



# Development of polystyrene-based films with temperature buffering capacity for smart food packaging



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## ABSTRACT

One of the main factors affecting the quality of perishable products is represented by temperature variations during storage and distribution stages. This can be attained through the incorporation of phase change materials (PCMs) into the packaging structures. PCMs are able to absorb or release a great amount of energy during their melting/crystallization process and, thus, they could provide thermal protection to the packaged food. Thus, the objective of this research was to develop polystyrene (PS)-based multilayer heat storage structures with energy storage and hence temperature buffering capacity for their application in refrigerated foods. To this end, polycaprolactone (PCL) was used as the encapsulating matrix of a phase change material (PCM) called RT5 (a commercial blend of paraffins with a transition temperature at 5 °C), by using high throughput electrohydrodynamic processing. The PCL/PCM fibrous mats were directly electrospun onto PS films and an additional PCL electrospun layer (without PCM) was also deposited in some experiments to improve the overall functionality of the PCM. The attained morphology, thickness, deposition time, temperature and multilayer structure played an important role on the energy storage capacity of the developed PS-based multilayer structures. Results obtained from a differential scanning calorimeter (DSC) show that RT5 can be properly encapsulated inside the PCL matrix and the encapsulation efficiency and, thus, the heat storage capacity was affected not only by the multilayer structure, but also by the storage time and temperature. The thermal energy storage/release capacity was of about 88–119 J/g. As a result, this work demonstrates the potential of these materials for an efficient temperature buffering effect of relevance in food packaging applications, in order to preserve the quality of refrigerated packaged food products.

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## 1. Introduction

Refrigeration plays an important role in food preservation, as low temperatures aid in preventing or slowing microbial, physiological and chemical changes in food produced by microbial and/or enzymatic activity. Temperature fluctuations in the cold chain during the storage and distribution stages have negative effects on food due to crystal ice growth, acceleration of chemical reactions and/or microorganisms growth, which could result in a reduction of quality and may shorten the shelf-life of the food products. Therefore, strategies to buffer potential temperature fluctuations during the commercialization of foods are highly desirable. Packaging can be designed to play an active role to maintain the food temperature within desired limits and, thus, to ensure the quality, safety and increase the shelf-life of the products (James et al., 2006). Usually, the limited thermal insulation and poor

thermal buffering capacity of standard packaging do not provide any protection for maintaining the cold chain. But there are different strategies that could contribute to improving the thermal buffering capacity of a package, being one of them the development of thermal energy storage (TES) structures through the addition of phase change materials (PCMs) (Gin and Farid, 2010; Oró et al., 2012) within the polymeric structures (Oró et al., 2013). This strategy has been recognized as one of the most preferred forms of energy storage, mainly due to the high energy storage density provided by the PCMs and the nearly isothermal heat storage characteristics that can be attained (Melone et al., 2012). Phase change materials (PCMs) are substances that undergo a phase transition at a specific temperature and, as a result, they are able to absorb and release the latent heat with a very small variation in temperature (Jin et al., 2010). PCMs could be used during transport, storage and distribution stages to maintain the cold chain of solid food, beverages, pharmaceutical products, textile industry, blood derivatives, electronic circuits, cooked food, biomedical products and many others (Oró et al., 2012). The most commonly used phase

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change materials are paraffin waxes, fatty acids, eutectics and hydrated salts (Farid et al., 2004). The paraffin compounds fulfill most of the requirements for being used as PCMs, as they are reliable, predictable, non-toxic, chemically inert and stable below 500 °C. They also show little volume changes on melting and have low vapour pressure in the melt form (Sharma et al., 2009). Direct applications of PCMs are difficult since they have weak thermal stability, low thermal conductivity and some of them are liquid at ambient temperature and, thus, are not easy to handle or to be directly incorporated into packaging structures (Fang et al., 2009). Therefore, the encapsulation of the PCM in a shell material is a plausible solution to avoid all these problems. Microencapsulated PCM is a form of PCM encapsulated in natural or synthetic polymeric capsules, which range in size from less than 1 µm to more than 1000 µm. The microcapsules protect the PCM against the influences of the outside environment, increasing the heat-transfer area, and permitting the core material to withstand changes in volume of the PCM, as the phase change occurs allowing small and portable thermal energy storage (TES) systems (Alkan et al., 2011).

One technique increasingly being used for the microencapsulation of materials is electrohydrodynamic processing. This technique that comprehending electrospinning and electrospraying has proven to be a suitable method for the encapsulation of several components, including biomedical substances, functional food ingredients, PCMs and others substances within polymer matrices (Goldberg et al., 2007; Perez-Masia et al., 2013). Electrospinning is a technique whereby long non-woven ultrafine fibres with diameters of several tens to several hundreds of nanometers may be formed by applying a high-voltage electric field to a polymeric solution (Teo and Ramakrishna, 2006). As a result of the applied electric field, a polymer jet is being ejected from the tip of a capillary through which a polymer solution is pumped, accelerated towards a grounded target and deposited thereon (Arecchi et al., 2010).

The aim of this work was, on the one hand, to develop heat storage materials by means of incorporation of a PCM substance with a first order thermal transition at 5 °C (RT5) in polystyrene (PS) films and, on the other hand, to study the effects of storage temperature and ageing on the performance of these films. It is important to understand how these materials will behave as a function of temperature and time, since these encapsulated structures are aimed to be introduced into packaging structures, in order to counteract temperature fluctuations and better maintain the cold chain, respectively.

## 2. Materials and methods

### 2.1. Materials

Rubitherm RT5, a technical grade paraffin wax consisting of a blend of normal paraffin waxes (C14–C18), was purchased from Rubitherm Technologies GmbH (Berlin, Germany). Polystyrene (PS) commercial grade foam was supplied by NBM (Valencia, Spain). The Polycaprolactone (PCL) grade FB100 was supplied by Solvay Chemicals (Belgium). N,N-dimethylformamide (DMF) with 99% purity and trichloromethane (99% purity) were purchased from Panreac Quimica S.A. (Barcelona, Spain). All products were used as received without further purification.

### 2.2. Preparation of polystyrene-based multilayer structures

#### 2.2.1. Preparation of polystyrene films

For obtaining the polystyrene (PS) films, pellets of PS were first melt-mixed by means of a mixer (Plastograph®, Brabender®,

Germany) at 175 °C and 100 rpm for 3 min. Once the melted product was obtained, films were prepared by compression-moulding using a hydraulic press (Carver 4122, USA), at 175 °C and 60,000 psi of pressure during 3 min.

#### 2.2.2. Preparation of PCL/PCM electrospinning solution

The electrospinning solution was prepared by dissolving the required amount of PCL, under magnetic stirring, in a solvent prepared with a mixture of trichloromethane:N,N-dimethylformamide (70:30 w/w) in order to reach a 13% in weight (wt.%) of PCL. Afterwards, 45 wt.% of PCM (Rubitherm 5) with respect to the polymer weight was added to the polymer solution, and stirred at room temperature until it was completely dissolved.

#### 2.2.3. Electrospinning process

The full process of PCM encapsulation via electrohydrodynamic processing is a proprietary method previously described (patent application number: P201131063). According to this process, PCL/PCM solutions were prepared as described in Perez-Masia et al. (2013), in order to produce fibrillar structures. Thus, the PCM was submicroencapsulated within polycaprolactone (polymer matrix) by means of a multinozzle high throughput Fluidnatek® electrohydrodynamic pilot processing tool commercialized by Bioinicia S.L. (Valencia, Spain). This tool is equipped with a variable high-voltage 0–60 kV power supply. The PCL/PCM solution was electrospun under a steady flow-rate using a multinozzle injector. The electrospinning conditions for obtaining PCM-containing polymer structures was optimized and fixed at 55 ml/h of flow-rate, tip-collector distance of 26 cm and the voltage of the collector and injector were set at 26 kV and 22 kV, respectively. The PCL/PCM nanofibres were directly electrospun over the polystyrene film. Two different deposition times (15 and 45 min) were evaluated in the PS-multilayer structures to see how deposition time affected thermal, tensile, optical and barrier properties. A second series of PS-multilayer systems were prepared by electrospinning a PCL layer (15 min) onto the electrospun PCL/PCM layer in order to assess the protection of this additional layer on the heat storage capacity.

#### 2.2.4. Preparation of polystyrene multilayer structures

Once the PCL/PCM and/or PCL nanofibres were collected onto the polystyrene compression moulded films, a curing step at 60 °C for 1 min was applied in an oven to enhance adhesion.

#### 2.2.5. Polystyrene multilayer conditioning and storage

Samples were equilibrated in desiccators stored at 0% RH by using silica gel and at two different temperatures (4 and 25 °C) for three months. Polystyrene multilayers were taken from the desiccators at different time intervals (0, 7, 15, 30, 45, 60 and 90 days) and analysed by DSC and FTIR.

## 2.3. Characterization of polystyrene PS-multilayer structures

### 2.3.1. Scanning Electron Microscopy (SEM)

The morphology of polystyrene multilayers structures was examined using SEM on a Hitachi microscope (Hitachi S-4100). Samples were frozen in liquid N<sub>2</sub> and cryofractured to observe the cross-section of the samples. Then, they were fixed on copper stubs using double side adhesive tape, coated with a mixture of gold–palladium, and observed using an accelerating voltage of 10 kV.

### 2.3.2. Differential Scanning Calorimetry (DSC)

Thermal analyses of PS multilayer structures were carried out on a DSC-7 calorimeter (Perkin Elmer Inc., USA) from –20 to 20 °C in a nitrogen atmosphere using a refrigerating cooling

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