Preparation, Properties and Encapsulation of High Thermostability Phase-change Material

Guo-Qing Zhang\textsuperscript{a}, Chang-Wei Cai\textsuperscript{a}, Guo-Cheng Zhu\textsuperscript{a}, Lan Zhou\textsuperscript{a}, Guo-Jin Liu\textsuperscript{a}, Jiri Militky\textsuperscript{b}, Jaromir Marek\textsuperscript{c}, Mohanapriya Venkataraman\textsuperscript{b}, Ju-Ming Yao\textsuperscript{a,*}

\textsuperscript{a}Key Laboratory of Advanced Textile Materials and Manufacturing Technology, Ministry of Education, Zhejiang Sci-Tech University, Hangzhou 310018, China
\textsuperscript{b}Department of Material Engineering, Technical University of Liberec, Studentská 1402/2, Liberec, 46117, Czech Republic
\textsuperscript{c}Institute for Nanomaterials, Advanced Technologies and Innovations, Technical University of Liberec, Studentská 1402/2, Liberec, 46117, Czech Republic

Abstract

Microcapsules containing phase change materials (PCM) have been attracting much attention due to their applications in many energy storage fields. However, most PCM microcapsules have relatively low thermostability with an onset decomposition temperature of about 150 °C, which to some extent restricts their applications. In this study, high chain ester of dodecanol laurate was first synthesized with raw materials of 1-dodecanol and lauric acid by esterification reaction under catalysis, and then the ester as core material was encapsulated using PMMA by emulsion polymerization. The resultant products, including the ester and the PCM microcapsules, were respectively characterized by using infrared spectroscopy (IR), differential scanning calorimeter (DSC), thermogravimetry (TG), laser particle size analyzer and scanning electron microscope (SEM). The synthesized dodecanol laurate have a high purity according to IR spectrum analysis and suitable phase temperature range of 22-30 °C from DSC measurement. In addition, the ester also showed good thermal properties with a latent heat of 206 J/g, small super-cooling degree of 0.5 °C and high thermal evaporation temperature of 220 °C, which would be very suitable for application in PCM energy storage materials. Using the above ester as core material, the PCM microcapsules were successfully fabricated by emulsification and polymerization processes. The prepared microcapsules showed perfect spherical shape with size about 865 nm and high heat storage performance with a latent energy of 118 J/g. Owing to high evaporation temperature of ester core material itself and further encapsulation, the prepared PCM microcapsules showed higher thermostability. TG results suggested that the microcapsules had a high onset weightless temperature which was over 252 °C, it was a significant increment comparing to those PCM microcapsules reported by most literatures. Moreover, thermo-regulating cotton fibers were fabricated by using the above PCM microcapsules. It’s seen that the PCM microcapsules deposited on the fibers uniformly and the fibers had a latent heat of 20.18 J/g.

Keywords: Esterification; PCM Microcapsules; Thermostability; Latent Heat
1 Introduction

In recent decades, the use of phase change materials (PCMs) for thermal energy storage has attracted extensive attention owing to increasing energy consumption and environment problems [1]. Because they can store and release great amounts of energy when undergoing phase change process, PCMs showed great potential in applications for solar energy storage, thermo-sensors, food and medicine package and intelligent textiles or fabrics, etc [2-5]. Especially in smart textiles, since early 1980s PCMs were first used in astronaut suits to against the extreme temperature fluctuations in outer space [6, 7], thermo-regulating textiles have been successfully developed for they are able to create a much more effective barrier than conventional textiles to protect the human body from harsh conditions.

During the processing and usage of the thermo-regulating textiles, it is very important to prevent leakage of the PCMs in the phase change process. After much researches and practices, microencapsulation of PCMs is regarded as a considerably feasible solution [8, 9]. A lot of studies have been performed on microencapsulation of PCMs using some polymers or inorganic materials as shell [10-14]. The resultant microcapsules or its emulsion can be used to fabricate the temperature-regulating textiles by finishing or solution spinning.

With increasingly application fields, thermal properties of the PCM microcapsules, especially thermostability, has become an important criterion. In fact, many synthesized PCM microcapsules show lower onset decomposition temperature, which limited the use of the energy storage systems. A lot of researches reported the preparation and properties of the PCM microcapsules with paraffin or fatty acids as core and polymer or SiO$_2$ as shell. However the thermogravimetric testing results suggest that the volatilization of n-octadecane from the capsules starts before 150 $^\circ$C [15-24]. As well known, most fabrics will undergo heat treatment under temperature of over 150 $^\circ$C during finishing, which leads to remarkable loss of PCM from microcapsule.

The low thermostability of PCM microcapsules significantly cannot meet the high temperature heat treatment of textiles, the main reason for the poor thermostability of the PCM microcapsules is due to the lower thermal volatilization temperature of PCMs. So it is necessary to further develop new high thermostability PCMs. Up to now, lots of PCMs have been discovered, but very few of them can be used for textile applications since the PCMs in textiles are required to have suitable temperature range, high latent heat capacity, stable chemical property, to be non-toxic and comfort. According to the above requirements, a few kinds of PCMs, such as paraffin, fatty acid and alcohol or their eutectics, are widely used in the fabrication of thermo-regulating textiles. Among these organic PCMs, fatty acids are preferred due to theirs high latent heat, broad phase change temperature range and low cost. But most of fatty acids including their eutectics also show low heat volatility temperature of about 140 $^\circ$C.

Recent years, fatty acid esters, as new PCMs, gradually draw many researchers’ attention because of their high latent heat and thermostability. Most fatty acid esters have an onset decomposition of above 200 $^\circ$C due to their higher chain length comparing to relevant fatty acid, which is significantly more suitable for those high temperature application conditions.

The objective of this work is dedicated to fabricate the high thermostability fatty acid ester and further encapsulation with it as core material. It is expected the prepared PCM microcapsules have good thermostability, high latent heat and small size to meet the application in the solution spinning. For the fabrication of thermo-regulating fibers, solution spinning is currently the most feasible method. In general, the thermo-regulating fibers can be prepared by adding