EFFECTS OF CALCIUM CARBONATE OF DIFFERENT COMPOSITIONS AND PARTICLE SIZE DISTRIBUTIONS ON THE MECHANICAL PROPERTIES OF FLEXIBLE POLYURETHANE FOAM

Ganiyu K. Latinwo*[1], David S. Aribike[2], Layioye O. Oyekunle[2], Akpoveta A. Susu[3], Semiu A. Kareem[4]

1. Department of Chemical Engineering, Ladoke Akintola University of Technology, P.M.B. 4000, Ogbomoso, Nigeria.
2. Department of Chemical Engineering, University of Lagos, Nigeria.
3. Department of Chemical and Polymer Engineering, Lagos State University, Epe Campus, Lagos, Nigeria.
4. Department of Chemical Engineering, Federal University of Technology, Yola, Adamawa, Nigeria.

kayodelatinwo@yahoo.com

Abstract: Effects of calcium carbonate of different compositions (0 – 40 wt%) and particle size distributions (0.06, 0.5, 3.5, 10, 20, and 841μm) on the mechanical properties of flexible polyurethane foam were investigated in this study. The experiments were conducted in a foaming plant (ISOBLOCK IB 150/4F-SS) and the mechanical properties determined such Indentation Force Deflection, Tensile strength, and Elongation at break were measured with the aid of a Hampden testing machine. The finely divided filler material increased the hardness characteristics of the foam to compositions of up to 35wt%, while coarse filler did not show any appreciable improvement in the property. For all particle sizes, the tensile strength and elongation at break decreased while increasing the load of the filler weight percentages. However, the tendency with which the coarse filler reduce the properties is not as much as for the fine fillers. Scanning Electron Microphotography of the foam samples shows how the filler affect the foam cellular structure and cell size distributions. [Nature and Science 2010;8(9):92-101]. (ISSN: 1545-0740).

Keywords: Flexible polyurethane foam, calcium carbonate, Indentation Force Deflection, Tensile strength, Elongation-at-break.

Introduction

Flexible polyurethane foams are versatile polymeric materials due to the many ways they can be applied in the fabrication of a wide range of materials for different uses in domestic and industrial applications. The production of flexible PUR foam typically involves the step-growth polymerization of a polyisocyanate of functionality 2.7 with a polyalcohol (polyol) of molecular weights from 2000 – 10000 and functionality 2 – 3, and the blowing reaction of two parts polyisocyanate with one part water in the presence of an amine and tin compound catalysts and other additives. Some notable applications of this foam material include mattresses, upholstery, furniture, footwear and textiles in our homes and offices, and as packaging, appliances, electronics, machinery and foundry, and as cushioning materials in automobiles, in the industries.

In 2007, the global consumption of flexible polyurethane foam was above 7 million metric tons and the average annual growth rate is about 5% (Avar, 1978). Usage of flexible polyurethane foam in Sub-Sahara Africa is as mainly mattresses and furniture and recently, there has been a growing demand for durable and high hardness characteristic (i.e. high compression resistant) foam at low cost (Latinwo, 2009). Foams with these qualities are of interest to many economic sectors, and therefore their preparation, characteristics, and applications are also an interesting subject. For these reasons, the use of several fillers to achieve improved properties in foam has been widely studied (Nunes et al., 2000). Some notable ones include inorganic materials such as calcium carbonate, dolomite, aluminum silica, titanium dioxide, and talc (Nunes et al., 2000) while some of the organic materials used as filler are carbon black and natural fibers (Mothé et al., 2002; Mothé and Araújo, 2000). Further, the cost of petro-chemical based polyol, which constitute the largest percentage of materials used in foam production and which possess the characteristics to induce superior mechanical properties in foam currently commands a high value due to the rising costs of the petro-chemical feed stocks (Niemeyer et al., 2006).

Mechanical properties of foamed plastics essentially depend on their bulk density (Kaewmesri et al., 2006). High density foams possess high hardness characteristics (Saint-Michel et al., 2006). They have fine cell structure of sizes less than 100 μm and cell density population in the order of $10^9 – 10^{15}$ cells/cm^3 (Suh, 2003). Foams with very fine cells and uniform cells distributions exhibit better mechanical properties such as hardness, impact strength, and rebound resilience (Suh, 2003). Conversely, the volume expansion ratio of high density foam is extremely low making it expensive, not patronized by consumers, and economically unattractive to manufacturing industries.

Particulate fillers are used in many composites to improve mechanical properties, impart color, or reduce material cost (Boyle et al., 2004). Particularly, in flexible polyurethane foam, presence of filler can create void fractions and enhance nucleation sites, on the polymer/filler interfaces, where gasses may be trapped (Chen et al., 2002). The filler serve as solid surfaces for heterogeneous cell nucleation. This nucleation process is promoted by the surface geometry and surface energies between solid-liquid, solid-gas, and liquid-gas present in the system (Klempner and Frisch, 1991). When the free energy for gas mass formation on the surface of a nucleating agent is less than for homogeneous nucleation in the polymer, heterogeneous nucleation is induced. It is expected that fillers as nucleating agent can influence the cellular structure of flexible polyurethane foam and promote increase in density and hardness characteristics. However, they reduce other properties such as resiliency and tear strength and contribute to the increase in permanent deformation (Osman et al., 2004). Accordingly, it is necessary to know the end-use of the material in order to use the correct concentration in the polymer matrix, obtaining a product of quality.

For instance, micro (1.5μm) and nano (12 nm) silica were used as filler to reinforce mechanical properties of both rigid and flexible polyurethane foam of densities 30 kg/m^3 and 60 kg/m^3, respectively, by Javni and co-workers (Javni et al., 2002). The filler concentrations were varied from 0 – 20 wt%. The nano- silica filler increased density of both types of foams, while micro- silica filler did not show any effect. Compression strength of both types of foams was lowered with nano- silica filler. On the other hand, hardness in the flexible polyurethane foam was improved with the nano- silica filler and rebound resilience was decreased. Chen et al. (2002) reported the use of Hi-flex (3.5 μm) and ultra-flex (0.07 μm) calcium carbonate to influence the properties of High Density Polyethylene plastic. 25 – 30 wt% of the filler materials were used. It was found that the ultra-flex CaCO$_3$ led to increase in the cell density of foam at a high saturation pressure but give a lower cell density at a low saturation pressure. Other fillers such as talc in High Density Polyethylene Plastic foam (Chen et al., 2002), and in High-melt-strength Polypropylene foam (Kaewmesri et al., 2006), clay in Poly (methyl methacrylate) foam (Fu and Naguib, 2006), wood fibre in Polypropylene foam (Bledzki and Faruk, 2006), and TiO$_2$ in High Density Polyethylene foam have been reported. The fillers were found to strongly affect the cell structure and mechanical properties of these foams.

Following from the literature survey, it is evident that foam properties are optimized at specific filler composition and particle size distribution. Industries that produce flexible polyurethane foam in Nigeria use fillers to modify the material's properties in some ways. However, the quantity used is defined randomly and there was no available data on the influence of this filler on the foam, nor was there any methodology to define the ideal quantity of calcium carbonate that should be added without causing damage to the mechanical properties of the final product. Calcium carbonate has large use in these industries because of its lower cost, non-toxicity, and non-abrasiveness.

In this work, a 2^k factorial (2^2) experiment was performed on calcium carbonate of particle size distributions (0.06, 0.5, 3.5, 10, 20, and 841 μm) and composition ranging from 0 – 40 wt% added to flexible polyurethane foam formulation to study their effects on mechanical properties such as Indentation Force Deflection, Tensile strength, and Elongation-at-break. It is intended to find the composition and particle size distribution that is most suitable for best foaming process without deleterious effects on the mechanical properties.

Experimental
Materials
Particulate filler (calcium carbonate) was purchased from West African Solid Mineral Company, Lagos, Nigeria. The average grain sizes of the calcium carbonate are 0.06, 0.5, 3.5, 10, 20, and 841 μm. Conventional polyether polyol was purchased from Korea Polyol Company, Ulsan, South Korea. The product name code is Konix FA-717. It is a 3500 molecular weight polyol of 1000 kg/m^3 density and hydroxyl value of 44. Toluene diisocyanate T-80 which is an 80-20 mixture of the 2,4- and 2,6- isomers of toluene diisocyanate was purchased from Lyondell Chemical Company, Texas, U.S.A. Other materials used are catalysts: Niax Stannous octoate (Dow chemical) and Dimethyl ethanol amine (DABCO, DMEA, Air Products), surfactant: Niax Silicone Oil L-580 (Dow chemical), Mould release agent: (Quatro HD X 40 Engine oil), and distilled water.

Experimental Setup
A foaming plant on a pilot scale (model ISOLBLOCK, IB 150/4F-SS) was used. The plant consist of a mixing chamber (0.15 m^3) into which is inserted a stirrer fitted with three phases motor 7.5 hp. The stirrer is operated at two speeds (700 and 1400 rpm). The mixing chamber is connected to a set of dosing cylinders with network of pipes. The dosing cylinders are for components with maximum capacity of polyol 0.1 m^3, isocyanate 0.06 m^3, tin catalyst 6 x 10^{-4} m^3, and blowing agent 0.01 m^3. The plant is operated by a control panel that includes the sequence for manual and automatic controls. Figure 1 shows the schematic diagram of the experimental setup.
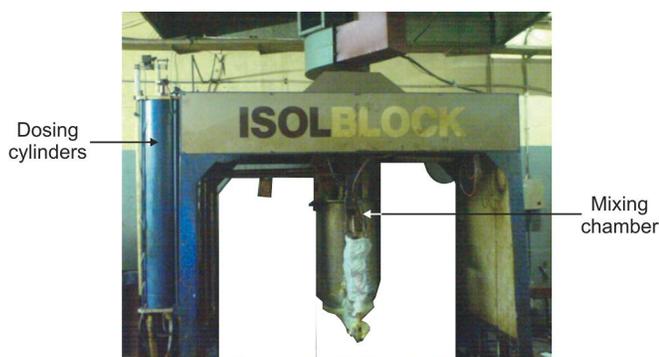
Figure 1: Foaming Plant (ISOLBLOCK IB 150/4F-SS)

Experimental Procedure

Inorganic filler reinforced flexible polyurethane foams were synthesized with the amounts of each chemical component chosen to obtain a target density of 25 kg/m^3. In the first step, 40 % of the total polyol required from formulation was weighed and mixed with the filler until there is complete homogenization. The volume fractions of the calcium carbonate used ranged from 0 – 40 wt%. Each of these composition ranges were investigated at an average particle size distributions (0.06, 0.5, 3.5, 10, 20, and 841 μm) representing ultrafine, lightly-fine and coarse fillers.

The 60 % of the polyol that remained from formulation was charged into the mixing chamber directly from the holding tank by means of automatic control on the foam plant control panel. Next, 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 the other chemical materials (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 (1.93 x 1.25 x 1.25 m), which was treated with mold release agent (Quatro HD X 40) to produce the free-rise foam. Two foam batches for each filler volume fractions 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.

Scanning Electron Microphotograph of randomly 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.

Design of Experiments

This study is based on the hypothesis that Indentation force deflection, tensile strength, and Elongation at break characteristics of polyurethane foams are functionally related to specific filler composition. To facilitate the analysis of variance of the experimental results, a 2^k factorial (2^2) design of experiment was conducted. The two factors are filler compositions and fillers particle size distributions with levels chosen to be within the range of reasonable formulations, and the ranges were carefully selected, since interpretation of the results was valid only within the experimental limits.

Indentation force deflection of the foam was measured by depressing a standard 380 x 380 x 50 mm sample in an indentometer (Hampden EC30, V2.48/024, testing machine) according to the procedures of ASTMD 3574 with the objective of evaluating the hardness characteristic of the foam with increasing carbonate concentration. The Indentation Force Deflection was quoted at 65 % indentation of the foam samples. This value give an indication of the force exerted by a seated adult and is attributed to the support characteristics of the foam. The Tensile strength and elongation-at-break of the foam samples were tested with the Hampden Indentometer following the procedure of ASTMD 3489-81. Tensile strength and elongation-at-break are important to the durability of the foam because any changes made to improve the hardness of the foam by addition of fillers do deteriorate these properties (Barma et al., 1978).

For all measurements three samples were collected from the top, middle, and bottom sections of the foam blocks. All samples were cut from the same location along the foam rise direction and placed in the test room 12 hours prior to tests. All tests were conducted at 23 ± 2 ^{O}C temperature. For all experiments, the values within a standard deviation of less than 10 % were used to calculate the mechanical properties.

Results and Discussions

In this study, the effects of filler compositions (0 – 40 wt%) and particle size distributions (0.06, 0.5, 3.5, 10, 20, and 841 μm) on the mechanical properties of flexible polyurethane foam such as Indentation force deflection, Tensile strength, and Elongation-at-break were investigated to determine the filler's composition and particle size distribution suitable for the best foaming process. Also studies of these factors on the

morphology of the foams were examined. Figure 2 depict Indentation force deflection at 65 % indentations versus filler compositions for all filler particle size distributions. The analysis of variance for this response is shown in Table 1. The analysis show that the main effects of filler compositions and particle size distributions are not meaningful, and cannot be interpreted on their own because, of the significant interaction effect between these factors at the 95 % level of significance considered in this work. It is therefore important to interpret the result by observing the effect of the filler composition at fixed levels of the particle size distributions. Figures 3 and 4 show the Tensile strength and Elongation at break versus filler compositions for all particle size distributions, while their analyses of variance are shown in Tables 2 and 3. The tables indicate the presence of non-significant interaction effects in the filler compositions and particle size distributions. The analyses revealed that the results of the tests on the main effects of filler compositions and particle size distributions on tensile strength and elongation at break are meaningful and could be interpreted. Microphotograph of selected foam samples were shown in Figures 5 – 7.

Table 1: The analysis of variance of filler compositions and particle size distributions to Indentation Force Deflection at 65% indentation.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	Computed f	F-Value
Filler Composition	24493.92	7	3499.13	24.99	2.25
Filler Particle Size Distribution	32209.15	5	6441.83	45.09	2.45
Interaction	37842.23	35	1081.21	7.57	1.67
Error	6845.39	48	142.86		
Total	101402.69	95			

Table 2: The analysis of variance of filler compositions and particle size distributions to Tensile strength.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	Computed f	F-Value
Filler Composition	12409.37	7	1772.77	16.1	2.25
Filler Particle Size Distribution	4337.58	5	867.52	7.88	2.45
Interaction	5900.21	35	168.58	1.53	1.67
Error	5284.07	48	110.08		
Total	27931.23	95			

Table 3: The analysis of variance of filler compositions and particle size distributions to Elongation-at-break.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	Computed f	F-Value
Filler Composition	143475.35	7	20496.48	31.12	2.25
Filler Particle Size Distribution	79410.28	5	15882.06	23.66	2.45
Interaction	37624.07	35	1074.97	1.63	1.67
Error	31617.38	48			
Total	292127.07	95			
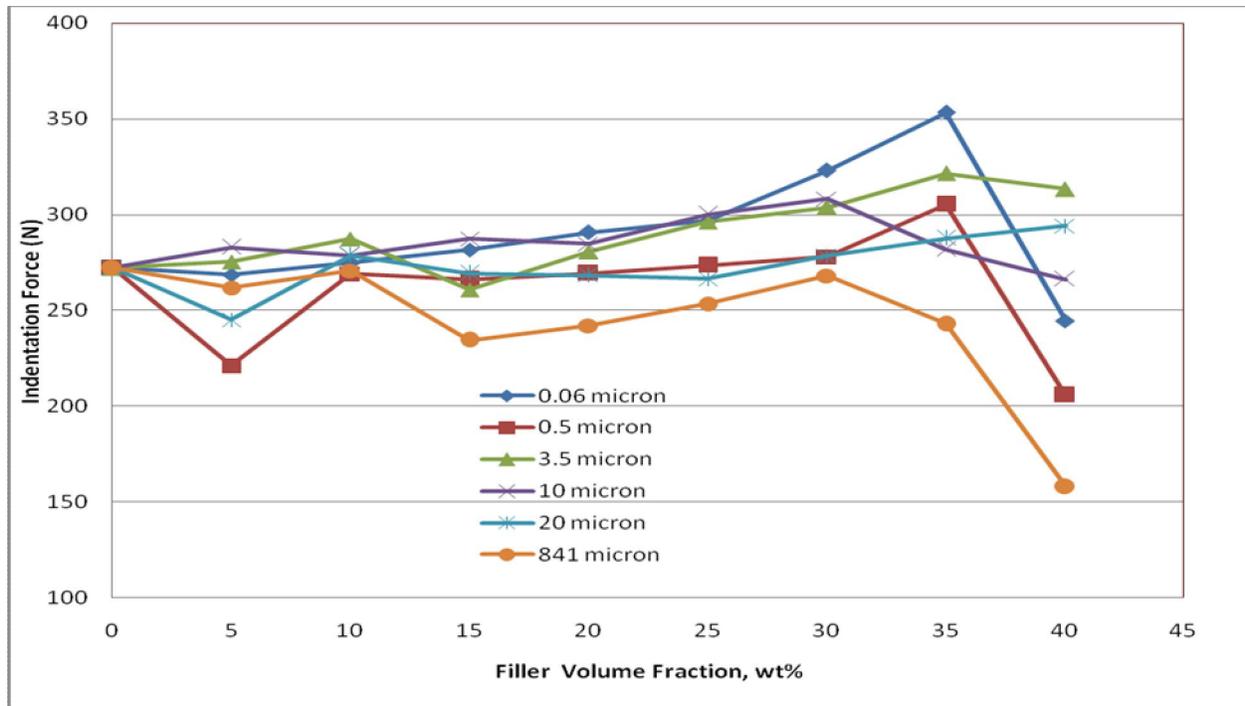
Figure 2: Effects of calcium carbonate on the 65% Indentation Force Deflection
Characteristics of Flexible Polyurethane Foam.

Effects of Filler Compositions and Particle size distributions on Indentation Force Deflection

At 5 wt% concentration, the indentation force for all of the particle sizes (except 10 μm) drops compared to the unfilled foam. At 10 wt% concentration, all the particle sizes seem to have the same effects on the indentation force. From 15 to 35 wt% filler concentrations, the indentation force increased gradually for all the particle sizes, with fine fillers (0.06, 3.5 μm) showing improved property compared to unfilled foam, while the coarse fillers (10, 20, 841 μm) deteriorate the indentation force.

The Coarse fillers (10, 20 μm) at low concentration (5 wt %) nucleate more bubbles at the bubbles/polymer interface and generate very small cells compared to fine fillers. The coarse fillers during foam growth are deposited at the plateau border area having a diameter of 50 μm. With continued expansion of the cells, the wall thinning process takes place and the reacting polymer is drained from the windows and struts into the plateau border area. Presence of coarse filler particles in this area can cause additional drainage. This could cause further thinning of the cell struts that would decrease the indentation force. Consequently, above 5 wt%, indentation force decreases with coarse fillers.

Fine filler particles (0.06, 0.5, 3.5 μm) on the other hand are collected on the cell windows which is

0.5 μm in diameter. Here, they interact with the soft segment in the polyurethane matrix decreasing their mobility and the ease with which they are drained into plateau border area, and thus increase the modulus and load bearing ability of the foam. However, the indentation force is not as much as for coarse filler at 5 wt% because the fine filler created fewer bubbles.

Filler particle size of 841 μm is substantially larger than the plateau border area and the cell windows. These fillers are randomly dispersed in the foam and destroy the formation of complete plateau border area and cell windows by draining excessive liquid polymer contributing to the foam poor structure and poor indentation force observed at all filler concentrations.

Calcium carbonate at all particle sizes investigated seem to have the same effects on indentation force as when the foam is unfilled at 10 wt% concentration, and signify the onset of transition of the effects of both fine and coarse fillers to that of enhancing the property and decreasing the property, respectively. The reason for this is unclear, but it could be that the combined effect of the filler composition and particle size has masked the individual effects that could have been expressed at this composition. However, from 15 to 35 wt%, the fillers has a gradual increase on the indentation force as the filler content is increased, with the fine fillers at (0.06, 3.5μm) showing improvement in the property

compared to unfilled foam. The fine fillers have a large surface area to volume ratio. At these quantities, the fillers create a large number of small cells and can interact with a lot more soft segment on the numerous polymer cell windows. This considerably improves the hardness as the filler content progresses from 15 to 35 wt%. It is interesting to note that coarse filler did not show any improvement in the foam indentation property in the filler content range of 15 to 35 wt%. Large quantity of coarse filler in this range did not allow a complete formation of plateau border area and cell windows by draining excessive liquid polymers from the cell struts thereby weakening the hardness strength of the foam. At 40 wt% filler content both fine and coarse fillers deteriorate hardness property of the foam.
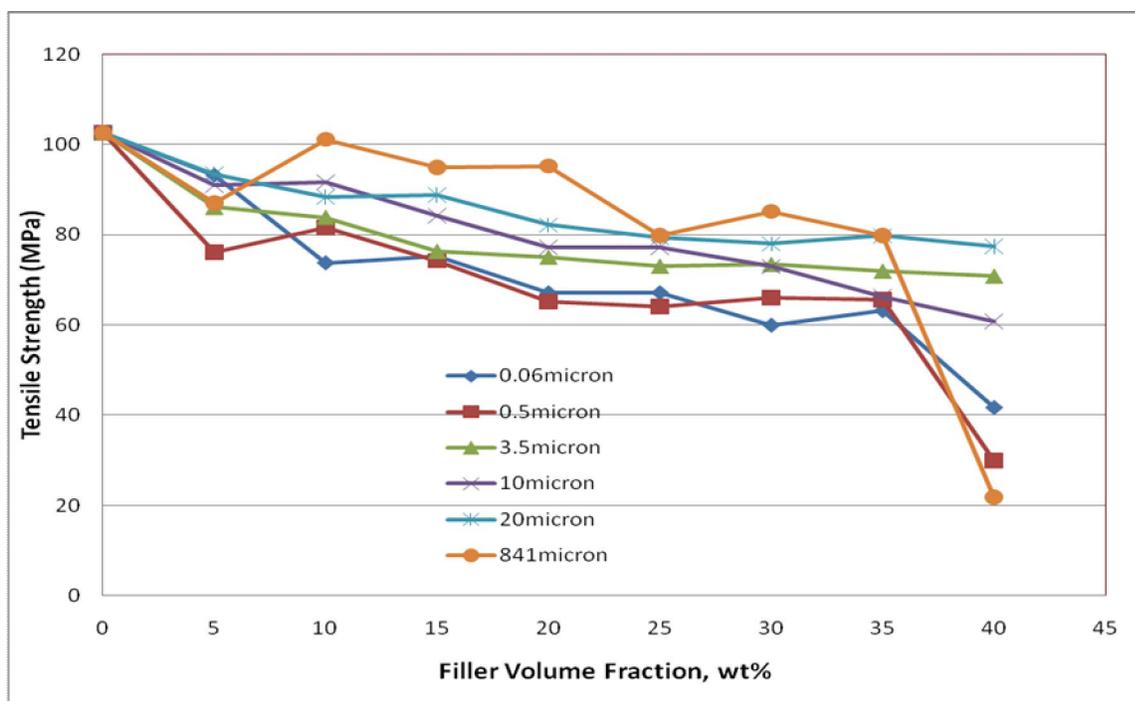
Figure 3: Effects of calcium carbonate of different particle sizes and compositions on the Tensile Strength of Flexible Polyurethane Foam.

Effects of Filler Compositions and Particle size distributions on Tensile Strength and Elongation at break.
    Figures 3 and 4 present the effect of calcium carbonate compositions and particle sizes on the tensile strength and elongation-at-break of filled flexible polyurethane foam. For all particle sizes, the tensile strength and elongation-at-break decreased while increasing the load of the filler weight percentages. However, the tendency with which the coarse filler reduce the properties is not as much as for the fine fillers. The decreased mechanical properties of the filled foam are probably due to the following factors:
- In the initial stage of foam reaction, when several chemical species are been formed, the dispersed fillers disturb the ease with which the randomly moving atoms of the reactant materials find themselves to form bonds. Because of the low surface area of the coarse fillers, the tendency with which they disturb the reaction is lower than for the fine fillers.   The reaction becomes incomplete and the foam structure becomes weakened.
- Secondly, agglomerated fillers, as a result of excessive particles that were not well dispersed in the polymer create stress concentrations in the polymer matrix and decreased the tensile strength and elongation at break.

    Control of foam density was generally good based on estimates of added water and TDI needed to compensate for the added fillers.
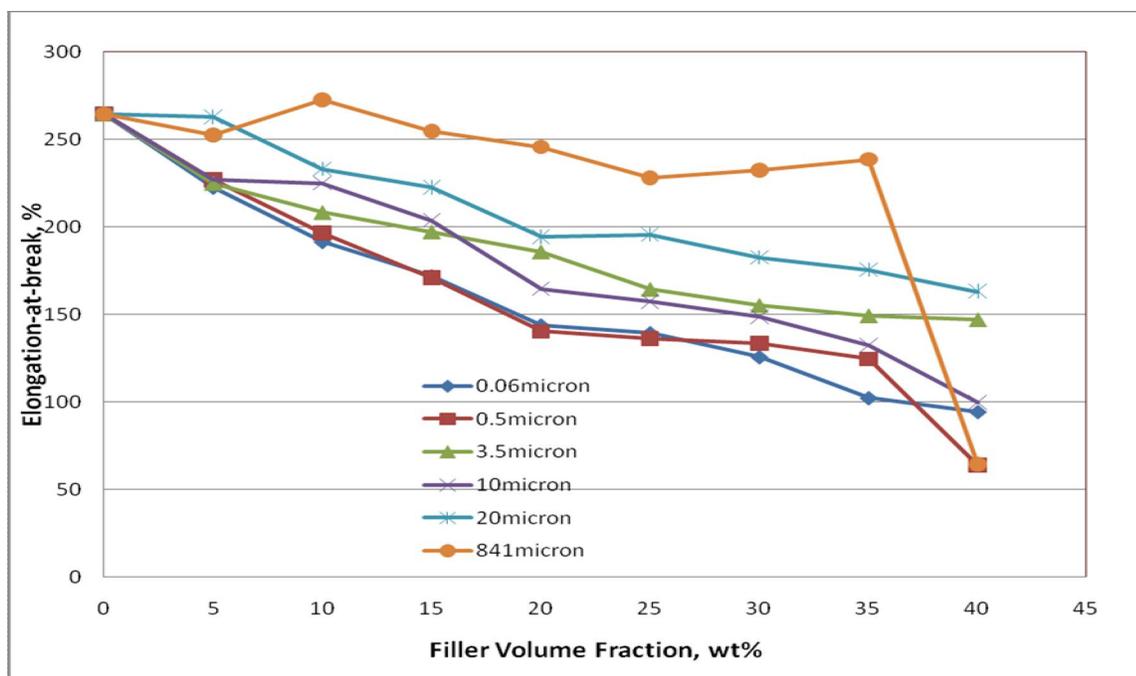
Figure 4: Effects of calcium carbonate of different particle sizes and composition on the
Elongation-at-break of Flexible Polyurethane Foam

Effects of Filler Compositions and Particle size distributions on Cell Nucleation.
    Figures 5 – 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 5. Morphologies of foam samples with formulation containing 15 wt% of fillers of particle size distributions 0.06, 3.5, and 841 μm are depicted in Figure 6. With a 40 wt% filler composition of particle size distribution 0.06 μm, the morphology is presented in Figure 7. Table 4 present the cell structure description, the average cell size per linear centimeter measured, and the corresponding compression set determined.

Table 4: Measurement of compression set, cell count, and microphotography (a: polyurethane foam containing 15 wt% of CaCO$_3$ of 0.06 μm size; b: polyurethane foam containing 15 wt% of CaCO$_3$ of 3.5 μm size; c: polyurethane foam containing 15 wt% of CaCO$_3$ of 841 μm size).

Properties	Ca15[a]	Cc15[b]	Cf15[c]	Ca40[d]	Unfilled
Density(kg/m^3)	25.7	25.9	25.7	27.1	20.4
Compression Set 75% (%)	7.6	8.0	7.6	24.7	10.7
Cell/cm (average)	14	14	13	15	11
Description of cell structure	Mainly big cells	Mainly regular but with clusters of smaller cells	Mainly big cells	Mainly regular but with clusters of smaller cells	Very irregular
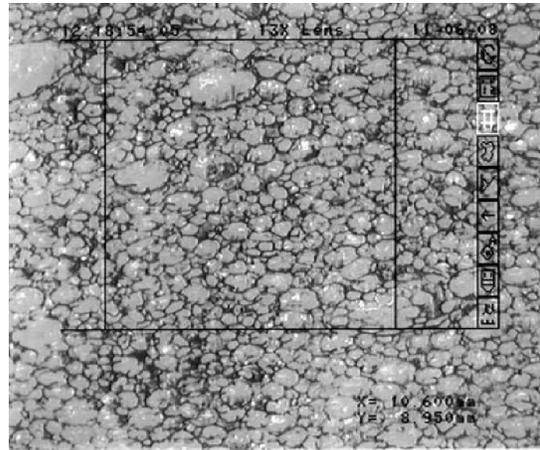
A

Figure 5: Cell morphology of unfilled flexible polyurethane foam
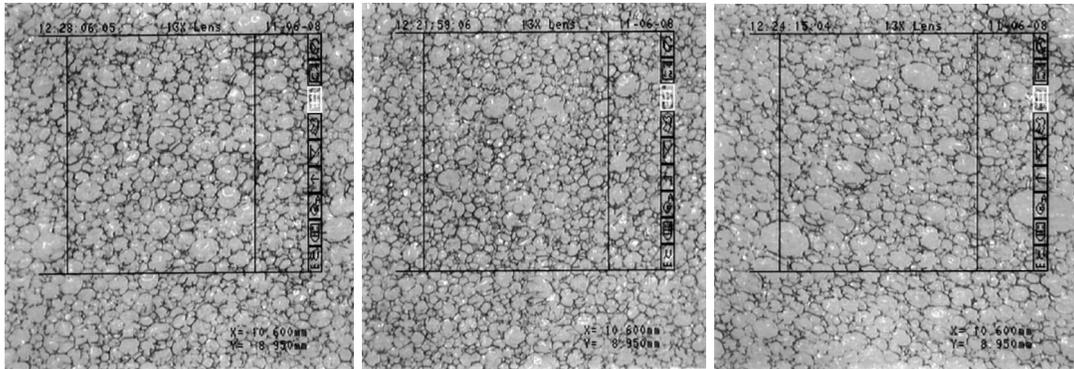
B: 0.06μm, 15wt% (Ca15)   C: 3.5μm, 15wt% (Cc15)   D: 841μm, 15wt% (Cf15)

Figure 6: Cell morphology of reinforced flexible polyurethane foam with calcium carbonate of different particle sizes.
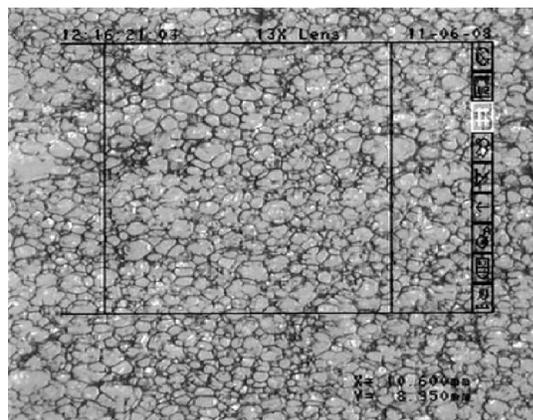
E: 0.06 μm, 40 wt% (Ca40)

Figure 7: Cell morphology of reinforced flexible polyurethane foam with calcium carbonate of different particle sizes.

It was observed in Figure 5, 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. In comparison with heterogeneous nucleation, the required activation energy for homogeneous nucleation is much higher (Klempner and Sendijarevic, 2004). 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.

Addition of filler in flexible polyurethane foams (Figure 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 (Chen, 2001). However, with different filler particle sizes of the same compositions, the foams cellular structures were more uniform at 0.06 and 3.5 μm particle sizes than for fillers of 841 μm. Even though the cell structure of foam with 40 wt% filler composition of particle size 0.06 μm is uniform and fine, the mechanical properties determined are low indicating that too much filler addition worked negatively to destroy the foams mechanical properties.

Conclusion

The use of purely inorganic filler as reinforcement materials in flexible polyurethane foams has been investigated. Calcium carbonate of six different particle size distributions (0.06, 0.5, 3.5, 10, 20, and 841μm) was used as fillers in the study. Concentration range of 0 – 40 wt% of these fillers was added in the foam formulation, with other chemical ingredients adjusted to obtain a target density of 25 kg/m^3. The essence was to reduce the quantity of polyol in the formulation to the extent it would not deteriorate the quality of the foam. Effects of the filler contents and particle sizes on the mechanical properties of the filled foam composites were investigated. The following observations were made in this study:

1. This study confirmed the technical feasibility of producing a variety of flexible polyurethane foam grades, using fine and coarse fillers.
2. The filler content and particle sizes strongly affected the cell geometry structures of the foam.
3. At 5 wt% concentration, the indentation force deflection at 65 % indentation for the entire fillers particle sizes (except 10 μm) drops compared to the unfilled foam. At 10 wt% concentration, all the particle sizes have the same effects on the indentation force. From 15 to 35 wt% filler concentrations, the indentation force increased gradually for all the particle sizes, with fine filler (0.06, 3.5 μm) showing improved property compared to unfilled foam, while the coarse fillers (10, 20, 841 μm) deteriorate the indentation force.
4. The inorganic filler- flexible polyurethane foam composites showed reduced tensile strength and elongation properties with the addition of fillers at all concentrations and at all particle sizes. This was due to cell geometry imperfection and expansion of the foam bulk, which pull apart the foam cell struts.

Acknowledgement

Correspondence to:

Dr. Ganiyu Kayode Latinwo
Department of Chemical Engineering
Ladoke Akintola University of Technology
P.M.B 4000, Ogbomoso, Oyo State, 210001, Nigeria.
Cellular Telephone: +2347039418289; +2348053623422
Email: kayodelatinwo@yahoo.com

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