

Study on the moisture absorption of pyridine containing polyurethane for moisture-responsive shape memory effects

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Abstract Supramolecular polymers are attractive in recent years. In this article, a series of pyridine containing polyurethanes (PUPys) with various pyridine contents and various MDI-BDO contents were synthesized from 1,6-hexamethylene diisocyanate (HDI), 1,4-butanediol (BDO), *N,N*-bis(2-hydroxyethyl) isonicotinamine (BINA) and diphenylmethane diisocyanate (MDI). Thereafter, the moisture absorption of PUPys was mainly investigated systematically from the effect of temperature, relative humidity (RH), pyridine content, MDI-BDO content, the mechanism, and the kinetic of moisture absorption. Results show that the moisture absorption process of PUPys matches with Fick's second law in the initial stage. The moisture absorption is dependent on the content of *N,N*-bis(2-hydroxyethyl) isonicotinamine (BINA), e.g., the moisture absorption decreases with the increase of MDI-BDO content as well as the decrease of BINA content. In addition, the moisture absorption increases with the increase of temperature and relative humidity (RH). Accordingly, the moisture absorption process of PUPy45 at RH = 65% and $T = 25^\circ$ can be expressed with the equation: $\ln M_t = 8.88 - 2975(1/T) + 0.5\ln t$. On the basis of the moisture absorption, the shape recovery process of PUPys film under the moisture

condition support that PUPys show excellent moisture-responsive shape memory effects.

Introductions

Aiming at constructing highly complex chemical systems and advanced materials through intermolecular force, supramolecular chemistry has drawn wide attention in the last decade [1–4]. Noncovalent hydrogen bonding is the dominant type of intermolecular force [5, 6]. A typical example of supramolecular polymer system is supramolecular complex formed with pyridine moieties as hydrogen bonding acceptors [7, 8]. So far, many kinds of supramolecular polymer networks including functional supramolecular polymers and liquid crystalline polymers have been achieved through the strong hydrogen bonding between carboxyl or phenol and pyridine moieties [9–11]. Most recently, shape memory effects (SMEs), which show the ability to fix a temporary shape and recover their original shape upon application of an external stimulus, were also achieved in the supramolecular polyurethane networks containing pyridine moieties [12–14]. In the previous reports, it was found that the dissociation of hydrogen bonding in the pyridine ring of PUPys resulted in the thermal-induced strain recovery by increasing the temperature above dissociation temperature. The moisture absorption of PUPy resulted in the moisture-responsive shape recovery by immersing in moisture condition [13]. It was suggested that the supramolecular polyurethane networks containing pyridine moieties would be a novel kind of advanced functional materials.

On the other hand, the moisture absorption in polymer is very important for a variety of industries ranging from

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microelectronics to adhesives and coatings. A significant number of studies concentrate on the investigation of moisture absorption of polymer systems. For example, Li et al. had studied the effect of temperature on moisture absorption in a bismleimide resin and its carbon fiber composites. It was found that an increase in temperature accelerated the moisture absorption [15]. Vogt et al. [16] investigated the moisture absorption of ultrathin poly(vinyl pyrrolidone) films on different substrate surfaces. It was reported that the equilibrium absorption of silicon oxide surface treated with hydrophobic hexamethyldisilazane decreased with the decrease of film thickness, while the equilibrium absorption of hydrophilic silicon oxide surface was insensitive to the changing of thickness. Vlasveld et al. [17] reported the moisture absorption of polyamide-6 silicate nanocomposites and its influence on the mechanical properties. They found that the modulus of nanocomposites decreases with the increase of absorbed moisture. Karad and Jones [18] also reported the moisture absorption mechanism of cyanate ester modified epoxy resin matrices. It was proposed that the thermal spiking enhanced moisture absorption can be explained by an equilibrium between cluster formation and declustering associated with network relaxation at the spike-temperature. In addition, to understand the water-driven SMEs of shape memory polyurethanes (SMPUs) [19–23], the influence of moisture absorption has been widely studied by Yang and co-workers [24–26]. It was proposed that the moisture absorption is the main reason for the decrease of T_g in the SMPUs which results in the shape recovery at room temperature. Recently, they had also proposed the concept and mechanism of water-responsive shape memory hybrid [27]. During the investigation of humidity and solvent effects of polythiophene and poly(vinyl pyridine), the humidity absorption of poly(vinyl pyridine) was observed to be a few times high [28]. It was proposed that the pyridine unit was sensitive to the moisture absorption. The structure and morphology of PUPys have been investigated systematically in another report. A preliminary investigation shows that the PUPys have moisture-responsive SMEs [29]. However, the moisture absorption behavior of PUPys has not been reported as far as the authors know. Aiming at understanding the moisture-responsive SMEs of PUPys, it is necessary to study their moisture absorption behaviors.

Therefore, a series of PUPys with various pyridine contents and various MDI-BDO contents were synthesized from 1,6-hexamethylene diisocyanate (HDI), 1,4-butanediol (BDO), *N,N*-bis(2-hydroxyethyl) isonicotinamine (BINA) and diphenylmethane diisocyanate (MDI). The moisture absorption of PUPy was investigated systematically from the effect of temperature, relative humidity (RH), pyridine content, MDI-BDO content, the mechanism, and the kinetic of moisture absorption in this article.

Experimental part

Materials

Extra-pure-grade HDI, BDO, BINA, and MDI (all from Sigma-Aldrich Chemical Co., St. Louis, MO) were used directly. Dimethylformamide (DMF) (from Ajax Finechem Ltd., Auckland, New Zealand) was dehydrated with 4-Å molecular sieves for several days before its use as a solvent.

Synthesis of PUPy

The composition and codes of each sample are provided in Table 1. The sample of PUPys was synthesized according to the literature [14]. Typically, the reaction was carried out in a 500-mL flask filled with nitrogen and equipped with a mechanical stirrer, a thermal meter, and a condenser. First, BINA powder and DMF was added to the flask according to the composition. After dissolving the BINA powder under the mechanical stirring, an equal molar HDI was added to the flask. Second, the oil temperature was raised to about 50 °C and 0.02 wt% catalyst (Dibutyltin dilaurate) was added to the reaction. During the reaction process, 10 mL DMF each time was added to the reaction to control the viscosity of solution occasionally. After 2 h, MDI and/or BDO were added to the reaction and the reaction was kept for another 4 h at 60 °C. Finally, the polymer solution diluted to 10 wt% solution was obtained. After casting on the PTFE mould and putting on the 100 °C oven for 12 h, PUPy films were prepared. Before testing, all films were put on the 100 °C vacuum oven with ca. 0.1 Pa pressure again for 12 h to get rid of the water completely. In this study, samples of PUPys containing MDI-BDO segment were coded as PUPyMB**, where the “**” delegates the MDI-BDO content, e.g., PUPyMB15; and samples of PUPys without MDI-BDO segment were coded as PUPy##, where the “##” delegates the BINA content.

Characterization

The moisture absorption was determined by weighing the specimens on a balance. Before testing, the specimens with a thickness of 1.0 mm were dried completely. The specimen was then put on the moisture condition with a certain RH and a certain temperature. The experimental processes of moisturizing and measurement interval with respect to time (t) are illustrated in Fig. 1. This illustration provides a typical duration for both moisturizing time and measuring time. For example, on the first stage, the duration of moisturizing time is short (e.g., $\Delta T = 5$ min (min)). After 30 min, the duration is increased gradually

Table 1 Composition of PUPys used in this study

Samples	BINA (g)	HDI (g)	BDO (g)	MDI (g)	^a BINA wt%	^b MB wt%	^c Mn ($\times 10^4$)	PDI
PUPy53	5.24	4.52	0	0	53.7	0	15.38	1.63
PUPy45	4.5	5.22	0.66	0	43.3	0	10.37	1.63
PUPy40	4.0	5.4	1.0	0	38.5	0	8.63	1.31
PUPy30	3.0	5.8	1.6	0	28.8	0	6.35	1.39
PUPy20	2.0	6.22	2.23	0	19.1	0	11.16	1.63
PUPy10	1.0	6.63	2.86	0	9.5	0	9.54	1.49
PUPyMB15	5.24	4.52	0.46	1.29	45.5	15.2	12.37	1.40
PUPyMB25	5.24	4.52	0.86	2.38	40.3	24.9	19.51	1.31
PUPyMB35	5.24	4.52	1.39	3.86	34.9	35.0	11.76	1.40
PUPyMB45	5.24	4.52	2.12	5.87	29.5	45.0	10.40	1.44
PUPyMB55	5.24	4.52	3.17	8.74	24.2	55.0	8.53	1.51

^a BINA wt% is the weight fraction of BINA, which is calculated by $W_{BINA}/W_{total} \times 100\%$

^b MB wt% is the weight fraction of MDI + BDO, which is calculated by $(W_{HDI} + W_{BDO})/W_{total} \times 100\%$

^c Mn is the number-average molecular weight, PDI is the polydispersity index

(e.g., $\Delta T = 10$ min) on the second stage. After 2 h, the duration time is further prolonged (e.g., $\Delta T = 30$ or 60 min) before the weight of specimen is constant. The measuring time is about 5 min. Finally, the moisture absorption in percentage at any time (M_t) is calculated by the following equation [30]:

$$M_t = [(W_t - W_d) / (W_d)] \times 100 \%$$

where W_d and W_t refer to the weight of the dry specimen and the wet specimen, respectively.

Results and discussions

Effect of temperature on moisture absorption

Figure 2 presents the dependencies of moisture absorption of PUPy45 on time under a moisture condition with various temperatures and a constant RH (RH = 65%). In Fig. 2, it

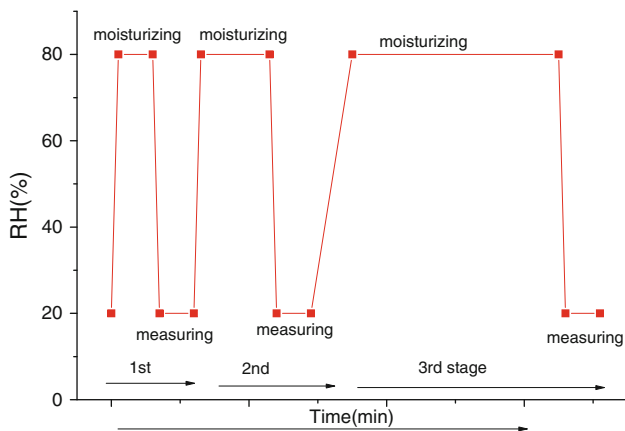


Fig. 1 Process of moisturizing and measurement interval

can be observed that the sample absorbs moisture very quickly within the first 90 min. It then slows down and tends to reach its saturated state after 1500 min within the temperature range of 22°–37°. It indicates that this moisture absorption process matches with Fick’s second law in the initial stage [16]. In addition, it was known from the Shen–Springer equation that the diffusion coefficient increased with increasing immersion temperature [31]. Thus, it is observed in Fig. 2 that the moisture content of PUPys at any time increases with the increase of immersion temperature. For example, within 90 min, the moisture absorption at 22° is only 0.7% while it reaches to 2.1% at 37°. Finally, it is found that the maximum moisture absorption also increases with the increase of immersion

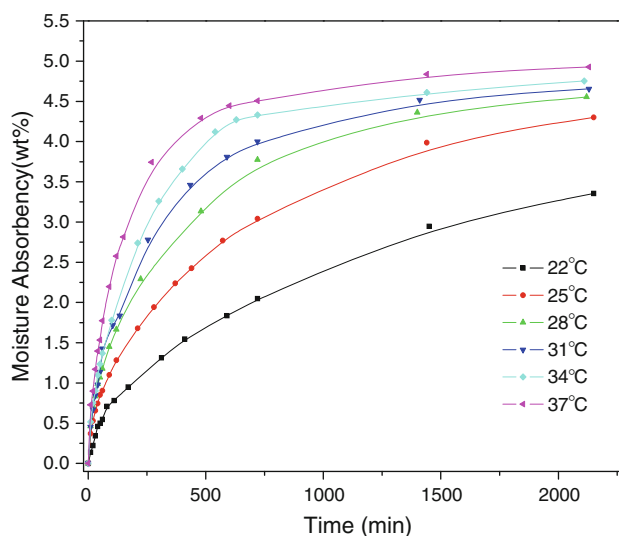


Fig. 2 Dependencies of moisture absorption on time at different temperature under RH = 65%

temperature particularly at the temperature below 28°. The maximum moisture absorption increases a little when the temperature is raised to above 28°. This is due to the fact that the moisture absorption is mainly determined by the moisture content between immersion environment and materials [16]. In addition, it is observed in Fig. 2 that the slope of curve for moisture absorption at 37 °C is much higher as compared with the moisture absorption at 22 °C during the first 480 min. It implies that the moisture absorption speed is faster at the higher immersion temperature. This is due to the fact that the diffusion speed is the main driving force to the moisture absorption due to its higher diffusion coefficient at higher temperature.

Effect of relative humidity on moisture absorption

Figure 3 presents the dependencies of moisture absorption of PUPy45 on time under a moisture condition with various RH and a constant temperature ($T = 34^\circ$). Similar to the moisture absorption behavior as described above, it is also observed in Fig. 3 that the moisture absorption enters into its equilibrium state after it has been exposed to moisture condition for a certain time, e.g., 300 min at RH = 90%, 450 min at RH = 70%, and 700 min at RH = 60%. However, before the sample is saturated with the moisture absorption, the moisture absorption kept increasing with respect to the time. Finally, it is found that the maximum moisture absorption increases with the increase of RH particularly at the RH above 70%. For example, the maximum moisture absorption is only 4.5% at RH = 70% while it reaches 7.23% at RH = 80; and 10.51% at RH = 90%. It is confirmed that the moisture absorption at equilibrium is dependent on the RH and the higher RH

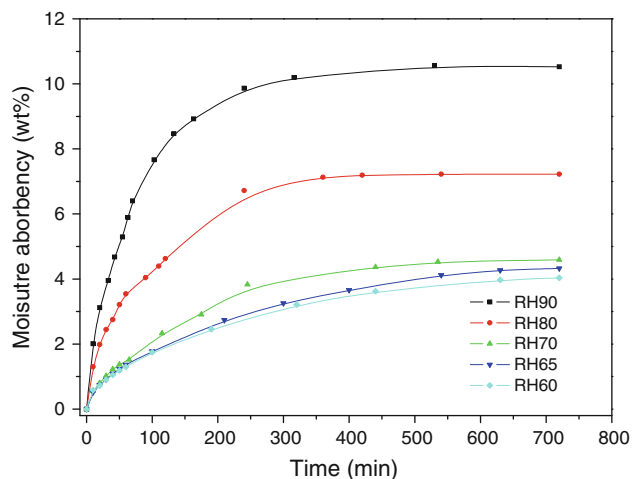


Fig. 3 Dependencies of moisture absorption on time at different RH under $T = 34^\circ\text{C}$

results in a higher moisture absorption speed. It indicates that the PUPy is sensitive to the moisture. The temperature as well as RH influences moisture absorption speed and maximum moisture absorption greatly. Thus, high moisture absorption can be obtained in PUPy by controlling the RH and temperature.

Effect of pyridine content on moisture absorption

In this experiment, the fraction of BINA unit reflects the pyridine content as each BINA unit contains one pyridine ring. Figure 4 presents the dependency of moisture absorption on time in PUPys with various BINA contents. It shows the effect of pyridine content on the moisture absorption behavior. It is observed in Fig. 4 that the PUPys with different BINA content show different moisture absorption under the same immersion moisture condition with $T = 32.5^\circ$ and RH = 85%. Though all PUPys show the similar moisture absorption process as described above, great differences are found after the moisture absorption reaches its saturation state. It is found that the moisture absorption tends to decrease slightly as the time increases particularly in the PUPys with high BINA content like PUPy53 and PUPy45. Most importantly, it is found that the PUPys with higher BINA content have higher moisture absorption at any time as compared with the PUPys with lower BINA content. In addition, it is observed that the maximum moisture absorption as well as the moisture absorption within 24, 48, and 65 h all increase with the increase of BINA content. These results imply that the BINA or pyridine ring unit has great influence on the moisture absorption of PUPys.

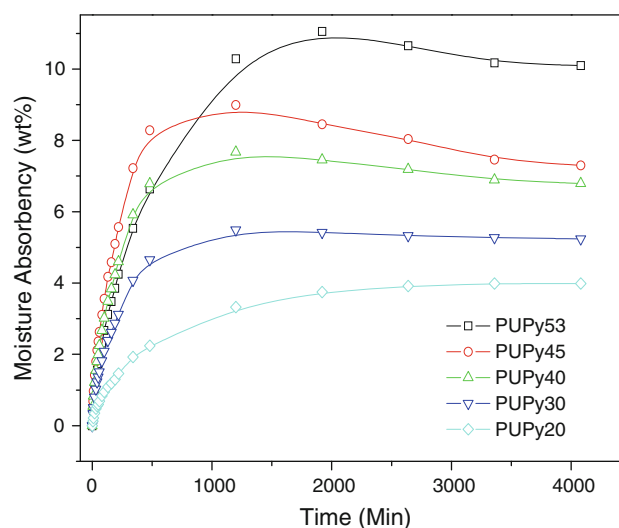


Fig. 4 Dependencies of moisture absorption on time for PUPy with various BINA contents

Effect of MDI-BDO content on moisture absorption

Figure 5 presents the dependencies of moisture absorption on time in PUPys with various MDI-BDO contents. It is observed in Fig. 5 that the pure PUPys without MDI-BDO segment (e.g., PUPy53) show the maximum moisture absorption of 10.2 wt% within 354 min; the PUPyMB15 has the maximum moisture absorption of 9.12 wt% within 1238 min; and the maximum moisture absorption in PUPyMB25 is also higher than 8.0 wt%. However, the PUPyMB35 and the PUPyMB55 take 2000 and 2600 min to reach the maximum moisture absorption of 6.78 and 5.45 wt%, respectively. It should be pointed out that the maximum moisture absorption may be not the saturated moisture absorption of PUPys in this system. It is found in Fig. 5 that the moisture absorption decrease a little after it reaches its maximum moisture absorption. It was reported that the absorbed moisture molecule can be divided into two parts. One is the free water molecules and another is the hydrogen bonded water molecule [25]. When the moisture absorption reaches their saturated state, one possible reason is that the free water molecule continues to exchange with the environment, another possible reason is that the hydrogen bonded water molecule is dissociated dynamically according to the mechanism of dynamic combinatorial chemistry or constitutional dynamic chemistry proposed by Lehn [2]. The exact mechanism should be investigated in future study. In addition, it is also found in Fig. 5 that the maximum moisture absorption as well as the saturated moisture absorption decreases with the increase of MDI-BDO content. It is known in the BINA-HDI-MDI-BDO system, the increase of MDI-BDO result in the decrease of BINA content as shown in Table 1. Therefore, it is confirmed that the moisture absorption of PUPys is mainly dependent on the BINA content, i.e., the

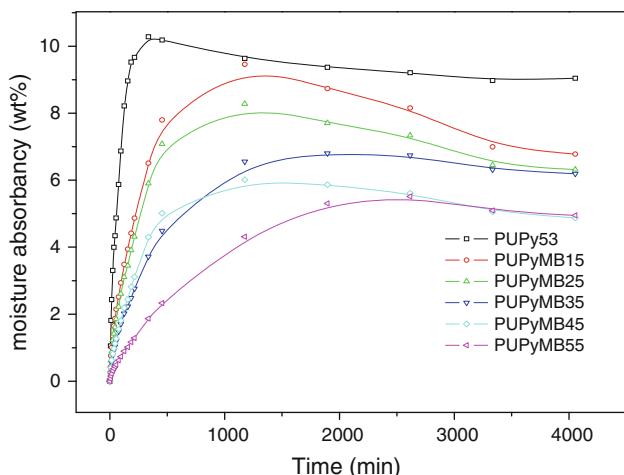


Fig. 5 Dependencies of moisture absorption on times for PUPy with various MDI-BDO contents

pyridine ring plays an important role on the moisture absorption in the PUPy.

Kinetic of moisture absorption

Since 1970s, tremendous efforts have been made to understand the mechanism of moisture absorption in polymers and composites. First of all, it is reported that diffusion is a thermally activated process and diffusivity is very sensitive to temperature in polymers. Diffusion coefficient obeys the activated transition state theory and its temperature dependence can be expressed by the Arrhenius equation (Eq. 1) [15]

$$D = D_0 e^{-E_a/RT} \tag{1}$$

In which E_a is the active energy of diffusion, D_0 is the maximum diffusion coefficient (at infinite temperature), and R is the gas constant. The exponential correlation of diffusivity with $1/T$ results in a very strong temperature dependence. Typical activation energies of moisture diffusion in thermosets range from 35 to 50 kJ/mol [15].

Being different from the diffusivity, the temperature dependency of equilibrium uptake is not very well-established. Many authors have reported the equilibrium moisture content to be independent of temperature; while other observes either positive or negative temperature dependences. There is little theoretical consideration on the temperature dependence of equilibrium uptake. However, it is important to understand the kinetics of moisture absorption into polymer films upon temperature. It was widely accepted that the simplest case for water absorption into a polymer is a Fickian process where the time resolved mass increase for a thin film on an impermeable substrate (as seen in Eq. 2) [16]:

$$\frac{M_t}{M_\infty} = 2 \left(\frac{D_t}{h^2} \right)^{0.5} \left(\frac{1}{\pi^{0.5} + 2} \sum_{n=1}^{\infty} (-1)^n \operatorname{erfc} \frac{nh}{2(D_t)^{0.5}} \right) \tag{2}$$

where M_t and M_∞ are the mass gains at time t and at equilibrium, respectively. D is the diffusivity of the penetrant, and h is the film thickness. It is assumed that the penetrate absorption is one dimensional, which should be valid given the large surface area to volume ratio and the impermeable substrate. At short times, this expression can be simplified to Eq. 3:

$$\frac{M_t}{M_\infty} = \frac{2}{h} \sqrt{\frac{D_t}{\pi}} \tag{3}$$

According to Eqs. 1 and 3, the dependency of M_t on time can be expressed with Eq. 4:

$$\frac{M_t}{M_\infty} = \frac{2}{h} \left[\left(D_0 e^{(-E_a/RT)} \frac{t}{\pi} \right) \right]^{1/2} \tag{4}$$

In addition, it was reported that glassy polymers show different absorption behavior at low- and high-relative

humidities when exposed to a range of relative humidities. At lower RH, sorption of gases and vapors into glassy polymers is successfully described by a dual mode sorption theory [18]. The equilibrium component of the theory is expressed by Eq. 5.

$$C = K_D P + C'_H b P / (1 + b P) \quad (5)$$

where C is the total solubility in polymer, K_D is Henry's law dissolution constant, b is the hole affinity constant in polymer, P is the pressure related to the RH, C'_H is the hole saturation constant in polymer. Recent reports by Vlasveld et al. [17] also confirmed that the maximum water content increased with the increase of RH within the RH range of 55–100%. Especially, it increased significantly at RH above 90%. Thus, it is believable that the total moisture absorption which is equal to the mass gains at equilibrium (M_∞) is insensitive to the temperature, but depends on the moisture content of the environment. Accordingly, it is generally accepted that the temperature and RH are the main factors to influence the moisture absorption.

Thus, according to the Eq. 4, the dependency of M_t on time can be expressed with Eq. 6:

$$\begin{aligned} \ln M_t &= \\ &= 0.5 \ln 2/h + .5 \ln D_0 - 0.5 \ln \pi + \ln M_\infty \\ &\quad - 0.5 E_a / RT + 0.5 \ln t \end{aligned} \quad (6)$$

For the same sample, the Eq. 6 can be simplified to Eq. 7:

$$\ln M_t = C + \ln M_\infty - 0.5 E_a / RT + 0.5 \ln t \quad (7)$$

where

$$C = 0.5 (\ln 2/h + \ln D_0 - \ln \pi) \quad (8)$$

At a certain temperature, the Eq. 7 can be simplified to Eq. 9.

$$\ln M_t = K + 0.5 \ln t \quad (9)$$

where

$$K = C + \ln M_\infty - 0.5 E_a / RT \quad (10)$$

The Eq. 10 can be further expressed with Eq. 11

$$K = C' - 0.5 E_a / RT \quad (11)$$

In which

$$C' = C + \ln M_\infty \quad (12)$$

Thus,

$$dK/d(1/T) = -0.5 E_a / R \quad (13)$$

According to the dependencies of moisture absorption of PUPy45 on time under a moisture condition with various temperatures and a constant RH (RH = 65%) as shown in Fig. 1. Under different immersion temperature (T), the dependency of $\ln M_t$ on $\ln t^{1/2}$ is obtained as shown in Fig. 6.

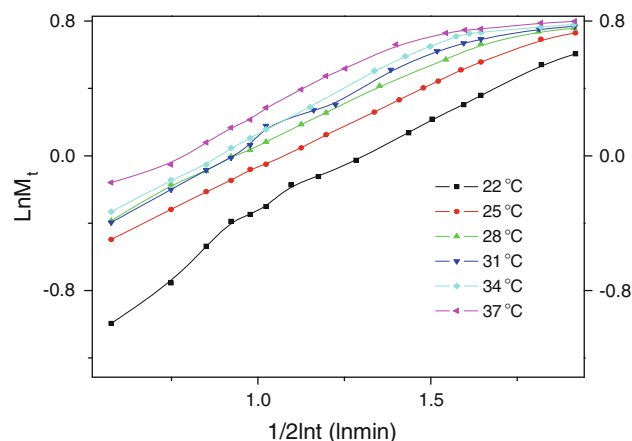


Fig. 6 Dependency of $\ln M_t$ on $\ln t^{1/2}$

It shows that the moisture absorption process of PUPy45 matches with Fick's second law (Eq. 2) in the initial stage. According to the Fig. 6, the K at different temperature (T) can be calculated, and the dependency of K on $1/T$ can be obtained as shown in Fig. 7. According to the Fig. 7, the slope $A = -2975$ and the intercept $B = 8.88$ can be calculated.

Thus,

$$0.5 E_a / R = 2975, \text{ and } C' = 8.88$$

Then

$$E_a = 49.44 \text{ KJ mol}^{-1}$$

Supposing the M_∞ is independent of temperature, and then M_∞ is the saturated moisture absorption at 37°. Thus, it can be obtained from the Fig. 7 that:

$$\ln M_\infty = 0.8$$

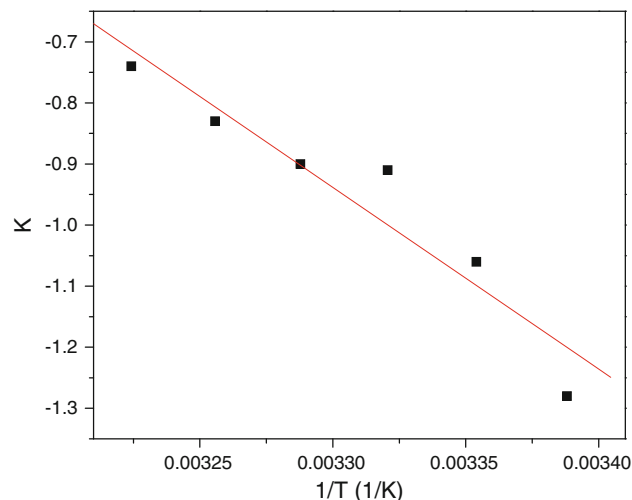


Fig. 7 Dependency of K on $1/T$

According to Eq. 12,

$$C = 8.08$$

The thickness of testing film in this experiment is 1.0 mm, i.e., $h = 1.0$ mm, According Eq. 8,

$$D_0 = 1.63 \times 10^{10}$$

Finally, the relationship between moisture absorption at any time (M_t) and time (t) at various temperatures (T) for PUPy45 under the moisture condition of RH = 65% can be expressed with the following equation:

$$\ln M_t = 8.88 - 2975(1/T) + 0.5 \ln t \quad (14)$$

Moisture-responsive SMEs of PUPys

Figure 8 presents the shape recovery process of PUPy53 film under the moisture condition of RH = 85% and $T = 30^\circ$. In this experiment, the PUPy53 film with a length of $L_0 = 34$ mm and thickness of 0.5 mm was elongated to more than 100% easily at 80 °C. The temporary rectangle film was fixed immediately after it was cooled down to room temperature. The shape fixity was very high, being close to 100% as reported in literature [14]. When the elongated rectangle film was put on the moisture condition with RH = 85% and $T = 30$ °C, it was observed in Fig. 8 that the fixed film with maximum length $L_{\max} = 75$ mm shrinks to $L_{t1} = 61$ mm after immersion for 20 min, and shrinks to $L_{t2} = 56$ mm after immersion for 30 min, and shrinks to $L_{t3} = 52$ mm after immersion 60 min, and shrinks to $L_{t4} = 45$ mm after immersion 80 min. Finally, the length is only 39 mm ($L_{t5} = 39.5$ mm) and it tends to change little ($L_e = 39$ mm). Thus, it can be calculated that the final shape recovery triggered by moisture is about 92.7%. It is believable that shape recovery can be

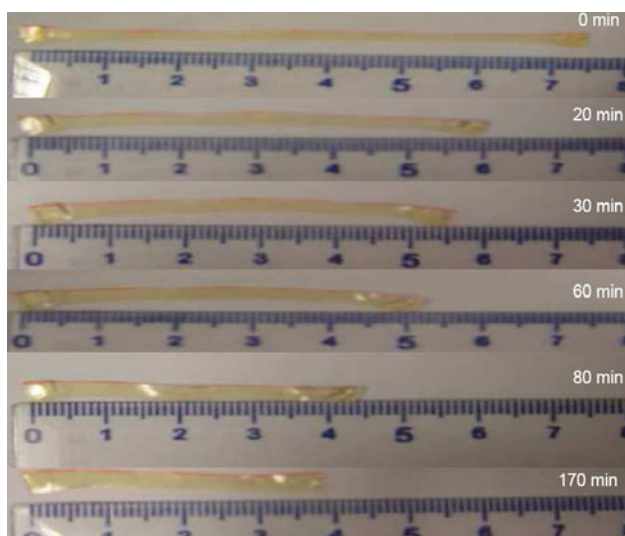


Fig. 8 Moisture-responsive shape recovery process of PUPy53 film

triggered by the moisture stimulus in the PUPys, i.e., the PUPys show excellent moisture-responsive shape recovery behavior.

Conclusions

In this study, a series of PUPys with various BINA contents were synthesized from BINA, HDI, MDI, and BDO. On the basis of the investigation of moisture absorption systematically, the following conclusions can be summarized:

1. The moisture absorption process of PUPys matches with Fick's second law in the initial stage.
2. The moisture absorption of PUPys is influenced greatly by immersion temperature and RH. As the temperature increases, the moisture absorption at any time including maximum moisture absorption increases particularly at the temperature below 28°. As the RH increases, the moisture absorption at any time including moisture absorption at equilibrium increases.
3. The moisture absorption of PUPys is mainly dependent on the BINA content. The maximum moisture absorption as well as the saturated moisture absorption decreases with the increase of MDI-BDO content as well as the decrease of BINA content.
4. The moisture absorption process of PUPy45 at RH = 65% can be expressed with the following equation: $\ln M_t = 8.88 - 2975(1/T) + 0.5 \ln t$.
5. Finally, the shape recovery process under moisture condition supports that the PUPys have excellent moisture-responsive SMEs.

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