

EFFECTS OF DIFFERENT FILLER TREATMENTS ON THE MORPHOLOGY AND MECHANICAL PROPERTIES OF FLEXIBLE POLYURETHANE FOAM COMPOSITES

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ABSTRACT

A discontinuous foaming process has been employed to investigate the effects of two types of fillers – calcite (CaCO_3) and dolomite ($\text{CaMg}(\text{CO}_3)_2$) on the morphological and mechanical properties of flexible polyurethane foam. The particle sizes of the fillers: 6 nm, 3.5 μm , and 0.84 mm vary from composition ranges of 0 – 40 wt%. Indentation hardness, Tensile strength, and Elongation at break of the polyurethane composites were examined as a function of the filler content in the polyurethane matrix. It was found that the influence of the fillers on the Indentation hardness of polyurethane composite depend strongly on the content and particle size of the fillers. The addition of filler of upto 20 – 35wt% and of nanosizes improves the Indentation hardness of the foam much more than the micro- and macro-scaled fillers. The tensile strength and elongation at break were however, reduced with an increasing amount of

filler in the polyurethane matrix for all particle sizes. Morphological examination of the foam revealed that the cell geometry characteristics exert a significant influence on the observed mechanical characteristics.

Key words: Polyurethane, calcite, dolomite, Indentation hardness, tensile strength, elongation at break.

INTRODUCTION

Flexible polyurethane foam is a class of cellular plastic that is becoming increasingly important as a cushioning material in domestic and industrial applications (Klempner and Sendijarevic, 2004), because of its excellent lightweight, strength/weight ratio, and superior comfort characteristics (Klempner and Sendijarevic, 2004; Woods, 1987).

The comfort characteristics (determined as mechanical properties) displayed by flexible polyurethane foam which possesses a cellular structure that is created by the expansion of a blowing agent depend essentially on the matrix structure and shape (morphology) of the cells (Comstock, 1981; Javni et al., 2002), which in turn depend on the foam bulk density. Foamed plastics have been provided in a wide range of bulk densities (i.e., 3 – 900 kg/m³). Low density foams (i.e. less than 80 kg/m³) are primarily used for insulation, packaging, and cushioning applications, and high density foams are primarily used for load bearing applications such as structural parts (Herrington and Hock, 1991). In recent time, the versatile use of flexible polyurethane foam material has made its world consumption to grow by 4 -5 % per annum and reached 7 million tons (Raffel and Loevenich, 2006).

Due to the rapid increase in the production volume of flexible polyurethane foam, as a result of new development areas (such as packaging, furniture, and automotive industries applying safety cushion for maximum energy absorption), methods have been initiated to improve mechanical properties of flexible polyurethane foam. Usually, properties of foam are varied by changing their densities, but the price which has to be paid for higher rigidity is an additional weight and thus a higher cost.

Various experiments aimed at improving both the mechanical properties and cost effectiveness of polyurethane foam has been conducted. One attempt to achieving this involved the chemical modification of its structure (Spitler and Lindsey, 1981; Abdul-Rani et al., 2004). Another modification involved the use of fillers to achieve flexible polyurethane /filler composite formation. Inorganic filler materials are available as nano-, micro-, and macro-scale crystals. Their effects on plastic foam materials have been known to strongly depend on their sizes, aspect ratio, hybrid morphology, and dispersion quality (Fu and Naguib, 2006; Boyle et al., 2004). It is also well known that fillers increase cell density and decrease cell size. By affecting the macroscopic cell geometry in this way they act as reinforcement materials in polyurethane foam composites (Javni et al., 2002; Chen et al., 2002). In general, because of the ultrafine phase dimensions involved, nanocomposites exhibit improvement in some properties of foam in comparison to their micro or macrocomposite counterparts, while in some other properties nanocomposites can exhibit deleterious effects (Giannelis, 1996). For the composite foam materials, the filler must be uniformly dispersed in the polymer matrix rather than be aggregated. Once uniform dispersion is achieved in the form of intercalation, improvements can manifest themselves in mechanical properties (Xu et al., 2001). On the other hand, because of the high surface to volume ratio in nanofillers used at high concentrations which can lead to incomplete

foam reaction (Aribike et al., 2007), nanocomposite can manifest deleterious effects on properties such as tensile strength and elongation at break.

The maximum benefit from nanolayer dispersal and reinforcement was demonstrated by Javni and co-workers (2002). In their study, they found that nano-silica used as filler in flexible polyurethane foam increased the hardness and compression strength, but decreased the rebound resilience of the foam. In the same study, micro-silica filler was found to decrease all the mechanical properties of the foam.

Nanolayered silicate has been used in poly (urethane-urea) composite (Xu et al., 2001) as filler material. The poly (urethane-urea) chains were found to have strong favorable interactions with the silicate surface resulting in a significant increase in modulus, and strength of the foam material.

Effects of various organoclays of nanosizes on the thermomechanical properties and morphology of polyurethane foam have been investigated by Chang and An, (2002). The study shows that most clay layers were dispersed homogeneously into the matrix polymer. Moreover, the addition of only a small amount of organoclay was enough to improve the thermal stabilities and mechanical properties of the foam.

The favorable interaction between the fillers and the materials used in polyurethane chemistry necessitated the transfer of this knowledge by intercalation and exfoliation chemistry from polyurethane /filler composites to a thermoplastic system.

In the work of Chen et al. (2002), nano- and micro-scaled calcium carbonate (calcite) were used to influence cell nucleation in the foaming process of polyethylene plastic. The nano-scaled calcite (ultra-flex CaCO_3) increased substantially the cell density of the foam thereby improving

the mechanical properties, while the micro-scaled calcite (Hi-flex CaCO_3) has an effect that was less significant in reinforcing the strength characteristics of the foam.

From the literature survey, it has become apparent that what is of great interest to researchers is the improvement in the mechanical properties of polymers, mostly with the use of silica filler. However, the high absorption property of silica which makes it vulnerable to rearrangement of its particle structures (Boyle et al., 2004) and exfoliation in polyurethane foam can limit its use especially at high concentrations, and confers on it some undesirable influence of the foam at these concentrations. It has therefore become imperative to consider other inorganic substances suitable as reinforcement agent in polyurethane foam.

In this study, we have used materials such as calcite (CaCO_3) and dolomite ($\text{CaMg}(\text{CO}_3)_2$) to influence the mechanical properties of flexible polyurethane foams. Properties of these composites were studied as functions of filler contents in the matrix polyurethane foam. The structures of the composites were studied with scanning electron microscopy (SEM).

EXPERIMENTAL

Materials

The source fillers used in this study – calcite and dolomite were obtained from West African Solid Mineral Company. The grain sizes of the fillers are 6 nm, 3.5 μm , and 0.84 mm. The flexible polyurethane foams were prepared from conventional polyol (Konix FA-717, triol, MW 3500) and crude diisocyanate, scuranate T80 (Lyondell Chemical Company). Water was used as a blowing agent in all cases.

Methods

Flexible polyurethane foams reinforced with the inorganic fillers were synthesized with the amounts of each chemical component chosen to obtain a target density of 25kg/m^3 . The experiment was carried out in a foaming plant (ISOLBLOCK, IB 150/4F-SS) (see Figure 1) consisting of a mixing chamber into which is inserted a stirrer that operates at two speeds (700 and 1400 rpm) and fitted with three phases 7.5 HP motor. The chemical materials are metered into the mixing chamber by automated control of the cylinders that transferred dosages of the formulated components. The procedure for preparing the composite foams involved two steps. In the first step, 40 % of the total polyol was weighed and used to infiltrate the filler particles until they are swollen and separated from the periphery of the agglomerate creating a complete dispersion of the fillers in the polyol. Calcite and dolomite of particle sizes (6 nm, 3.5 μm , and 0.84 mm) and compositions from 0 – 40 wt% was investigated. The remaining 60 % of the polyol was charged directly into the mixing chamber from the holding tank by means of automatic control on the control panel. Thereafter, the mixture of polyol with filler was added to the content of the mixing chamber and stirred for 6 s. In the second step, all other ingredients (surfactant, catalysts and water as the blowing agent) were added to the mixture of polyol and filler in the mixing chamber and thoroughly premixed. The diisocyanate was weighed directly into this mixture, and the completed formulation was stirred with an overhead mechanical mixer for 4 s. The foam formulation was then immediately poured into an open mold, which was treated with mold release agent (Quatro HD X 40) to produce the free-rise foam. Three foam batches for each filler concentrations were produced with identical chemical formulation. After 10 min, the foams were removed from the mold and left to cure for at least 7 days.

Characterization

X-ray diffraction experiments were performed on the pure polyurethane, the pristine fillers, and the polyurethane/filler composites on an x-ray diffractometer operating in a θ - θ geometry using Cu K α ($\lambda = 0.154\text{nm}$) radiation. Samples were scanned at a rate of $1^\circ/\text{min}$ from 2° to 40° in 2θ .

Hardness properties of the polyurethane/filler composite foams were determined with a Hampden mechanical tester (Hampden EC30). The property was measured following the procedure of ASTM D3574 by depressing a standard (38 x 38 x 5 cm) piece of the foam with a compression anvil at a loading rate of 30 mm/min.

Evaluation of tensile strength and elongation at break properties were carried out on the Hampden mechanical tester. A crosshead speed of 20 mm/min was used for all experiments. An average of 3 individual determinations was obtained for all experiments and the values with standard deviations of less than 10 % were used.

Scanning Electron Microphotograph of selected samples was conducted to evaluate the cell morphology of the filler reinforced flexible polyurethane foam. Thin slices fractured foam samples, that has been treated with liquid nitrogen and coated in gold was used for this examination. Images were taken on the scanning electron microscope (SEM) operated in the secondary electron mode at a 15 kV accelerating voltage. The average cell count per linear centimeter and description of the cell structure were the structural foam parameters measured. Relationships of the SEM study to mechanical properties such as density and compression set were determined.

RESULTS

Range of dispersion of the filler materials in polyurethane foam

Figures 2-3 show the x-ray diffractogram of the fillers, pure polyurethane, and selected polyurethane/filler nanocomposites. The figures show the presence of strong interaction of the filler galleries with the polymer matrix. This can be explained by observing the interlayer spacing of the fillers in the order: dolomite ($d = 27.82\text{\AA}$) > calcite ($d = 17.95\text{\AA}$) and which when compared to the polyurethane nanocomposites, the obvious peaks of the fillers in the x-ray diffraction curves indicated that the fillers were intercalated and not homogeneously dispersed in the polyurethane matrix. For example, the d-spacing, after interaction by the filler increased from 27.82\AA for dolomite to 31.22\AA for the hybrid, and from 17.95\AA for pristine calcite to 28.12\AA for the polyurethane-calcite hybrid. Other researchers working with the use of fillers in polymer have confirmed that the spacing suggests the intercalating of polymer chains into the filler galleries (Hsiao et al., 2001; Zilig et al., 1999) and so influence the chemical structure of the polymer. Sharp peaks exist and increase in intensity in the region in which higher filler loading have been used in the polyurethane suggesting that some portion of the fillers agglomerate.

Morphology

Figures 4 – 7 show the Scanning Electron Microphotograph images of selected foam samples. Without the addition of filler, the flexible polyurethane foam morphology is shown in Figure 4. Morphologies of foam samples with formulation containing 20 wt% of calcite of particle size distributions 6 nm, 3.5 μm , and 0.84 mm are depicted in Figure 5 and in Figure 6 is shown the morphology of 20 wt% of dolomite of particle size distributions 6 nm, 3.5 μm , and 0.84 mm. With a 20 and 40 wt% filler (calcite) composition of particle size distribution 6 nm, the

morphology is presented in Figure 7. Table 1 present the cell structure description, the average cell size per linear centimeter measured, and the corresponding compression set determined.

It was observed in Figure 4, that when no filler was used, the foam cell structures were irregular with mainly large cells. The dominant cell nucleation mechanism in unfilled flexible polyurethane foam is assumed to be homogeneous nucleation (Klempner and Sendjarevic, 2004; Kaewmesri et al., 2006). In comparison with heterogeneous nucleation, the required activation energy for homogeneous nucleation is much higher. Hence, cell nucleation tends to occur within a relatively longer period of time. This resulted in a smaller number of cells of larger and variable sizes (Table 1).

Addition of filler in flexible polyurethane foams (Figures 5 and 6) creates a filler/polymer interface unto which gasses are trapped. Here, microvoids are formed inside the foam. These microvoids lower the activation energy required for cell nucleation. This negligible nucleation energy and the presence of numerous preexisting microvoids made it possible for fast cell nucleation, and the spontaneous formation of a large number of cells led to more uniform cell size within the foam (Table 1). However, with different filler particle sizes of the same compositions, the foams cellular structures were more uniform at 6 nm and 3.5 μm particle sizes than for fillers of 0.84 μm (Figures 5 and 6). Even though the cell structure of foam with 40 wt% filler volume fraction of particle size 6 nm is uniform and fine, and of the same density as the foam reinforced with 20 wt% filler of 6 nm size (Figure 7), the mechanical properties determined is low indicating that too much filler addition worked negatively to destroy the foams mechanical properties.

Effects of Filler Compositions and Particle Sizes on Indentation Hardness

The variations of hardness of flexible polyurethane foams reinforced with fillers (calcite and dolomite) of particle sizes 6 nm, 3.5 μm , and 0.84 mm are plotted against filler contents of 0 – 40 wt% in Figures 8 and 9, respectively. In Figure 8, we see that the hardness of polyurethane foam reinforced with calcite increased gradually as the filler load is increased from 0 – 35 wt% for the nano (6 nm)- and micro (3.5 μm)-sized fillers reaching a maximum at 35 wt% for the nanocomposites, while at 15 wt% there is decrease in the hardness and at 40 wt% the hardness is extremely low. The macro (0.84 mm)-composite foam did not show any improvement in its hardness for all compositions of the filler.

Figure 9 show the hardness of polyurethane foam hybrid with dolomite. The hardness was lowered initially with 5 wt% of the filler for both the nano- and microcomposites, before increasing gradually with the filler contents up to 25 wt% for the nanocomposite and 35 wt% for the microcomposite, and then afterwards dropped gradually to 40 wt%. As with the calcite filled foam, the polyurethane-dolomite hybrid macrocomposite did not add to the reinforcement of the hardness of the foam.

The hardness of the hybrids increased from 134.94 N to a maximum of 166 N for the nanocomposite polyurethane foam reinforced with calcite of 35 wt% and to a maximum of 160.2 N for the nanocomposite polyurethane foam reinforced with dolomite of 25 wt%, while the micro filler calcite increased the hardness to a maximum of 152 N at 35 wt% and the dolomite filler was to achieve a maximum of 148 N. The increase in the hardness of the foam by nano and micro fillers is due mainly to the random dispersion of the fillers into the polyurethane matrix. The interaction between the filler particles and the polymer matrix as a result of intercalation of

the polymer chains into the filler galleries influence the chemical structures of the polymer chains.

Specifically, the changes in the chemical structure of the polymer chain are apparent from the SEM images of the foam morphology (see Figures in section on morphology and Table 1). The hardness values at 20 wt% of all hybrids indicate that the hardness increases as the sizes of particles are reduced from 0.84 mm to 3.5 μm to 6 nm (see Figure 8 and 9). A similar result was observed at all other composition of the fillers. The SEM images show that the nano and micro sized fillers created more cell densities and reduced cell sizes than the cell structure of neat polyurethane foam. This indicates that hardness increases with reduced cell sizes.

The observed fall in the indentation hardness of the foam at 40 wt% for both fillers at all particle sizes can be explained by the agglomeration of the excessive filler particles on the plateau border areas of the foam cellular structures. As can be seen in Figure 7 the cell structure of foam with 40 wt% filler volume fraction of particle size 6nm is uniform and fine and of the same density as the foam reinforced with 20 wt% of 6 nm filler, however, the hardness property determined for the 40 wt% is low indicating that too much addition of filler have worked negatively to destroy the foams mechanical properties.

Effects of Filler Compositions and Particle Sizes on Tensile Strength and Elongation at Break

Figures 10 and 11 represent the tensile strength of the hybrid foams with different contents of calcite and dolomite, respectively. Also Figures 12 and 13 represent the elongation at break of the hybrid foams with different contents of calcite and dolomite, respectively. In all systems, the

tensile strength and elongation at break was decreased as the contents of the fillers were increased. This response is characteristic of materials reinforced with stiff inorganic materials and is particularly noteworthy for its intercalated morphology. However, another noteworthy phenomenon is that contrary to what was obtained with indentation hardness, dolomite and calcite of 0.84 mm particle size had higher tensile strength and elongation at break than the nano and micro-sized composites. This may be attributed to the good dispersion of the macro-sized particles of the fillers in the polymer matrix with respect to other particles of the fillers. Because the macroparticles are not substantially infiltrated by the liquid polymer film, there is less agglomeration of the particles in the polymer matrix. The improved tensile strength and elongation at break of the macroparticles over all other particles can be ascribed to the resistance exerted by the macro fillers to agglomeration, as well as the orientation and aspect ratio of the filler layers. Additionally, the stretching resistance of the oriented backbone of the polymer chain also contributed to the observed trend in the macro, micro, and nanoparticles effects on tensile strength and elongation at break.

Following from these results, it can be said that there is an optimal amount and size of fillers needed to achieve the greatest Indentation hardness in polyurethane/filler composites. Further, the tensile strength and elongation at break properties were better with fillers of macroparticle sizes than those of nano and micro-sizes because of the uninfiltration of the internal periphery of the macroparticles by the liquid polymer resin.

Conclusion

This study has investigated the various properties of polyurethane nano, micro, and macrocomposites obtained when calcite and dolomite of particle sizes 6 nm, 3.5 μm , and 0.84 mm were used as fillers in the polyurethane systems. The polyurethane/filler composites were synthesized with filler contents ranging from 0 – 40 wt%. Indentation hardness, tensile strength, elongation at break, and morphologies of the composite foams have been compared in great detail. Results show that these properties are dependent on the filler type and content in the polymer matrix. For the 35 wt% calcite and 25 wt% dolomite of nanosizes the indentation hardness showed maximum values and then decreased with further filler loading, whereas the tensile strength and elongation at break were higher with macroparticle sized fillers though the properties were less than for neat polyurethane.

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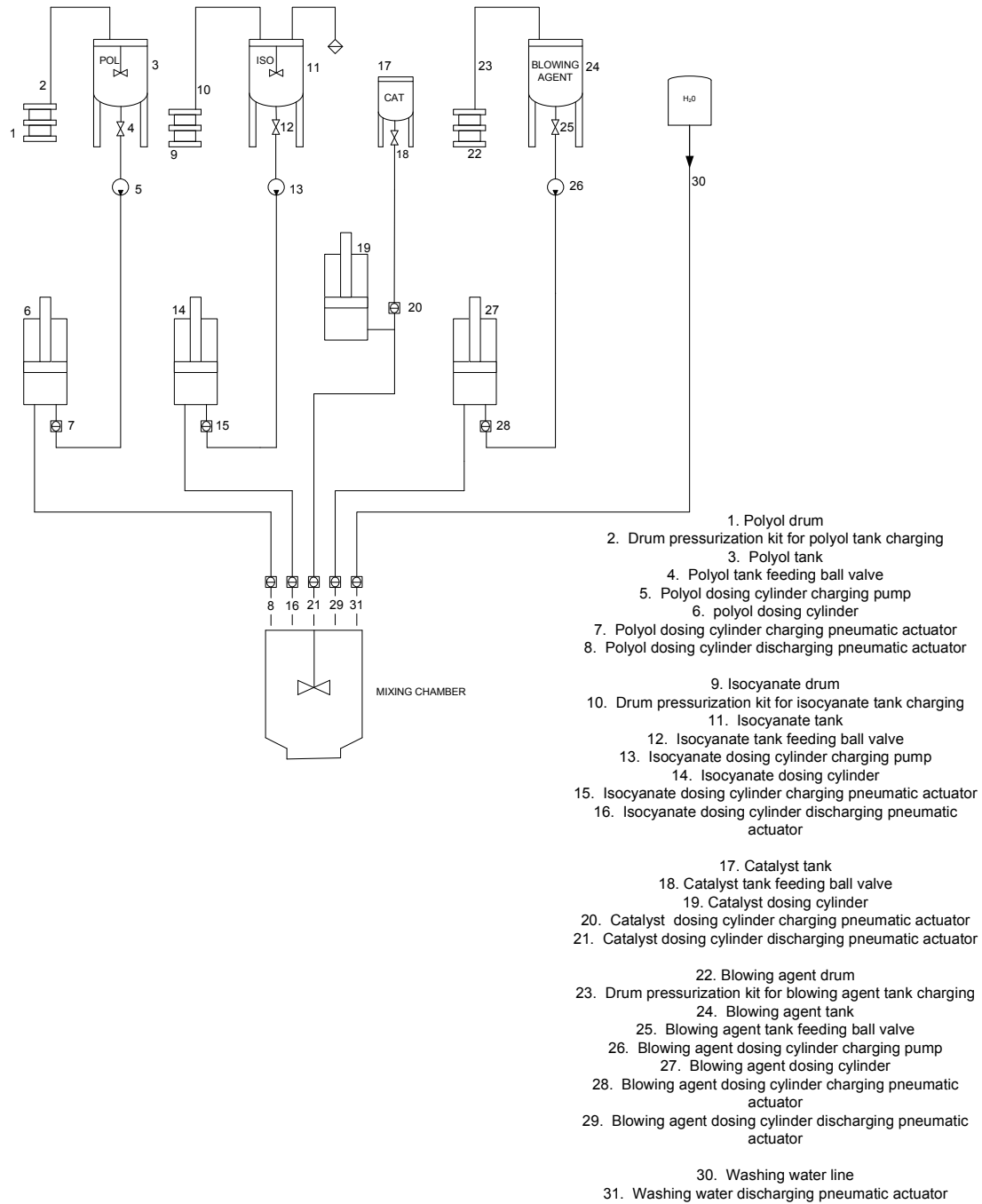


Figure 1: Schematic diagram of the Experimental Setup

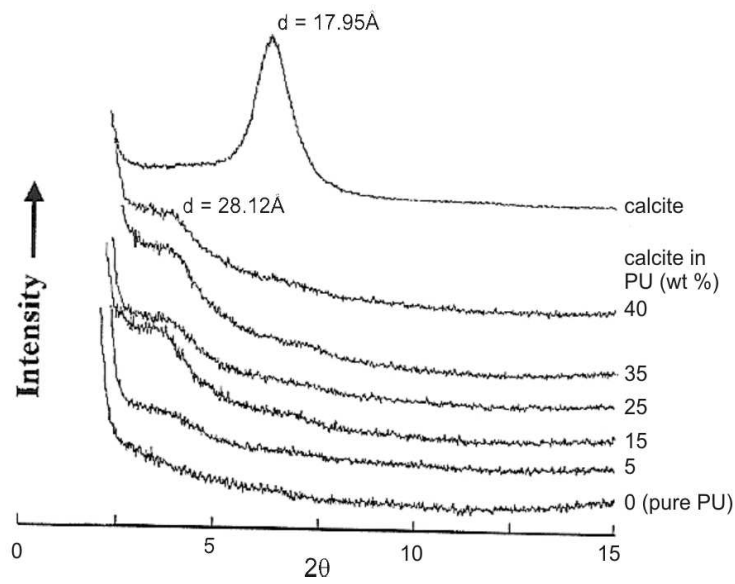


Figure 2: Patterns of X-ray diffraction of calcite and selected polyurethane nanocomposites

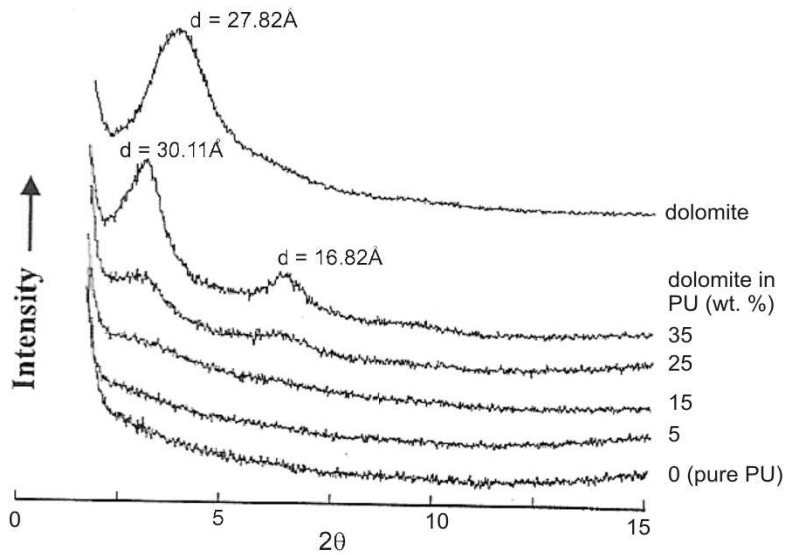
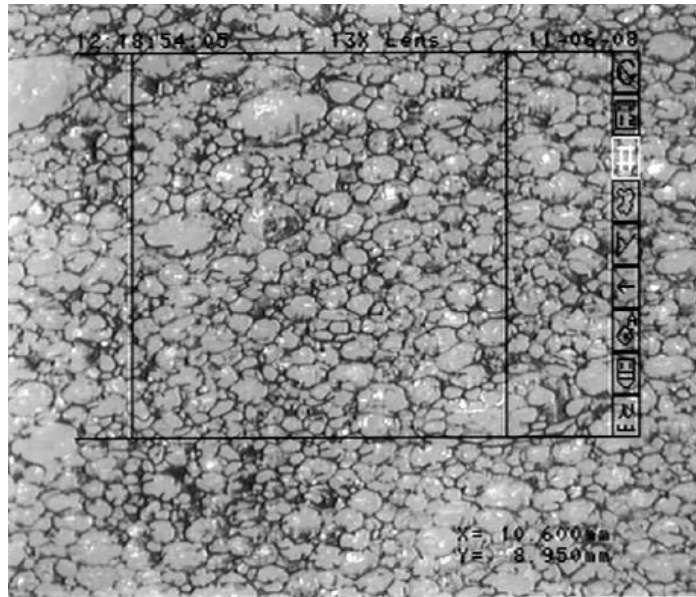
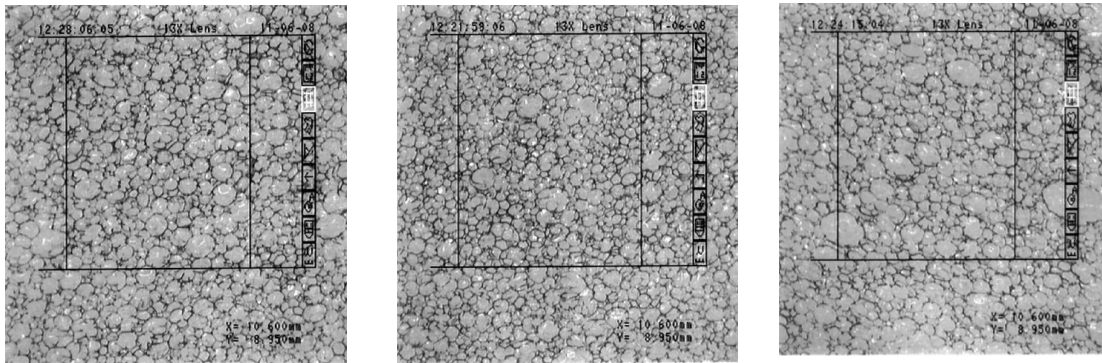


Figure 3: Patterns of X-ray diffraction of dolomite and selected polyurethane nanocomposites



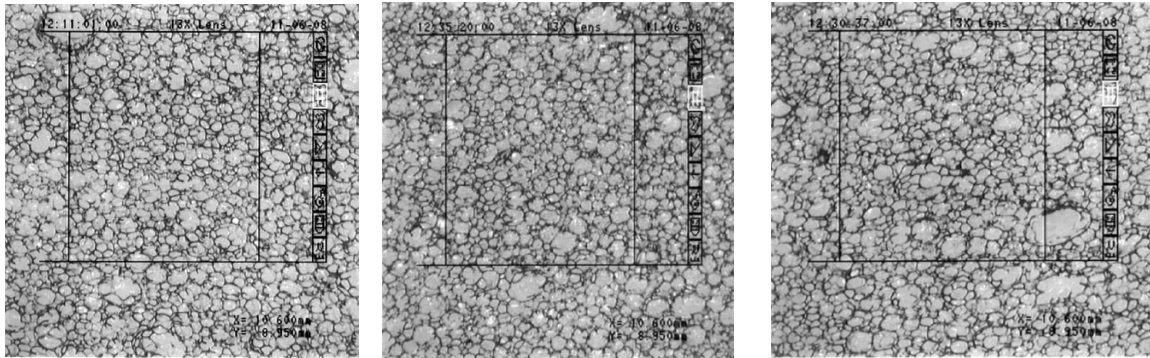
A: unfilled foam with polyol content short of 20wt%

Figure 4: Cell morphology of neat flexible polyurethane foam with polyol content short of 20wt%.



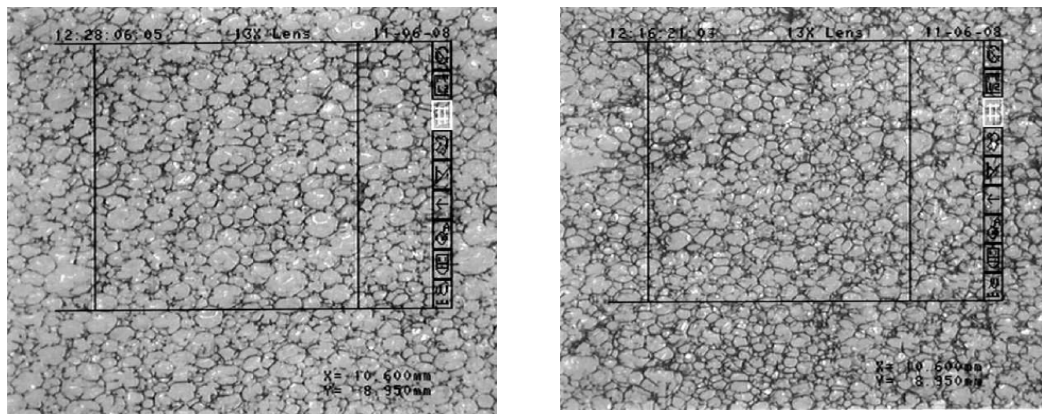
B: Calcite, 0.06 μ m, 20wt% (Ca31) **C:** Calcite, 3.5 μ m, 20wt% (Cc31) **D:** Calcite, 841 μ m, 20wt% (Cf31)

Fig 5: Cell morphology of reinforced flexible polyurethane foam with calcite of different particle sizes.



E: Dolomite, 0.06 μ m, 20wt% (Da31) **F:** Dolomite, 3.5 μ m, 20wt% (Dc31) **G:** Dolomite, 841 μ m, 20wt% (Df31)

Figure 6: Cell morphology of reinforced flexible polyurethane foam with dolomite of different particle sizes.



H: Calcite, 0.06 μ m, 20wt% (Ca31) **I:** Calcite, 0.06 μ m, 40wt% (Ca81)

Figure 7: Cell morphology of reinforced flexible polyurethane foam with calcite of different weight compositions.

Table 1: Measurement of compression set, cell count, and microphotography.

Properties	Ca31	Cc31	Cf31	Ca81	Da31	Dc31	Df31	Unfilled
Density(kg/m³)	25.7	25.9	25.7	26.1	26.3	25.6	24.8	23.4
Compression Set 75% (%)	7.6	8.0	7.6	24.7	5.4	7.3	7.0	10.7
Cell/cm (average)	14	14	13	15	13	13	12	11
Description of cell structure	Mainly big cells	Mainly regular but with cluster of smaller cells	Mainly big cells	Mainly regular but with cluster of smaller cells	Mainly regular but with cluster of smaller cells	Mainly regular but with cluster of smaller cells	Very irregular	Very irregular

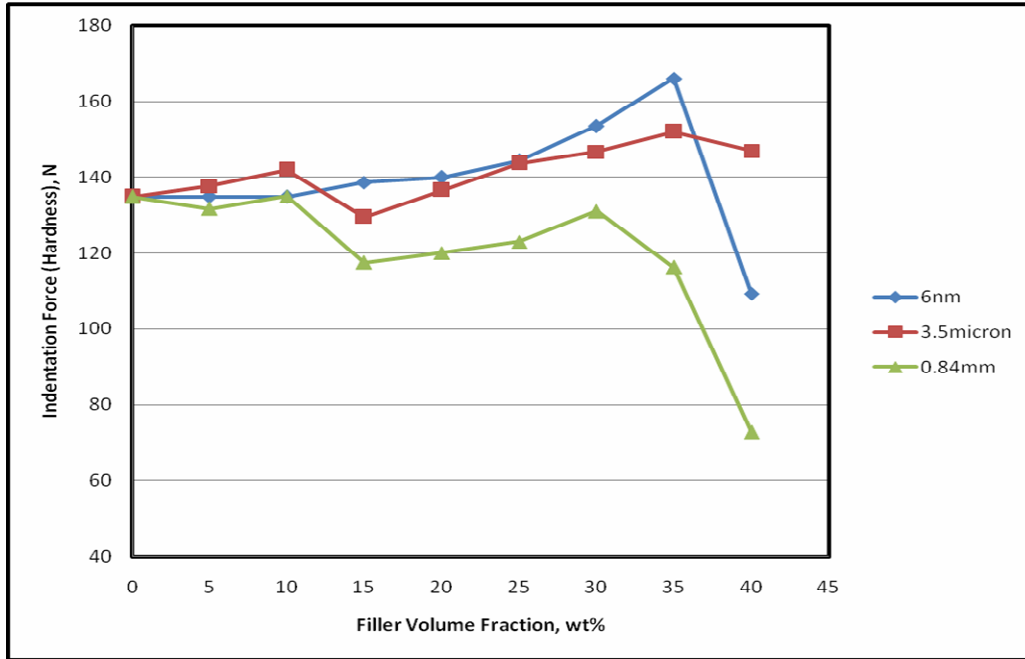


Figure 8: Effects of Filler (calcite) of different compositions and particle sizes on Indentation hardness of Flexible Polyurethane foam.

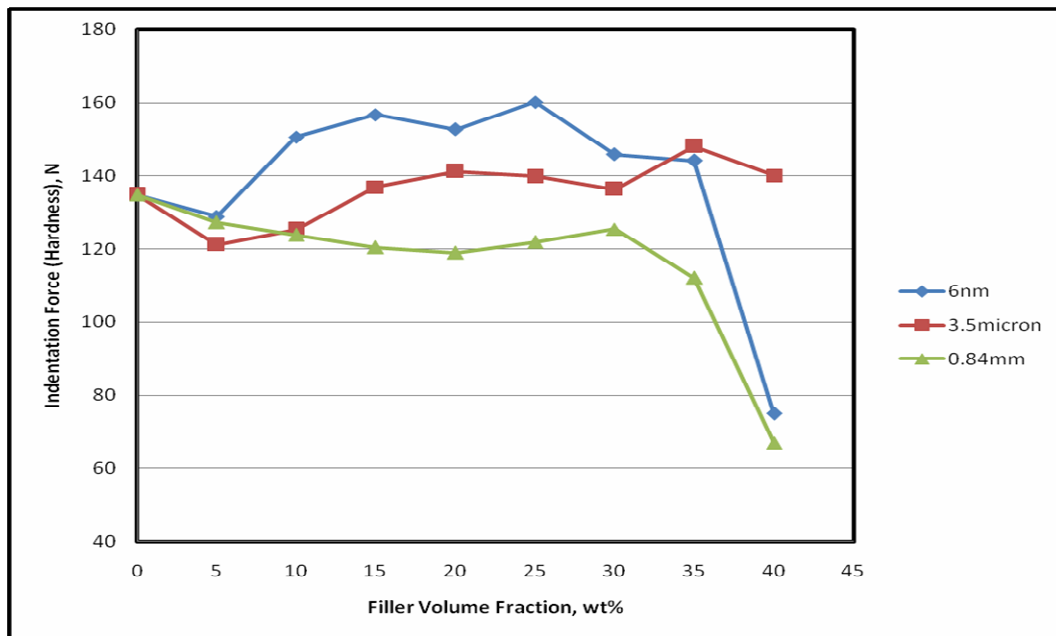


Figure 9: Effects of Filler (dolomite) of different compositions and particle sizes on Indentation hardness of Flexible Polyurethane foam.

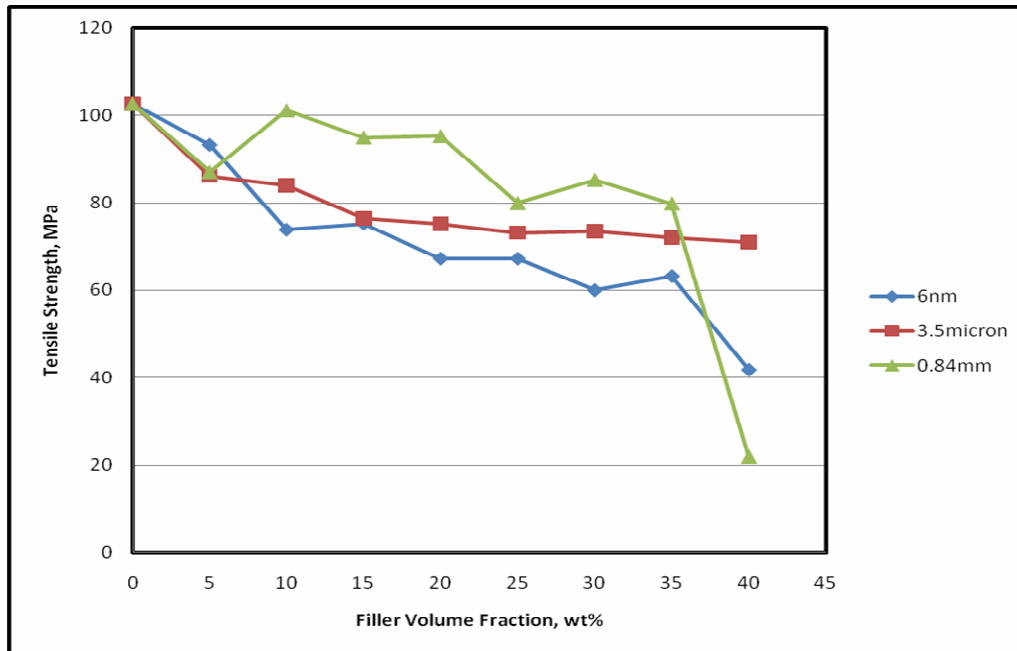


Figure 10: Effects of Filler (calcite) of different compositions and particle sizes on Tensile Strength of Flexible Polyurethane foam.

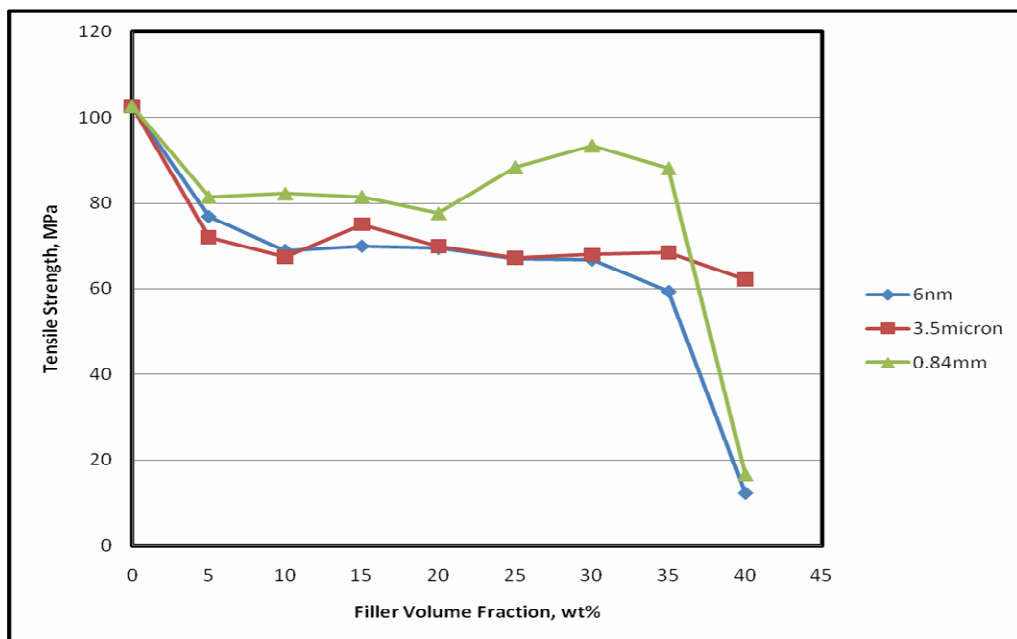


Figure 11: Effects of Filler (dolomite) of different compositions and particle sizes on Tensile Strength of Flexible Polyurethane foam.

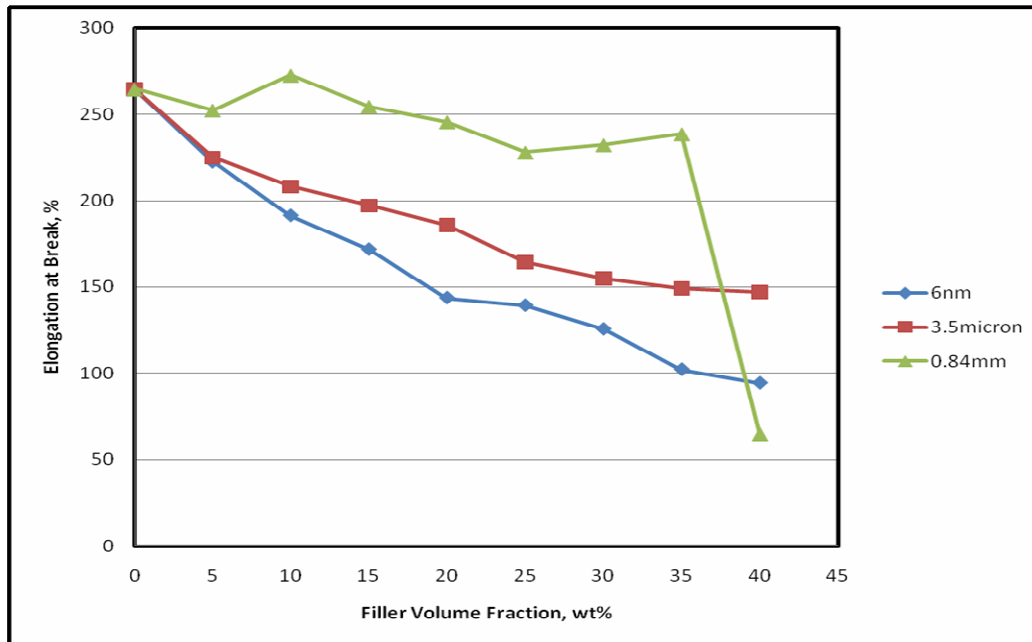


Figure 12: Effects of Filler (calcite) of different compositions and particle sizes on Elongation at Break of Flexible Polyurethane foam.

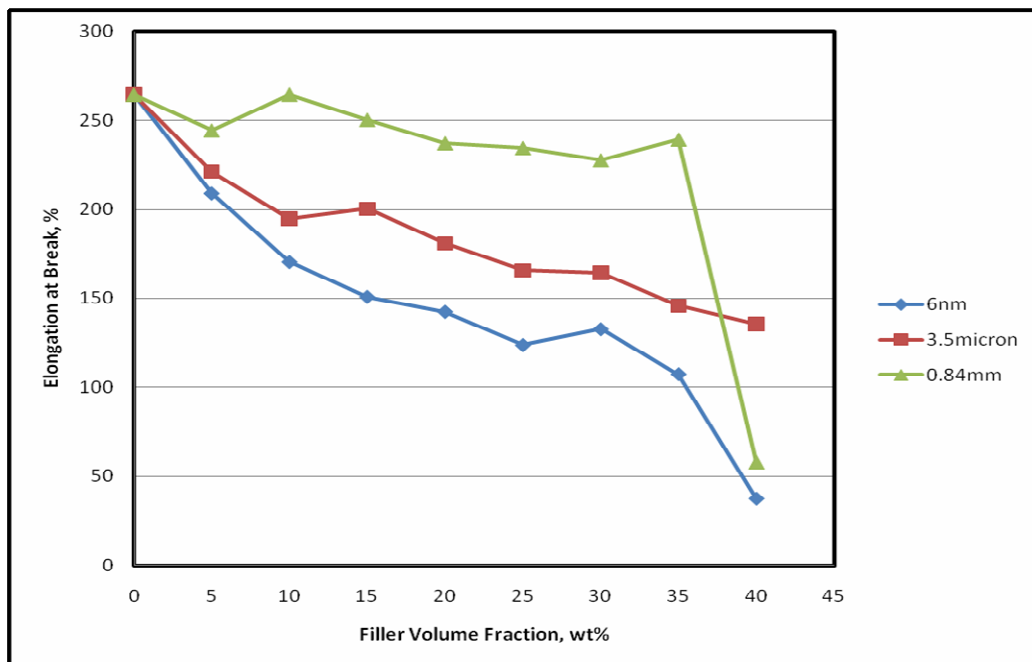


Figure 13: Effects of Filler (dolomite) of different compositions and particle sizes on Elongation at Break of Flexible Polyurethane foam.