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Improvement of thermal inertia of styrene–ethylene/butylene–styrene (SEBS) polymers by addition of microencapsulated phase change materials (PCMs)

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ABSTRACT

In this work, microencapsulated phase change materials (PCMs) with a melting temperature of 52 °C have been used to improve thermal inertia phenomena on an elastomeric matrix of styrene–ethylene/butylene–styrene (SEBS) material. The amount of PCMs has varied in the 1–10 wt.% and these materials have been processed by conventional injection molding without PCM degradation. Mechanical characterization of SEBS–PCM compounds has been carried out and the obtained results show good maintenance of both resistant and ductile properties for PCM amounts comprised in the 1–5 wt.% range. Scanning electron microscopy (SEM) analysis has revealed good wetting properties of PCM microcapsules with the SEBS matrix which is a key factor to obtain good mechanical performance. The effect of PCM addition on thermal inertia has been evaluated by active infrared thermography (IRT), showing a remarkable effect on thermal regulation of SEBS in the temperature range close to the melting point of the PCM (52 °C). This thermoregulation effect is more accurate as the PCM content increases. Also, cooling curves have been constructed in order to quantify the thermal inertia effect in a cooling process.

1. Introduction

Among the wide variety of thermoplastics which are useful in many industrial applications, thermoplastic elastomers are characterized by excellent balance between processability (typical of commodity plastics) with excellent physical properties (typical of vulcanized elastomers) and are increasingly used in automotive and medical applications as liquid silicone rubbers.[1–5] Styrene–ethylene/butylene–styrene (SEBS) polymers derive from styrene–butadiene–styrene (SBS). SEBS is obtained by hydrogenation of SBS polymers; this process allows removing of insaturations typical of the butadiene components (carbon–carbon double bonds are saturated with hydrogen) and this has a positive effect on environmental, thermal and UV radiation resistance maintaining thermoplastic behavior, so that SEBS is useful in applications in which the use of SBS is restricted due to its sensitiveness to degradation.[6,7] SEBS polymers successfully combine elastomeric properties with low processing costs typical of commodity plastics. The excellent aging resistance of SEBS polymers is due to the absence of carbon–carbon double bonds. By varying the relative ratio of the components (styrene, ethylene and butylene) on SEBS formulations it is possible to obtain a wide variety of elastic and hardness values with different applications in industry. SEBS polymers are commercially available in white color or even in transparent grades. They can be processed at relatively low temperatures and show excellent resistance to intermediate temperatures.

Many research works have focused on SEBS blends and its potential use as compatibilizer for blends of polyolefins and styrene derivatives. Some studies show the effects of SEBS formulation on chemical properties, thermal behavior and stabilization [8,9] and others have focused on the use of additives for improved mechanical properties. As it can be injection molded, some works have focused on the study of rheological properties of powdered SEBS and

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mechanical relaxation has been followed at low flux frequencies.[10] Some studies have proved the excellent behavior against aging and the usefulness of employing stabilizers to prevent photo-oxidation.[11,12] Despite some research works have focused on individual SEBS polymers, many authors have worked on blends with commodity and engineering plastics (polypropylene-PP, polystyrene-PS, polyamide 6-PA6…).[13–16]. Some aspects such as compatibility, morphology, mechanical performance, influence of particle fillers (including nanoparticles) and additives, etc. have been widely studied in the literature.[17–20] Even, potential use of SEBS as matrix for composites has been studied.[21–23] As compatibilizer, SEBS shows interesting properties due to the nature of its structure with polar moieties (styrene) and non polar moieties (ethylene/butylene) and this allows compatibilizing a wide variety of plastics [24–26].

Recently, energy storage phenomena have attracted growing attention. The use of phase change materials (PCMs) has grown in a remarkable way in the last years so that they find applications as thermal energy storage in building applications.[27–29] PCMs are materials with a well defined melting point and a relatively high heat of fusion. These materials absorb energy to melt, and release heat to crystallize so that, they can be used as energy storage materials. PCMs can be organic materials (waxes, paraffin, fatty acids, etc.), inorganic compounds or low melting point mixtures (eutectics). [30–32] Also, PCMs can be used at a macro-scale range or at a micro-scale range. Anyway, encapsulation is needed to keep absorbed heat. This encapsulation can be obtained by dispersing the PCM powder into a polymer matrix and subsequent coating of the finished part.[33,34] On other hand, encapsulation can be carried out at micro-scale range. In this case, PCM microparticles are covered with a protective coating characterized by high thermal stability which allows great number of melt-crystallization cycles. The use of PCMs can be useful to optimize thermal inertia which can be defined as the capacity of a material to preserve thermal energy with a slow and progressive release of it. It depends on the mass, density and specific heat. Optimizing thermal inertia leads to good thermal damping efficacy which is of great importance in building applications [35,36].

In this work we have used microencapsulated phase change materials (PCMs) with a melting point of 52 °C to optimize thermal inertia of parts made of styrene–ethylene/butylene–styrene polymers for uses in industrial applications in which thermal regulation is a key factor. SEBS–PCM compounds with different PCM to SEBS ratio have been prepared by conventional injection molding and mechanical properties of compounds have been determined. Dispersion of PCM microcapsules has been studied by SEM analysis. In addition, thermal inertia of SEBS–PCM compounds has been studied by cooling curves obtained by active infrared thermography (IRT).

2. Experimental

2.1. Materials

Two different styrene–ethylene/butylene–styrene (SEBS) commercial grades with different hardness and elongation values were used to obtain a silicone rubber like material. An extremely soft (Shore A hardness = 5) grade Megol TA 5® Neutral and a relatively rigid elastomer (Shore A hardness = 90) Megol TA 90® Neutral supplied by Applicazioni Plastiche Industriali (Api SpA, Vicenza, Italy) were blended in a 70/30 weight ratio respectively to obtain similar properties to silicone rubber to evaluate potential substitution. Table 1 summarizes main properties of MEGOL TA® commercial grade product line.

Phase change materials microcapsules (micro PCMs) with a melting point of 52 °C, MPCM 52-D were supplied by Microtek Labs (Microtek Laboratories Inc., Dayton, USA). These microcapsules are composed of a paraffin blend core (85–90 wt.%) with tetracosane as the main component and a melamine–formaldehyde inert polymer shell (10–15 wt.%) which is stable up to 250 °C heating thus enabling processing with conventional injection molding at intermediate temperatures. Microcapsules are supplied in powder form with an average diameter of 17–20 μm.

2.2. Processing of SEBS–PCM compounds

In a first step, MPCM 52-D microcapsules were mechanically mixed with the SEBS blend. The weight percentage of MPCM 52-D microcapsules added to SEBS matrix was set to 1%, 2%, 5% and 10%. Over 10 wt.% microcapsules form aggregates and are not homogeneously dispersed in the SEBS matrix with mechanical mixing. Once these mixtures

Table 1
Main characteristics ranges for different properties of SEBS commercial grade Megol TA® polymers.

Shore hardness range	5–90 A
Compatibility	Polypropylene (PP), polyethylene (PE), ethylene–vinyl acetate copolymer (EVA)
Aging resistance ozone (72 h – 40 °C – 200 ppcm) tension = 20%	Excellent
Density (g cm^{-3})	0.88–0.89
Tear strength (kN/m)	22–44
Tensile modulus 100% elongation (MPa)	1.1–4.2
Tensile strength (MPa)	6.0–7.2
Elongation at break (%)	700–550

Table 2
Main parameters of the injection molding process at 180 °C of SEBS–PCM compounds.

Injection rate	35%
Injection distance	294 dmm
Injection shot	150 dmm
Filling	144 dmm
Holding pressure	15%
Holding time	10 s
Shutoff nozzle	1610 offset
Shot distance	294 dmm
Suction distance	0 dmm
Mold temperature	40 °C
Cooling time	11 s
Clamp pressure	54 Tm

were prepared, tensile test standard samples were obtained with an injection molding machine Meteor 270/75 (Mateu and Solé, Barcelona, Spain). The injection temperature was 180 °C and the main injection molding parameters are summarized in Table 2.

2.3. Mechanical characterization of SEBS–PCM compounds

Mechanical properties of SEBS–PCM compounds with different PCM content were obtained with a universal tensile test machine ELIB 30 (S.A.E. Ibertest, Madrid, Spain) following the guidelines of the ISO 527. All samples were tested at 25 °C and a relative humidity of 50% using a crosshead rate of 50 mm min^{-1} with a load cell of 100 N. A minimum of ten samples were tested and average values of elongation at break (ductile mechanical property) and tensile strength (resistance mechanical property) were calculated.

Hardness of SEBS–PCM compounds was determined using a shore durometer (Instruments J. Bot S.A., Barcelona, Spain) with the A scale following the guidelines of the ISO 868.

2.4. Microscopic characterization of SEBS–PCM compounds

Morphology analysis of PCM microcapsules and fractured surfaces of SEBS–PCM compounds was carried out by using a scanning electron microscope JEOL JSM-6300 (Jeol USA, Peabody) operated at an acceleration voltage of 15 kV. Prior to observation, samples were covered with a 5–7 nm Au layer in vacuum conditions.

2.5. Thermal characterization of SEBS–PCM compounds

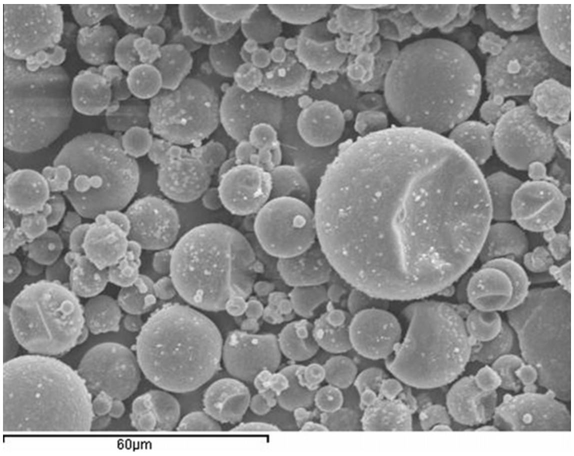

Fig. 1. SEM micrograph of phase change materials microcapsules MPCM 52-D with a melt point of 52 °C (1000×).

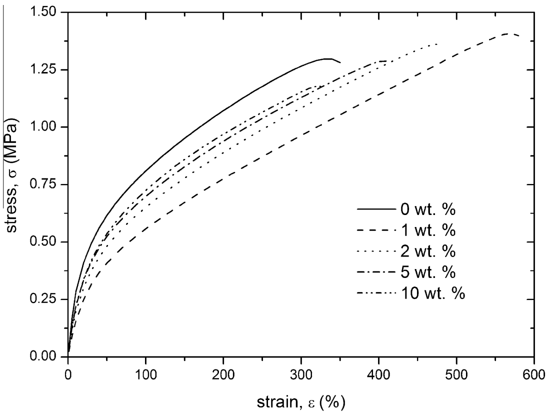

Fig. 2. Stress–strain ($\sigma - \varepsilon$) curves of SEBS–PCM compounds with different weight% of PCM (52 °C) microcapsules.

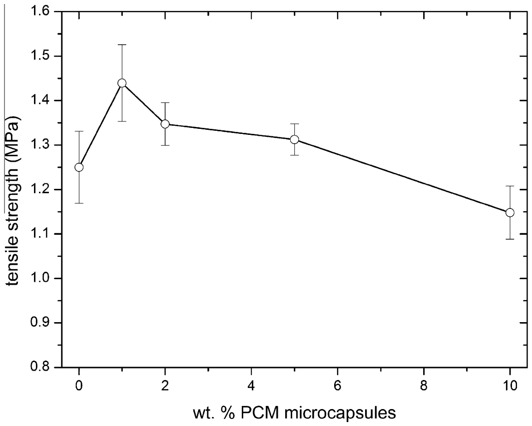

Fig. 3. Variation of the tensile strength of SEBS–PCM compounds in terms of the wt.% of PCM (52 °C) microcapsules.

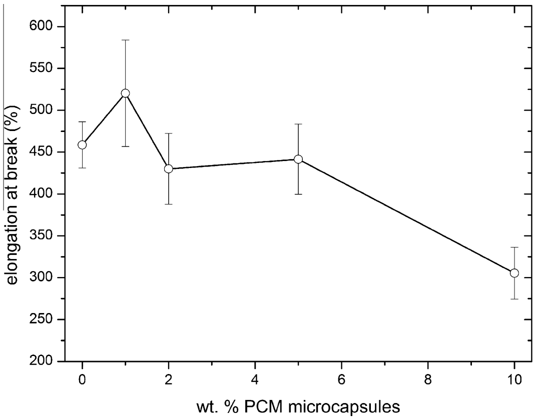

Fig. 4. Variation of the elongation at break of SEBS–PCM compounds in terms of the wt.% of PCM (52 °C) microcapsules.

Thermal properties of PCM microcapsules were determined with differential scanning calorimetry (DSC) using a Mettler-Toledo 821 (Mettler-Toledo Inc., Schwerzenbach, Switzerland). Test was carried out in nitrogen atmosphere (40 mL min^{-1}) and the temperature program was the following: a first heating from −30 to 100 °C at 10 °C min^{-1} was followed by a cooling from 100 to −30 °C at −10 °C min^{-1}; finally, a second heating from −30 up to 350 °C at 10 °C min^{-1} was carried out to evaluate thermal degradation at high temperatures. The phase change point was determined as the minimum of the peak and the heat of fusion was calculated by integrating the peak and normalizing it to the sample size.

2.6. Characterization of thermal inertia of SEBS–PCM compounds

Thermal inertia of SEBS–PCM compounds was studied by step heating infrared thermography. An infrared camera CANTRONIC IR980-600 (Cantronic Systems Inc., Coquitlam, Canada) was used for thermal image capture. This IR camera is equipped with a focal plane array (FPA)-uncooled microbolometer with a resolution of 320 × 240 pixel, a spectral range of 8–14 μm and a thermal time constant of 4 ms. The temperature range for measurements is from −20 to 600 °C.

Samples were subjected to a step heating from room temperature to 62 °C until constant temperature was obtained for all samples. After that, samples were placed on a paper shelve at room temperature to measure evolution of temperatures during the cooling process. Emissivity of SEBS–PCM compounds was set to 0.98 as other rubbers and automatic captures at intervals of 10 s were taken. After that, IR images were evaluated with the CMView SE Reporter 1.0 supplied with the camera.

3. Results and discussion

3.1. Mechanical properties of SEBS–PCM compounds

Fig. 1 shows a SEM micrograph of MPCM 52-D microcapsules powder. Addition of PCM microcapsules can lead to a change in mechanical response of SEBS–PCM compounds. Tensile strength is highly sensitive to the presence of additives, mainly at high additive amounts. Poor interaction between additive particles and the polymer matrix produces more pronounced stress concentration effects which lead to early fracture. On other hand, if there exists good interaction between filled particles and the polymer

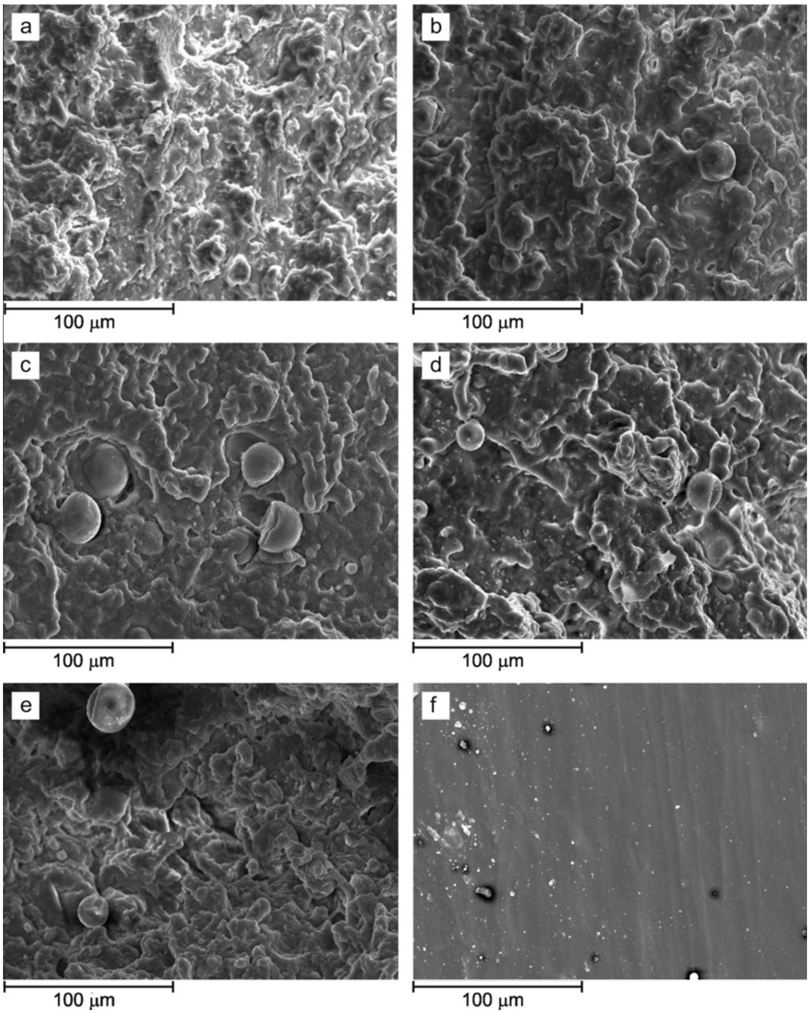

Fig. 5. (a–e) SEM micrographs of fractured surfaces from tensile tests of SEBS–PCM compounds with different wt.% of PCM microcapsules (500×); (a) 0 wt.%, (b) 1 wt.%, (c) 2 wt.%, (d) 5 wt.% and (e) 10 wt.%. (f) SEM micrograph of injection molded sample surface (500×).

matrix, stress concentration effects also occur but in less extent and it is possible to achieve an increase in mechanical properties.

Fig. 2 shows the stress–strain curves for unfilled SEBS blend and SEBS with different amounts of PCMs. We can clearly observe that presence of small amounts of PCMs (less than 5 wt.%) lead to good mechanical properties (high tensile strength and elongation at break) while higher amounts of PCM (10 wt.%) produce a decrease on mechanical performance. In the case of SEBS–PCM compounds, we can see an increase in tensile strength with presence of PCM microcapsules (Fig. 3). For 1 wt.% PCM, tensile strength changes from 1.25 MPa (unfilled SEBS) up to values of about 1.45 MPa (which represents a percentage increase of about 16%). So that, we could think that good interaction between PCM microparticles and SEBS matrix is obtained with mixing in the injection molding machine. In these conditions, PCM microparticles act as interlock points thus restricting polymer chain motion. As the PCM content increases, tensile strength decreases in a slight way but it still remains at similar values to unfilled material. For 10 wt.%, tensile strength slightly decreases as a consequence of the high amount of microcapsules which can form aggregates.

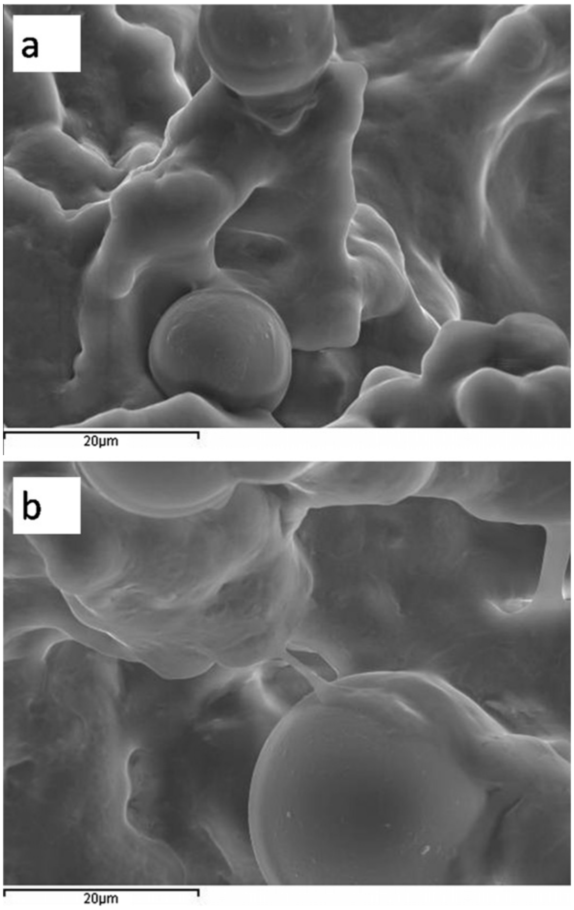

Fig. 6. SEM micrographs of fractured surfaces from tensile tests of SEBS–PCM compounds with different wt.% of PCM microcapsules showing good interaction between the SEBS matrix and PCM microcapsules (2000×) (a) 1 wt.% and (b) 2 wt.%.

Together with tensile strength, elongation at break is also highly sensitive to presence of stress concentrators. If the added particles do not interact with the polymer matrix, a strong stress concentration effect is expected thus leading to a decrease in both tensile strength and elongation at break. In contrast, if there is good microparticle–matrix interaction, stress concentration is less pronounced; it is possible to maintain elongation at break at high levels, similar to the unfilled material (or a slight decrease). Fig. 4 shows the evolution of the elongation at break in terms of the wt.% of PCM added to the SEBS mixture matrix. The initial elongation at break of the unfilled material is about 455%. As it can be observed, for PCM contents in the 1–5 wt.%. range, the elongation at break remains at high values (higher than 425%) but for higher PCM amount (10 wt.%), presence of aggregates leads to a decrease in elongation at break up to values of 300%.

SEM analysis of tensile fractured surfaces (Fig. 5) clearly shows presence of some PCM microcapsules in the fracture region. As the PCM content increases, presence of more PCM microcapsules can be detected in the fracture region. Nevertheless it is important to remark that even for high weight percentages of PCM, few microcapsules can be observed. This could be related to the fact that many PCM microcapsules are hidden or embedded in the SEBS matrix, so that they do not take part in the fracture process as stress concentrators. So that, it is possible to expect good interaction between PCM microparticles and the SEBS matrix as mechanical characterization has suggested. Also, good smooth surface is obtained after injection molding as observed in Fig. 5f.

This interaction can be clearly seen if we observe detailed zoom images of fractured surface of tensile samples. Potential lack of interaction between the disperse component (filler or additive) and polymer chains can be detected by SEM analysis by a small gap among the particle–matrix interface. As we can see in Fig. 6, this gap is not present. In fact, we can clearly observe good embedding of the PMC microcapsules on the SEBS matrix. All this is representative for good wetting of PCM microcapsules by the SEBS mixture. This good interface interaction/adhesion avoids stress concentration phenomena thus leading to formation of interlock points that restrict polymer chain motion and this has a positive effect on both tensile strength and elongation at break as described previously.

If we take into account that this material is widely used in applications with high flexibility, hardness is widely used to characterize mechanical performance of these

Table 3
Variation of the hardness Shore A of SEBS–PCM compounds in terms of the wt.% of PCM microcapsules.

Wt.% PCM	Hardness shore A
0	37
1	35
2	36
5	38
10	40

materials. Table 3 shows variation of the Shore A hardness values of SEBS–PCM compounds in terms of PCM weight%. The initial Shore A hardness of the unfilled material is about 37 and this remains with values in the 35–40 range for all compositions studied, so that, we can conclude that no significant changes in hardness is achieved by PCM addition thus, same final applications can be achieved with SEBS–PCM compounds.

3.2. Thermal inertia of SEBS–PCM compounds

The heat of fusion of PCMs is about 131.9 J g^{-1} as calculated by DSC (Fig. 7) which is in total accordance with the technical data supplied by the provider. As it can be observed in Fig. 7, the main melt/crystallization peak is located at 52 °C and a small peak can be detected at 30 °C. The usefulness of PCM additives in polymer matrices is based on the thermal regulation effects they can provide. Therefore, in heating process, PCM microcapsules will absorb heat in order to melt and this will have a retarding effect on the material temperature, stabilizing the temperature in a range near the PCM melt point. On the other hand, in a cooling process, PCM microcapsules will release heat in order to crystallize and this will also delay the cooling process. So that, PCMs are useful to regulate temperature in heating and cooling processes in a temperature range near to the melt/crystallization temperature of PCM (in this case, around 52 °C).

Infrared thermography (IRT) techniques could be useful to evaluate the improvement of the thermal inertia phenomena achieved by addition of PCMs to a SEBS matrix. Fig. 8 shows IRT images taken at different moments of the cooling process from 62 °C to room temperature of samples with different PCM (52 °C) content. At high temperatures, far from phase change of PCMs (52 °C), the surface temperature of all samples is practically identical (same coloration in Fig. 8a). This situation is clearer observable in Fig. 8b which shows a temperature profile of a line that crosses all five samples. As we can see surface temperature is practically identical and independent of the PCMs weight%. Once the phase change point (52 °C) has been reached, we can observe a slight difference in surface temperature for the different SEBS–PCM samples. Obviously, as the PCM weight% increases, the retarding effect is more evident. Fig. 8d shows the thermal profile for an intermediate point in the cooling process and we clearly detect gradual differences in surface temperature as the PCM increases. In this case we observe a temperature difference of about 1.5 °C between the unfilled SEBS (with a maximum temperature of 45.9 °C) and the SEBS–PCM compound with 10 wt.% PCM (with a maximum temperature of 47.3 °C). If we consider a point below the phase change temperature in which the temperature gap is maximum (after 325 s cooling time) we can see in a qualitative way (Fig. 8e) that the temperature gap has considerably increased (marked color difference). If we observe the temperature profile for the marked line that crosses all samples, the temperature gap is about 3.5 °C (Fig. 8f). In this region we can clearly observe the thermo regulating effect of the PCMs as they allow a soft cooling process thus maintaining temperature as PCMs release heat in the phase change. As PCMs are dispersed in a polymeric matrix, characterized by low thermal conductivity, the thermal inertial phenomenon is more pronounced. As the cooling process goes on, the temperature gap tends to diminish as temperature is reaching values far from the phase change point located at 52 °C. For a cooling time of 573 s, the temperature gap is again lower than 2 °C thus indicating that as we move away from the phase change point, the effects of the PCM are less pronounced.

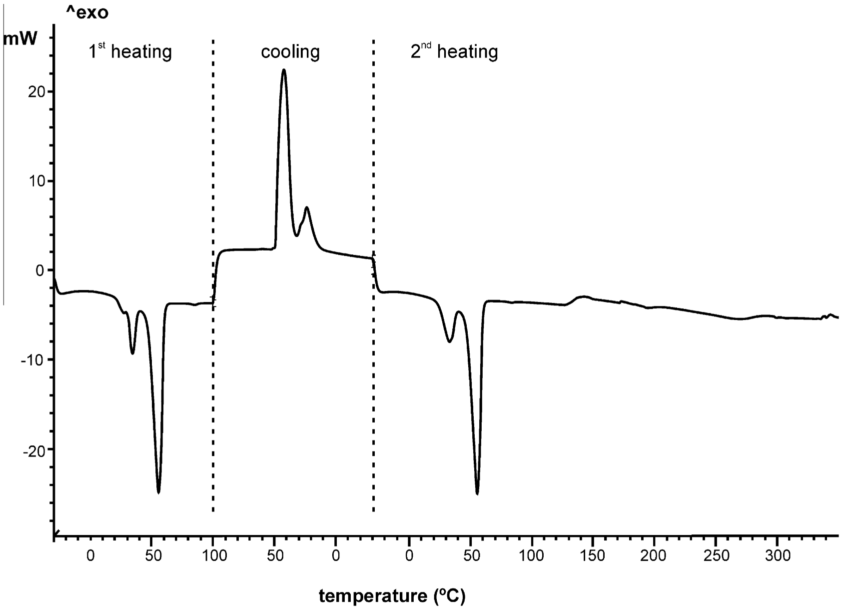

Fig. 7. DSC calorimetric curve of PCM (52 °C) microcapsules MPCM 52-D subjected to a heating–cooling–heating cycle.

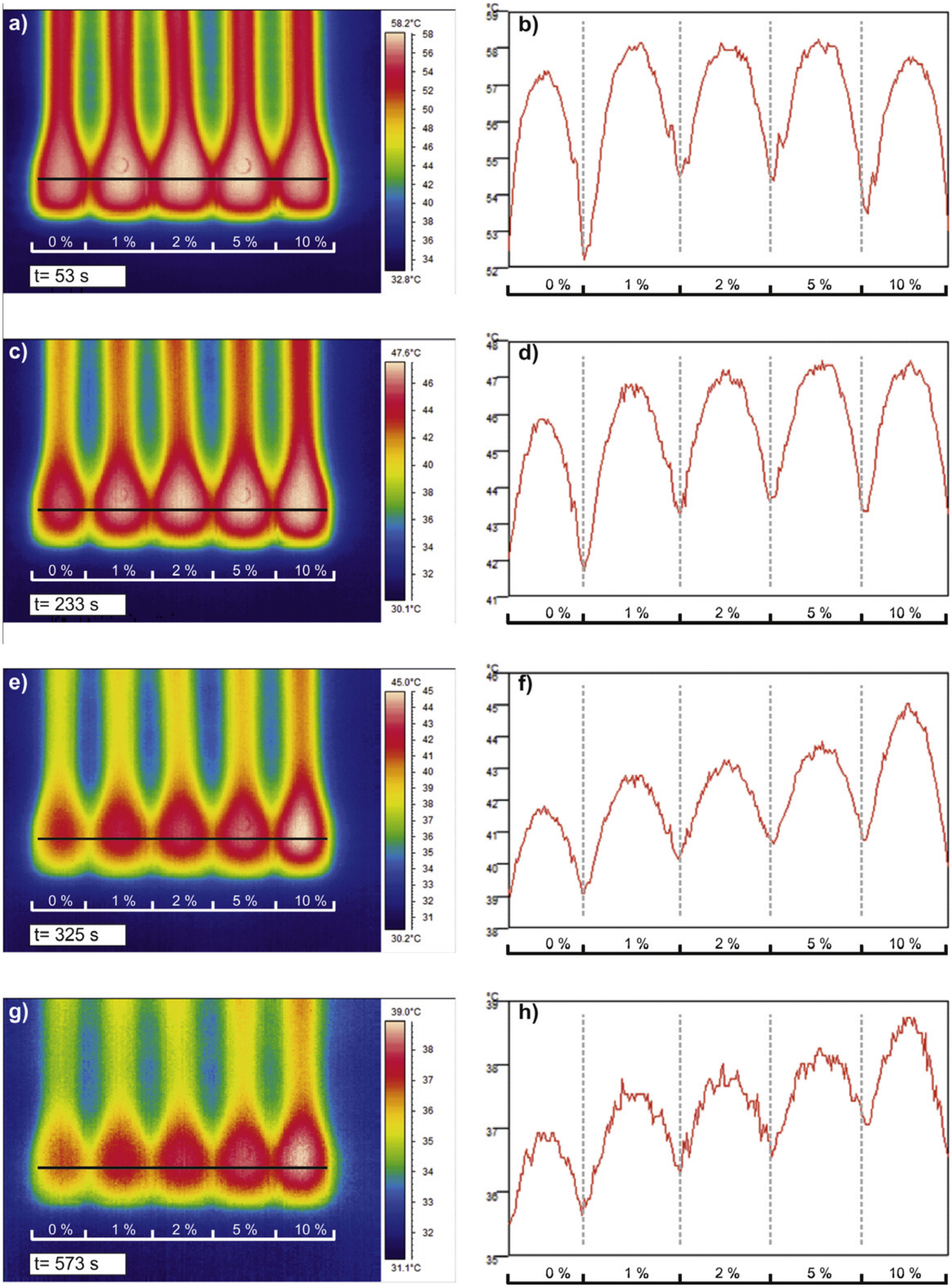

Fig. 8. Infrared thermography images (left) and temperature profile for a marked line (horizontal black line) that crosses all samples (right) at different moments in the cooling process of SEBS–PCM compounds with different PCM amount in the 1–10 wt%. range, (a and b) 53 s, (c and d) 233 s, (e and f) 325 s and (g and h) 573 s.

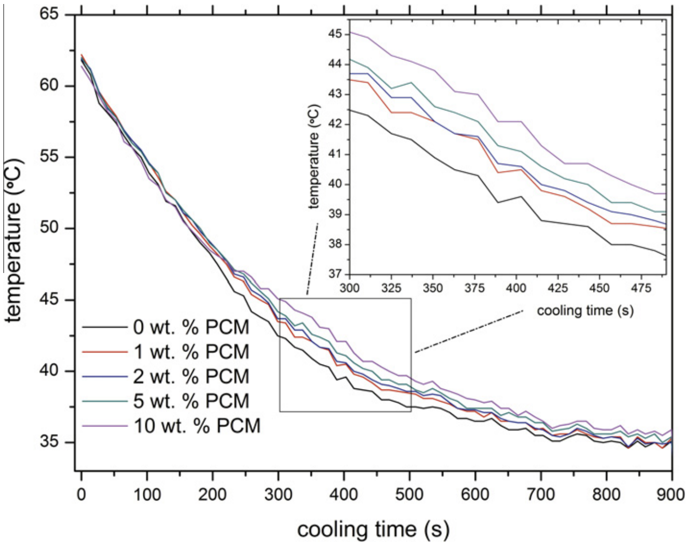

Fig. 9. Cooling curves for SEBS–PCM compounds with different wt.% of PCM microcapsules in terms of the cooling time.

In addition to IRT images, it is possible to observe the thermo regulating effect (thermal inertia) by representing the cooling curve for a surface point in terms of the cooling time. Fig. 9 shows the cooling curve corresponding to the central point located at the base of the sample. At the beginning of the cooling process, around 65 °C, we can see that similar temperatures are obtained for all samples independently of the PCM amount. When the surface temperature decreases below 52 °C, the phase change occurs and PCM's microcapsules release heat which is retained in the polymeric matrix thus having a thermo regulating effect (tendency to maintain temperature invariable), so that the decrease in surface temperature is less pronounced as the PCM amount increases. Furthermore, as the SEBS matrix is characterized by low thermal conductivity, it also contributes to maintaining temperature at high levels, so that, the thermo regulating effect can be observed in a large temperature range below the phase change located at 52 °C. As we move far from the phase change point (lower temperatures), the thermo regulating effect is less pronounced ad we observe a decrease in the temperature gap between the unfilled SEBS matrix and the SEBS–PCM compounds (for all PCMs amounts). In the zoom region of Fig. 9, we can see that the cooling curves are shifted to high temperatures as the PCM content in SEBS–PCM compounds increases.

4. Conclusions

The use of microencapsulated phase change materials (PCMs) is an efficient method to obtain thermo regulating effects in both heating and cooling processes on polymer matrices based on styrene–ethylene/butylene–styrene (SEBS) polymers. Addition of 2–5 wt.% of PCMs (with a phase change point located at 52 °C) leads to good equilibrium of mechanical performance and thermal inertia effects. For these compositions, tensile strength and elongation at break remains at similar values of those of the unfilled SEBS matrix. Scanning electron microscopy (SEM) shows in a clear way the good wetting properties of PCM microcapsules with the polymer matrix, and this has a positive effect on overall mechanical properties. Over 10 wt.% PCM, the mixing process is difficult and great amount of aggregates can be formed and this could lead to stress concentration effects. Compositions with 1 wt.% PCMs are interesting from a mechanical point of view but the thermo regulating effect is very low. On other hand, samples with 10 wt.% PCMs show a great thermo regulating effect but the overall mechanical properties are decreased as a consequence of the stress concentration effects of aggregates. So that, we can conclude that compositions with 2–5 wt.% PCM are interesting for industrial purposes since they offer good equilibrium between mechanical properties and thermal inertia phenomena.

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