

# Karanja Oil Polyol and Rigid Polyurethane Biofoams for Thermal Insulation

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**ABSTRACT:** Rigid polyurethane biofoams were prepared from karanja polyol which was derived by ring-opening reaction of epoxidized karanja oil. The polyol, which had a hydroxyl value of 186 mg KOH/g, was thoroughly characterized and the structure confirmed by spectral techniques. The foam formulations were developed to achieve shrinkage-free foams with water used as the blowing agent. The resulting foams were characterized for their mechanical properties like density, compression strength and flexural strength. The densities and mechanical properties, such as compression and flexural strength, varied with the amount of methylene diphenyl diisocyanate (MDI) for a fixed amount of polyol and other additives as a result of side reactions leading to allophanate and urea linkages. Scanning electron microscopy (SEM) results indicated that the cells are spherical and samples are isotropic. However, the cell size distribution varied with MDI content. The thermal conductivity was found to be in the 0.036–0.042 W/m.K range, which is well suited for insulation purposes.

**KEYWORDS:** Karanja oil, biofoam, compression strength, thermal conductivity

## 1 INTRODUCTION

A wide variety of oilseeds and, in particular, tree-borne oil seeds, are available in India. Due to the large population, edible oils are in short supply and non-edible tree-borne oilseeds (TBOs) of karanja, jatropha, mahua and neem are being considered as a source of straight vegetable oil (SVO) and biodiesel. Karanja oil, extracted from seeds of *Pongamia glabra*, is one of the least exploited and highly potent sources of non-edible oil in the country. The present production of karanja oil in India is approximately 200 million tons per annum and it is ranked next to neem oil ([www.novodboard.com](http://www.novodboard.com)). Karanja oil has a fatty acid composition of oleic acid (18:1; 61.65%) and linoleic acid (18:2; 18.52%) [1]. It has been used in the tanning industry for dressing leather, and as a raw material for manufacturing soaps, candles, shampoos and many types of cosmetic products [2]. It is also used as a biofuel, lubricant, and water-paint binder and finds application in insecticide

products and agro chemicals. The oil is known to have value in herbal medicine for the treatment of rheumatism, as well as human and animal skin diseases [3]. Research work on karanja oil has been restricted to development of sulfurized karanja oil, co-sulfurized karanja oil, use of EP additive, lubricity booster in metal working fluids, and 2-ethylhexyl esters of karanja oil fatty acids in two-stroke gasoline engines [4, 5]. The performance of karanja oil as an alternative to linseed oil in the formulation of offset printing ink has also been investigated by Bhattacharjee *et al.* [6].

Many vegetable oils have been extensively used in the development of polyurethane foams. Vegetable oils like palm oil [4, 7–11], soya bean oil [12–15], castor oil [5, 16–21] and rapeseed oil have been utilized for the development of polyols and their polyurethane (PU) foams. Several types of polyols have been developed from palm oil by the Malaysian Palm Oil Board for flexible, rigid and semi-rigid foams. These foams were found suitable for industrial sectors like building products, furniture, decorative items and automotive parts. Rigid urethane foams from tallow- and lard-based polyols [22–24] have also been reported. However, there are no reports in the literature on the use of karanja oil products in the preparation of PU foams except our previous work [25] where

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we prepared rigid PU foams using diethanolamides of karanja oil. The present study aims at developing rigid foams from karanja polyol derived through the ring opening of epoxidized karanja oil with ethylene glycol. The foam characteristics such as mechanical, thermal insulating properties and microstructure, i.e., cell morphology, have been investigated.

## 2 EXPERIMENTAL

### 2.1 Materials and Methods

Karanja seeds were procured from Sanjeevani Herbal Health Society (Hyderabad, India). Hydrogen peroxide (30%), formic acid (85%), sulphuric acid, acetic acid, 4N hydrobromic acid, iodine monochloride, potassium hydroxide, hydrochloric acid, n-butanol, pyridine, chloroform, acetic anhydride, ethylene glycol and sodium sulphate were procured from S.D. Fine Chemicals Pvt. Ltd. (Mumbai, India). Catalyst (DMCHA) was purchased from Fluka Chemie GmbH (Germany). Tegostab® B 8450 was procured from Evonik India Ltd. Polymeric diphenylmethane diisocyanate (PMDI, Suprasec® 5090) was acquired from Huntsman (Belgium).

The  $^1\text{H}$  NMR spectra were recorded on a 400 MHz Bruker ARX 400 spectrometer (Wissembourg, France) with  $\text{CDCl}_3$  as solvent. Infrared (IR) spectra were obtained on a Perkin Elmer 1600 Series FT-IR spectrometer (Norwalk, CT) with a liquid film between NaCl cells. Density (mass per unit volume) of the foam samples were calculated using the formula.

$$\text{Density} = \frac{\text{weight of the sample (g)}}{\text{Length (cm)} \times \text{breadth (cm)} \times \text{thickness (cm)}}$$

The PU foams were cut into specimens with dimensions of about  $5 \times 5 \times 3$  cm (length  $\times$  breadth  $\times$  thickness). The dimensions were accurately measured to determine their densities using the above equation. The density of foam was the average value from six specimens.

Compression strength tests of the foam samples were carried out using a Shimadzu Autograph AGS-10kN model Universal Testing Machine (Japan) at a crosshead speed of 1 mm/min. Cuboidal specimens of the foam of dimension  $5 \times 5 \times 3$  cm (length  $\times$  breadth  $\times$  thickness) were prepared for compression test. A minimum of 3 blocks were used from each foam sample. The blocks were compressed in the direction parallel and perpendicular to the foam rise during foaming at a rate of 1 mm/min. The load at 10% deformation was determined to calculate the compression strength. A three-point bending test was used to determine the flexural strength by ASTM D790 method. The test specimens (6 in number) were prepared according

to ASTM D790 as 1/800, 1/200 and 500. A Shimadzu Autograph AGS-10kN model Universal Testing Machine was used for this purpose and test speed was set to 1 mm/min. The flexural strength (FS) was calculated based on the following equation:

$$FS = PL/bd^2$$

where  $P$  is the ultimate load of the specimen,  $L$  is the span,  $b$  is the specimen width, and  $d$  the specimen thickness. Thermal conductivity of the samples was measured using specimens of size 200 mm  $\times$  200 mm  $\times$  20 mm using a LaserComp Fox 200 heat flow meter at an upper plate temperature of 10 °C, lower plate of 35 °C and mean temperature of 25 °C.

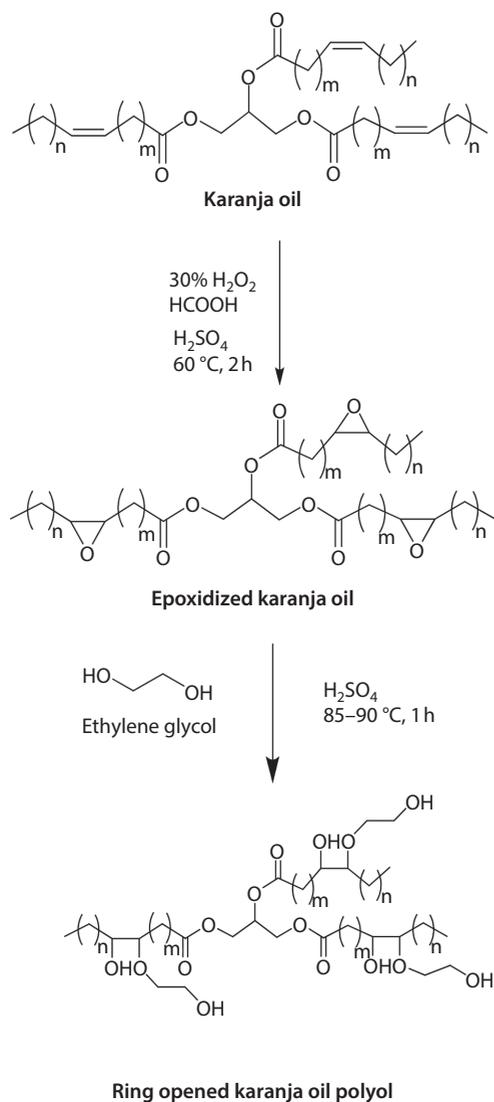
### 2.2 Preparation of Epoxidized Karanja Oil (EKO)

Karanja oil having iodine value of 86.5 g/100 g (100 g, 0.12 mol), formic acid (13.0 g, 0.28 mol) and sulfuric acid (1.1 mL, 2 wt% of  $\text{HCOOH}$  and  $\text{H}_2\text{O}_2$ ) were placed in a three-necked round-bottom flask and the temperature was maintained at 5–10 °C. Hydrogen peroxide solution of 30% concentration (80.8 mL, 0.85 mol) was added slowly to the contents through an addition funnel under mechanical stirring at 5–10 °C over a 1 h period. After complete addition of  $\text{H}_2\text{O}_2$ , the contents were stirred at 60 °C for 3 h. The progress of the reaction was monitored by withdrawing aliquots of the reaction mixture at various time intervals. The samples were extracted with ethyl acetate and then washed with water until they were acid free. The extent of epoxidation and unsaturation was analyzed by oxirane and iodine values. Maximum oxirane value of 4.15 and iodine value 3.77 were obtained, which indicated the completion of epoxidation. This was confirmed by IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and ESI-MS spectroscopy. The various steps involved are given in Scheme 1.

### 2.3 Preparation of Karanja Polyol from Epoxidized Karanja Oil

Ethylene glycol (24 mL, 0.4 mol) and sulfuric acid (0.5 mL, 1 wt% of the sample) were placed into a two-necked round-bottom flask and the temperature of the contents was maintained at 85–90 °C. Epoxy karanja oil (100 g, 0.10 mol) was added dropwise by using a dropping funnel under mechanical stirring for 1 h. The completion of the reaction was monitored using thin-layer chromatography (TLC) and eluted with hexane and ethyl acetate (90:10, v/v). After the completion of the reaction, the sample was extracted with ethyl acetate and washed with distilled water to remove acid. The compound was dried over sodium sulphate ( $\text{Na}_2\text{SO}_4$ ) and desolventized using a rotary evaporator

and dried under reduced pressure (2–3 mm Hg) to obtain dried karanja polyol. The degree of epoxidation and unsaturation was analyzed by oxirane and iodine values. Oxirane value of 0.38 and iodine value of 0.3 were obtained, which indicated the completion of the reaction. The hydroxyl value of the compound was found to be 186 mg KOH/g. The structure of the karanja polyol was characterized by FTIR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and ESI-MS spectroscopy.



**Scheme 1** Steps involved in the synthesis of karanja polyols.

**Table 1** Foam formulations and foaming characteristics.

S. no	Sample code	Foam composition (g)					Cream time (s)	Foam rise time (s)	Tack free time (s)
		Polyol	Surfactant	Catalyst (DMCHA)	MDI	Water			
1	KF-1	50	2.0	1.0	80	1.0	18	117	240
2	KF-2	50	2.0	1.0	70	1.0	14–15	110	200
3	KF-3	50	2.0	1.0	50	0.5	16	117	180

## 2.4 PU Foam Formulation

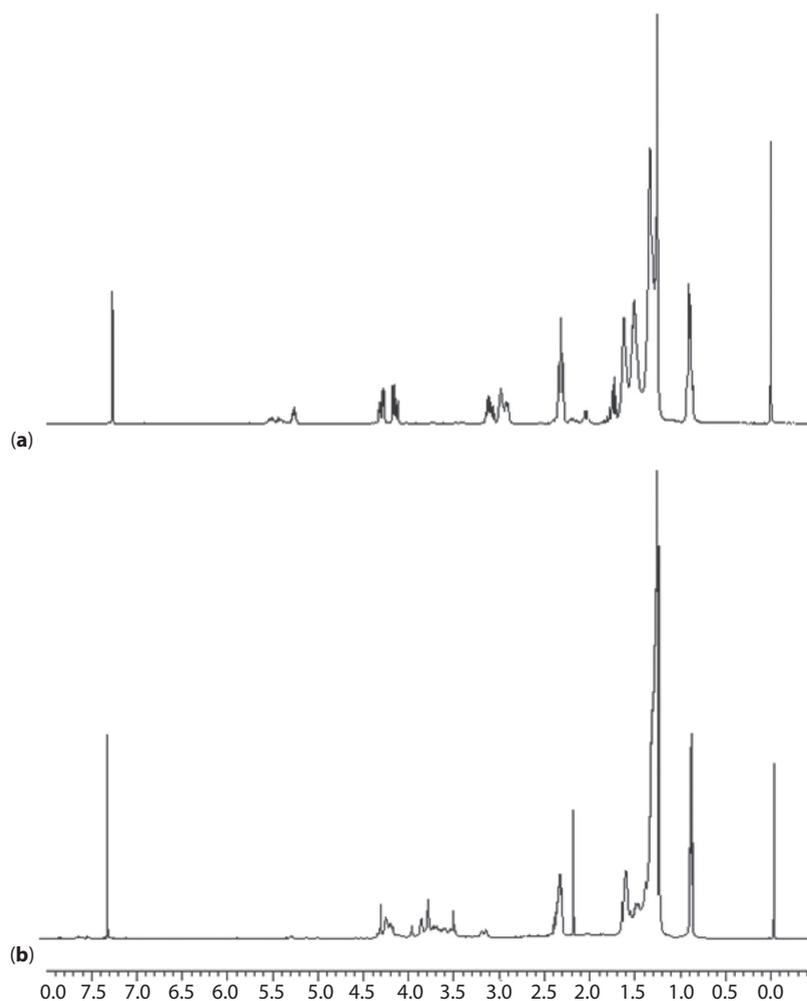
The nature and amount of the catalyst, surfactant, blowing agent (water) and polymeric MDI were varied to get shrinkage-free foams with good foam height. The following parameters were taken into consideration: good foam height, moderate cream time, tack-free and shrinkage-free foams which had closed cell structure. Tegostab 8404 (surfactant), dimethyl cyclohexylamine (DMCHA, catalyst), and Suprasec 5090 (polymeric MDI) were chosen to develop the formulations. Recipes for the formulations are given in Table 1. The PU foams were prepared by adding PMDI to the polyol mixture, which consisted of a karanja polyol, distilled water, catalyst and Tegostab (surfactant), with stirring. At the creamy stage, the mixture was poured into a paper box and left undisturbed to rise freely. The full rise time (the time from mixing to full expansion of foaming) was recorded. Later, the foam was removed from the mold and allowed to post cure for 2 days at room temperature before cutting it into test specimens.

## 3 RESULTS AND DISCUSSION

### 3.1 Structure and Properties of Epoxidized Karanja Oil and Karanja Polyol

The structure of epoxidized oil and polyol were confirmed by IR, NMR and mass spectral studies and the data are compiled in the supplementary data.

**Epoxidized karanja oil:** The epoxy karanja oil obtained as a light yellowish liquid had an oxirane value of 4.15. Formation of epoxy ring was further confirmed by  $^1\text{H}$  NMR spectra, as shown in Figure 1a, of the epoxy karanja oil, where the absence of peaks at 1.9–2.0 ppm ( $-\text{CH}_2-$  protons adjacent to the unsaturation) indicated complete disappearance of unsaturation; and appearance of new peaks at 2.9 to 3.15 ppm corresponding to  $-\text{CH}-\text{O}-\text{CH}-$  protons of the epoxy carbon and at 1.49 to 1.52 ppm corresponding to  $\text{CH}_2$  proton adjacent to two epoxy bearing carbons confirmed the presence of epoxy group.



**Figure 1** The  $^1\text{H}$  NMR spectrum of (a) epoxy karanja oil and (b) karanja polyol.

**Karanja polyol:** The polyol obtained was a viscous reddish-yellow liquid with a HV of 186 mg KOH/g. The structure was confirmed by  $^1\text{H}$ NMR spectroscopy (Figure 1b) and supported by IR,  $^{13}\text{C}$  NMR and LC-MS data (see supplementary data). The multiplet at chemical shift 3.42–3.98 ppm corresponding to  $>\text{CH}_2\text{-OH}$ ,  $-\text{CH}_2\text{-OH}$ ,  $-\text{O}-\text{CH}_2\text{-CH}_2\text{-OH}$  and a singlet at 2.17 corresponding to  $-\text{OH}$ , characteristic of ethanoloxy hydroxyl functionality, as seen in  $^1\text{H}$  NMR spectrum Figure 1b, confirmed the formation of ethanoloxy hydroxyl derivative of epoxy karanja oil. The appearance of epoxidized oils, polyol and foam samples is depicted in Figure 2 and the properties of the polyol are given in Table 2.

### 3.2 Foaming

The foam rise time (FRT) affects the density and therefore the other mechanical properties of the foam and is considered to be an important parameter. In the



**Figure 2** Appearance of karanja oil derivatives and PU foam.

**Table 2** Physicochemical properties of karanja polyol.

Property	Value
Hydroxyl value	186 mg KOH/g
Acid value	0.5 mg KOH/g
Moisture content	0.1%
Density/sp gravity	0.9724 g/cc
Viscosity (at 40 °C)	20.5 cSt

present case, it was observed that the FRT depended on the composition of the foam formulation. The cream time was found to be in the 15–18 s range and the foam rise time around 110–120 s. The curing of the foams was confirmed by determining the tack-free time wherein formulations with higher amount of MDI showed higher tack-free time.

### 3.3 Density Measurements

In the present case, the foams prepared were found to be very rigid. The variations in amounts of isocyanate and blowing agent affected the height and, hence, the densities. The densities of different formulations are shown in Table 3; they are around 52–54 g/cc and around 62 g/cc when the blowing agent is reduced to half. The reaction between NCO and H<sub>2</sub>O leads to evolution of CO<sub>2</sub>, which in turn affects the foam height and the density. Higher amount of blowing agent (water) led to high shrinkage in the case of KF-3. However, for KF-1 and KF-2, higher amount of blowing agent led to shrinkage-free foams with good foam height and lower densities.

### 3.4 Thermal Conductivity

The insulating property of the foams depends on its thermal conductivity and in turn is related to foam density and cell morphology. The thermal conductivity of rigid polyurethane foam is dependent on many other factors like cell gas, temperature, presence of moisture and time of measurement. In the case of low density PU foams, the solid material makes up only 3–5% of the volume and is mainly found in the cell struts and cell walls. However, they can bear mechanical loads due to rigidity and anti-buckling properties.

According to Nieuwenhuyse [26], the increase in thermal conductivity does not increase in proportion to the increase in density; the thermal conductivity of rigid polyurethane foam changes little in the density range of 30 to 100 kg/m<sup>3</sup> relevant for building. In the present case, the K values are found to be in the 0.036–0.042 (W/m.k) range, as listed in the Table 3, and the thermal conductivity was found to decrease with a slight decrease in the density. The use of a lesser

amount of MDI has led to lower values of thermal conductivity but higher densities led to higher K values. Owing to the high closed-cell content of rigid polyurethane foam (PUR/PIR) (proportion of closed cells > 90%), the blowing agents remain in the insulation material over the long term.

### 3.5 Mechanical Properties

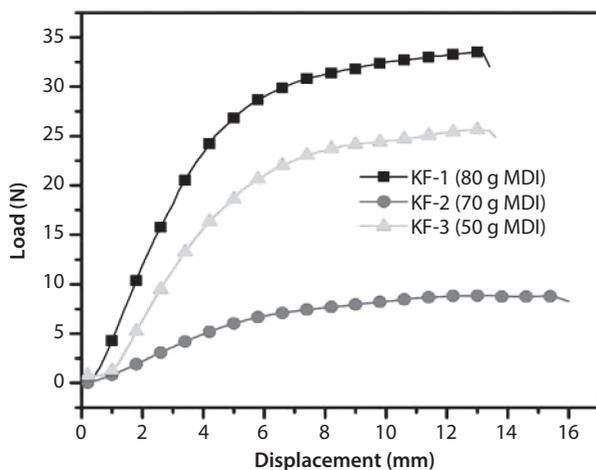
Flexural strength is the measure of the ability of the material to withstand the load applied perpendicularly to its longitudinal axis. Flexural tests were done in the direction parallel to the foam rise. The initial linear portion of three curves is due to the linear elastic deformation of the cell structures (within the elastic limit). As the material further stretches, the cells begin to rupture and the slope of the load-deflection curve changes, denoting the deformation of the foam, and at the maximum load the sample breaks. The cell stretching in the tension side of the specimen leads to multiple cracks below the central loading point which coalesce, leading to the eventual failure of the foam [5]. In the present case, the reason for higher FS value in KF-1 than KF-2 is the higher strength of cell walls in KF-1 compared to KF-2, despite similar density values. The flexural strength of closed-cell foam derives from its edges, walls, faces and the entrapped gas. For formulation 3, i.e., KF-3, the flexural strength was the same as that of KF-1 because of its closely packed cells and higher density. Higher MDI content led to higher flexural strength. This is due to the side reactions of isocyanates leading to formation of carbodimides and allophanates, which in turn reinforce the cell struts. The enhanced flexural strength is due to the strengthening of the cell walls. Generally, high density foams have higher flexural strength, so is the present case. In the case of the sample with equal amounts of polyol and MDI (KF-3) the flexural strength was closer to that of KF-1, indicating the influence of density on the mechanical property. Load vs. displacement graphs for samples with varying MDI amounts are shown in Figure 3.

Compressive strength of rigid polyurethane foam is basically a function of its density [27]. Compressive stress is generally determined at 10% deformation and this is known as short-term behavior. All the

**Table 3** Mechanical and thermal properties of karanja foams.

Sample code	Density (g/cc)	Thermal conductivity (W/m.k)	Compression strength (parallel) (N/m <sup>2</sup> )	Compression strength (perpendicular) (N/m <sup>2</sup> )	Flexural strength (N/m <sup>2</sup> )
KF-1	54–55	0.0415–0.0426	0.264–0.286	0.266–0.335	0.573–0.608
KF-2	52	0.0365–0.0377	0.211–0.254	0.232–0.165	0.293–0.374
KF-3	63–64	0.0400	0.184–0.217	0.229–0.332	0.496–0.668

specimens exhibit curves with an initial linear region corresponding to elastic deformation and a hump corresponding to the yield. The curves show a plateau region with a slight increase in the load upon further compression. Compression strength was found to be the same when tested in directions parallel and perpendicular to the foam rise (Figure 4a and 4b; Table 3). This is because of the spherical shape of the cells and isotropic nature of the foam samples. Composition with more MDI exhibits higher compression strength values for samples tested in directions parallel and perpendicular to the foam rise. When excess PMDI is used, it can react with formed urethane groups, generating allophanates, which introduce additional networks to the PUF sample, increasing the firmness and stiffness. In addition, it can react with amines (generated by reaction of NCO group with water) to form



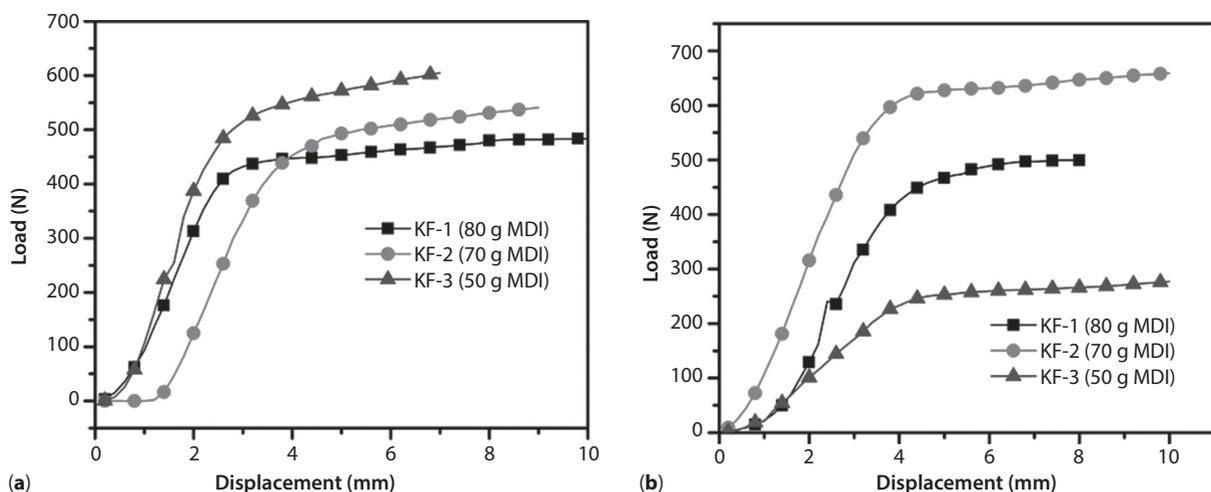
**Figure 3** Representative Load Vs. displacement graphs for karanja foams (KF) during flexural strength test.

urea groups [28–31]. Due to rigid crosslinks within the foam structure, elastic recovery is restricted, thus making foams rigid and stiff. Higher level of covalent crosslinking increases the modulus or stiffness of the polymer. Therefore, as the NCO index increases, the degree of crosslinking and mechanical strength increases and correspondingly compressive strength of the PUF samples increases. Figure 3 shows the load vs. displacement graphs during compression tests for samples with varying MDI amounts.

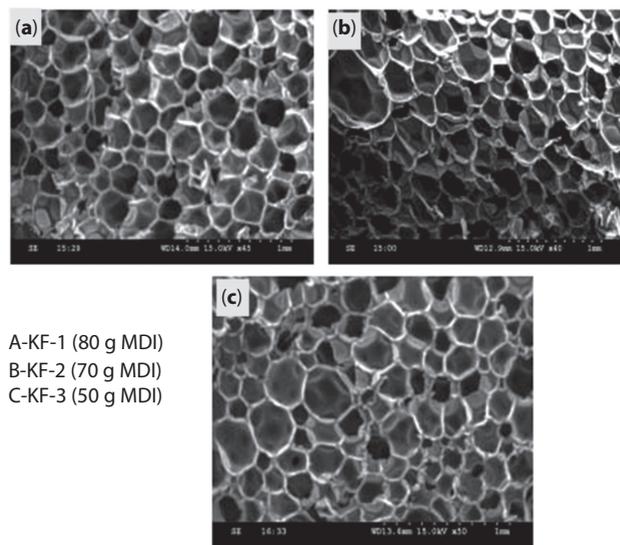
Generally, the density in the cellular materials is the main factor for determining the properties. As the density of the cellular materials increases, the mechanical strength increases. The compressive strength of the PUF sample prepared with less MDI is significantly lower in spite of its higher density. This is because of two reasons: the amount of MDI and blowing agent (water). Crosslinking is reduced and the side reactions leading to allophanate and urea linkages are absent, leading to poor mechanical properties.

### 3.7 Microstructure

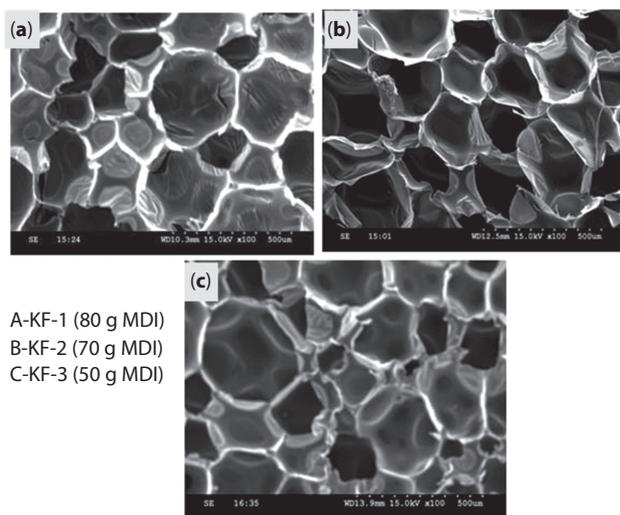
Thin slices of the foam samples were subjected to SEM analysis. The SEM images (Figures 5 and 6) show that the cells are spherical without any cell wall breakage. In the case of KF1 and KF2, with increasing amount of MDI or increasing NCO/OH ratio, we observe that the cells are no longer uniform and the struts and cell walls become thinner. However, the cell size (KF-1 = 0.23 and KF-2 = 0.29 microns) and density do not change significantly. In sample KF-3, cells had an average diameter of 0.315 microns. The presence of less amount of water compared to the other formulations might be the reason for this behavior, since water



**Figure 4** Representative Load Vs. displacement graphs for karanja foams (KF) during compression test in (a) parallel direction (b) perpendicular direction.



**Figure 5** SEM images of cross sections of foam samples of different composition at a magnification of 40x.



**Figure 6** SEM images of the foam samples taken at magnification of 100x.

aids in the liberation of  $\text{CO}_2$ , which in turn leads to bubble formation and foaming. Liberation of lesser amount of  $\text{CO}_2$  leads to fewer and larger bubbles.

## 4 CONCLUSIONS

The viability of karanja oil as a source of polyol for polyurethane foam development has been explored in this work. The two-step synthesis involved epoxidation of karanja oil followed by alcoholysis using ethylene glycol, which led to the polyol with a hydroxyl value of 186 mg KOH/g. The polyurethane foams were prepared by a water-blown process using a mixture of

karanja oil-based polyol and polymeric MDI with other additives. These cellular materials were found to have good mechanical properties which varied with isocyanate index. The cells had a spherical shape without any cell wall damage, thereby exhibiting a high percentage of closed cell content. As a result, the thermal conductivity values were also in a range which would be suitable for insulation purposes. This study has led to the development of rigid polyurethane foams using karanja oil, a non-edible oil, as a renewable source.

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