

# Effect of Foam Density on the Properties of Water Blown Rigid Polyurethane Foam

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**ABSTRACT:** Density is an important parameter that influences the properties and performances of rigid polyurethane foam (PUF). Rigid PUF with different densities were prepared by varying the amount of distilled water as blowing agent. This investigation reports the mechanical, morphological, water absorption, thermal conductivity, and thermal behavior of rigid PUF varying with the density, which controls the foam architecture. The density of the PUF decreased from 116 to 42 kg/m<sup>3</sup> with an increase in the amount of water from 0.1 to 3.0 parts per hundred polyol by weight (phr), respectively. It was found that the mechanical properties of the PUFs changed with the foam density. The results of water absorption of the PUFs showed that water absorption

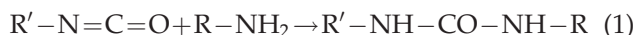
increased with decrease in density, due to increase in the cell size and decrease in the cell-wall thickness. The thermal conductivity measurements showed that the thermal conductivity decreased with increase in density. It was due to the decrease in cell size. The thermal analysis of the PUFs shows that the glass transition temperature increases with the decrease in foam density, but the thermal stability decreases with the decrease in foam density. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 108: 1810–1817, 2008

**Key words:** rigid polyurethane foam; foam density; thermal conductivity; compressive stress; morphology; polyurea

## INTRODUCTION

Rigid polyurethane foam (PUF) is recognized as an outstanding material for different applications. It has many desirable properties such as low thermal conductivity, low density, excellent dimensional stability, high strength-to-weight ratio, low moisture permeability and low water absorption. So, it is suitable for the application in household refrigerators, construction and industrial insulation, flotation in boats, life preservers, buoys, and other marine equipment. In aircraft, PUFs are used as stiffening and rigidizing agents and fillers in wing and fuselage structures.<sup>1</sup> The specific properties of PUF can be varied within limits by choice of raw materials, density, and method of applications. Density, however, is one of the main variables controlling most of the mechanical properties and has an effect much larger than that of other possible changes in polymer and foam structure. Hence, the study of the effect of density on the properties of rigid PUF has acquired importance for the broad range of its applications. Physical as well as chemical-blowing agents can be used in the preparation of PUFs. The distilled water

is one of the most widely used chemical-blowing agents. It reacts with diisocyanate and then generates gaseous carbon dioxide and polyurea [eq. (1)].



The carbon dioxide inflates reactant mixture; as a result, a cellular structure is formed. Therefore, some additional diisocyanate is needed that reacts with water.<sup>2,3</sup> The widely used physical-blowing agents are chlorofluorocarbons (CFCs) and hydrochlorofluorocarbons (HCFCs). The formation of PUF is based on the reaction of diisocyanate with polyol. The reaction is exothermic in nature, and the reaction heat can be used to form a cellular structure by the rough evaporation of physical-blowing agents present in the system. The foaming process can be explained by the nucleation and growth mechanism as discussed in the available literature.<sup>4,5</sup>

Nowadays, the use of CFCs and HCFCs was considered to be undesirable mainly because of environmental problems such as depletion of ozone layer and global warming. The emerging substitutes for PUF-blowing agents are several hydrofluorocarbons (HFCs), hydrocarbons (HCs), cyclopentane, and water,<sup>6</sup> because they show zero ozone depletion potential and reduce global warming problem. When water is used

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**TABLE I**  
**Characteristics of the Used Materials**

Materials	Equivalent weight	Functionality	Role
Polymeric methane diphenyl diisocyanate <sup>a</sup> (PMDI)	136.6	2.7	NCO content = 30.8%
Polyether polyol <sup>b</sup>	127.5	3.0	OH value = 440 mg KOH/g
<i>N,N,N,N</i> -penta methyl diethylene triamine <sup>c</sup> (PMDETA)	–	–	Catalyst
Polyether dimethyl siloxane <sup>d</sup>	–	–	Surfactant
Distilled water <sup>e</sup>	9.0	2.0	Blowing agent

<sup>a,b</sup> Supplied by Huntsman International Pvt, Ltd., Mumbai, India.

<sup>c</sup> Supplied by Aldrich.

<sup>d</sup> Supplied by Goldschmidt.

<sup>e</sup> Purified in our laboratory.

as blowing agent, carbon dioxide is the by-product, which is free of CFC. So, water is considered to be very ecofriendly blowing agent in the preparation of PUF. However, there are some disadvantages of water as a blowing agent; such as the system viscosity is high, forms more amount of heat (exotherm) while preparing the foam, which may lead risk of fire and scorch the foam. Sometimes, the system cost is high due to the increase in the use of excess of isocyanate.<sup>7</sup> Nevertheless, water and mixture of water and HCFC are used in many cases because of its environment-friendly characteristics. The final structure and properties of PUFs depend on the molecular architecture of the constituent polymer and processing condition.<sup>8</sup> Many authors have focused on the mechanical behavior related to the mechanics of cell deformation. Several research works have been carried out to identify the change in the properties of the rigid PUF with respect to the density. Generally, it has been found that the density affects the mechanical, morphological, insulation, and dimensional stability of PUF.<sup>9–13</sup> Kim and coworkers<sup>14</sup> have reported on the properties of the water-blown PUF, using different raw materials. Goods et al.<sup>15</sup> have prepared rigid PU foam and studied their mechanical properties. Jung et al.<sup>16</sup> reported on the properties of foam blown by HCFC and distilled water. Badri et al.<sup>17</sup> studied their mechanical properties and reported that PU foam can be prepared from palm-oil resources. Jian et al.<sup>18</sup> have prepared the rigid PUF from Rosin-based polyol and studied the structure and physical properties. In none of these cases, the concentration of blowing agent was optimized to obtain optimized properties.

This study investigation reports the optimization of the blowing agent to obtain desired density of rigid PUF, which gives optimized mechanical properties. The effect of foam density on the mechanical, morphological, water absorption, thermal insulation (thermal conductivity), and thermal properties of the water blown rigid PUF was investigated. In this case, scanning electron microscopy (SEM), UTM, TGA,

DTG, and DSC were used to characterize the properties of the PUF materials and to correlate them with its density. Importantly, all these properties of rigid PUF have been compared with those of the neat PUF, that is, prepared without any blowing agent.

## EXPERIMENTAL

### Materials

The polymeric methane diphenyl diisocyanate (PMDI, Suprasec 5005) and sucrose-based polyether polyol (Daltolac R 180) were obtained from Huntsman International, Mumbai, India (Daltolac R180 and Suprasec 5005 are trademarks of Huntsman). Distilled water was generated in our laboratory and was used as a chemical-blowing agent. *N,N,N,N*-pentamethyl diethylene triamine (PMDETA) was supplied from Aldrich and used as a catalyst. Polyether dimethyl siloxane (TEGOSTAB B 8460), supplied by Goldschmidt, Germany, was used as a surfactant. The characteristics of different materials used in the preparation of PUF are shown in Table I. All the chemicals were used as received.

### Preparation of foam

The PUF samples with various densities were prepared by one-shot and free-rising method, and their chemical compositions were shown in Table II. The

**TABLE II**  
**Chemical Compositions of Rigid Polyurethane Foam (PUF) Blown by Water**

Materials	phr <sup>a</sup>
Polyether polyol	100
PMDETA	0.5
Tegostab B 8460	2.0
Distilled water	(0.0 <sup>b</sup> , 0.1, 0.3, 1.0, 1.5, 3.0)
PMDI	(118, 119, 122, 134, 143, 167)

<sup>a</sup> Represents as parts per hundred of polyols by weight.

<sup>b</sup> PUF prepared without any blowing agent has been designated as WOBA.

foam density of free-rising method depends on the temperature of the reaction and thus will vary slightly depending on the size of the batch. The mixing of the components followed the same time for all the foams. All the raw materials, except PMDI, were first well mixed in a plastic beaker. Then PMDI was added into the beaker with vigorous stirring for 10 s. The resulting mixture was immediately poured into an open paper mold ( $30 \times 25 \times 15$  cm) to produce free-rise foams. After the preparation, the foams were kept in an oven at  $70^\circ\text{C}$  for 24 h to complete the polymerization reaction. Samples were cut after curing into specific shapes by rubbed with fine emery paper, and then the different properties of the foams were characterized. The amount of PMDI required for the reaction with polyether polyol and distilled water was calculated from their equivalent weights. For the completion of the reaction, excess PMDI (NCO/OH = 1.1) was used.

#### Measurement of different properties

The apparent density of the PUF samples was measured according to ASTM D 1622-03. The size of the specimen was  $25 \times 25 \times 25$  mm (length  $\times$  width  $\times$  thickness). The apparent densities of three specimens per sample were measured, and then the average values are reported. The mechanical properties of the PUF samples were measured at ambient condition with Instron UTM Hounsfield testing equipment (model H10KS). A compressive stress at 10% strain and compressive modulus (initial linear slope of the stress–strain curve) in parallel to foam rise direction were performed according to ASTM D 1621-00. The size of the specimen was  $55 \times 55 \times 30$  mm (length  $\times$  width  $\times$  thickness), and the rate of crosshead movement was fixed at 2.5 mm/min for each sample. The strength of five specimens per sample was measured, and the average of these values was reported. The energy absorption of the PUF was calculated from the area under the stress–strain compressive curves, and the average of five values was reported. The hardness (shore A) was tested on the durometer hardness tester, and the average of five values was reported. The morphology of the PUF samples was studied with a JEOL, JSM 5800 scanning electron microscope, Japan. The samples were gold-coated before scanning to provide an electrically conductive surface. The accelerating voltage was 20 kV to avoid degradation of the sample. The JSM 5800 was used to observe the size of the cells on the PUR samples and averaged. The water absorption of the PUFs samples was measured according to ASTM D 2842-01. The size of the specimen was  $40 \times 40 \times 20$  mm (length  $\times$  width  $\times$  thickness). The water absorption of three specimens per sample was measured and averaged. The thermal

conductivity of the PUFs was tested between two plates on a guarded hot-plate apparatus as per ASTM C 177-97. The size of the specimen was  $100 \times 100 \times 25$  mm (length  $\times$  width  $\times$  thickness). The thermal properties of the foams were examined on a differential scanning calorimeter (Q100, TA Instruments, USA) and thermogravimeter (Q50, TA Instruments, USA) under nitrogen environment at a heating rate of  $20^\circ\text{C}/\text{min}$ .

## RESULTS AND DISCUSSION

Rigid PUF was prepared by reaction with polymeric diphenyl methane diisocyanate (PMDI) as an isocyanate and a polyether polyol in the presence of PMDETA, an amine catalyst as shown in Table I. In this case, the density of the PUF varied by varying the concentration of water as blowing agent (WOBA). Figure 1 shows the variation in density of PUF with amount of distilled water concentration 0–3.0% based on the weight of polyol. With the increase of distilled water from 0.1 to 3.0 phr, the densities of the PUF decreased from 116 to  $42 \text{ kg}/\text{m}^3$ . The density of the foam without WOBA was  $118 \text{ kg}/\text{m}^3$ . The density of WOBA sample is higher due to more amount of polymer present per unit volume of PUF. In this case, a small amount of water or moisture, which remains as an inherent impurity in polyether polyol, probably acts as a blowing agent, and also the isocyanate (PMDI) tends to vaporize due to the high-exothermic reaction between polyol and isocyanate. This exothermic temperature is different from water containing formulation and WOBA formulation; the latter form pores within the polymer. Such pores, acting as stress concentrators, can significantly reduce the properties of the PUF. Figure 1

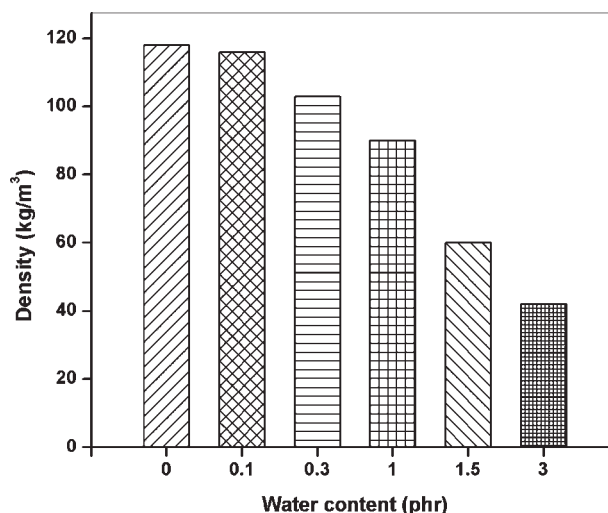
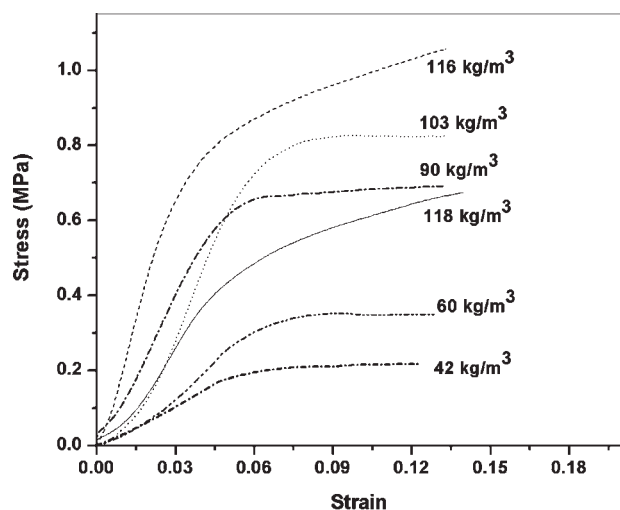


Figure 1 Density of the PUF as a function of water content.



**Figure 2** Stress–strain behavior of the PUF at different density.

clearly shows that the densities of the PUFs decreased with an increase in water content.

### Mechanical properties

Figure 2 shows the stress–strain curves of the PUF with different densities. It indicates that, to achieve a particular strain, stress increases, but it remains plateau after 10% strain. It may be due to cell-wall buckling of most of the cells in PUF. Table III shows the effect of density on the compressive stress at 10% strain and compressive modulus. When the density increases from 42 to 116 kg/m<sup>3</sup>, the compressive stress at 10% strain of the PUFs decreases from 8.7 to 2.0 kg/cm<sup>2</sup>. The compressive stress for WOBA foam was 4.7 kg/cm<sup>2</sup>. The compressive modulus also changed in a similar fashion.

Toughness is the resistance of the material to fracture or to undergo deformation and is calculated as the energy absorption from the area under the curve in the stress–strain curve.<sup>15</sup> Figure 3 shows the effect of density on toughness or energy absorption of the PUF. The results show that the toughness or energy absorption increases with the increase in density. Figure 4 shows the change in hardness of the PUFs with density. It shows that the hardness of the PUFs

increases from 15 to 45 with an increase in density from 42 to 116 kg/m<sup>3</sup>. Hardness is defined as the resistance to indentation. When the density increases, the cell size of PUF decreases and cell structure becomes smaller. Hence, more force is necessary to cause indentation in the PUF.

It is reported<sup>19</sup> that the Power law can be used to depict the relationship between the mechanical properties (compressive strength and modulus) and the density.

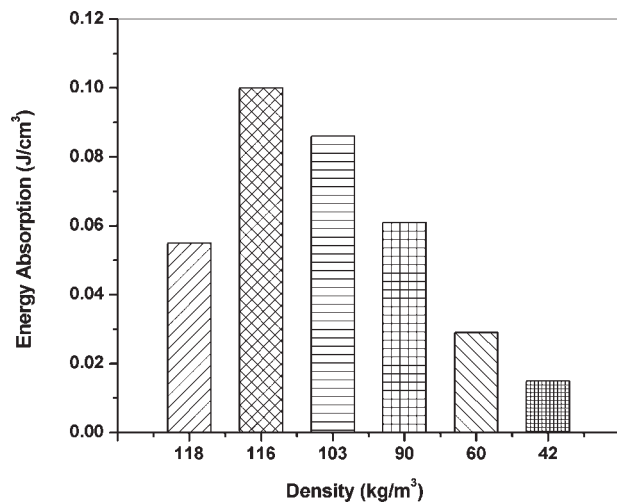
$$\log(\text{strength property}) = \log A + B \log(\text{density}) \quad (2)$$

where  $A$  is a constant related to the temperature and physical properties of the resin and  $B$  is related to the deformation mechanics of cellular materials.<sup>15,19</sup> The compressive strength and modulus of PUF exhibit the dependence of Power law with respect to the foam density. The slope of the plot of  $\log(\text{property})$  versus  $\log(\text{density})$  was used for the determination of the density exponent value and was calculated as 1.5 for compressive strength and 1.72 for compressive modulus as shown in Figures 5 and 6. These values are quite similar to the values reported (in the range 1–2) in the literature.<sup>15,19</sup> Goods et al.<sup>15</sup> reported the density exponent of 2.10 and 1.60 for compressive strength and modulus, respectively, for the water blown rigid PUF. In this case, they prepared PUF with much higher packed density via molding technique from sucrose-based polyether polyol (OH number 490 mg KOH/equiv per gram) and MDI in the presence of a diamine catalyst. In our case, we prepared PUF by free-rise method from similar type of polyols (but with lower OH number) and a triamine as catalyst. However, the strength and modulus of PUF exhibit Power law dependence with respect to the foam density although the strength density exponent is somewhat different from the density exponent for the modulus.

Distilled water used as chemical-blowing agent reacts with the isocyanate group to generate carbon dioxide and polyurea with the release of the heat (exothermic reaction). Polyurea is known to be more rigid than polyurethane. The urea group can further reacts with isocyanate group to generate biuret, which introduces additional networks inside the

**TABLE III**  
Effect of Density on the Compressive Stress and Compressive Modulus of PUF

Sr. No.	Sample name	Water content (phr)	Density (kg/m <sup>3</sup> )	Compressive stress at 10% strain (kg/cm <sup>2</sup> )	Compressive modulus (MPa)
1	WOBA	0	118	4.7	9.4
2	PUF-1	0.1	116	8.7	21.6
3	PUF-2	0.3	103	8.1	15.2
4	PUF-3	1.0	90	6.1	11.7
5	PUF-4	1.5	60	3.5	6.5
6	PUF-5	3.0	42	2.0	3.2

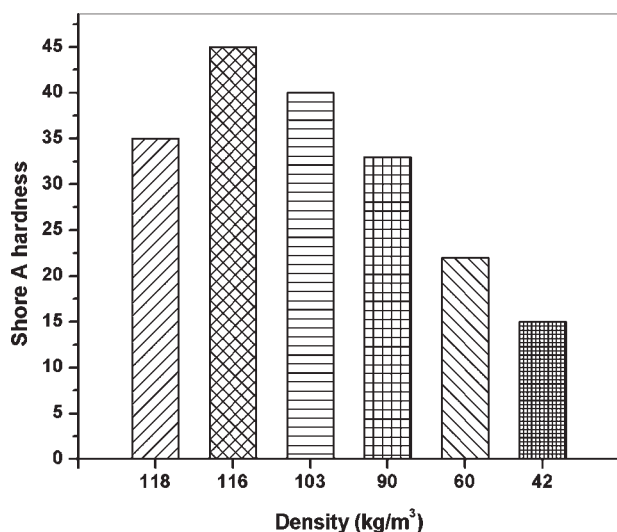


**Figure 3** Effect of density on energy absorption of the PUF.

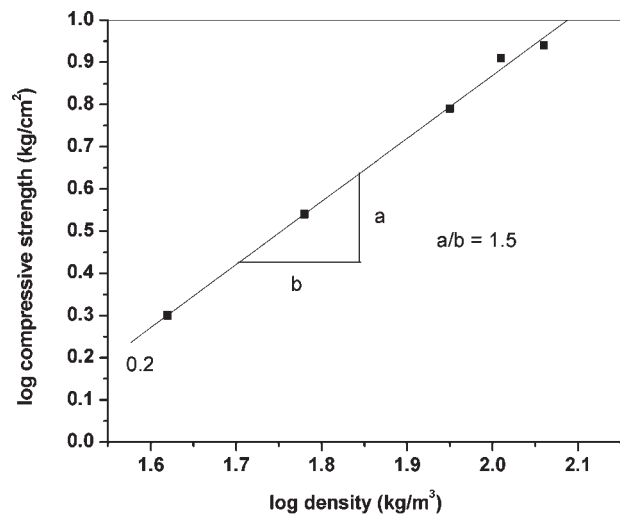
PUF.<sup>20</sup> Results of the mechanical properties of the PUFs reveal that the strength properties increase with the increase in density of the PUFs. This can be explained by the Power law [eq. (2)]. The results so far explained that the mechanical strength properties are poor in the WOBA samples. This is due to the fact that most of the cells were broken [as observed by SEM Fig. 7(d)] and did not form the uniform cell structure.

### Morphology

In general, the physical properties of foam not only depend on the rigidity of the polymer matrix, but also relate to the cellular structures of the foam. Thus, it is of interest to observe the fracture of the foam specimen using SEM. Figure 7(a–d) shows the

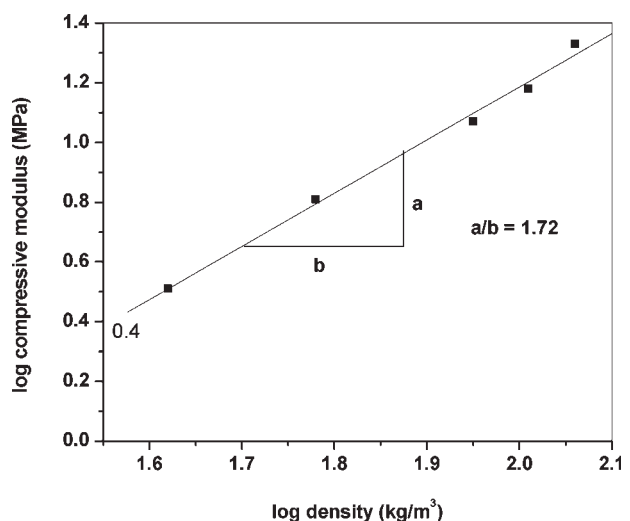


**Figure 4** Effect of density on hardness of the PUF.

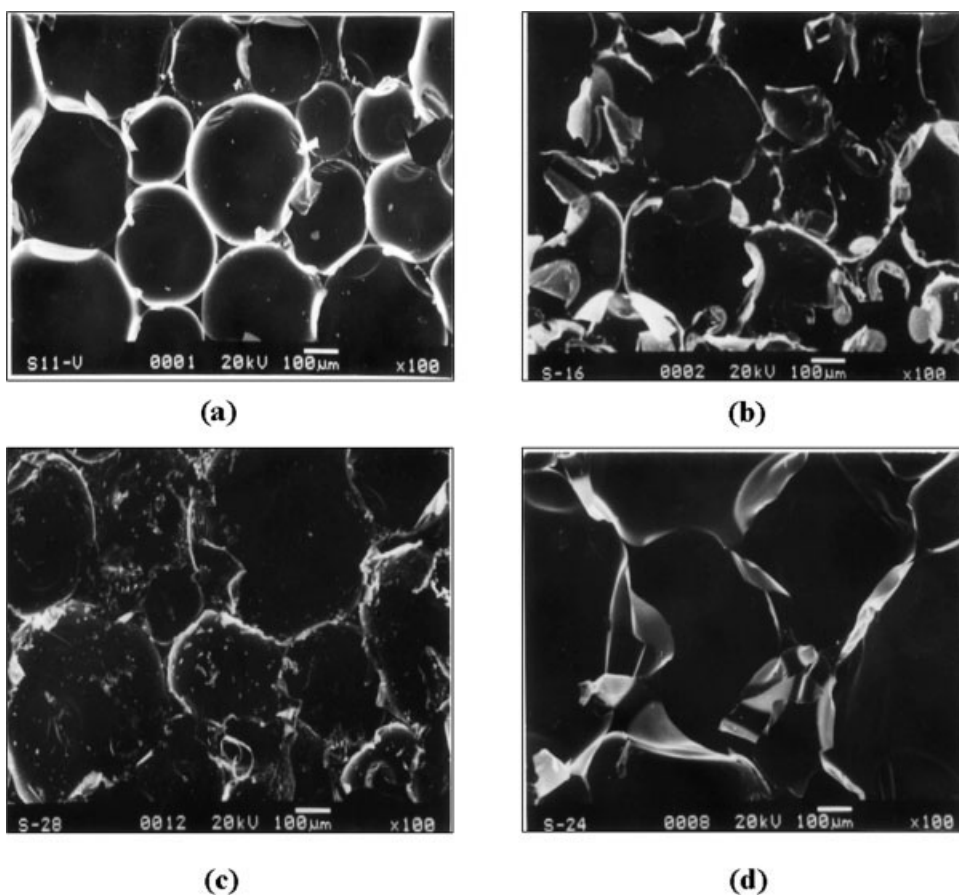


**Figure 5** Density dependence on the compressive strength of the PUF.

microphotographs of the fracture surfaces for PUF with different densities. In case of high density PUF, that is, with less amount of WOBA, the cells are more uniform, well-defined, and the cell-wall is thinner [Fig. 7(a)]. When the blowing agent increases, the foam density decreases, and the cells are not uniform, and, in most cases, they are broken [Fig. 7(b,c)]. In case of WOBA, the cells are much larger and broken probably because of nonuniform distribution of moisture (an inherent impurity in polyether polyol) as blowing agent. The average cell size was calculated from the SEM photographs. Figure 8 shows the effect of foam density on the average cell size of the PUFs. The average cell size of the PUFs increases from 233 to 488  $\mu\text{m}$  with the decrease in density from 116 to 42  $\text{kg/m}^3$ , and for WOBA sample cell size was found to be 367  $\mu\text{m}$ .



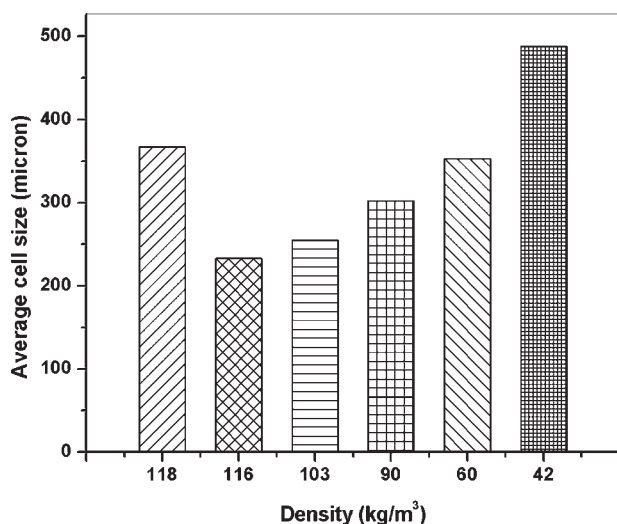
**Figure 6** Density dependence on the compressive modulus of the PUF.



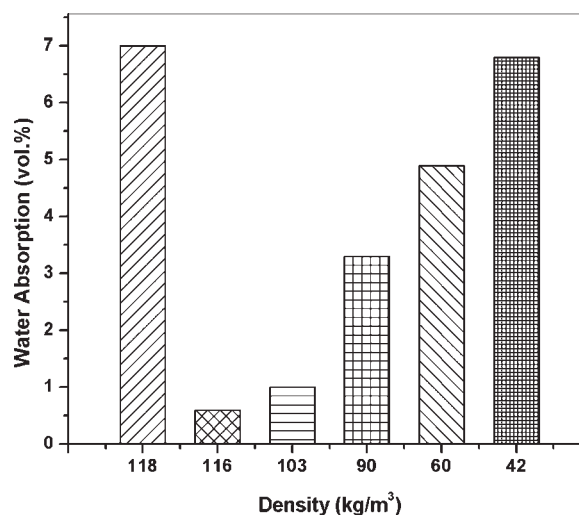
**Figure 7** Microphotographs of PUF samples of different density: (a) PUF with  $103 \text{ kg/m}^3$ ; (b) PUF with  $60 \text{ kg/m}^3$ ; (c) PUF with  $42 \text{ kg/m}^3$ ; and (d) PUF (WOBA) with  $118 \text{ kg/m}^3$ .

The size of the cell is important in controlling the mechanical and thermal insulation properties of PUF. A chemical-blowing agent such as distilled water generates carbon dioxide through the chemical reaction with diisocyanate accompanying the exo-

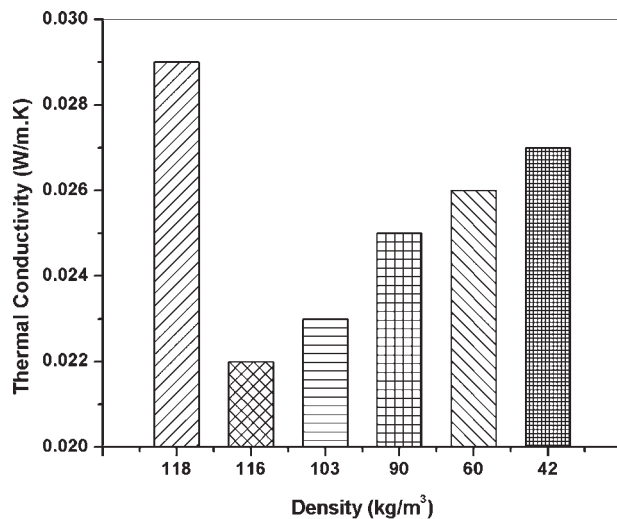
thermic reaction heat. Because of the increase in temperature, the concentration of blowing gas in the mixture exceeds its limit of solubility, and thus the nucleation process of bubbles begins. With the increase of WOBA, number of such bubbles formed



**Figure 8** Effect of density on average cell size of the PUF.



**Figure 9** Effect of density on water absorption of the PUF.



**Figure 10** Effect of density on thermal conductivity of the PUF.

increase and later they coalesce resulting in the increase in cell size.

### Water absorption

The mass and volume of the PUF samples were taken and designated as  $M$  and  $V$ , respectively. After immersing into water for 96 h, the weight of samples was taken again. If the mass of the samples after water soaking is  $N$ , the water absorption ( $W_A$ ) of the samples in volume percentage is calculated as

$$W_A = (N - M)/V \times 100 \quad (3)$$

Figure 9 shows the effect of density on the water absorption of the PUFs. It is clear from the figure that the water absorption of the PUFs decreases from 6.8 to 0.6 vol % with an increase in density from 42 to 116 kg/m<sup>3</sup>, in the WOBA it was 7.0 vol % at a density of 118 kg/m<sup>3</sup>. It is generally known that the amount of water absorption of a cellular material mainly depends on the cell structure of foam whether it is closed or open and also on cell wall (struts) thickness.<sup>21</sup> Figure 9 indicates that high density foam absorbs less amount of water. Because the cells become smaller in size as the density of foam increases. This was already confirmed from the SEM studies (Fig. 7). WOBA sample absorbed more water; in this case, the cell size is much larger, the cells are broken and are interconnected to accommodate more amount of water.

### Thermal conductivity

The thermal insulation properties of materials are mostly expressed in terms of their  $K$ -factor, which are determined by the following equation,

$$K = Q \times S/A \times \Delta T \quad (4)$$

where  $K$  is the thermal conductivity (W/m K),  $Q$  is the heat flow (W),  $S$  is the sample thickness (m),  $A$  is the sample area (m<sup>2</sup>), and  $\Delta T$  is the temperature difference across the plate (K). The insulating capacity increases as the  $K$  value decreases. The thermal conductivity is defined as the rate of heat transfer through unit thickness, across unit area, for unit difference in temperature. In general, the thermal conductivity of PUF depends on the foam density, cell size, cell orientation, ratio of close to open cell content and on the thermal conductivity of the gases (blowing agent), and filling materials. Figure 10 shows the effect of density on the thermal conductivity of the PUFs. It indicates that as the density of the foam increases, the thermal conductivity decreases. It may be due to the decreased radiant heat transfer via trapped gases in lower foam cells in the higher density PUF.<sup>22</sup>

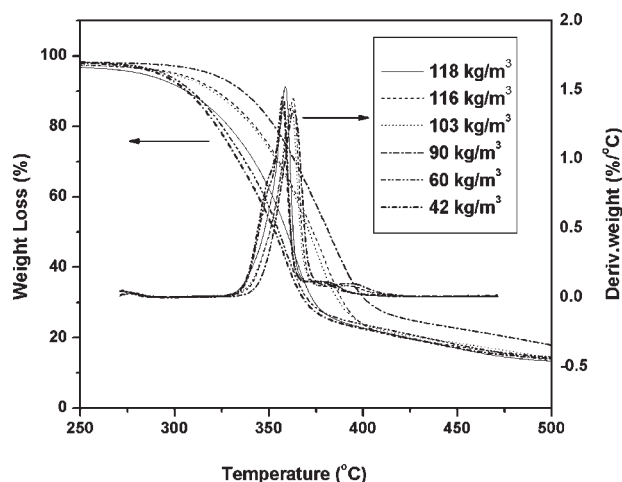
### Thermal analysis of PUF

The glass transition temperatures ( $T_g$ ) of the PUF samples measured by differential scanning calorimetry (DSC) are shown in Table IV. The results indicate that the  $T_g$  of the PUF samples increases slightly with the increase in water content and decrease in density. It is due to increase in the crosslink density by the formation of polyurea, which is known to be more rigid than polyurethane. The mobility of the polymer chains decreased and hence the  $T_g$  of the PUF increases. Seo et al.<sup>20</sup> also observed that the  $T_g$  of PUF increases with the increase in water content.

Figure 11 shows the effect of density on the weight loss and degradation temperature of the PUF. The degradation temperature ( $T_{max}$ ) varies with the amount of water content and foam density. In samples PUF 1–3 has higher thermal stability, due to sufficient amount of water reacts with excess of PMDI to form polyurea linkage. WOBA has medium thermal stability because it has insufficient water content to form polyurethane linkage. PUF 4 and 5 have lower thermal stability. It might be due to

**TABLE IV**  
Effect of Density on the Glass Transition Temperature ( $T_g$ ) and Thermal Stability ( $T_{max}$ ) of the PUF

Sr. No.	Sample name	Water content (phr)	Density (kg/m <sup>3</sup> )	$T_g$ (°C)	$T_{max}$ (°C)
1	WOBA	0	118	91	363
2	PUF-1	0.1	116	101	374
3	PUF-2	0.3	103	105	375
4	PUF-3	1.0	90	107	371
5	PUF-4	1.5	60	106	360
6	PUF-5	3.0	42	110	359



**Figure 11** Effect of density on the weight loss and degradation temperature of the PUF.

more amount of water reacts with excess PMDI to form biuret and allophanate linkages. It follows the general route of the thermal stability of polyurethane linkages. Biuret and allophanate are the thermally weakest links in the PU network, next in thermal stability trends are the urethanes, closely followed by ureas.<sup>23</sup>

## CONCLUSIONS

Rigid PUFs with varying density using water as a blowing agent were prepared via free-rise method. The results of the mechanical properties of this PUF indicated that they change with the density of PUF and follow the Power law. The increase in the mechanical properties of the PUF was due to the increase in the crosslinks and formation of additional crosslinks that arose from biuret formation by a reaction between distilled water and PMDI. The studies of morphology by SEM showed that the cell size of the PUF increased with the decrease in density. It was due to the evolution of more blowing gas and with increase in distilled water. Interestingly, the thermal conductivity of the PUF increased with the decrease in the density, which is due to convective transfer by trapped gases in large cells. The glass transition temperature of the PUF slightly increased with the increase in water content and decrease in density due to the rigid polyurea and additional

crosslinks arising from biuret formation. The thermal stability of the PUF decreased with the increase in water content and decrease in density, due to the formation of thermally weakest links such as biuret and allophanate in lowest density and higher water content.

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