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Development and Characterization of Flexible Polyurethane Foam: Part I- Physicochemical and Mechanochemical Properties

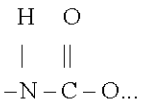
M.O. Edoga and E.A. Egila
Department of Chemical Engineering, Federal University of Technology, Minna, Nigeria

Abstract: The study of development and characterization of flexible polyurethane foams has been carried out principally by reacting non-conventional polyether polyol (which contained some amount of CaC_2) and toluene diisocyanate with small proportions of silicon oil, stannous octoate, dimethylethylamine, methylene chloride and distilled water in a glass reactor. At subsequent mixing stages, the reactants were thoroughly mixed using an electrically operated mechanical stirrer for 30 min. At the end of the reaction, the polyurethane resins were formulated by simplistic unifactor approach into four foam samples (A-D) at a mould temperature of 60°C and atmospheric pressure. The physicochemical and mechanochemical properties of the various samples were determined. The results show that sample B possessed the best quality whose properties are given as follows: density -15.90 kg m^{-3}; water absorption -998.4%; tensile strength -50.72 k N m^{-2}; shear (tear) strength -373 N m^{-2}; compression set -10% and elongation at break -271.42%.

Key words: Development, characterization, polyurethane foams, simplistic unifactor approach, physicochemical, mechanochemical, properties

INTRODUCTION

Polyurethanes are chemically complex polymeric materials principally formed by the reaction of bischloroformates with diamines. From industrial perspective, they are formed by the reaction of diisocyanates and dihydroxyl compounds (polyols) of either polyester or polyether family. Polyurethanes contain the



group linkage. Isocyanates are compounds that have one or more of the highly reactive group (N = C = O) and this group easily react with the polyols that are active hydrogen containing compounds to form either flexible or rigid plastic foams (Boden *et al*., 1985).

Polyurethane foams whether flexible or rigid are plastic materials in which a proportion of solid phase is replaced by gas in form of numerous small bubbles (cells). This gas may be in continuous phase to give an open cell material or it may be discontinuous to give non-communicating cells (Herington, 1979). Talk of flexible foams, the reaction is between diisocyanates and carboxyl terminated polyesters or commonly by the reaction of dihydroxyl polyethers or polyesters with diisocyanate (Woods, 1982). Generally, foams are prepared using either the chemical, mechanical or physical methods. Generally, polyurethanes are used in a wide variety of applications as adhesives, coatings, elastomers and as foams of flexible and rigid types. Presently, flexible foams have gained so much popularity because of their wide variety of products suitable for various applications (Technical Bulletin, 1981). They are used as insulators including textile laminates for cold weather clothing, automobile crash panels, upholstery, bedding, carpet underlay, synthetic sponges and as absorbent in separation processes (e.g., cleaning of oil spill on the sea).

Flexible polyurethane foam was first produced in the laboratory in 1941, worldwide and later in the 1960's when it became an industrial project (Woods, 1982). In Nigeria, foam industry is a relatively young one when compared to other well-established chemical process industries. Today, the foam industry requires urgent attention and expansion so as to meet up with the unlimited market demands in Nigeria. This research is one such attempt.

The object of this research, therefore was to develop polyurethane foams locally and conventionally by chemical method (blowing by *in situ*-chemical reaction) comparable to international standard. Specifically, the aim

Corresponding Author: M.O. Edoga, Department of Chemical Engineering, Federal University of Technology, Minna, Nigeria

was to polycondense toluene diisocyanate with polyether polyol of which the product was subsequently formulated with other chemical additives, such as dimethlethylamine, silicone, stannous octoate, methylene chloride, distilled water and Kaolin (clay) into foam.

MATERIALS AND METHODS

Conventional or the research code names of the chemicals/materials are used in the present study Table 1. The various experiments were done in chemical engineering laboratory, chemical engineering department, Federal University of Technology (FUT), Minna-Nigeria.

Development of flexible polyurethane foam samples: Twenty five grams of polyether polyol was first charged into the steel foam reactor. In the same reactor, a mixture of 0.8 g water, 0.4 g silicone oil and 0.1 g climethyle-thylamine that was previously stirred for 10 min was charged and was thoroughly stirred for about 30 sec. Subsequently, 5.7 g methylene chloride was added and mixed for 15 sec. Later, stannous octoate was also added to the mixture and was stirred for about 10 sec. Finally, 9.5 g toluene diisocyanate was charged into the steel foam reactor and was stirred thoroughly for 3-5 sec after which the mixture was poured into a plastic mould immediately the creaming was observed, a process that simultaneously tallied with the rising of the foam. The entire stirring operation was carried out using an electrically operated mechanical stirrer.

The mixture was allowed to attain full rise and kept to cure for about 5 h of 60°C (mold temperature at pour).

The foam block samples were discharged from the moulds for trimming and other necessary evaluation tests (physicochemical and mechnochemical properties).

The mechanochemical tests were performed according to the standard methods of testing, flexible cellular materials by Stone (1983).

Physicochemical/mechanochemical tests

Density test: The density of the foam sample was determined by first finding the weight of a regular shaped foam block and subsequently the volume of the foam block. The volume was determined by measuring the length, breadth and height of the foam. Their product equals the volume. From the ratio of 1 weight/mass to volume, the density was obtained.

Compression set: The compression set was determined when a foam sample was in between two flat metal plates and was subsequently compressed to 50% of the original thickness. That was left for 72 h and later allowed to recover for 30 min. The compressed foam sample was remeasured to obtain the percentage change in original thickness as shown in Table 2.

Table 1: The basic chemical/materials used for the experiment

Material	Code	Structure	Source	Comment
Polyether polyol	PEP	R-OH, R-CH_2-OH -NH-C-O ‖ O	Dow chemical company (SPECFLEX)	Soluble in water melthing pt = 116.3°C Bp = 34.6°C b.p.°C/Tor -121/10
Toluene Diisocyanate	TDI	CH_3 NCO NCO	Dow chemical company (OORANOL)	m.p. = 22°C Bonds are hydrozyable
Silicone oil	S_{iOI}	S_i-O-C	Goldschmidt company	m.p. = 246.8°C
Stannous octoate	STOC	S_n	Air product and chemical Inc. (DABCO)	Soluble in water but not in alcohol and ether
Dimethylethlamine	DMEA	R-NH_2	Goldschmidt company	b.p. 40°C, heat of vaporization 142, mol.wt. 84.9
Methylene chloride	MECH	Cl Cl - C - H H	INDEDS chloro Ltd. U.K.	Universal solvent, b.p. = 100°C
Distilled water	DIWA	O H H	F.U.T., Minna Nigeria	m.p. 0°C Specific gravity = 1.000
Clay kaoline				

Table 2: Composition of various foam samples from various formulation

Materials (g)	A	B	C	D
PEP	25.00	25.0	25.0	25.0
TDI	9.40	9.5	9.2	9.6
DIWA	0.80	0.8	0.8	0.8
DMEA	0.10	0.2	0.2	0.1
MECH	5.60	5.5	5.8	5.7
STOC	0.01	0.1	0.2	0.2
S_iO_l	0.40	0.6	0.5	0.5

Table 3: Dimensions of various foam samples

Dimension (m)	A	B	C	D
Length	0.1300	0.1270	0.1130	0.0950
Breadth	0.0995	0.0900	0.0810	0.0800
Height	0.0220	0.0270	0.0180	0.0200

Table 4: Densities of the various foam samples

Sample type	Average weight	Volume×10^{-4} (m^2)	Mass (kg)	Density (kg m^{-3})
A	33500	2.840	0.0047	16.55
B	31500	3.142	0.0050	15.90
C	3250	1.648	0.0027	16.38
D	3380	1.520	0.0026	17.11

Table 5: Mechanochemical properties of various foam samples

Mechanochemical property	A	B	C	D
Tensile strength (KN m^{-2})	49.28	50.72	46.42	43.57
Elongation at break (%)	285.71	271.42	252.14	242.86
Tear strength (N/m)	345.00	373.00	361.00	358.00
Water absorption (%)	1117.20	998.40	1536.80	1284.30
Compression set (90%)	8.10	8.15	7.50	7.0
Resilience (%)	4.85	4.84	4.00	4.01
Hysteresis return (%)	75.00	90.00	-	-
4-inch load bearing at 25% lb 150 m^2	8.00	8.05	7.40	7.01
4-inch load bearing at 50% lb 150 m^2	11.00	11.05	10.50	10.15
4-inch load bearing at 65% lb 150 m^2	15.00	15.05	14.45	14.25

Tension test: The foam block sample used for this test was shaped dog bone and the sample was then pulled at a constant rate until it broke using tensile testing machine. The force breaking point per unit cross-sectional area gave the strength of the foam.

Tear test: This test was carried out using shear testing machine by continuously trying to tear a foam block sample after a split or break has been started. It involved the measurement of the force required to continuously tear the foam block sample.

Identation force deflection test: The test was carried out on a sample by dropping a 1 kg weight load upon it. Subsequently, other varying loads were placed upon the foam samples to obtained 35, 50 and 65% indentation hardness, respectively, as shown in Table 3 and 4.

Elongation at break test: This test was carried out using the tension testing machine to obtain the change in length as a result of elongation of the dog bone shape foam sample. The reading was subsequently taken just before the breakage so as to obtain the percentage elongation of the various samples.

Water absorption test: The water absorption capacity of the foam sample was determined by first weighing the foam sample after which it was submerged in water for 24 h. When the sample was removed and blown free of the surface water, the foam sample was reweighed. The difference in weight multiplied by 100 gives the percentage of the absorption water.

Other tests such as the average molecular weight, hysteresis return percentage, modulus of rupture resilience percentage and the air flow at the middle were also determined according to the standard methods of testing flexible cellular materials (Egila, 2003).

RESULTS AND DISCUSSION

Physicochemical properties: The results of applying modified simplistic unifactor approach in obtaining composition of various foam samples from various formulations and their corresponding dimensions, densities and water absorption are presented in Table 2-5.

From Table 2 and 4, the effects of varying some constituents of the foam samples are reflected on the physicochemical properties of the samples. For instance, varying the amount of toluene diisocyanate (TDI) controlled the hardness of the foam since increasing the amount correspondly increased the density as observed in sample D which was formulated with the largest amount of TDI. On the other hand, increasing the amount of dimethylethylamine caused a decrease in the density of foam samples (Table 2 and 4) whereas a decrease in DMEA caused the reaction between TDI and DIWA to increase in the production of CO_2 gas during foaming which could be accountable for the increase in the volume of sample A.

Considering, the effect of stannous octoate on the foam sample, increasing the quantity also increased the hardness of the foam, thereby increasing the density of the foam samples as observed in samples C and D. This is plausibly explained by the singular fact that stannous octoate enhances foam cohension and hence the crosslinking ability of the foam which invariably will influence water absorption capacity as reflected in samples C and D (Table 2 and 4).

The effects of TDI and STOC on the average molecular weight of the foam samples showed the same trend as that of density. The values of the densities,

molecular weight and water absorption are conformity with the standard values (Stone, 1983; Knibbe, 1985).

Mechanochemical properties: The results of the mechanochemical tests are presented in Table 5.

From Table 5, it can be seen that sample B possessed the best quality in terms of the tensile strength (50.72_k N m^{-2}) when compared to other samples whose tensile strength values were slightly below the literature value (Table 5). For elongation at break sample A and B possessed considerable elongation ability when compared to other samples. They are comparable to the literature value (Table 5). Regarding the tear strength, sample A possessed the closest value of 345 N m^{-1} to that of the international standard of 289 N m^{-1}. In the same vein, sample A was able to reach the specified requirement for indentation since it could carry a load of 10 kg to obtain a 65% indentation when compared to its weight of 0.0049 g.

Also, it can be seen from Table 5 that sample B exhibited a compression of 0.02 m over a period of 72 h, which apparently signifies a very good durability.

Above all, it can be seen from Table 5 that samples A and B possessed good tensile strength, enduring elongation at break, tear strength, indentation and compression, resilience and hysteresis return comparable to the literature values.

CONCLUSION

The overall conclusions emerging from the conventional development of flexible polyurethane foam by reacting polyether polyol and toluene diisocyanate with small proportions of silicone oil, stannous octoate, dimethylethylamine, methylene chloride and distilled water in a glass foam reactor at mould temperature at pour of 60°C are that:

- Flexible foams of comparable characteristics quality with the international standard can conventionally and locally be developed in our laboratory typical of samples A and B of 16.55 and 15.90 kg m^{-3} density (super soft foam samples), respectively.
- The quality of foam could easily be manipulated by properly varying the amounts/concentrations of some of the principal reactants typical of TDI, DEP, DMEA as can be seen in Table 2.
- Finally, it can be concluded that the cost of production of flexible foam to a large extent depends on the cost of PEP since PEP is the ingredient that is required in the largest proportion.

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