



One-step synthesis and characteristics of LiOH-castor oil based stable polyurethane foam

Aabid Hussain Shaik¹ · Swapnil Banerjee¹ · Ariful Rahaman² · Snehalata Agashe³ · Afzal Khan⁴ · Mohammed Rehaan Chandan¹

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Abstract

This work reports one-step synthesis process of LiOH-castor oil based polyurethane foam (PU) by completely replacing synthetic polyol. The reaction involved reacting varying weight percentage of lithium hydroxide with castor oil along with isocyanate. The successfully synthesized stable PU foams were characterized for density, sol fraction, cellular morphology, polymer phase morphologies and thermo-mechanical properties. The properties of the LiOH-castor oil based PU foams were compared with the conventional PU foam. Flexibility of the synthesized foams was observed to be lower than conventional PU foam for 0.05%, 0.1% and 0.5% LiOH due to the presence of closed cells with pin holes. Whereas, presence of open cellular structure in 0.3% LiOH-castor oil based PU foams showed flexible nature. Sol fraction of all the synthesized foams was found low (~5%) indicating improved reactivity. Thermal conductivity, glass transition temperature, thermal stability and mechanical properties of the synthesized PU foams suggest the possibility of replacing conventional PU foams for various applications.

Keywords Polyurethane foam · Castor oil · Lithium hydroxide · Polymer phase morphology

Introduction

Polyurethanes (PU) foams are widely used polymers which find applications in upholstery, automotive seating, mattresses, and heat insulations etc.[1]. Other novel applications were addressed by enhancing the properties of PU foam [2–4] upon adding nanoparticles in the polymer matrix, such as in oil–water separation, electromagnetic shielding, medical cushioning, etc.[5–7]. Vegetable oils

and their derivatives are being used as an alternative polyol in polyurethane foam synthesis since last 5 decades [1, 8, 9]. Although, vegetable oil derivatives, such as fatty acids, fatty acid esters, etc.can be obtained by transesterification or hydrolysis of vegetable oils, these modified oils have failed to replace the synthetic polyol completely in the polyurethane foam formulation. PU foam synthesis from renewable resources such as soybean oil, castor oil, palm oil, rapeseed oil, cardanol oil etc. [10–25] suggests possibility of partial replacement of synthetic polyol. Among these vegetable oils, castor oil is the only naturally hydroxylated seed oil which makes it a feasible candidate for the complete replacement of synthetic polyol without further modification [26]. Synthetic polyol when completely replaced with castor oil in PU foam synthesis results in collapsed foam [18]. Various reasons have been attributed to the instability of foam; among them some major points are slow reaction between castor oil and isocyanate, low transient viscosity build-up during polymerization reaction, hard domain aggregation in the polymer phase, and slow modulus development of the reacting foaming blend [18]. Only few works has been reported in the literature addressing the issues mentioned above to overcome the causes of instability in the PU foam.

✉ Mohammed Rehaan Chandan
chandan1816@gmail.com

¹ Colloids and Polymers Research Group, School of Chemical Engineering, Vellore Institute of Technology, Vellore 632014, Tamilnadu, India
² Department of Manufacturing Engineering, School of Mechanical Engineering, Vellore Institute of Technology, Vellore 632014, Tamilnadu, India
³ Indian Polyurethane Association Technical Centre, Pune, Maharashtra 411088, India
⁴ State Key Laboratory of Silicon Materials, School of Materials Science and Engineering, Zhejiang University, Hangzhou-310027, China

Sharma et. al. initially investigated the foam stability upon gradual introduction of castor oil in the foaming blend. It shows that the complete replacement of synthetic polyol with castor oil resulted in collapsed foam [18]. Shaik et al. has reported the synthesis of stable PU foam using castor oil as a polyol and glycerol as crosslinking agent [27]. This work shows that the stability of the foam was restored by adding glycerol at varying concentration in the castor oil blend. Introduction of glycerol has found to increase the reactivity as it has high hydroxyl value. Foams synthesized were found rigid in nature with closed cells as observed in the cellular morphology. Sharma et. al. has reported the use of n-butyl lithium for the modification of castor oil before being used as a bio based polyol in the synthesis of PU foam [28]. The study reports the successful synthesis of stable PU foam by reacting castor oil with n-butyl lithium at sub-zero temperature resulting in splitting the ricinoleic acid into two fragments thereby increasing the hydroxyl index of the modified castor oil. This resulted in the faster and complete reaction of castor oil with isocyanate producing stable PU foam. Also, the Li^+ ions produced during the reaction were found to disrupt the H-bonding between the urea-urea hard domains, thereby, overcoming the hard domain aggregation [29]. Hence, literature suggests that a probable solution to overcome instability of vegetable oil-based PU foams is to introduce Li^+ ion in the foaming blend. The earlier work [28] involves multiple and complex steps of modification of castor oil with n-butyl lithium which makes it difficult to process at large scale. Therefore, coming up with a simple process of synthesis can possibly make the technology feasible at commercial scale.

In this work, we report a successful one-step synthesis process of PU foam production using LiOH and castor oil blend for the generation of Li^+ ion in the foaming blend. The foams were synthesized and were characterized for bulk density, sol fraction, cellular morphology, polymer phase morphology and thermo-mechanical properties and were compared with the conventional PU foam.

Materials and methods

Chemicals used

Isocyanate (polymeric 4,4 diphenyl methane diisocyanate, Suprasec 6456) was taken from Huntsman. Conventional polyol used (polypropylene glycol based, Konix FA 505, KPX Chemicals, S. Korea) was having approximate functionality, hydroxyl value and molecular weight of 3.0, 35 mg KOH/g polyol, and 4700 g/mol, respectively. Castor oil used (Molychem) had an approximate functionality of 2.7, hydroxyl value of 163 mg KOH/g oil and molecular weight of 930. Foam additives used was a blend of surfactant

(Tegostab 87348734 LF, Evonik), catalysts which contains bis(2-dimethylaminoethyl) ether (Niax A1, Momentive), diethanolamine (Sigma-Aldrich) and a mixture of triethylenediamine, and dipropyleneglycol (Dabco 33LV, Air Products). Lithium hydroxide monohydrate used (Molychem) having a molecular weight of 41.95 and purity 99%. Distilled water was used as a chemical blowing agent.

Blending of lithium hydroxide and castor oil

Before starting the foaming process, castor oil was blended with lithium hydroxide. Castor oil blend contains 0.05%, 0.1%, 0.3% and 0.5% (w/w of Castor oil) of LiOH which were stirred using magnetic stirrer at 2000 rpm for 5 min. The resultant mixture was used as a polyol in the PU foam synthesis.

PU foam synthesis

The lithium hydroxide blend castor oil (93.3 parts by weight (pbw)), distilled water (4.2 pbw), catalyst blend (2.0 pbw) and surfactant (0.5 pbw) are poured into a paper/plastic cup of volume 300 mL. The blend was homogenized with a high speed mechanical stirrer at an approximate speed of 3000 rpm till it turns cream in colour. Further, isocyanate was added to the blend maintaining NCO index as 1 and stirred at high speed (3000 rpm) for around 20 s. The foam was then allowed to expand freely inside the paper cup acting as the mould and was then kept for curing at room temperature for 2 h.

Characterizations

Foam density was measured by taking foam of size $1 \times 1 \times 1 \text{ cm}^3$ and their weights were measured using a high precision weighing machine. For each foam, five samples of mentioned dimension were taken from different areas of foam and their average was considered. The sol fraction in the polymeric phase was estimated by solvent extraction. Initially three samples were cut from each synthesized foams and their weight was measured. Further the dried samples were immersed in dimethyl formamide (DMF) for a period of 3 days. The samples were then kept in a vacuum oven at 80°C for 24 h. Finally the sol fraction was calculated based on the ratio of difference in weight to that of the original weight. The cellular foam morphology was imaged by scanning electron microscope (ZEISS SEM). Samples of dimension $5 \times 5 \times 2 \text{ mm}^3$ were cut and placed on sample holder. It is then placed into gold sputtering machine for about 30 min for coating the sample with gold. Further, the sample is placed into SEM and the micrographs were obtained. Finally, mean cell diameter (MCD), cell number density

(CND), and mean strut thickness (MST) were calculated from scanning electron micrographs, using image analysis software (ImageJ). The thermal conductivity of the synthesized foam samples are carried out by a thermal properties analyser (KD2 Pro, Decagon Devices, Inc.). Foam samples of dimension $10 \times 3 \times 3 \text{ cm}^3$ were cut and the probe of the instrument was inserted vertically into the samples. A differential scanning calorimeter (Q20, TA Instruments) was used to find the glass transition temperature. Approximately 2 mg of crushed PU foam sample was kept into an aluminum pan before inserting into the instrument. Samples were cooled from $80 \text{ }^\circ\text{C}$ to $-60 \text{ }^\circ\text{C}$ at a rate of $-5 \text{ }^\circ\text{C}/\text{min}$. Thermogravimetric analysis of the foam samples were performed under inert atmosphere (by using nitrogen gas) using TGA instrument (SDT Q600, TA Instruments, USA). Approximately 3 mg of crushed PU foam samples were heated from room temperature to $800 \text{ }^\circ\text{C}$ at a rate of $10 \text{ }^\circ\text{C}/\text{min}$. Final weight of the samples was noted upon completion for measuring the derivative weights of the foam samples. The tensile strength of the synthesized foam samples are carried out by a Universal Testing Machine (INSTRON 5960, dual column). Minimum of three foam samples of dimension specified under ASTM D3574 were cut and clamped in UTM. Further the loading is done at the rate of $10 \text{ mm}/\text{min}$ till fracture point of foam is attained.

Results and discussion

Foam density, sol fraction and cellular morphology

Bulk density of PU foams may vary slightly according to the type of foam prepared. Rigid foams have a higher density than flexible foams as they have closed cells [30]. The

density of the conventional PU foam is $0.032 \pm 0.3 \text{ g/cc}$ [31]. Fig. 1(a) shows the bulk density of foams prepared from castor oil. 0.05% and 0.1% LiOH based foams have high densities of around 0.13 g/cc and 0.18 g/cc respectively. Furthermore, the foams prepared from 0.3% and 0.5% LiOH have densities around 0.04 g/cc and 0.06 g/cc respectively. This implies that 0.3% LiOH based foams have bulk density comparable to that of conventional PU foams due to the presence of open cells as verified by the SEM image (Fig. 2(d)). On the other hand, due to closed cell structure in other samples (Fig. 2(b),(c),(e)), the bulk density is found relatively higher. The variation of bulk density can also occur due to the variation of cell number density (CND) of the foams [18]. Higher the CND more will be the bulk density of the foam. In order to find the percentage of loose molecules present after the reaction in the samples sol fraction was measured. It is well known in the literature that the reactivity of castor oil with isocyanate is very low, which contributes to the instability of PU foams [18]. Therefore, sol fraction is an important measurable parameter which indicates the extent of the reaction between the reactants. Sol fraction of conventional PU foam is around 1.4% [18]. Fig. 1(b) indicates the sol fraction of the synthesized PU foams. Sol fraction of foam with pure castor oil was found to be around 36% which is in agreement with the literature. However, sol fraction of LiOH based PU foams is found to have drastically reduced (around 5%) as shown in Fig. 1(b). This indicates the improved reactivity between castor oil and isocyanate in presence of LiOH.

Figure 2 shows the scanning electron micrographs of foam specimens. The scanning electron micrographs of 0.3% LiOH-castor oil based foams (Fig. 2(d)) show open cellular structure implying flexible nature of foam similar to the micrograph obtained for petro based PU foam

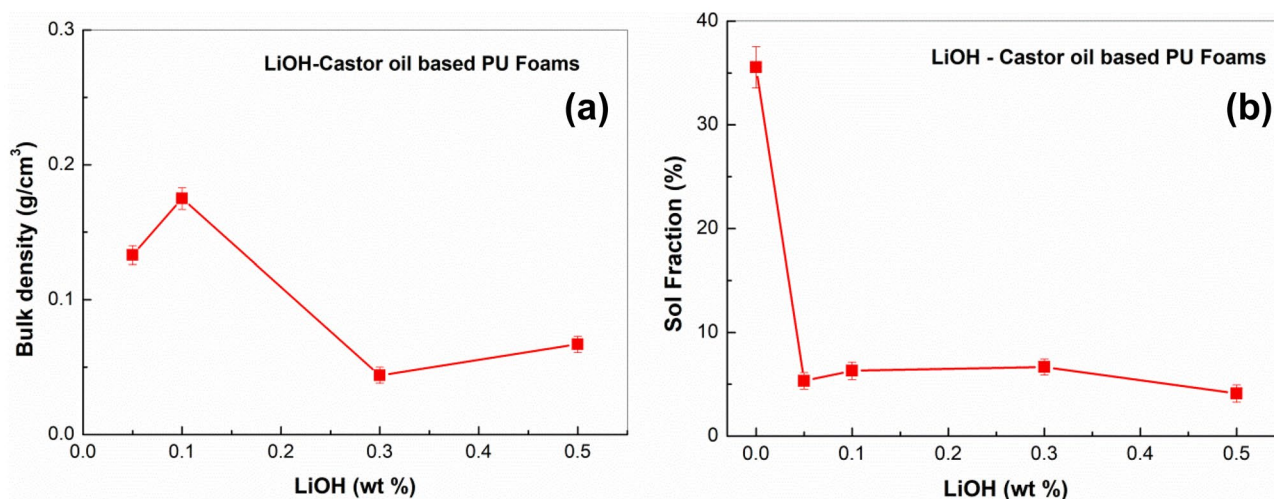


Fig. 1 (a) Bulk density and (b) sol fraction of LiOH-Castor oil based PU foams (trend line is guide to eyes)

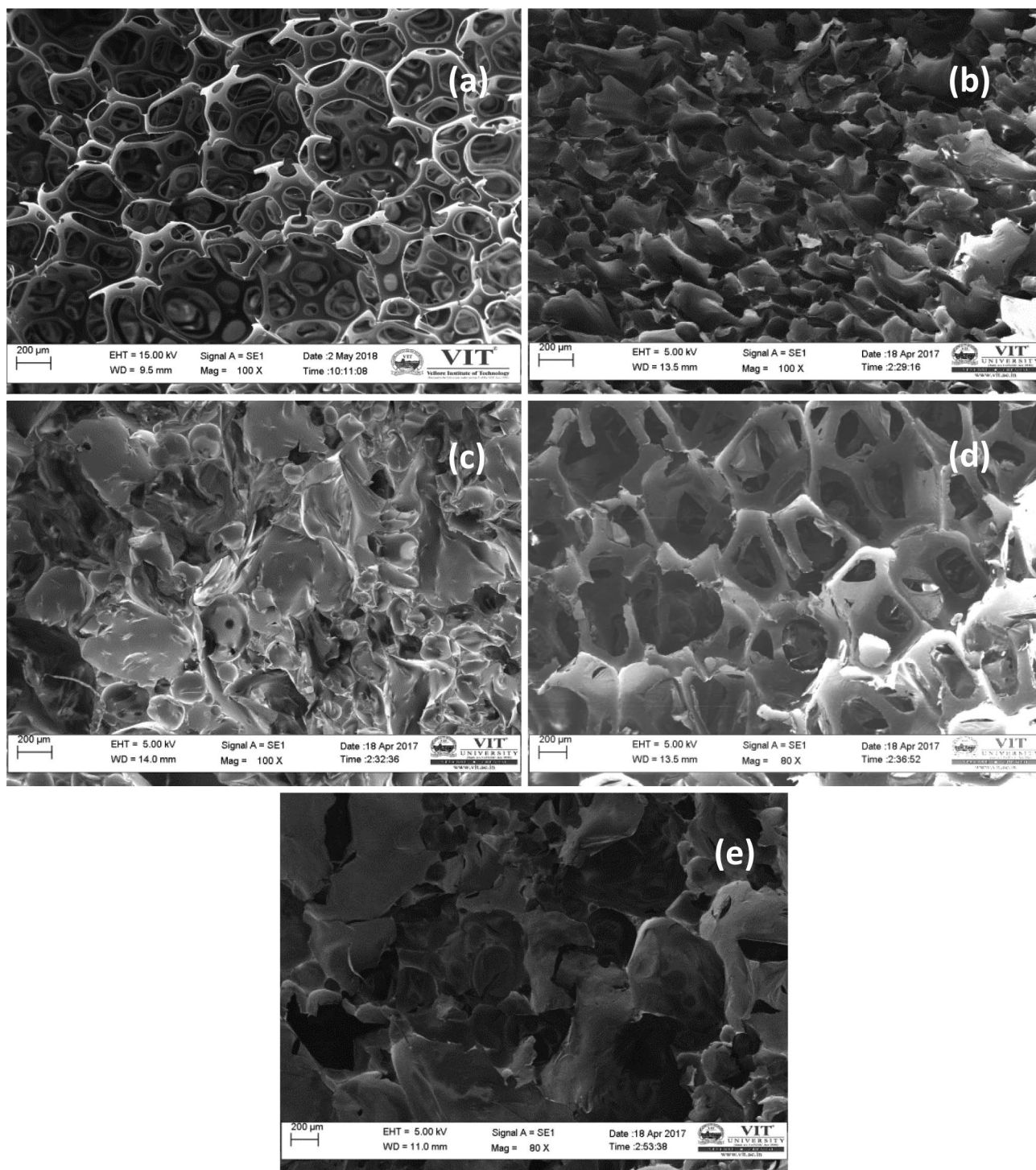


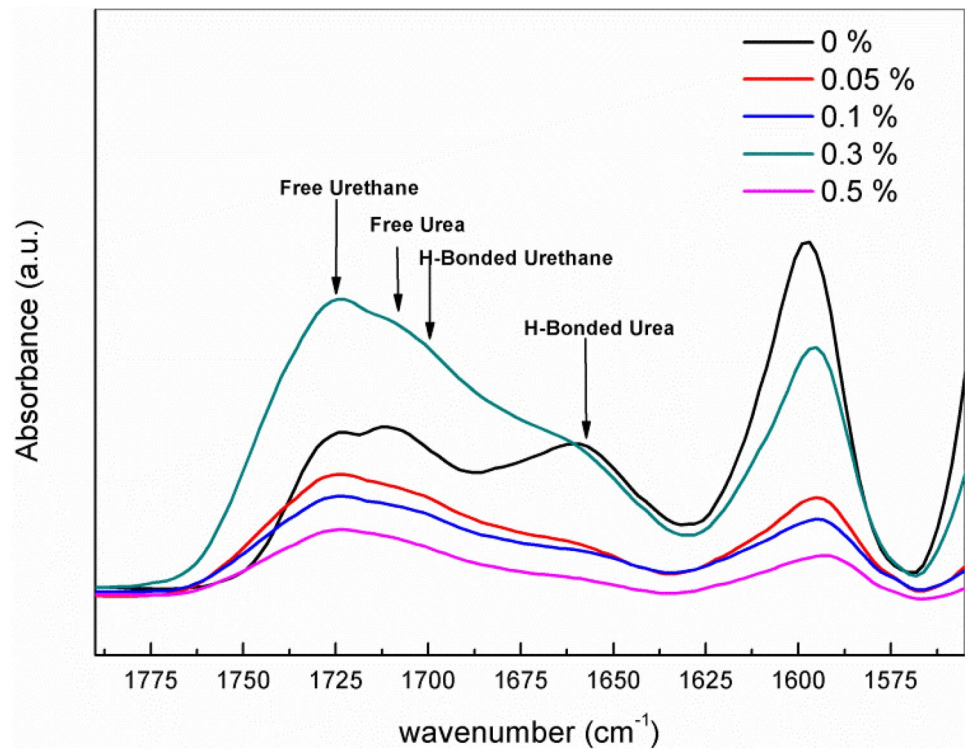
Fig. 2 Scanning electron micrographs of (a) Conventional PU foam, (b) 0.05%, (c) 0.1%, (d) 0.3% and (e) 0.5% LiOH based PU foams

(Fig. 2(a)). On the other hand, scanning electron micrographs of 0.05%, 0.1% and 0.5% LiOH-castor oil (shown in Fig. 2(b), 2(c), 2(e) respectively) based PU foams show closed cellular structure with presence of pin holes implying semi-rigid nature of foam [28].

Segmental polymer phase morphology

The stability of the foam also depends on the polymer phase morphology, which in turn decides the modulus development of the stretching polymer film while the

Fig. 3 FTIR of LiOH-castor oil based PU foams



foaming takes place [32]. The polyurethane consists of two phases namely urethane soft phase and poly-urea hard phase. Poly-urea (thermodynamically originated) disperses in the soft urethane matrix. A well dispersed hard phase causes uniform modulus development throughout the polyurethane film during cell growth, resulting in stable polyurethane foams [18, 33]. Hydrogen bonding in

polyurethanes plays a significant role in determining the phase segregation [34, 35]. Aggregation of hard phase is the result of hydrogen bonding between the urea-urea phases, which causes uneven stretching film modulus resulting in rupture or failure of foam [36]. In order to evaluate the hydrogen bonding between urea-urea hard domains FTIR was performed on the foam samples.

Fig. 4 Thermal gravimetric analysis of LiOH-castor oil based PU foams

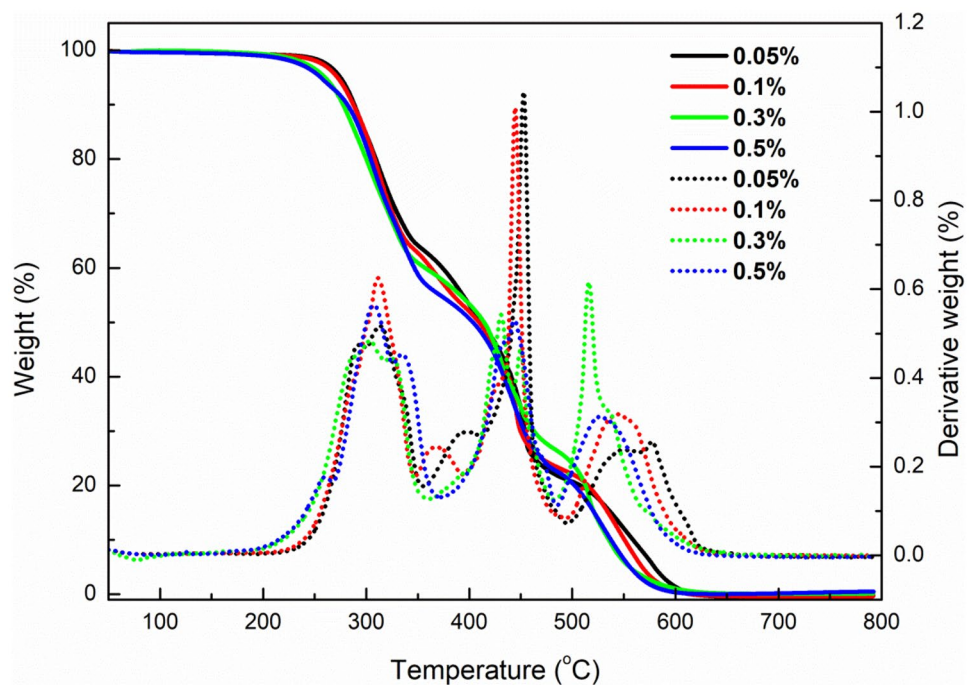


Table 1 Thermal conductivity of PU foam with varying % of LiOH

| Sl.No | PU foam sample wt% LiOH | Thermal Conductivity (W/mK) at room temperature ~ 28 °C |
|-------|-------------------------|---|
| 1 | 0.05 | 0.032 ± 0.004 |
| 2 | 0.1 | 0.037 ± 0.005 |
| 3 | 0.3 | 0.027 ± 0.003 |
| 4 | 0.5 | 0.030 ± 0.006 |

Figure 3 indicates that FTIR spectra of foams, where H-bonded urea peak appears around 1660 cm^{-1} [18]. Pure castor oil based PU foam (0%) shows a prominent peak at 1660 cm^{-1} indicating the presence of hydrogen bonding between the hard domains, causing foams to collapse. On the other hand, introduction of Li^+ molecules in the castor oil blend has significantly reduced the H-bonding between the hard domains as indicated by the disappeared peak at 1660 cm^{-1} . This disruption of hydrogen bonding by Li^+ ion is due to its smaller molar volume and

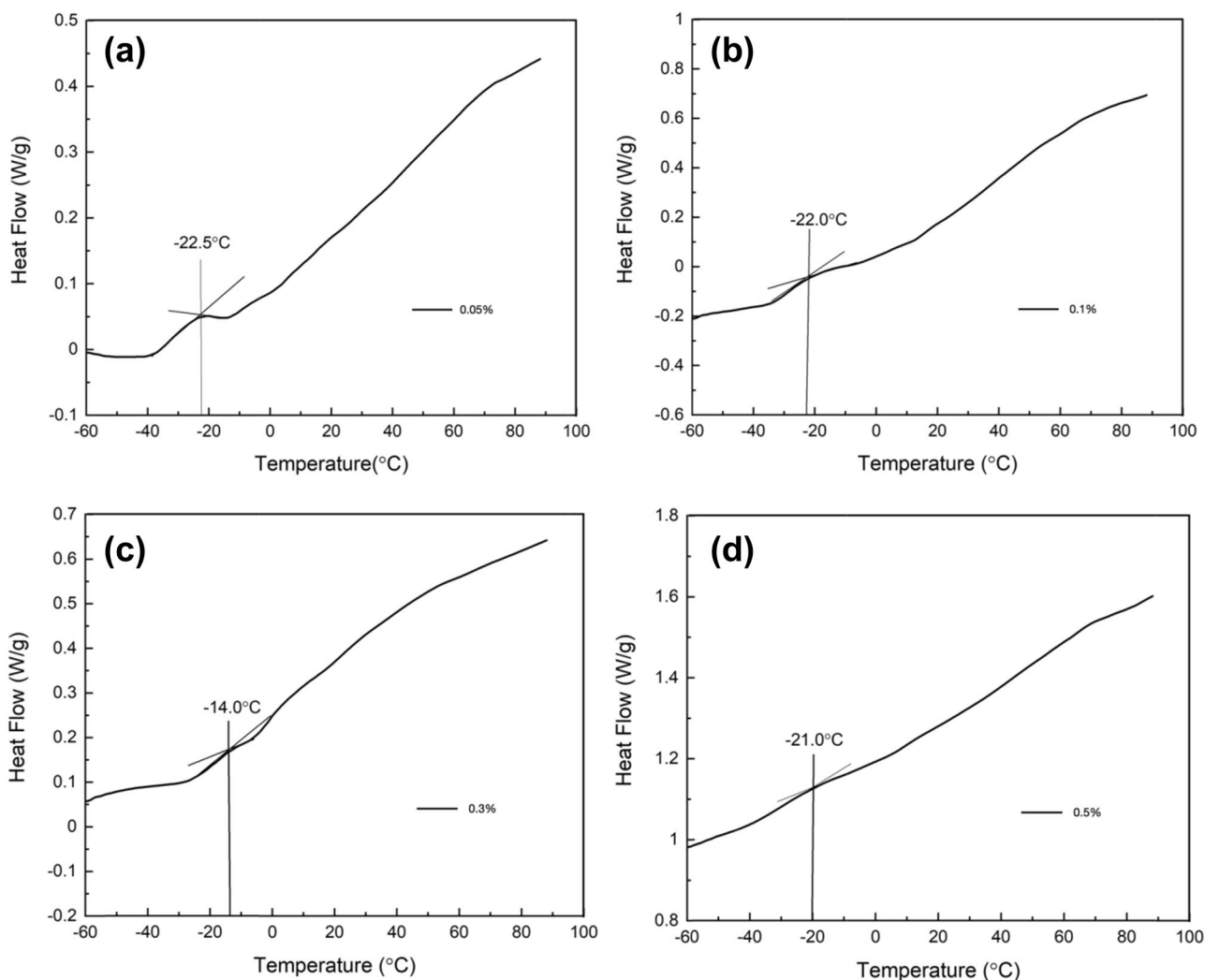
Table 2 Mechanical properties of PU foam with varying % of LiOH

| PU foam sample wt% LiOH | Tensile Modulus (MPa) | Tensile Stress at break (MPa) | % Elongation at break |
|-------------------------|-----------------------|-------------------------------|-----------------------|
| 0.05 | 1.04 | 0.26 | 25 |
| 0.1 | 0.89 | 0.42 | 47 |
| 0.3 | 0.31 | 0.18 | 58 |
| 0.5 | 0.15 | 0.06 | 40 |

stabilization energy of O-- Li^+ complex [29, 37]. Thus, all the foams were found stable.

Thermogravimetric analysis

From the analysis of the TGA results of LiOH-castor oil based PU foams shown in Fig. 4, it is observed that there

**Fig. 5** Differential scanning calorimetry of (a) 0.05%, (b) 0.1%, (c) 0.3% and (d) 0.5% LiOH based PU foam samples

are three peaks in the mid abscissa range of the data curve. These illustrate three degradation zones. Each of the peaks corresponds to breakage of certain bond present in the polyurethane foam. The first peak refers to the breakage of C-H bonds in the range of 230 °C – 350 °C. At this temperature the foam starts to break up into smaller fragments from the original body. The second peak signifies the breakage of the C=O bond in the urea segment and the (N-H) linkages of the polymer matrix. This happens at a temperature range of 410 °C – 460 °C. The final peak represents the breakage of the C-C bonds in the polymer, which results in the total collapse of the polymer [38]. A significant result can be obtained from the thermogravimetric analysis is the derivative weight loss. It throws light on various temperature range where significant apparent weight is lost due to thermal degradation. A material is considered thermally stable as long as its weight does not change considerably with the increase in temperature. From Fig. 4 it is observed that the synthesized PU foams were found stable till 225 °C which is comparable with respect to the synthetic PU foam, where the 1st degradation occurs at ~217 °C [39], beyond which thermal disintegration begins.

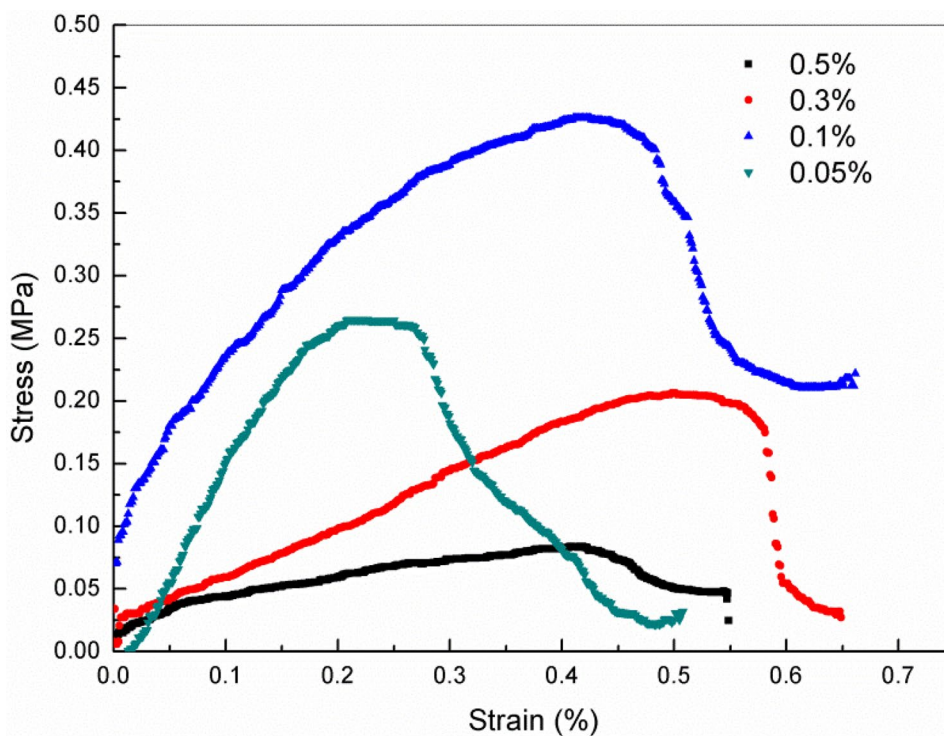
Thermal and mechanical properties

PU foams find wide application as a thermally insulating material in various industries [40, 41]. Conventional flexible and rigid PU foams have thermal conductivity of ~0.026 W/mK and ~0.033 W/mK (approximately) respectively

[42–44]. The thermal conductivities of the prepared foams were measured and tabulated in Table 1. The thermal conductivity of 0.3% LiOH-castor oil based foam is found to be 0.027 W/mK which is comparable to that of the conventional flexible PU foam. On the other hand, thermal conductivity of 0.05%, 0.1% and 0.5% lithium modified castor oil based foam is found to be 0.032, 0.037 and 0.030 W/mK respectively which is comparable to that of conventional rigid PU foam. Higher values of thermal conductivity were obtained for 0.05%, 0.1% and 0.5% LiOH based samples is due to the presence of closed cells. Variation of the thermal conductivity values among these samples can be correlated with the bulk density values. Wherein, a sample with higher bulk density is showing higher thermal conductivity. Thus, LiOH-castor oil based PU foams can be used as potential thermal insulating material for suitable applications.

Glass transition temperature (T_g) of the synthesized PU foams were extracted from the DSC curves shown in Fig. 5. The figure indicates the temperature below which the polymer chains become completely crystalline. Thus, for flexible PU foam it indicates the temperature below which the foams become stiff. It can be observed from the plots, that with increase in LiOH in the sample, the peaks observed continued to flatten, indicating that the degree of crystallization of the LiOH based PU foam to decrease. The sharp peaks indicate a higher degree of cross-linking between the polymer chains [45]. As the T_g of all the synthesized samples are well below 0 °C. These foams can be suitably used under different ambience with varying temperatures. A variation

Fig. 6 Stress vs strain plot for PU foams



in T_g values of 0.05%, 0.1% and 0.5% with respect to 0.3% LiOH based samples can be attributed to the type of the foam produced (i.e. rigid and flexible). The rigid foams have a T_g values $\sim 22^\circ\text{C}$, whereas flexible foam is having a value of 14°C .

Table 2 showing tensile modulus, tensile strength at break and percentage elongation at break of all LiOH-castor oil PU foam samples were determined using the tensile stress (MPa) versus strain (%) curve (shown in Fig. 6). Percentage elongation of 0.3% lithium modified castor oil foam is found to be 57% which is lower than that of conventional PU foam having a value of 79% [18]. This reduction in percentage elongation is because of low molecular weight of castor oil as compared to that of synthetic polyol [46]. On the other hand, tensile strength for 0.3% LiOH-castor oil PU foam is found to be 0.31 MPa which is much higher than that of conventional PU foam having a value of 0.048 MPa [18]. Thus, the mechanical properties of 0.3% LiOH-castor oil based PU foam can be compared to conventional flexible PU foam which is a probable alternative against conventional PU foams.

On the other hand, tensile strength of 0.5% Li modified castor oil is found to be 0.15 MPa which is much lower than that of conventional rigid PU foam. The tensile stress of 0.05% and 0.1% LiOH-castor oil based foams are 1.04 MPa and 0.89 MPa respectively is comparably higher with respect to the conventional rigid foam (0.4255 MPa). Thus it can be concluded that 0.05%, 0.1% and 0.5% LiOH-castor oil foam has much lower tensile strength than that of conventional rigid PU foam.

Conclusion

Stable polyurethane foam was successfully synthesized via one-step process using LiOH-castor oil blend by completely replacing synthetic polyol. The synthesized PU foams were characterized for density, sol fraction, cellular morphology, polymer phase morphologies and thermo-mechanical properties. Sol fraction of the foams indicates improved reactivity. Density of the foams was relatively higher as compared to conventional flexible polyurethane foam. Cellular morphology suggests presence of closed cells with pin holes for 0.05%, 0.1% and 0.5% LiOH-castor oil based PU foams and open cellular structure for 0.3% LiOH-castor oil based PU foams. Thermal conductivities of the foams were approximately same as compared to both flexible and rigid conventional PU foams. Glass transition temperature of all the foams were found below -10°C . Thermogravimetric analysis shows foams were stable till 225°C . The tensile strength of LiOH-castor oil based PU foam was found to be much higher than the conventional foams (tensile strength of 0.3% Li based PU foam is 0.31 MPa). Therefore, one step synthesis process of the LiOH-castor oil based PU foams

suggests the possibility of replacing synthetic polyols in the synthesis process of PU foams at commercial scale.

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