

# Hexamethylene dilauroyl, dimyristoyl, and dipalmytoyl amides as phase change materials for thermal energy storage

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## Abstract

Hexamethylene dilauroyl, dimyristoyl, and dipalmytoyl amides have been produced as solid–liquid phase change materials via condensation of hexamethylene diamine with the respective acyl chlorides (lauroyl chloride, myristoyl chloride, and palmytoyl chloride) and were characterized by FT-IR, NMR, DSC, and TG analysis. Hexamethylene dilauroyl, dimyristoyl, and dipalmytoyl amides crystallized due to structural symmetry and flexibility of long alkyl groups. They were characterized by DSC and FT-IR spectroscopy before and after thermal cycling to determine their thermal reliability. Phase change enthalpies were found 110.1 and  $-103.3 \text{ J g}^{-1}$  for hexamethylene dilauroyl amide (*N,N'*-hexamethylene didodecanamide), 116.9 and  $-110.4 \text{ J g}^{-1}$  for hexamethylene dimyristoyl amide (*N,N'*-hexamethylene ditetradecanamide), and 144.5 and  $-140.5 \text{ J g}^{-1}$  for hexamethylene dipalmytoyl amides (*N,N'*-hexamethylene dihexadecanamide) by DSC. The endurance of hexamethylene dilauroyl, dimyristoyl, and dipalmytoyl amides was studied by TG analysis.

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## 1. Introduction

Phase change materials are organic or inorganic materials able to store or release thermal energy under controlled circumstances. Typically these materials should have a large melting latent heat, high thermal conductivity, a melting temperature in the desired working range, melt congruently with no subcooling, be low in cost, chemically stable, non-toxic and non-corrosive. Most organic Phase Change Materials (PCMs) like paraffins, fatty acids, low melting esters, and polymers corresponds to majority of these properties. Latent heat storage is a relatively new area of research and pioneered by Dr. Telkes in the 1940s (Lane, 1983). It did not receive much attention, however, until the energy crisis of the late 1970s and early 1980s.

The use of PCMs is an effective way of storing thermal energy like solar energy and industrial waste heat due to the advantages of high thermal storage density and a smaller temperature difference between storing and releasing heat (Sharma and Sagara, 2005).

Synthetic PCMs have attracted considerable attention (Zalba et al., 2003; Arndt et al., 1984; Farid et al., 2004; Alkan, 2006; Sarier and Onder, 2007; Li and Ding, 2007a,b) for the past decades. The PCMs have a lot of applications as solar energy storage, waste heat recovery, smart air-conditioning buildings, temperature-adaptable greenhouses, electric appliances with thermostatic regulator, energy storage kitchen utensil, and insulation clothing (Gschwander et al., 2005; Liu et al., 2006; Su et al., 2005; Chen et al., 2007; Wang et al., 2008).

Fatty acids are one of the commonly used solid–liquid phase change heat storage material with excellent heat storage density and low price in spite of their poor thermal stability, corrosivity, bad odor, and especially sublimating during heating. There are also series of different derivatives

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of fatty acids synthesized to obtain PCMs having more preferable properties (Suppes et al., 2003).

In this study, synthesis and determination of thermal performance of a series of novel phase change materials by condensation of some of fatty acid chlorides with hexamethylene diamine compound was aimed. The resultant materials were with the amide functionality and had a probably different potential application range.

Hexamethylene diamine (1,6-hexanediamine) is produced from adiponitrile and used widely in chemical industry. Its main uses are as a raw material in the production of nylon polymers, hexamethylene diisocyanate for use as monomer feedstock in polyurethane production, and cross-link in epoxy resins. It is chemically and thermally stable at room temperature. The fatty acid chlorides were lauroyl chloride, myristoyl chloride, and palmitoyl chloride synthesized from lauric acid, myristic acid, and palmitic acid, respectively. The synthesized hexamethylene dilauroyl, dimyristoyl, and dipalmitoyl amides were called as *N,N'*-hexamethylene didodecanamide, *N,N'*-hexamethylene ditetradecanamide, and *N,N'*-hexamethylene dihexadecanamide according to IUPAC nomenclature. The selection of the fatty acids was performed to get a series of materials working in different temperature ranges.

In order to investigate the thermal properties of hexamethylene dilauroyl, dimyristoyl, and dipalmitoyl amides, Differential Scanning Calorimetry (DSC) measurements were conducted before and after thermal cycling tests. It provides information for the suitability of the materials to be used as solid–liquid PCMs. Structural stability of

the hexamethylene dilauroyl, dimyristoyl, and dipalmitoyl amides were checked using Fourier Transform Infrared (FT-IR) Spectroscopy taken before and after thermal cycling. The thermal endurance of the materials was determined by Thermal Gravimetric (TG) analysis.

## 2. Experimental procedures

### 2.1. Materials

Hexamethylene diamine, sulphurous oxychloride, toluene, lauric acid, myristic acid, palmitic acid, and pyridine are all of Merck reagent grade. Except  $\text{SOCl}_2$ , all materials were used with further purification to remove trace water.

### 2.2. Synthesis of hexamethylene dilauroyl, dimyristoyl, and dipalmitoyl amides

A four-necked flask equipped with an agitator, a thermometer and a condenser was charged with the calculated amount of one of fatty acids (lauric, myristic and palmitic acids), sulphurous oxychloride, pyridine and toluene (the molar ratio of fatty acid/sulphurous oxychloride is 1/1). The flask was immersed to an oil bath at 50 °C for 2 h, and then the toluene solution of hexamethylene diamine with the molar ratio 2/1 of fatty acid/hexamethylene diamine was dropped to the vigorously mixing reaction vessel and the reaction was continued for another 4 h to ensure completeness. After the reaction was over, a milky solid product was obtained by removing low-weight ingredients

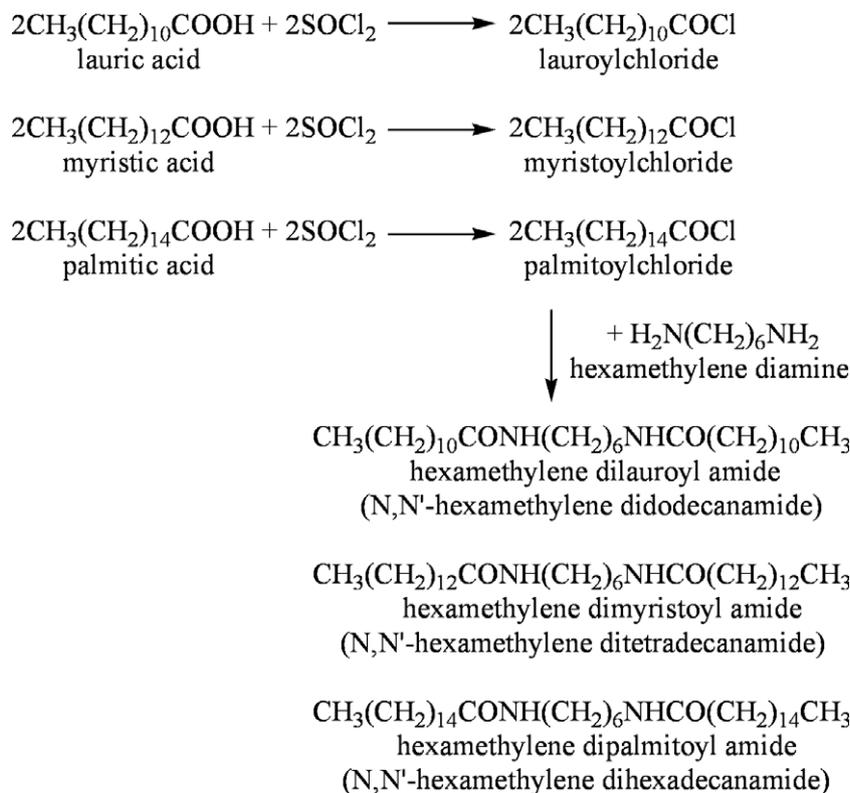


Fig. 1. Synthetic scheme for the preparation of hexamethylene dilauroyl, dimyristoyl, and dipalmitoyl amides.

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