

# Hexadecanol/phase change polyurethane composite as form-stable phase change material for thermal energy storage



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## ABSTRACT

A novel phase-change composite based on hexadecanol and dye-linked polyurethane (dye-PU) was obtained in the present paper. Hexadecanol functions as a phase-change material (PCM) while dye-PU functions as both a supporting material and PCM, unlike in previous phase-change composites where it is used only as a general supporting material. FT-IR, XRD, DSC, and TG were employed to determine the structure and basic properties of the composite. The phase change enthalpy of the composite reached 229.5 J/g, which was higher than that reported for most form-stable PCMs. The maximum weight percentage of hexadecanol that could be added to the composite without leakage was 63.8%. The linked dye absorbed visible light and converted it into thermal energy that could be stored by the phase-change material.

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## 1. Introduction

Thermal utility is an effective system of solar energy radiation and presents great significance in energy savings and environmental protection [1–5]. In particular, phase-change materials (PCMs) are key components of promoting the efficient and versatile use of solar energy [6–10]. Among the various PCMs available, form-stable PCMs have highly promising applications because of their excellent form-stable effect during phase transition [11–15]. Thus, development of form-stable PCMs is a popular research topic [16–21]. Photothermal conversion for thermal utility is another key approach to harness solar energy because of the low thermal efficiency of visible light, which accounts for approximately 40% of solar radiation, impedes direct or effective application [22,23]. To resolve this problem, direct collection and conversion of solar radiation into thermal energy for use in various technical applications has received significant attention [24–30]. In previous studies, we synthesized novel visible light-driven PCMs by integrating dye molecules into a poly(ethylene glycol) matrix to absorb visible light and convert it into heat [31–34]. At room temperature, dye-PU has the same crystal form with PEG. Under light irradiation, linked dye effectively absorbed visible light and then converted it to thermal energy through non-radiation thermal decay, raising the temperature. When the temperature reached the phase change temperature, the PEG crystalline domains converted to an amorphous liquid state at the microlevel.

Hard segment in dye-PU prevented the flow of PEG and maintained the dye-PU a solid state, thus dye-PU finished a form stable phase transition. This integration showed great potential for further development.

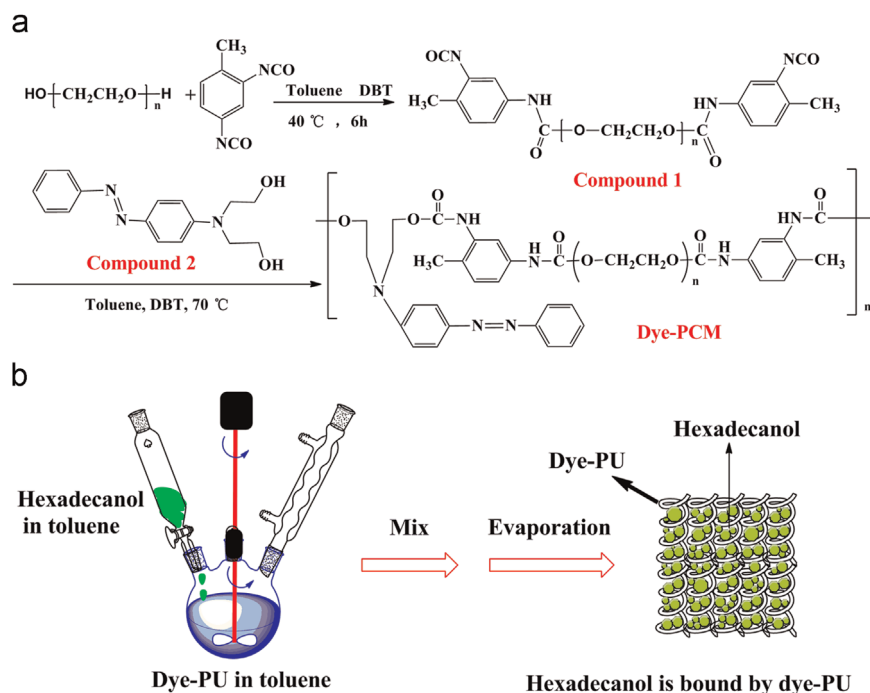
In the present work, we report the synthesis of novel hexadecanol/dye-polyurethane (dye-PU) composite by integrating hexadecanol into a visible light-driven PCM matrix (Scheme 1). The dye-PU functions as light-to-heat converter, supporting material and PCM, unlike in previous phase-change composites where it is used only as a general supporting material. Besides photothermal conversion and form-stable phase transition properties, the novel hexadecanol/dye-PU composite presents many other advantages compared with previously reported materials, including a high phase-change enthalpy and suitable phase change temperature. While the phase-change enthalpy of the composite was as high as 229.5 J/g, which was much higher than that reported for previous PCMs, one of the phase-transition temperatures was as low as 40 °C. These characteristics reveal the suitability of the composites for application in textile products, telecommunications, microprocessor equipment, and construction.

## 2. Experimental

### 2.1. Materials

Technical-grade hexadecanol was supplied by Bosheng Technology Ltd. (Guangzhou, China). Analytical-grade polyethylene glycol (PEG,  $M_n = 10,000$ , Shanghai National Medicines Co. Inc.,

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**Scheme 1.** Acquisition of the phase change composite.

China) and dye A [31] (2,2'-((4-(phenyldiazenyl) phenyl) azanediyl) diethanol) were dried at 80 °C under high-vacuum conditions (−0.1 kPa) for 48 h. Analytical-grade toluene (Tianjin National Medicines Co. Inc., China) was dried using a 5 Å molecular sieve and then distilled prior to use. Analytical-grade toluene-2, 4-diisocyanate (TDI, Tianjin Kemiu Chemical Reagent Co. Inc., China), and dibutyltin dilaurate (DBT, Tianjin Kemiu Chemical Reagent Co. Inc.) were used as received. All other reagents employed in this work were of analytical grade.

## 2.2. Preparation of composites

The synthetic route for the hexadecanol/dye-PU composite is illustrated in Scheme 1. First, the dye-linked phase-change PU was synthesized in a flame-dried glassware through a step-condensation reaction under an inert nitrogen ( $\text{N}_2$ ) atmosphere. Predetermined amounts of dried PEG10000, TDI, and DBT (used as catalyst) in freshly distilled toluene were mixed and stirred for 6 h under  $\text{N}_2$  at 40–45 °C to produce an NCO-terminated prepolymer (Compound 1). A stoichiometric amount of dye A (Compound 2) was added to this mixture, and the reactions were continued for 6 h at 100 °C. Next, a specific amount of hexadecanol and a known concentration of the reaction liquid obtained above were mixed. The solution containing the resultant hexadecanol/dye-PU composite was subsequently vacuum-evaporated and then dried for 48 h at 80 °C under vacuum (−0.1 kPa) prior to testing to produce the final hexadecanol/dye-PU composite.

## 2.3. Characterization

The Fourier-transform infrared (FT-IR) spectra of the samples were obtained using a NICOLET-460 FTIR spectrophotometer. The crystalline products were studied using a D/Max2400 (Rigaku, Japan) from 5 °C to 90 °C at room temperature. The thermal properties of form-stable hexadecanol/dye-PU composite (e.g., melting and crystallizing points and latent heats) were measured through the DSC technique using a 910S system (TA Instruments, USA). Analyses were performed at 5 °C/min heating rate under a constant stream of  $\text{N}_2$  and flow rate of 20 mL/min. Methods for the light irradiation experiments are reported

in our previous study [31]. The solar simulator used was a CHF-XM-500W system (Changkuo Technology, Beijing, China).

## 3. Results and discussion

### 3.1. Synthesis and characterization of hexadecanol/dye-PU composite

Scheme 1 shows a schematic of the preparation route for hexadecanol/dye-PU composite. First, dye-PU was synthesized through two-step polymerization from PEG 10000, TDI, and dye A (Scheme 1a) in toluene, and the peaks at 8.91–8.99 ppm in the  $^1\text{H}$  NMR (Fig. S1 in the Supporting information) of dye-PU corresponded to the proton peak in the  $-\text{CO}-\text{NH}-$  group, which showed that the polymerization of TDI with PEG and dye A took place. The solution of hexadecanol in toluene was subsequently added to the dye-PU solution, and the hexadecanol/dye-PU composite was obtained by evaporating the solvent (Scheme 1b).

The FT-IR spectra of hexadecanol, dye-PU, and the hexadecanol/dye-PU composite are shown in Fig. 1. In the spectrum of dye-PU (Fig. 1a), the peak at  $3308\text{ cm}^{-1}$  corresponds to the N–H stretching vibrations of  $-\text{CONH}-$ , the peaks observed at  $2848$  and  $2916\text{ cm}^{-1}$  represent the C–H stretching vibrations of  $-\text{CH}_2$  and  $-\text{CH}_3$ , respectively, and the peak at  $1725\text{ cm}^{-1}$  is attributed to C=O stretching vibrations. The peak detected at  $1599\text{ cm}^{-1}$  reveals the C=C stretching vibration bands of the benzene ring. In the spectrum of hexadecanol (Fig. 1c), the peak at  $3303\text{ cm}^{-1}$  corresponds to H–O stretching vibrations of hexadecanol, the peaks observed at  $2847$  and  $2915\text{ cm}^{-1}$  represent the stretching vibrations of  $-\text{CH}_2$  and  $-\text{CH}_3$ , respectively, the peaks at  $1471$  and  $1462\text{ cm}^{-1}$  result from the deformation vibrations of  $-\text{CH}_3$  and  $-\text{CH}_2$ , respectively, and the peak appearing at  $1058\text{ cm}^{-1}$  reveals C–O stretching vibrations. In the spectrum of hexadecanol/dye-PU composite (Fig. 1b), only the characteristic peaks of both hexadecanol and dye-PU may be seen. These results confirm the physical complex resulting from combination of hexadecanol and PU.

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