



Thermal insulation capability of PEG-containing polyurethane foams

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ABSTRACT

Polyethylene glycol (PEG) compounds and mixtures have many properties that make them suitable for thermal applications in buildings, such as having high heat of fusion, phase change repeatability, chemical stability, non-corrosive behavior, and low-cost. In this study, we developed a number of PU rigid foams incorporated with three types of PEGs, as new insulation materials provided with an enhanced thermal capacity, and sought their suitability for various applications such as layer of floor and ceiling coverings in constructions, insulations in controlled temperature transportation packaging, inner coverings of automobile seats, etc. In order to investigate the thermal properties of PEG-containing PU foams, differential scanning calorimeter (DSC) tests were conducted first. Then, a two-layer concrete–PU foam system was designed in the laboratory conditions to examine the insulation performances via using a computer-aided thermal measurement setup which was sensitive to the simulated environmental temperature changes. The PU–PEG composites produced here can be helpful for the design of thermal insulators. PUI, including 44% PEG 600, exhibited fairly efficient thermal regulation under moderate ambient temperature conditions, whereas PUII (49% PEG 1000) is suitable for temperature control in both mild and hot surroundings. PUIII, containing 53% PEG 1500, showed suitable heat storage and thermal stability characteristics. PUIV, containing 38% PEG 600/PEG 1000/PEG 1500, also confirmed good thermal and durability characteristics. The blend of three PEGs is suitable for preventing discontinuous thermal regulation when the external temperature increases or decreases. PU foams containing PEGs can be assumed to be leak-resistant, which is promising for their industrial applications.

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1. Introduction

On a worldwide basis, energy utilization is predicted to increase by 71% from 2003 to 2030 [1]. Energy resources are expected to be used much more efficiently to support this expansion in energy consumption for the next 20–25 years. In this context, number of attempts has been rising in a great diversity.

Energy requirements in industrial, commercial and residential sectors are, mainly for space heating and cooling in buildings, comprising approximately 60% of services [2]. Therefore, thermally competent building designs have become increasingly important for energy saving. Such approaches also help to improve thermal comfort conditions for residents and to protect building materials from constant fluctuations in outdoor climatic conditions [3].

Commercially, polyurethane (PU) rigid foams have been widely used for thermal insulation as excellent energy savers. They exhibit highly favorable characteristics. The air trapped within the

honeycomb-like structure confers quite low thermal conductivity. They offer a good mechanical and chemical stability and are suitable to form sandwich structures with various facer materials. If some heat storage materials are additionally incorporated into PU foams used in buildings, heat loss to environment or heat gain from surrounding will be reduced further and energy saving will be much more efficient despite of the large variations in outdoor temperature conditions [4].

Phase change materials (PCMs), which are capable of absorbing or releasing large amount of heat during phase transitions between two solid states and/or solid and liquid states, have become new components of composite materials in thermal isolation applications. They can provide an enhanced thermal capacity in addition to the existing passive insulation characteristic of the structure. In building applications, the phase transition temperatures of PCMs lie generally within a range from -10°C to 50°C . By selecting two or more different PCMs and forming a mixture thereof, phase transition temperature range can be widened for any desired application [5].

PCMs' use in buildings is limited to their direct applications in ventilation ducts or in gypsum board, plaster, concrete or other wall coverings. It has been reported that PCM wallboards could save up

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to 20% of the costs of residential space conditioning, emphasizing the importance of PCMs' contribution to the energy utilization [6,7].

PCMs investigated for the design of construction materials are primarily hydrated salts, e.g. $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, and paraffin waxes, $\text{CH}_3-(\text{CH}_2)_n-\text{CH}_3$. However, there are some limitations to the use of hydrated salts since they have corrosive effects on building components, and decompose after repeated heating/cooling cycles. On the other hand, paraffin waxes are costly to apply in buildings, and leakage may occur when directly adsorbed onto wallboard, which restricts their applications [8–16]. Although polyethylene glycols (PEGs) have many properties that make them suitable for thermal applications in buildings, such as having high heat of fusion, phase change repeatability, chemical stability, non-corrosive behavior, low-cost, etc., only a few limited studies on impregnating PEGs into construction materials such as lightweight concrete or wallboards have been published [6].

The aim of the present study is to develop a number of PU rigid foams incorporated with three types of PEGs, as new insulation materials provided with an enhanced thermal capacity, and to seek their suitability for various applications, e.g. layer of floor and ceiling coverings in constructions, insulations in controlled temperature transportation packaging, electronics, inner coverings of automobile seats, etc. [17]. In order to investigate the thermal properties of PEG-containing PU foams, differential scanning calorimeter (DSC) tests were conducted first to provide information for the practicable enthalpy enhancements of products in comparison of the correspondent PEG values. Then, a two-layer concrete–PU foam system was designed in the simulated laboratory conditions regarding possible interactions of room and its environment to examine the resultant insulation performances for different system combinations of such an application via using a computer-aided thermal measurement apparatus which is sensitive to the simulated environmental temperature changes. Furthermore, leakage of PU-PEG composites was also tested to explain their durability.

2. Experimental

2.1. Materials

Three types of PEGs, say PEG 600, PEG 1000, PEG 1500 used as PCMs, were technical grade chemicals from Merck (Fig. 1). PU foam as a sheet of 0.02 m in thickness and with the density of 18 kg m^{-3} was obtained from a commercial source. 0.1 M glycerinaldehyde was applied as binder [18]. Additionally, commercial types of Portland cement and aggregates were used for the design of concrete container.

2.2. Methods

2.2.1. Preparation of PEG-impregnated PU foam

Each PU foam sample was prepared to cover the lateral surface and the base of concrete container, and then held at 60°C to prevent it from moistening until PCM impregnation was complete. Aqueous PEG solutions were prepared as 30% by mass. An aliquot of 50 mL PEG was mixed well with 2 mL of 0.1 M glycerinaldehyde (GA) (aq), then added drop wise to the foam using an automatic pipette so that the liquid mixture spread evenly on the surface. PU-Control

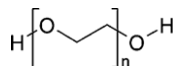


Fig. 1. Structural formula of PEGs, $(\text{C}_2\text{H}_4\text{O})_n \cdot \text{H}_2\text{O}$ where n is the number of monomer repeats.

samples were prepared by adding 50 mL of water and 2 mL of glycerinaldehyde solution. Uniform spreading of PEG solution into the PU foam structure was maintained by rolling a stainless steel cylinder of 10 N on the surface for 5 min. Afterwards, all specimens were kept at 5°C overnight to improve binding of GA in the structure. Next, samples put in coarse filter papers were waited under the same load of 10 N for 1 h to remove the excess of aqueous phase. Specimens were finally left in ambient air for 2 days at 20°C . Table 1 lists the specifications of the PU specimens prepared, including either a type of PEG or a mixture of PEGs used alternately to broaden the phase transition interval and to improve the temperature stabilization of the foams.

2.2.2. DSC measurements

To characterize the thermal behavior of pure PEGs and PEG-containing PU foams, analyses were conducted on a PerkinElmer Diamond type DSC; the instrument was recalibrated for measurements performed in different atmospheres in an accredited laboratory conditions.

During analyses of PEGs, test specimens were heated from 5 to 60°C at a rate of $10^\circ\text{C min}^{-1}$ under a nitrogen atmosphere (50 mL min^{-1}) in order to observe their actual phase change enthalpies under oxygen free environment. The DSC analyses of each PEG were conducted three times to determine its capacity of heat storage for repetitive heating cycles.

Four different PU-PEG composites were also tested by DSC. During those DSC measurements, specimens were heated from -10 to 60°C at $10^\circ\text{C min}^{-1}$, under ambient air similar to the ordinary using conditions. ΔH values indicating their phase change enthalpy were derived from the division of calculated area of energy in mJ, matching phase transition interval, by the sample weight in g, which was directly obtained from the DSC analysis.

2.2.3. Computer-aided thermal measurement setup

2.2.3.1. Molded concrete container. Portland cement, fine aggregates and water were mixed at a ratio of 2:6:1, then the mixture was decanted into a double-walled hollow cylindrical mold to construct the concrete container. After curing for 1 month, the concrete was obtained with a mass of 859.8 g, density of 2400 kg m^{-3} and interior volume of 232.3 cm^3 . The specific heat capacity of $c = 0.750 \text{ J g}^{-1} \text{ }^\circ\text{C}^{-1}$ and thermal conductivity of $\lambda = 1.6 \text{ W m}^{-1} \text{ K}^{-1}$ were taken from the literature for the concrete of a similar density [19]. The lid (62 g) for closing the container and the interior covering (17 g) to prevent water penetration into the concrete were both composed of PET, with $c = 1.03 \text{ J g}^{-1} \text{ }^\circ\text{C}^{-1}$ and $\lambda = 0.15 \text{ W m}^{-1} \text{ K}^{-1}$.

2.2.3.2. Thermal measurement setup. To test the thermal isolation efficiency of PU-PEG composites encircling concrete container, the computer-aided thermal measurement system shown in Fig. 2 was setup. The closed system consisted of a concrete cylinder with a plastic lid through which a water-resistant Teflon Thermocouple 1 was positioned at the center. The developed PU insulation layer was covered over the lateral surface as well as the base of the closed system. Then the Thermocouple 2 of stainless steel was settled tightly next to the outer surface of the container, aligned vertically with the Thermocouple 1. A data logger, TESTO 177-T4, running within the temperature range of -50 to 250°C with the precision of $\pm 0.5^\circ\text{C}$, was connected to a computer. Then, 220 g of water was put into the container, which was initially at $1-2^\circ\text{C}$ in order to avoid the loss of mass due to the evaporation with the increasing water temperature. The whole system was placed on a thermostat heater with a magnetic stirrer. Prior to heating, water in the system was stirred for 15 min, say, until thermocouples indicated temperature stabilities for both of measurement positions.

Table 1
Polyurethane specimens prepared and their specifications

Specimen	Density (kg m^{-3})	Thickness (m)	Area (m^2)	Added materials	Added mass/PU foam, %
PU-Control	18	0.02	0.085	50 mL pure water, 2 mL of 0.1 M GA	4
PUI	18	0.02	0.085	50 mL 30% PEG 600, 2 mL of 0.1 M GA	44
PUII	18	0.02	0.085	50 mL 30% PEG 1000, 2 mL of 0.1 M GA	49
PUIII	18	0.02	0.085	50 mL 30% PEG 1500, 2 mL of 0.1 M GA	53
PUIV	18	0.02	0.085	50 mL 30% PEG 600:PEG 1000:PEG 1500 (1:1:1), 2 mL of 0.1 M GA	38

To predict the contribution of the developed PU-PEG composite to the isolation of the system, in comparison of those achieved by concrete itself or concrete and PU-Control combination, the constant heating condition was applied at the rate of 2°C min^{-1} with stirring at 100 rpm. Thermocouple 2 was used to measure outer surface temperatures, in order to start and end each test at the same initial and final temperatures, which was almost equal to the ambient temperature at the beginning. Specifically, the test was initiated by recording temperatures of inner medium and outer surface simultaneously and conducted until arriving the predetermined final temperature of the latter. Based on the correspondent temperature difference of inner medium, specific heat capacity of water ($4.18 \text{ J g}^{-1} \text{ K}^{-1}$) and mass of it (220 g), the estimated enthalpy changes of inner medium were calculated. This approach allowed to compare the enthalpy changes of inner medium in terms of water equivalent values as a relevant and general process in calorimetric type experiments. Therefore, tests could be achieved under controlled conditions, i.e. constant heating and stirring, constant mass of water, exact positioning of thermocouples, and measuring for the predetermined outer surface temperature range under standard atmospheric conditions of $20 \pm 2^\circ\text{C}$ and $65 \pm 5\%$ relative humidity. Consequently, this system was designed as a special application

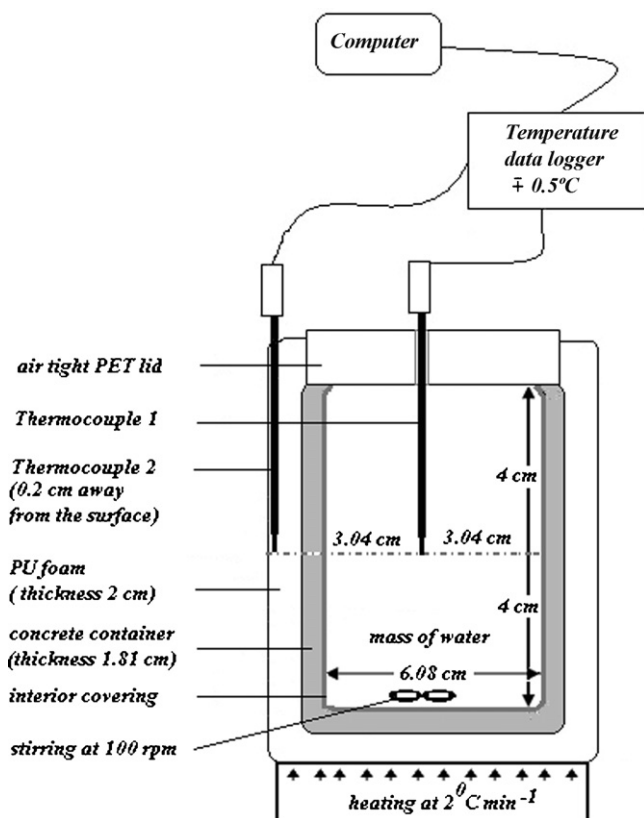


Fig. 2. Computer-aided thermal test system made of concrete and surrounded by PEG-incorporated PU foam.

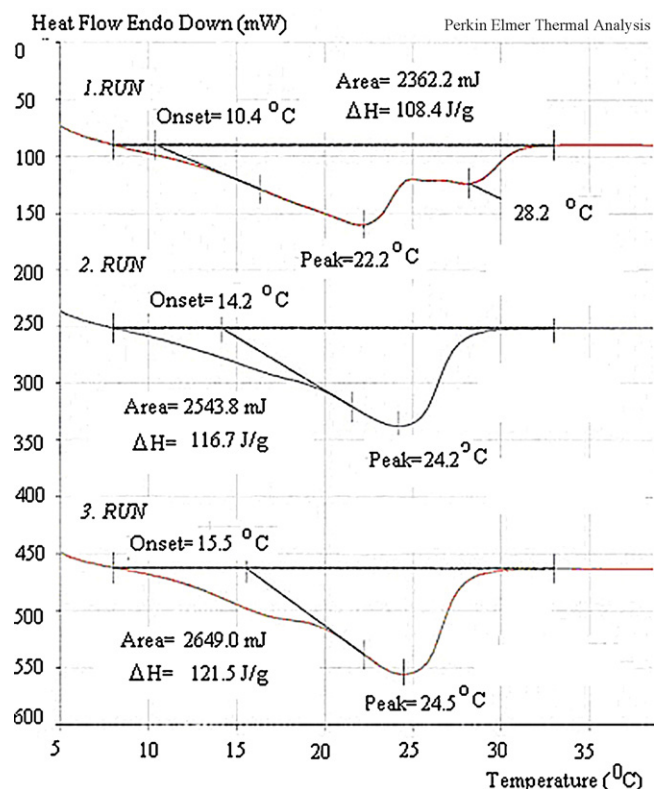


Fig. 3. DSC graphs of PEG 600 for three successive heating at $10^\circ\text{C min}^{-1}$ (sample mass is 21.8 mg).

similar to a building mantled from three front walls so that only one of them was heated whereas the fourth side was an interface with the outside.

2.2.4. Leakage tests

The leakage behavior of foams was investigated using a centrifugal shear force test [20]. PEG-containing PU sheets were cut into small pieces of approximately 2 g. The prepared sample and 10 mL of water were placed into a test tube, centrifuged at 400 rpm for 4 h at room temperature and then stored overnight in a refrigerator at 4°C . The supernatants were then examined visually whether there was any oil–water phase separation indicating the presence of PEG leakage.

3. Results and discussion

3.1. Thermal properties of PEGs

DSC results for PEG 600, PEG 1000 and PEG 1500 are presented in Figs. 3–5 and Table 2. ΔH values for their phase changes are all fairly large, and the PEGs are thus suitable for heat absorption or heat release. After three heating, values for the enthalpy of fusion and phase transition temperature for all three PEGs remained almost

Table 2
Phase transition temperature intervals and corresponding enthalpy changes of PEGs and PU-PEG composites obtained from DSC measurements during heating at $10\text{ }^{\circ}\text{C min}^{-1}$

Specimen	Temperature interval of phase transition ($^{\circ}\text{C}$)	T_{maximum} ($^{\circ}\text{C}$)	ΔH (J g^{-1})
PEG 600	10–32	22.2	108.4
PEG 1000	33–46	41.7	149.5
PEG 1500	44–55	51.2	176.3
PUI [50 mL 30% PEG 600 (aq) added]	0–28	18.1	62.3
PUII [50 mL 30% PEG 1000 (aq) added]	28–42	37.8	76.7
PUIII [50 mL 30% PEG 1500 (aq) added]	40–52	49.6	138.1
PUIV [50 mL 30% PEG 600:PEG 1000:PEG 1500 (1:1:1)(aq) added]	0–48	17–31–42.4	72.4

constant, indicating their thermal cycling stability that is essential for long-term use as PCM.

3.2. DSC analyses results of PU-PEG composites

Fig. 6a–d show the DSC results of PEG-containing PU foams. The DSC phase transition intervals obtained for PUI–PUIV all coincide with those for PEG 600, PEG 1000 and PEG 1500, and the enthalpy changes observed are distinctive. Shifts in phase change intervals compared to those of PEGs can be attributed to the response of a hybrid structure composed of materials of different characteristics. Table 2 summarizes their phase transition intervals and peak temperatures occurred as well as their enthalpy changes (ΔH values).

DSC curves of PUI–PUIII in Fig. 6a–c reveal considerable enthalpy values achieved, say 62.3 , 76.7 and 138.1 J g^{-1} , respectively, which are comparable with those for PEG 600, PEG 1000 and PEG 1500, say 108.4 , 149.5 and 176.3 J g^{-1} , respectively. Alternately, DSC curve of PUIV characterized by three peaks showed that mixing of three different PEGs in PU foam resulted in a phase transition interval covering a wide range from approximately $0\text{--}48\text{ }^{\circ}\text{C}$, where the enthalpy change was 72.4 J g^{-1} , succeeded on the same product. Thus, PEGs performed their phase changes successfully in the composite structure and determined the heat storage capacity of the ultimate product significantly.

3.3. Results of computer-aided thermal measurements

Fig. 7 summarizes the thermal behaviors of the developed PU-PEG composites in the simulated test system where T_{OS} is the temperature of the outer surface, corresponding to the mea-

surements of Thermocouple 2 shown in Fig. 2, T_{C} , T_{PU} and $T_{\text{PUI}}-T_{\text{PUII}}-T_{\text{PUIII}}-T_{\text{PUIV}}$ are temperatures of inner media obtained from the measurements of Thermocouple 1, which was surrounded by the different systems, i.e. concrete alone, PU-Control mantled concrete and PUI–PUIV foams mantled concretes, respectively.

When only the concrete was used as an interface between outer and inner media, the temperature of the inner medium showed a parallel increase to that of outer surface in relation to the thermal conductivity of the concrete. In the system surrounded by PU foam, temperature of water (T_{PU}) maintained within a considerably narrow range, say from 5.8 to $13.5\text{ }^{\circ}\text{C}$, against the large changes in external temperature, say from 22.3 to $51.6\text{ }^{\circ}\text{C}$, in dependence of the insulation function of the foam, entrapping great amount of still air, characterized by the slope of T_{PU} curve plotted versus time, which is lower than those of T_{OS} and T_{C} (see Fig. 7).

Furthermore, in all systems covered by PU-PEG composites, internal temperatures remained almost unchanged in a certain temperature interval while heating at a constant rate, indicated by their more or less parallel lines to the horizontal axis of time in Fig. 7. The provided thermal isolation became apparent in the test of PUI containing 44% PEG 600 as the temperature of the outer surface changed from 22.3 to $35.6\text{ }^{\circ}\text{C}$, coinciding to the phase change interval of the PCM itself as well as that of the composite PUI; the temperature of water remained at very low values ($0.9\text{--}1.0\text{ }^{\circ}\text{C}$) and slightly changed ($1.0\text{--}1.3\text{ }^{\circ}\text{C}$) during the further increases in outer surface temperature up to $51.6\text{ }^{\circ}\text{C}$ related to the PCM content in the structure. This could be achieved by means of latent heat of

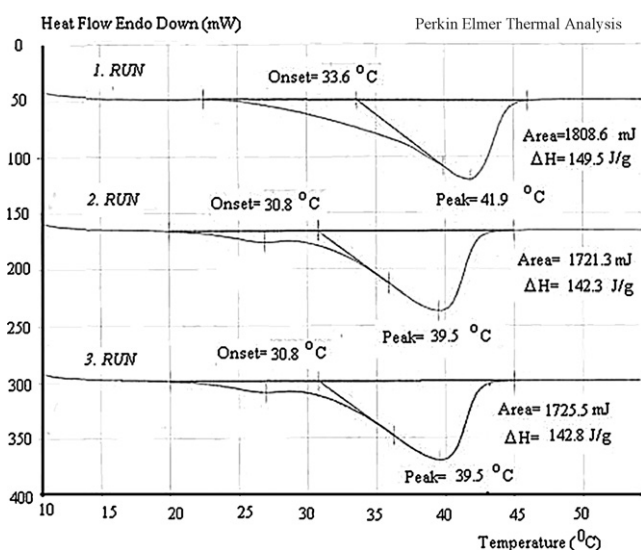


Fig. 4. DSC graphs of PEG 1000 for three successive heating at $10\text{ }^{\circ}\text{C min}^{-1}$ (sample mass is 12.1 mg).

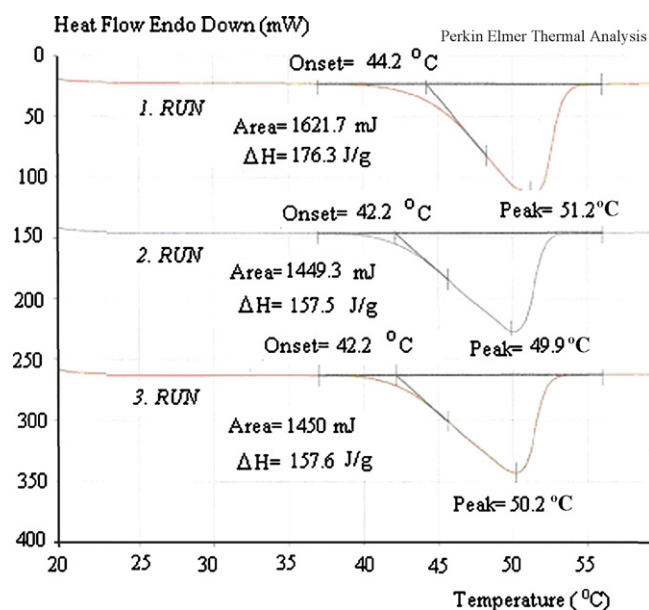


Fig. 5. DSC graphs of PEG 1500 for three successive heating at $10\text{ }^{\circ}\text{C min}^{-1}$ (sample mass is 9.2 mg).

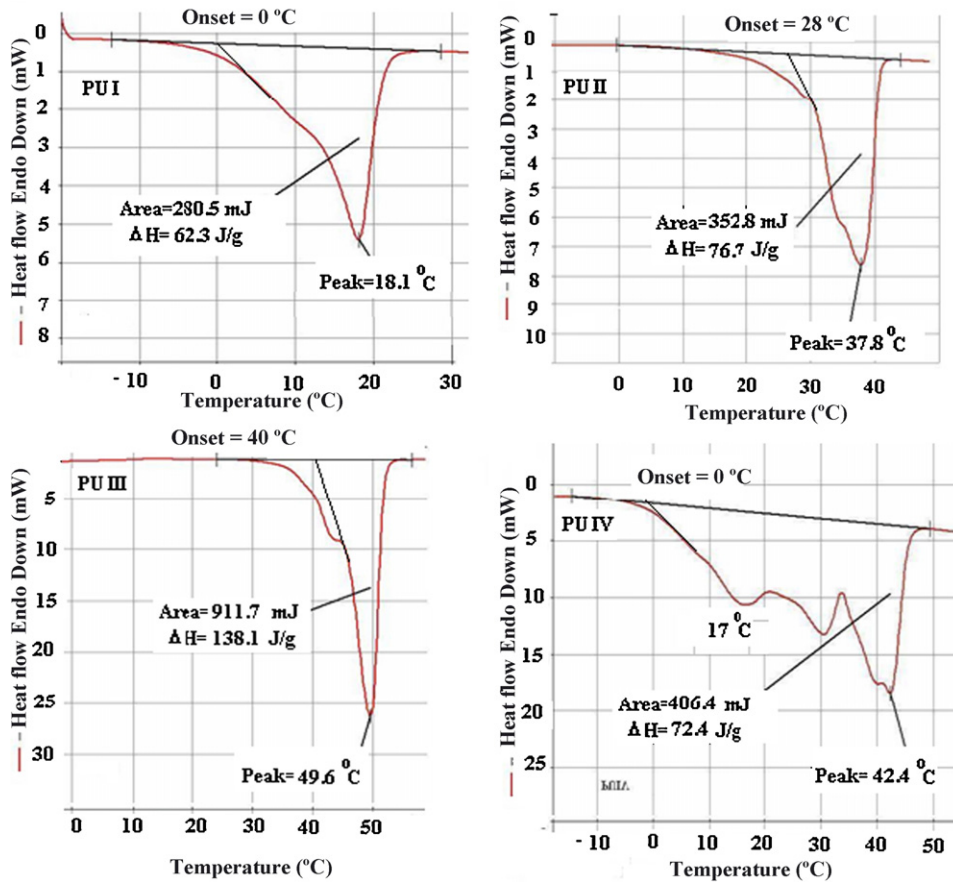


Fig. 6. DSC curves of PU-PEG composites containing: (a) PEG 600 (PUI, sample mass is 4.49 mg); (b) PEG 1000 (PUII, sample mass is 4.59 mg); (c) PEG 1500 (PUIII, sample mass is 6.60 mg); (d) PEG 600–PEG 1000–PEG 1500 (PUIV, sample mass is 5.60 mg) during heating at $10^{\circ}\text{C min}^{-1}$.

fusion taking place for solid to liquid phase changes of some PEG 600 molecules as well as the heat absorbed by its liquid phase. In the tests conducted for PUII–PUIV, the desired thermal buffering effects were even obvious for higher outer surface temperatures

from 35.6 to 51.6°C . These results were thus found in accordance with the DSC results indicating large latent heat of fusion above 22°C (see Fig. 6b–d).

Moreover, Table 3 illustrates the corresponding increases in water temperature while outer surface temperature changes in between 22.3 and 51.6°C . In the tests conducted with PU-PEG composites less temperature increases resulted in and equivalent enthalpy changes were quite small. For instance, in PU-Control test, increase in the temperature of water was 7.7°C and the enthalpy change was 7.08 kJ ; on the other hand, in PUI test, increase in the temperature of water was only 0.4°C whereas the enthalpy change was 0.37 kJ which was only 5.2% of the previous, indicating an improved thermal buffering effect. These results imply the achievement of the improved insulation of PU-PEG composites as compared to PU only. The system was therefore workable for certain temperature intervals and the test results supported to the DSC results of PU composites.

3.4. Leak testing of PU-PEG composites

The leakage behavior of foam samples is an important property in determining potential applications and durability. After the centrifugal shear force test and overnight incubation at 4°C , no oil–water phase separation was detected in the supernatants, verifying the leakage resistance of the composite samples. In addition, no significant change in mass was observed for PEG-containing PU foams and no PEG stain was evident on filter papers following compression of samples under a static load of 12 kPa for 2 days.

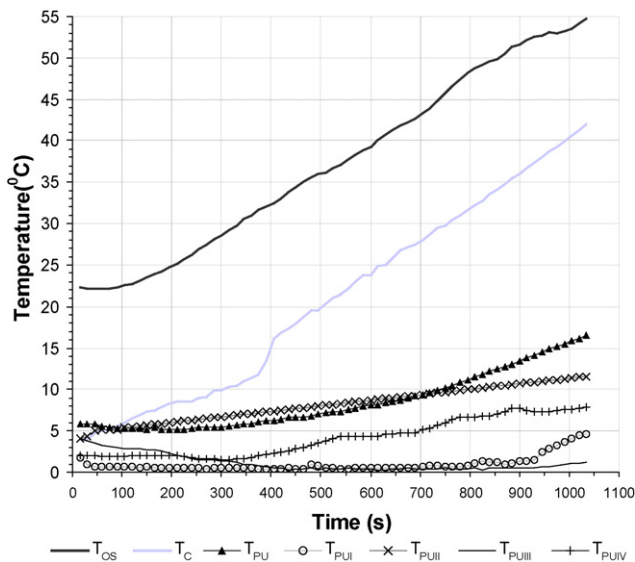


Fig. 7. The temperature versus time plots of PU-PEG composites enveloping concrete container in comparison to the concrete alone and PU-Control foam covered concrete while heating the container at $2^{\circ}\text{C min}^{-1}$.

Table 3Temperature and enthalpy changes in the inner medium of water in the test system while heating the container at 2 °C min⁻¹ (mass of water 220 g)

Experimental group	Outer surface temperature change (°C)	Inner medium (water) temperature change (°C)	ΔT (temperature difference of water) (°C)	Increase in water enthalpy ($\Delta H = mc \Delta T$) ^a (kJ)
Non-isolated concrete	22.3–51.6	3.9–36.5	32.6	30.09
PU-Control	22.3–51.6	5.8–13.5	7.7	7.08
PUI (44% PEG 600)	22.3–51.6	0.9–1.3	0.4	0.37
PII (49% PEG 1000)	22.3–51.6	3.9–10.7	6.8	6.25
PIII (53% PEG 1500)	22.3–51.6	0.4–0.6	0.2	0.18
PIV (38% PEG 600–1000–1500)	22.3–51.6	2.1–7.7	5.6	5.15

^a *m*, mass of water; *c*, specific heat capacity of water (4.18 J g⁻¹ K⁻¹).

4. Conclusions

In this study, we tested a new approach to integrate PEGs as PCM in insulation materials of foam type. Three different types of PEGs were used so that the different melting temperature ranges were possible for those developed materials. DSC analyses of PU-PEG composites yielded high enthalpies in certain temperature intervals indicating that the heat absorption/release capacities of PU foams could be improved by means of PEG incorporation. Thermal analyses of the new materials also prove that the contained PCMs are all active. The PU-PEG composites produced here can be helpful for the design of thermal insulators. Furthermore, PU foams containing PEGs can be assumed to be leak-resistant, which is promising for their industrial applications.

In our particular setup, enhanced thermal buffering compared to pure insulation material was observed. By enveloping the concrete with PU-PEG composites in a test system, the heat transfer from the external environment was lowered so that internal temperature changes could be minimized. While heating the system at 2 °C min⁻¹, inner medium temperature difference of ΔT was measured as 32.6 °C in the non-isolated container whereas temperature changes of the inner media in all other isolated containers were quite small, namely 7.7 °C for PU-Control (only passive insulation by means of entrapping great amount of still air), 0.4 °C for PUI (with 44% PEG 600), 6.8 °C for PUII (with 49% PEG 1000), 0.2 °C for PUIII (with 53% PEG 1500), for 5.6 °C for PUIV (with PEG 600–1000–1500).

We conclude that PUIII, containing 53% PEG 1500, showed satisfactory performance in all aspects and has suitable heat storage and thermal stability characteristics for further industrial applications. Sample PUIV containing 38% PEG 600/PEG 1000/PEG 1500, also showed good thermal and durability characteristics. The blend of three PEGs is suitable for preventing discontinuous thermal regulation when the external temperature increases or decreases. PUI, including 44% PEG 600, exhibited fairly efficient thermal regulation under moderate ambient temperature conditions, whereas PUII (49% PEG 1000) is suitable for temperature control in both mild and hot surroundings.

Continuous ambient phase transitions can also be achieved by a suitable choice of thermal insulation materials, for example, an outer layer of PU foam containing PEG 1000 or PEG 1500 as a collector of environmental heat and an inner layer of PU foam containing PEG 600 as a barrier to heat transfer from the external environment. PU foams containing PEGs can be assumed to be leak-resistant, which is promising for industrial production of PEG-containing PU. Heat capacity values measured in concrete wall simulation experiments verified the enhanced thermal capacity of these samples.

In conclusion, PU-PEG foams are thought to be suitable composites for the design of thermal insulation systems in different forms, i.e. as a single layer of a kind or layer upon layer of different kinds. Such materials could be satisfactorily used to reduce the energy demand for space cooling or heating as well as in

containers for special purposes like, controlled temperature transportation packaging for foods, medicines, electronic equipment, etc. Further investigations are necessary to quantify the effect of the new materials in particular applications. For instance, the computer-aided temperature measurement setup can be modified using containers other than concrete, e.g. aluminum or plastic, in order to experiment the utilization of PU-PEG composites for different applications such as temperature-controlled transport packaging, etc. Changing the rate and direction of heating for cold inner medium experiments, proceeding similar experiments during free/rapid cooling of warm inner media are also promising.

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