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Saturated linear diesters from stearic acid as renewable phase change materials



Michael C. Floros, Suresh S. Narine*

Trent Centre for Biomaterials Research, Departments of Physics & Astronomy and Chemistry, Trent University, Peterborough, ON, Canada K9J 7B8

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ABSTRACT

Renewable phase change materials have been gaining traction as a 'green' solution to store excess energy and therefore improving energy use efficiency. A series of diesters were synthesized in a solvent-free, melt condensation synthesis from the fatty acid derivative stearoyl chloride, and dialcohols between 2 and 10 carbons in length (18-*n*-18 diesters where *n*=2, 4, 6, 8 and 10). Characterization by FTIR, ¹H NMR, DSC and TGA reveals significant changes to the thermal performance and stability of these materials in the presence of contaminants. This may explain the poor performance previously reported in literature for this type of material. These diesters have phase change enthalpies between 230 and 247 J/g, elucidated for the first time, and all melt above 65 °C, with a single, narrow melting and crystallization point, contrary to what was previously reported.

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1. Introduction

Phase change materials (PCMs) are designed to store and release thermal energy by exploiting their melting/crystallization temperatures, and the energy of a latent heat transition. To achieve this, these compounds should undergo phase change over a narrow temperature range, and have a high enthalpy of phase change. PCMs have recently attracted significant attention for improving the efficiency of processes where thermal losses reduce efficiency and increase cost. These processes include power generation, heating and cooling of buildings, green houses, solar energy capture, and off-peak energy storage [1]. Currently, the most common materials used as PCMs are based on salts or paraffin waxes, neither of which is renewable [2].

In this work, we revisit a series of renewable, lipid based diesters of stearic acid with even carbon length dialcohols for use as PCMs. Synthesis of these materials for use as PCMs has already been reported within the literature, but in all instances the melting temperatures and enthalpies reported were much lower than those found in the present work [3–5]. We propose that the discrepancy lies in the purity of the PCMs, and present a simple, low cost method for rapid purification, allowing these PCMs to perform at their optimal levels. These PCMs possess superior

enthalpies compared to paraffin wax – the most commonly used organic PCM [6].

2. Materials and methods

All materials were purchased from Sigma-Aldrich and used as received. Purities for all materials were at least 98%, with the exception of stearoyl chloride (97%). The dialcohols used to synthesize the diesters of the form 18-*n*-18 were ethylene glycol (*n*=2), 1,4-butanediol (*n*=4), 1,6-hexanediol (*n*=6), 1,8-octanediol (*n*=8) and 1,10-decanediol (*n*=10).

A 3-neck flask containing approximately 10 g stearoyl chloride was flushed with nitrogen. About 0.5 equivalents of dialcohol were added to give a 1:1 ratio of alcohol to acid chloride groups. The flask was stoppered and a gentle stream of nitrogen was initiated. The outlet gas was bubbled through a saturated solution of sodium bicarbonate in water to neutralize the liberated hydrogen chloride. The reaction mixture was heated to 90 °C with stirring and held at this temperature for 6 h, after which the reaction was complete. The unpurified reaction mixture was then recrystallized from a hot mixture of anhydrous ethanol and acetone (1:1 by volume) to yield the pure diester. The use of ethanol ensured no unreacted acid chloride remained in the product. Yields were greater than 90 % for each diester.

Structures were confirmed by ¹H NMR recorded on a Varian Unity-INOVA at 499.695 MHz in CDCl₃ (Agilent Technologies, Santa Clara, CA, USA). ATR-FTIR spectra were collected using a Thermo

* Corresponding author. Tel.: +705 748 1011; fax: +705 750 2786.

E-mail address: sureshnarine@trentu.ca (S.S. Narine).

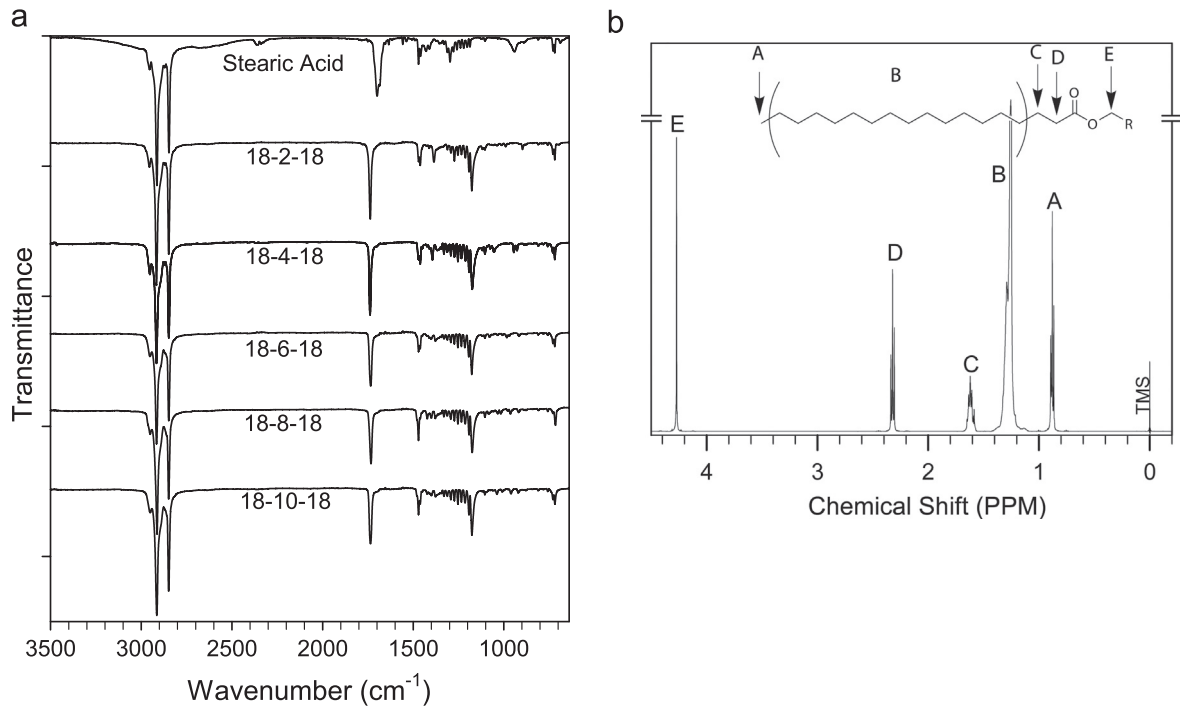


Fig. 1. (a) Stacked FTIR and (b) ^1H NMR of 18-2-18.

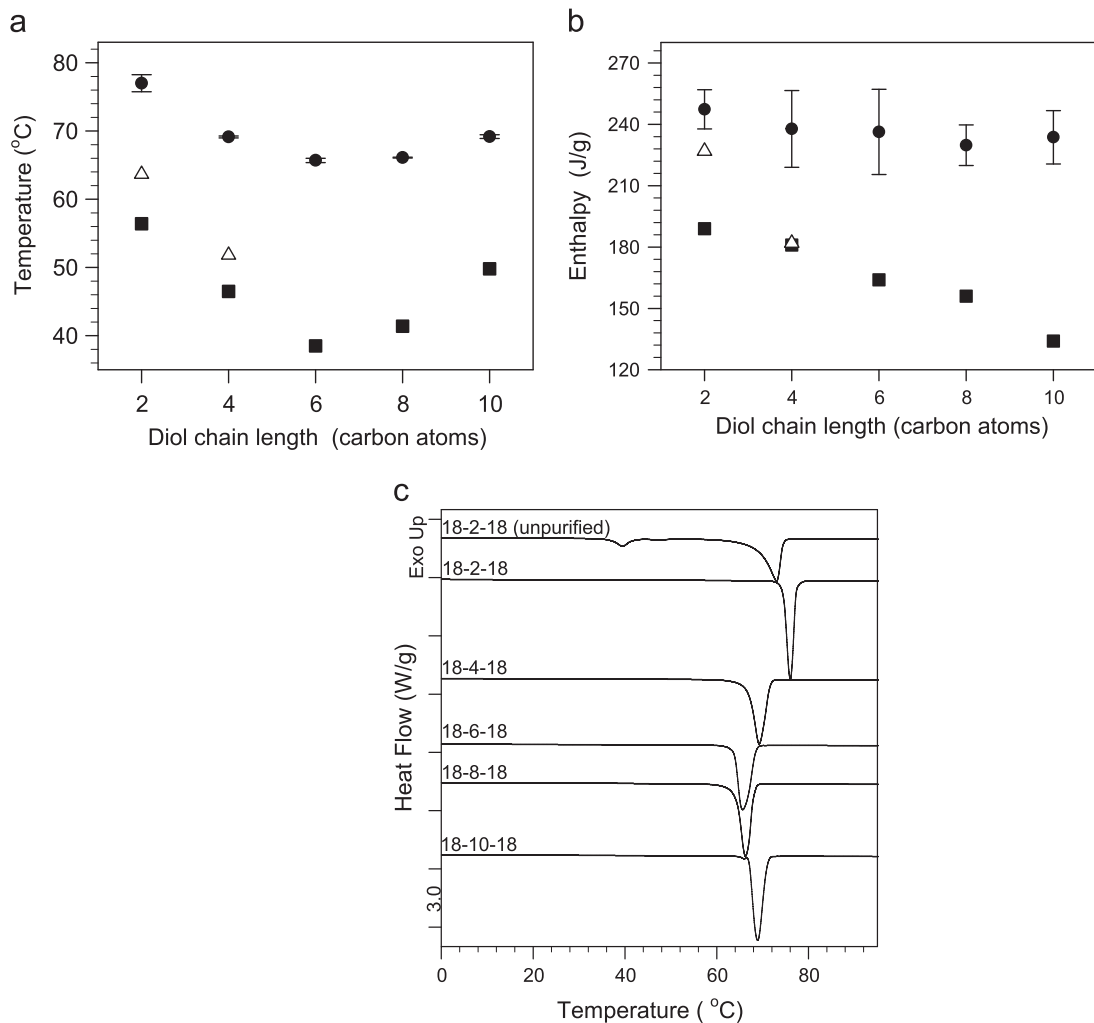


Fig. 2. Thermal characteristics showing (a) peak melting point, and (b) enthalpy of fusion for 18- n -18 saturated diesters, where n =diol chain length. Crystallization (not shown) and fusion enthalpies were not statistically different. Symbols are as follows: ●, this work; ■, ref [3]; ▲, ref [4,5]. Error bars represent standard deviations from triplicates. (c) DSC heating stack showing purified diesters and unpurified 18-2-18 for comparison.

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