating (kJ/kg)	Cooling (kJ/kg)	Calculated (kJ/kg)
0	0	0	0
5	3.278	3.230	3.331
10	6.220	6.130	6.661

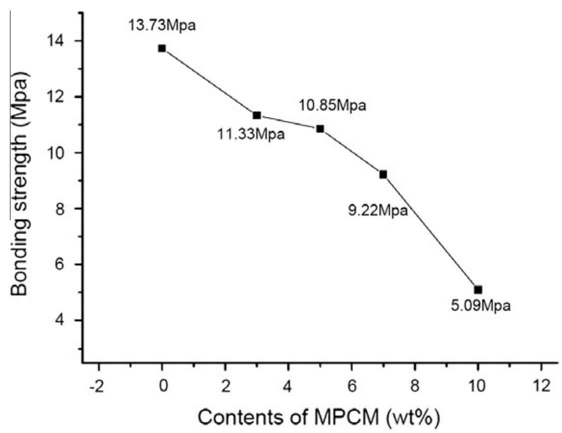

Fig. 6. The bonding strength of the adhesive and MWAC with increased loading contents of MPCM.

solidification. So, melting temperature is higher than solidification temperature in DSC analysis. As shown in Fig. 5, we confirmed that the adhesive has no DSC peak because the adhesive has no characteristic of heat storage. However, as MPCM loading contents increase in the adhesive structure, the DSC peaks also increase. The latent heat of the paraffin is obtained as the total area under the peaks of the solid–solid and solid–liquid transitions of the MPCM in the composite by numerical integration. In the case of the latent heat of 5 wt% of MPCM in MWAC, the 3.278 kJ/kg and 3.230 kJ/kg were measured by heating and cooling. Also, in the case of 10 wt% of MPCM in MWAC, the 6.220 kJ/kg and 6.103 kJ/kg were measured by heating and cooling. These results are similar to those of the calculated latent heat. Also, the numerical DSC peaks are shown in Table 3. Therefore, we confirmed that the performance of the heat storage capacity is increased when MPCP loading contents in MWAC increase. As expected, we confirm that the adhesive with performance of heat storage is useful for application in a wood-based heating system.

3.5. Analysis of bonding strength of MWAC

To determine the bonding strength between the adhesive and MWAC, we performed a test with a universal testing machine (Zwick Co.). Fig. 6 shows the change of the bonding strength with increasing loading contents of the MW. The bonding strength of adhesive is 13.73 MPa as seen in Fig. 6. However, the bonding strength of MWAC is reduced by increasing the loading contents of the MPCM. In the case of the 3 wt% and 5 wt%, these bonding strengths indicate the 11.33 MPa and 10.85 MPa, respectively. This shows that there are no significant differences of bonding strength compared to the adhesive. However the bonding strength strongly decreased as from beyond the 7 wt% of loading contents of MPCM. Also, the bonding strength of the MWAC which has 10 wt% of MPCM did not attain half of the adhesive. The reduction of bonding strength caused the reduction of the durability of wood flooring. And we estimate that this research will be supplied as reference and over the 7 wt% of MPCM loading contents bring decrease of bonding strength drastically. Also we confirmed that the bonding strength of MWAC which has 10 wt% of MPCM loading contents is 5 MPa. It is more than 0.8 MPa which is the minimum adhesive bonding strength in the Korea Housing corporation standard to apply wood flooring.

4. Conclusions

Recently, many studies have been carried out on the reduction of energy consumption. We applied the PCM to wood-based floor for the reduction of energy saving to improve the thermal efficiency through latent heat storage. In this study, the properties of MWAC that had been used in the test were measured and an MWAC was made by stirring the epoxy resin, hardener and MPCM, having a latent heat storage performance, with MPCM based on mass ratio. In order to check the micro structure and chemical reaction of the MWAC between the MPCM and adhesive, SEM and FTIR tests were performed. A DSC analysis and UTM analysis were also performed to measure the heat storage and bonding strength of the MWAC. According to the morphology test results, the MWAC with over 7 wt% of loading contents of MPCM did not show a form similar to the shape of the mold, because the viscosity of the MWAC is high. Also, we confirmed that the spherical micro particle of MPCM was well-dispersed in the adhesive from SEM analysis. Furthermore, we checked the properties of heat storage revealed in MWAC through the DSC analysis. According to the FTIR test results, the MWAC consists of the MPCM and adhesive by physical reaction not chemical reaction. Finally, the increasing of loading contents of MPCM caused the reduction of bonding strength.

Actually, MWAC was consists of maximum 10 wt% of MPCM, but it has a poor latent heat capacity. However we confirmed that MWAC has heat storage properties which are not existence to adhesive. Also we checked that MWAC with 10 wt% of MPCM could apply to wood-based flooring as adhesive because its bonding strength is strong enough as 5 MPa. A minimum adhesive bonding strength is 0.8 MPa according to the Korea Housing corporation standard to apply wood-flooring. Therefore if MWAC could get minimum workability, MWAC with 10 wt% of MPCM loading contents could be a good material as adhesive has with heat storage properties.

Acknowledgements

This research was supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (No. 2011-0004181).

References

[1] Castellón C, Medrano M, Roca J, Cabeza LF, Navarro ME, Fernández AI, et al. Effect of microencapsulated phase change material in sandwich panels. Renew Energy 2010;35:2370–4.
[2] Tyagi VV, Kaushik SC, Tyagi SK, Akiyama T. Development of phase change materials based microencapsulated technology for buildings: a review. Renew Sustain Energy Rev 2011;15:1373–91.
[3] Athienitis AK, Chen TY. Experimental and theoretical investigation of floor heating with thermal storage. ASHRAE Trans 1993;99(1):1049–57.
[4] Lafontaine LH. Radiant heating and cooling. Heat Piping Air Cond 1990;62(3):71–8.
[5] Fontana L. Thermal performance of radiant heating floors in furnished enclosed spaces. Appl Therm Eng 2011;31(10):1547–55.
[6] Braun J. Reducing energy costs and peak electrical demand through optimal control of building thermal storage. ASHRAE Trans 1990;96(2):876–88.
[7] Lin K, Zhang Y, Xu X, Di H, Yang R, Qin P. Experimental study of under-floor electric heating system with shape-stabilized PCM plates. Energy Build 2005;37:215–20.
[8] Wang X, Zhang YP, Xiao W, Zeng RL, Zhang QL, Di HF. Review on thermal performance of phase change energy storage building envelope. J China Sci Bull 2009;54:920–8.

[9] Tomlinson JJ, Kannberg LD. Thermal-energy storage. J Mech Eng 1990;112:68–72.
[10] Tyagi VV, Buddhi D. PCM thermal storage in buildings: a state of art. Renew Sustain Energy Rev 2007;11:1146–66.
[11] Cho JS, Kwon A, Cho CG. Microencapsulation of octadecane as a phase-change material by interfacial polymerization in an emulsion system. J Colloid Polym Sci 2002;280:260–6.
[12] Schossig P, Henning H, Gschwander S, Haussmann T. Micro-encapsulated phase-change materials integrated into construction materials. Solar Energy Mater Solar Cell 2005;89:297–306.
[13] Rong Y, Chen H, Wei D, Sun J, Wang M. Microcapsules with compact membrane structure from gelatin and styrene–maleic anhydride copolymer by complex coacervation. Colloids Surf Physicochem Eng Asp 2004;242:17–20.
[14] Hong K, Park S. Preparation of polyurea microcapsules with different composition ratios: structures and thermal properties. Mater Sci Eng A 1999;272:418–21.
[15] Zhang XX, Fan YF, Tao XM, Yick KL. Fabrication and properties of microcapsules and nanocapsules containing *n*-octadecane. Mater Chem Phys 2004;88:300–7.
[16] Su J, Wang L, Ren L. Fabrication and thermal properties of microPCMs: used melamine–formaldehyde resin as shell material. J Appl Polym Sci 2006;101:1522–8.
[17] Sun G, Zhang Z. Mechanical properties of melamine–formaldehyde microcap sules. J Microencap 2001;18(5):593–602.
[18] Li W, Zhang X, Wang X, Niu J. Preparation and characterization of microencapsulated phase change material with low remnant formaldehyde content. Mater Chem Phys 2007;106:437–42.
[19] Sarier N, Onder E. The manufacture of microencapsulated phase change materials suitable for the design of thermally enhanced fabrics. Thermochim Acta 2007;452:149–60.
[20] Fang G, Li H, Yang F, Liu X, Wu S. Preparation and characterization of nano-encapsulated *n*-tetradecane as phase change material for thermal energy storage. Chem Eng J 2009;153:217–21.
[21] Wang JP, Zhao XP, Guo HL. Preparation of microcapsules containing two phase core materials. Langmuir 2004;20:10845–50.
[22] Kim S, Drzal LT. High latent heat storage and high thermal conductive phase change materials using exfoliated graphite nanoplatelets. Solar Energy Mater Solar Cell 2009;93:136–42.
[23] Kim H, Lee B, Choi S, Kim S, Kim H. The effect of types of maleic anhydride-grafted polypropylene (MAPP) on the interfacial adhesion properties of bio-flour-filled polypropylene composites. Compos A Appl Sci Manuf 2007;38:1473–82.