Temperature-sensitive Glass Containing Non-polymeric N-isopropylacrylamide Aqueous Solutions

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Abstract. This paper reported a new kind of temperature-sensitive glass containing non-polymeric N-isopropylacrylamide (NIPAM) aqueous solutions, determined by transmittance spectrum and Fourier transform infrared (FT-IR) spectroscopy. It was found that the NIPAM monomer aqueous solutions showed thermal properties as well as poly N-isopropylacrylamide (PNIPAM). The concentration of NIPAM monomer aqueous solutions is a key factor. Critical temperature of light transmission ranges from about 30°C to 55°C under atmospheric pressure, which make it simple for different applications. The critical temperature (CT) of light transmission decreases, while the concentration increasing. The double-layer hollow quartz glass filled with non-polymeric NIPAM aqueous solutions is a recyclable and green material, and will be beneficial for various applications.

Introduction

Stimuli-responsive polymers and hydrogel show their responsive properties upon change in environmental conditions, for example, temperature, pH, light or electric field [1-5]. Among all the stimuli-responsive polymers, poly N-isopropylacrylamide (PNIPAM) is one of the most investigated thermos-responsive polymers [6], which exhibits a phase transformation at a certain temperature called lower critical solution temperature (LCST) [7]. LCST of PNIPAM differs from other thermo-responsive polymers, as it is independent of the molecular weight and the concentration [8]. However, sometimes it requires different LCST for various applications, and many researchers have done lots of work upon that [9-10].

Here we show for the first time that non-polymeric N-isopropylacrylamide (NIPAM) aqueous solutions were found to undergo phase transformation. The results demonstrate the relationship between LCST of non-polymeric NIPAM monomer aqueous solutions and its concentration. Since there is no polymeric progress in the experiment, the property of NIPAM does not change at all, which makes it a recyclable and green phase-transformation material. Controllable LCST will be beneficial for various applications, and the investigation of the non-polymeric NIPAM will have some reference for the fundamental research of PNIPAM.

Experiment Details

The NIPAM monomer aqueous solution is filled in the double-layer hollow quartz glass, as shown in Figure 1. NIPAM monomer (Sigma-Aldrich Co.) aqueous solutions with different concentrations were prepared by dissolution in distilled water. The phase transformation was traced by monitoring the transmittance of deuterium-tungsten light source through double-layer hollow quartz glass on spectrometers (HR4000, Ocean Optics Co.). The quartz glass were placed on the holder...
(CUV-UV-FL, Ocean Optics Co.) and filled with NIPAM monomer aqueous solutions. The temperature was controlled by heating slowly and cooling naturally.

Figure 1. Experimental setup of temperature-sensitive glass (1- quartz glass; 2- NIPAM monomer aqueous solutions).

The experiment started with 20.0 wt. % NIPAM monomer aqueous solutions, while at room temperature and under atmospheric pressure, NIPAM monomer aqueous solutions with higher concentration was saturated and precipitated crystal. On the other hand, NIPAM monomer aqueous solutions with concentration below 15.0 wt. % couldn’t show the phenomenon of phase transformation. Thus the concentrations of aqueous solutions in the experiments were chosen from 15.0 wt. % to 20.0 wt. % accordingly.

NIPAM monomer aqueous solutions with different concentrations were heated slowly and cooled naturally, and the spectra of various temperatures were recorded. The environment temperature was 18.0°C, and the experiment temperature could be perfectly controlled.

Figure 2 shows the experimental setup. Using deuterium tungsten light source, 200-410nm (deuterium lamp) and 360-2000nm (tungsten lamp), collecting light signal by fiber grating spectrometer (Ocean Optics Company HR4000 and S02827). The light transport through optical fiber and quartz cuvette into the spectrometer. Spectra of various temperatures were recorded by the computer.

**Result and Discussion**

The experiment started with 19.0% (wt.) NIPAM monomer aqueous solutions, while at room temperature and pressure, NIPAM monomer aqueous solutions with concentration above 19.0% (wt.) will be saturated and there is crystals precipitation. On the other hand, NIPAM monomer aqueous solutions with concentration below 15.0% (wt.) couldn’t show the thermosensitivity obviously, thus the concentrations of aqueous solutions in the experiments were chosen from 15.0% (wt.) to 19.0% (wt.) accordingly.

Figure 3 shows the transmittance of 19.0% (wt.) NIPAM monomer aqueous solutions in the process of heating, and the CT is about 30.1°C for 19.0% (wt.) monomer aqueous solution.

Figure 4 shows mean transmittance of NIPAM monomer aqueous solutions with different concentrations. The transmittance is obtained by the following formula:
\[ T(\lambda)(\%) = \frac{I_i(\lambda)}{I_0(\lambda)} \times 100 \]  
\[ \bar{T} = \frac{1}{m} \sum_{i=1}^{m} T(\lambda_i) \]  

Wherein \( m \) is the number of wavelength in range by system distinguish.

Figure 3. Transmittance spectra of 19.0% NIPAM monomer aqueous solutions in heating process.

Figure 4. Mean transmittance of NIPAM monomer aqueous solutions with different concentrations.

The CS becomes lower with the increasing of concentration as shown in Figure 4, obtained by
formula (2), wherein $400 \text{nm} < \lambda < 760 \text{nm}$. After natural cooling the NIPAM aqueous solutions returned to its original state, which is similar to the thermal properties of the poly-NIPAM.

After the whole progress, the NIPAM monomer aqueous solution was evaporated and tested by FT-IR spectrum. As shown in Figure 5, the groups of the substances obtained after evaporation of NIPAM aqueous solutions and NIPAM monomer are totally the same. The aqueous solutions were prepared with the evaporated NIPAM, resulting in temperature sensitivity as it used to. The results indicated that the NIPAM monomer did not change after the whole experiment, which make it green and recyclable phase-transformation material.

![Figure 5. FT-IR spectrum of the substances obtained after evaporation of NIPAM aqueous solutions (a) and NIPAM monomer (b).](image)

**Conclusion**

Thermal property was found in NIPAM monomer aqueous solutions. When the concentration was above a certain level, with the increase of temperature, there exists phase transformation in the NIPAM monomer solutions. Besides all the information mentioned above, further research of phase transformation in non-polymeric NIPAM monomer aqueous solution is required to understand its innate character. And the non-polymeric NIPAM will have some reference for the fundamental research of PNIPAM. In addition to its scientific value, the first observation presented in this report will make NIPAM a recyclable and green material, and will be beneficial for various applications.

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