

**Studies on Flexible Polyurethane Foam with improved Flame Retardancy****A Study on the effect of Flame Retardant in Flexible Polyurethane Foam**

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**Abstract** – Most of the domestic (interior decoration) and industrial systems use polyurethane (PU) based material. Flexible polyurethane foam ( $PUF_F$ ), a PU derivative is versatile because of its wide applications in many aspects and the main drawback of  $PUF_F$  is its inflammability. The combustibility nature may end up with the fire hazard and accidents. The fire related accidents may be prevented by incorporating a suitable flame retardant (FR) in  $PUF_F$ . The presence of flame retardants (FRs) mainly postpones the flashover which lessens the intensity of the burning rate. Brominated halogen based decabromodiphenyl ether (decaBDE) is selected as FR in this present study. decaBDE is incorporated as a FR to subsume the FR characteristics in  $PUF_F$  in order to overcome the fire related accidents which is alarming in current condition. The composition of decaBDE was varied from 1 % to 5 % in order to assay the effect of added FR in the laboratory based developed  $PUF_F$  using polyol and isocyanate as the precursors. The effect of addition of decaBDE in  $PUF_F$  was studied by analysing mechanical property (tensile strength, rebound resilience), physical property (density), thermal property (thermogravimetric analysis-TGA) and flammability test (UL 94). The cell structure (morphology) and the presence of functional groups in the developed  $PUF_F$  were confirmed by using optical microscope and Fourier Transform Infrared Spectroscopy (FTIR) respectively. The paper reports that the developed  $PUF_F$  confirms the improved FR characteristics than the unmodified (without addition of FR)  $PUF_F$ .

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**Keywords**-Halogen FR; decabromodiphenylether; flexible polyurethane foam; flame retardant; brominated FR

**I. INTRODUCTION**

Polyurethane (PU) is one of the attractive polymeric engineering material, promising in wide areas and used in different types of industries. These are linear polymers with  $-NHCO_2$  (carbamate) groups. This is basically developed by the addition of diisocyanates/polyisocyanates with diols/polyols. When polyol is introduced to the isocyanate, foam formed can be decoded by several parameters like cream, jel and rise time, recession and expansion factor [1]. Polyurethane foams (PUFs) in generally categorized into two types with respect to the cell structure, such as rigid PUF ( $PUF_R$ ) and flexible PUF ( $PUF_F$ ) with closed and open cell structure, respectively. The flexibility or rigidity nature of the PUFs product purely depends on the group of polyol and diisocyanates used for the preparation of PU. The development of  $PUF_F$  is generally a step-growth polymerization of isocyanate and polyol (molecular weights ranges from 2000-10000 g/mol) with additives [2]. The production and consumption of  $PUF_F$  is increasing worldwide. In 1960, 45,000 metric tons (mt) of  $PUF_F$  was produced. In 1985, about 1.4 million mt (mmt)  $PUF_F$  was produced and half of them were consumed by United States [3]. In 2007, it was increased to 7 mmt (5 times more than in 1985) and the average annual growth rate was increased to 5 % [4]. Further it has been reported recently that global polymeric foam market may reach 18 mmt by 2015 [5].

Polyurethanes (PUs) are widely used in the manufacture of furniture, bedding foams ( $PUF_F$  and  $PUF_R$ ), coatings adhesives/binders, footwear, building insulation, sealants, automobiles, refrigerators and freezers [6-9] etc. Though PU is combustible by nature, its applicability in domestic and industries play a vital role because of its innate qualities like durability, shape maintenance (memory foam), softness and good support. The end product of PU based materials can be designed in a creative way because of its versatility characteristics. The application of PUFs in seating (domestic/industry) as a filling material for cushioning purpose is inevitable. The ability to burn immediately with minimum flame source is one of the main drawback of the  $PUF_F$ . The indispensable usage in abundant quantity leads to the innovative, creative way of developing  $PUF_F$  considering the safety aspects currently.

Flame retardants (FRs) are mainly classified as inorganic, halogenated, non halogenated, nitrogen, phosphorous and sulphur based. In general, brominated FRs are cost effective and is used in abundant quality in FR category. decaBDE is a halogen based FR and is tagged as the second largest FR comes under the brominated group. In general, halogen based FR functions by interfering with the radical chain mechanism which occurs normally in the gas phase during combustion. The radicals (high energy OH and H) formed during burning will be removed by the bromine (escapes during combustion), which is present in brominated FRs [10]. decaBDE is used as FR in polyethylene wire and cable

application, high impact polystyrene (HIPS), PUF<sub>F</sub>, textiles etc. It is usually insoluble in water, melts at 304 °C-309 °C and considered as less toxic [11]. Registration, Evaluation, Authorization and restriction of Chemicals (REACH) status has identified decaBDE as the one among the main halogenated FRs with the identification in lack of risks with the usage [12]. The usage of the FRs in PUF<sub>F</sub> has been reported earlier [13]. PUF<sub>F</sub> added with FRs if ignited, FRs present in it will decrease the combustion rate and hence extinguishes the fire by spreading to the other adjacent materials. FRs may act in solid, liquid and gas phase either chemically or physically. During combustion, FRs may play role in any one of the processes like heating, decomposition, ignition/flame spread [14]. The addition of FRs in PUF<sub>F</sub> will alter the physical, chemical and thermal characteristics of PUF<sub>F</sub> [15]. It has been reported that the addition of FRs may also reduce the combustion rate [16].

The versatility of the product and addition of FRs in the modification of PUFs is gaining interest currently. Inflammability characteristics of PUF<sub>F</sub> leads to fire related accidents. This type of fire accidents can be prevented by the addition of FRs. The demand, requirement and inevitability paved the way for the present investigation by incorporating decaBDE, aiming modified, improved FR characteristics in PUF<sub>F</sub>.

## II. MATERIALS AND METHODS

### 2.1. Raw Materials

The basic precursors used in the present investigation are polyether polyol, isocyanate (toluene diisocyanate (TDI)) and decabromodiphenylether (decaBDE). Polyol and the decaBDE were stirred in order to homogenize the samples thoroughly. After stirring, isocyanate was introduced to the homogenized samples. This mixture (polyol, decaBDE and isocyanate) was poured into the mould made with respect to the required PUF<sub>F</sub> size. The developed PUF<sub>F</sub> at the laboratory level was left curing for atleast 24 h. After the curing period, the testings were done using the appropriate methods and standards.

### 2.2. Morphology

The cell structure of PUF<sub>F</sub> developed was analysed by using optical microscope. In this study, optical microscope was used to examine the morphology of the developed PUF<sub>F</sub>. The developed PUF<sub>F</sub> cell structure was studied with 50 X magnification. The optical microscope used in the present study was CARLL ZEISS (AX10 ERC5s), Germany. The images were read by using Axiovision Rel.4.8 software.

### 2.3. Fourier Transform Infrared (FTIR) Spectroscopy

PUF<sub>F</sub> samples for the FTIR spectra analysis was washed with the distilled water and dried in oven before the analysis. The FTIR spectrum of the PUF<sub>F</sub> developed was analysed in the range of 4000-400cm<sup>-1</sup> using JASCO 6300 model (Japan). This was used to confirm the presence of functional group present in the developed PUF<sub>F</sub>.

### 2.4. Thermogravimetric Analysis (TGA)

Thermo gravimetric analysis was done using EXSTAR 6000 (Japan). TGA was carried out in order to decipher the thermal stability of the developed PUF<sub>F</sub> samples. The developed PUF<sub>F</sub> test samples of 2-3 mg was placed in a specimen holder and was heated upto 800 °C at a uniform heating rate of 20 °C /min in an inert nitrogen (N<sub>2</sub>) atmosphere (N<sub>2</sub> flow rate 140 mL/min). The weight loss of the sample for a particular temperature range of 30-800 °C is noted. The weight loss is generally a indicator of the thermal stability, sample composition, inert and volatile content of the sample as well. The analysis was recorded by using muse measurement software.

### 2.5. Flammability Test (UL 94 Test)

The flammability test was usually carried out in order to check the FR property of the sample. In this study UL-94 tests was carried out with respect to the standard [17]. UL 94 horizontal burning (HB) test for foam materials (HBF-1 or -2) was done using a vertical burner with a blue flame at a height of 25 mm. The PUF<sub>F</sub> samples were subjected for the test. The PUF<sub>F</sub> sample specimen size used was 127 mm x 12.7 mm x 3.2 mm according to the standards.

### 2.6. Tensile Strength

The strength and the elasticity of the PUF<sub>F</sub> samples under tension were studied by the parameter tensile strength. It is one of the mechanical test methods, performed by using a universal testing machine (UTM) according to the standard IS 7888 [18]. The tests were performed on the developed PUF<sub>F</sub> using UTM machine with a cross head speed of

5.00 mm/min as per the standard. The DAK system inc (SERIES 9000), India was used to measure the tensile strength of the developed PUF<sub>F</sub> samples. The sample tensile strength was decoded by DAK run software.

### 2.7. Rebound Resilience

Rebound resilience (RR) is a procedure where surface resilience/elasticity/springiness of PUF<sub>F</sub> can be evaluated. RR of PUF<sub>F</sub> was determined by resilience tester using ASTM D 3574 standards. The tester used for the present study was Blue Steel Engineers PVT. LTD, India. A steel ball present in the tester of 16 mm diameter was released and made to fall from a particular height (assigned) on the PUF<sub>F</sub> sample. The % of the ball rebounds was calculated. From this RR values were calculated. The higher % indicates PUF<sub>F</sub> has better resilience.

### 2.8. Apparent Density

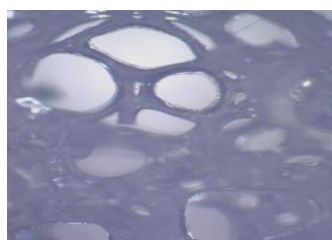
Apparent density test of the developed PUF<sub>F</sub> samples was assayed with respect to ISO 845:2006 (E) standards [19]. The apparent density of PUF<sub>F</sub> was calculated with respect to the mass and dimensions of the samples according to the procedure.

## III. RESULT AND DISCUSSION

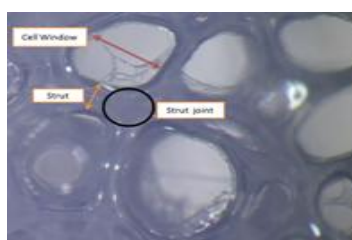
Decabromodiphenyl ether (decaBDE) comes under the group of polybrominated diphenyl ethers and is one among in the category of brominated flame retardant (BFR). This bromine based FR is used in this study to assay the effect of its addition in PUF<sub>F</sub>.

### 3.1. Morphology

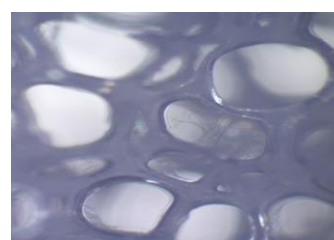
The cell structure of PUF<sub>F</sub> can be easily deciphered by assaying the sample under optical microscope. Figure 3.1 (a-f) shows the open cell structure (OCS) of the modified and unmodified PUF<sub>F</sub> samples with different % of decaBDE. The modified PUF<sub>F</sub> with decaBDE and the unmodified PUF<sub>F</sub> shows the OCS. This reveals that the developed PUF is flexible one. OCS, spherical shape was observed in all the samples with respect to the unmodified PUF<sub>F</sub>. Cell window, strut and strut joint were found in all the images with respect to the samples additionally confirms developed one at the laboratory level is PUF<sub>F</sub> [20].



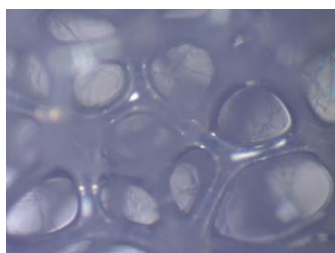
**Figure. 3.1.a. unmodified PUF<sub>F</sub>**



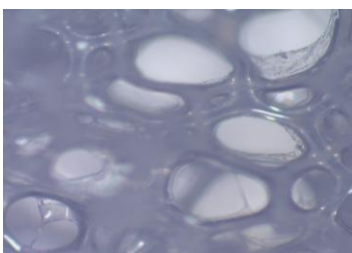
**Figure.3.1.b.1% decaBDE PUF<sub>F</sub>**



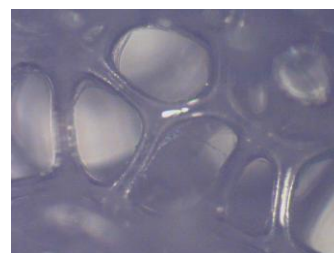
**Figure 3.1.c.2 % decaBDE PUF<sub>F</sub>**



**Figure.3.1.d. 4% decaBDE PUF<sub>F</sub>**



**Figure 3.1. e.4% decaBDE PUF<sub>F</sub>**



**Figure.3.1.f.5 % decaBDE PUF<sub>F</sub>**

### 3.2. Fourier Transform Infrared (FTIR) Spectroscopy

The functional group present in the material was evaluated by using FTIR spectroscopy. All the samples taken for the study was fixed in the scanning range from 4000-400 cm<sup>-1</sup>. The spectrum of the modified and unmodified PUF<sub>F</sub> was found to be similar. The peak at 1091.59 cm<sup>-1</sup>, 1086.69 cm<sup>-1</sup>, 1093.44 cm<sup>-1</sup>, 1091.51 cm<sup>-1</sup> and 1091.51 cm<sup>-1</sup> shows the presence of C-N stretch for 1 %, 2 %, 3 %, 4 % and 5 % of decaBDE samples, respectively which normally lies in the range 1250-1020 cm<sup>-1</sup>. The FTIR of the PUF<sub>F</sub> samples confirms the presence of carbonyl adsorption bond (C-O stretch:

presence of non-H bonded urea carbonyl stretch) at  $1708.62\text{ cm}^{-1}$ , for 1 % - 5 % of decaBDE samples, respectively. The frequency bands in the range  $2850\text{ cm}^{-1}$ - $3000\text{ cm}^{-1}$  confirms the presence of alkane C-H stretch. Figure 3.2 shows  $2902.34\text{ cm}^{-1}$ ,  $2937.70\text{ cm}^{-1}$ ,  $2902.34\text{ cm}^{-1}$ ,  $2971.77\text{ cm}^{-1}$  and  $2971.77\text{ cm}^{-1}$  stretch for the samples for 1 %, 2 %, 3 %, 4 % and 5 % of decaBDE respectively. The absorbance at  $3341.07\text{ cm}^{-1}$ ,  $3342.03\text{ cm}^{-1}$ ,  $3341.07\text{ cm}^{-1}$ ,  $3337.21\text{ cm}^{-1}$  and  $3340.10\text{ cm}^{-1}$  is the stretching of free NH band (hydrogen-bonded NH with oxygen (ether) and hydrogen-bonded NH with oxygen (carbonyl) in the urethane) for 1 %, 2 %, 3 %, 4 % and 5 % of decaBDE samples, respectively [21]. The band in this region, generally confirms the characteristics of urea and urethane groups. The same has been reported [21, 22]. In the FTIR spectrum the absorbance between  $2270\text{ cm}^{-1}$ - $2240\text{ cm}^{-1}$  was not observed. This shows and confirms that the isocyanate is converted completely to  $\text{PUF}_F$  i.e. urea and urethane bonds with the addition of polyol along with the decaBDE [23].

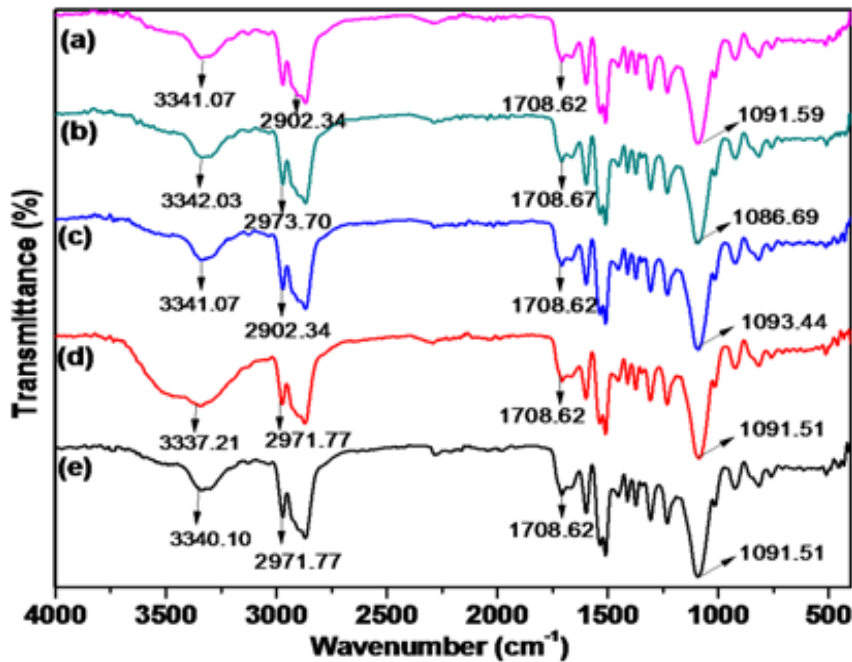


Figure. 3.2 FTIR Spectrum of the  $\text{PUF}_F$  with various % of decaBDE ((a)-1 % decaBDE, (b)-2 % decaBDE, (c)-3 % decaBDE, (d)-4 % decaBDE, (e)-5 % decaBDE)

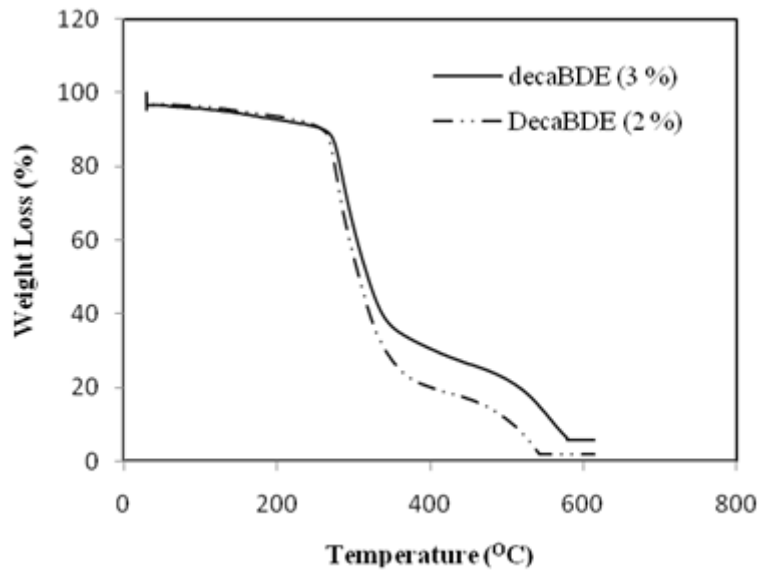
### 3.3. Flammability of $\text{PUF}_F$ -UL 94 Rating

The burning characteristics of  $\text{PUF}_F$  can be easily decoded by UL 94 rating test. Burning extinguishes, in a horizontal bar (HB) test of not more than 38.1 mm/min (0.64 mm/sec) for the sample of thickness 3 mm to 12.7 mm is classified as HB in UL 94 rating. The % of decaBDE varied was 1 % - 5 %. The time to extinguish the flame was observed on an average of about 2.19 mm/s, 1.97 mm/s, 0.45 mm/s, 1.51 mm/s and 1.25 mm/s for 1 %, 2 %, 3 %, 4 % and 5 % decaBDE samples, respectively. This burning time was found to be less when compared to the unmodified  $\text{PUF}_F$ . The best condition with respect to the testing method was found at 3 % decaBDE samples. With respect to the increase in % of decaBDE, the resistance to the flame was observed more in the case of 3 % decaBDE samples with the extinguish time of 0.45 mm/s.

### 3.4. Thermogravimetric Analysis (TGA) – Thermal Characteristics of $\text{PUF}_F$

TGA, one of the promising tools is used in this present study, in order to evaluate the effect of addition of decaBDE on the thermal property, i.e. thermal stability of the  $\text{PUF}_F$  developed at the laboratory level. The decomposition of developed  $\text{PUF}_F$  was performed in the temperature range from  $30\text{ }^\circ\text{C}$ - $800\text{ }^\circ\text{C}$ . The thermal stability was assayed at 5 % and 50 % weight loss of  $\text{PUF}_F$  samples. 5 % and 50 % weight loss corresponds to the initial and soft segments decomposition respectively [24]. Figure 3.3 shows 5% and 50 % weight loss of 2 % and 3 % decaBDE TGA. 5 % weight loss was observed at  $117.5\text{ }^\circ\text{C}$ ,  $136.6\text{ }^\circ\text{C}$ ,  $206.7\text{ }^\circ\text{C}$ ,  $242.4\text{ }^\circ\text{C}$ ,  $147\text{ }^\circ\text{C}$  and  $138\text{ }^\circ\text{C}$  for unmodified, 1 %, 2 %, 3 %, 4 % and 5 % decaBDE  $\text{PUF}_F$  samples, respectively. In case of 50 %, weight loss (decomposition) was observed at  $307.9\text{ }^\circ\text{C}$ ,  $318.5\text{ }^\circ\text{C}$ ,  $324.4\text{ }^\circ\text{C}$ ,  $354.9\text{ }^\circ\text{C}$ ,  $319.2\text{ }^\circ\text{C}$  and  $311.2\text{ }^\circ\text{C}$  for unmodified 1 %, 2 %, 3 %, 4 % and 5 % decaBDE  $\text{PUF}_F$  samples, respectively. The temperature at 3 % decaBDE samples was found to be  $242.4\text{ }^\circ\text{C}$  and  $354.9\text{ }^\circ\text{C}$  at 5 % and 50 % weight loss respectively. This shows the higher thermal stability than the other samples with respect to the different % of

decaBDE. This can be correlated with the UL 94 results where the 3 % decaBDE shows best UL 94 rating with flame extinguishing time of 0.45 mm/s when compared with the other samples. It was found that modified samples with the addition of various % of decaBDE shows higher stability when compared with the control (unmodified without the addition of decaBDE).



*Figure.3.3. Thermogram of decaBDE PUF<sub>F</sub> (2 % and 3 %)*

### 3.5. Apparent Density

The apparent density values ranges from 36 Kg/m<sup>3</sup> to 51 Kg/m<sup>3</sup>. Figure 3.4 shows that with the increase in % of decaBDE the density of the PUF<sub>F</sub> increases and its almost constant with increase in 3 % to 5 % decaBDE samples.

### 3.6. Rebound Resilience

Rebound Resilience (RR) is one important physical property in conforming the comfortness in the seating (cushion/foam) material. The comfort rating increases with respect to the increase in RR %. Figure 3.4 shows the effect of RR with respect to the different % of decaBDE in PUF<sub>F</sub>. RR was found to be in the range of 28.34 % to 32.14 % with the varying % of decaBDE in PUF<sub>F</sub>. This shows that addition of FR did not affect the RR of PUF<sub>F</sub>.

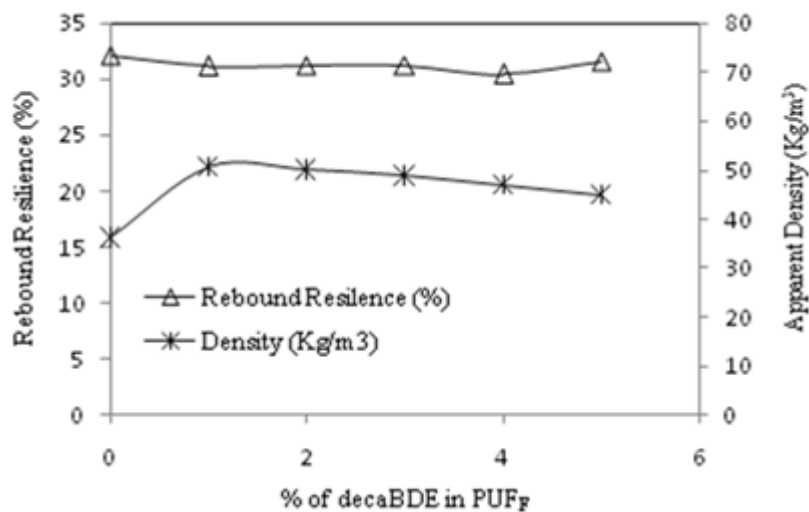


Figure.3.4. Rebound Resilience and Density of decabDE PUF<sub>F</sub> (2 % and 3 %)

### 3.7. Tensile strength-Mechanical properties

Figure 3.5 shows the effect of tensile strength with the various % of decabDE in the developed PUF<sub>F</sub>. Tensile strength test gives basic information on the elasticity and strength of the foam sample under tension. The tensile strength of modified PUF<sub>F</sub> was found to be 29 kPa and the elongation at break was found to be 93 %. Increase in the % of decabDE of 1 %, 2 %, 3 %, 4 % and 5 % results in 75 kPa, 65 kPa, 58 kPa, 57 kPa and 55 kPa tensile strength respectively. The elongation at break was found to be 93 %, 96.2 %, 86.2 %, 85.2 %, 80.7 % and 77.3 % for 1 %, 2 %, 3 %, 4 % and 5 % of decabDE respectively. It was found that at 1 %, tensile strength and elongation at break was found maximum and decreased with respect to the increase in % of decabDE. The increase in % of decabDE FR content decreases the tensile strength and elongation at break. The increase in FR % may lead to the agglomeration of FR while mixing in PUF<sub>F</sub>. During homogenization, the dispersion of FR may be difficult which may lead to the high stress concentration in the developed PUF<sub>F</sub> at the lab level. This may be one of the reasons in the decrease in tensile strength and elongation at break.

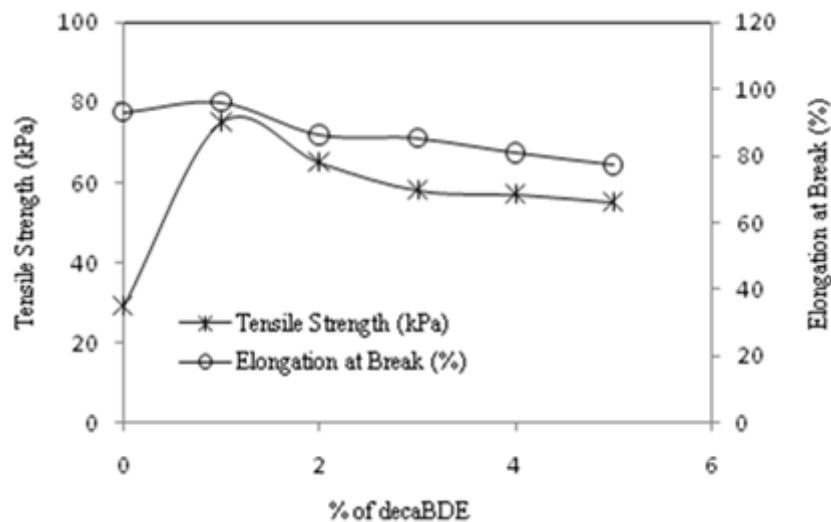


Figure.3.5. Tensile Strength and Elongation at Break (%) of decabDE PUF<sub>F</sub> (1 %, 2 %, 3 %, 4 % and 5 %)

## IV. CONCLUSION

PUF<sub>F</sub> incorporated with various % of decabDE (brominated halogen) as FR is assessed and evaluated in the present study. The morphology of the samples taken for the study reveals the presence of OCS with cell window, strut and strut joint. The presence of functional groups was decoded with FTIR spectrum analysis. The absence of absorbance between 2270 cm<sup>-1</sup>-2250 cm<sup>-1</sup> in all the modified samples with respect to the unmodified one confirms the complete conversion of isocyanate into PUF<sub>F</sub>. In TGA, the thermal stability of 3 % decabDE was found to be higher with the decomposition at 50 % was found at 354.9 °C, whereas the unmodified was found at 307.9 °C. UL 94 flammability test (HB) was found to be achieved for 3 % decabDE PUF<sub>F</sub> sample where the flame extinguishing time was found to be 0.45 mm/s. Density of the PUF<sub>F</sub> was found to be increased from 36 Kg/m<sup>3</sup>-51 Kg/m<sup>3</sup> with increase in different % of decabDE. Tensile strength was found to be in the range of 55-75 kPa for the different % of decabDE. The present study reports that incorporation of decabDE shows the enhanced FR property in the lab scale developed PUF<sub>F</sub>.

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