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Effects of Formulation Parameters on the Properties of Flexible Polyurethane Foam (FPF)

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Abstract: For flexible polyurethane foam production, the level of TCFM 11 (Freon) and carbon dioxide release from the reaction of di-isocyanate and polyol must be properly monitored and kept within a suitable value. The use of formulation with higher water level and no TCFM 11 blowing agent to produce low density foam is the most common sources of scorching problems. The higher the water level in the formulation, the higher is the exothermic temperature in the block. The maximum recommended water level used with polyol in the absence of TCFM 11 is 4.8 parts per 100 polyol. At higher water level, there may be a risk of scorching which may lead to fire. A suitable formulation was designed to accommodate for a reasonable level of both the primary blowing agent (CO_2) and the secondary blowing agent (TCFM 11), resulting in the production of a flexible polyurethane foam. ASTM test procedures were employed in characterizing the foam properties. Such properties as the foam density, load deflection test, compression set, porosity and flammability properties were carefully analyzed to ascertain the overall performance of the final foam. Results of flammability test reveals that incorporating some little amount of chemicals such as Bromine or Chlorine plus inert fillers such as antimony will reduce to a great extent the combustibility of foam materials, but care must be taking in adjusting the formulation to prevent the resulting effects of scorch.

Key word: Foam · Flammability · Compression · Density · Cell count and blowing agents

INTRODUCTION

Cellular plastics are produced by the dispersion of aqueous phase in to a fluid polymer. They can be prepared by a variety of methods. The most important process, consist of expanding a fluid polymer phase to a low density cellular state and then preserving this state. This is the foaming or expanding process. Other methods of producing the cellular state include leaching out solid or liquid materials that have been dispersed in a polymer, sintering small particles and dispersing small cellular particles in a polymer. The latter process however, is relatively straight forward processing techniques but is of minor importance [1].

The expansion processes consist of three steps:

- Creating small discontinuities or cells in a fluid or plastic phase.
- Causing these cells to grow to a desired volume.
- Stabilizing this cellular structure by physical or chemical means [2].

Cellular plastics and polymer have been prepared by a variety of process involving many methods of cell initiation, cell growth and cell stabilization. The most convenient method of classifying these methods appears to be based on the cell growth and stabilization processes. The growth of a cell in a fluid medium at

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equilibrium is controlled by the pressure difference (ΔP) between the inside and outside of the cell, the surface tension of the fluid phase (λ) and the radius (r) of the cell. [3] Such pressure difference may be generated by lowering the external pressure (decompression) or by increasing the internal pressure in the cells (pressure generation). Other methods of generating the cellular structure are by dispersion of gas or liquid in the fluid state and stabilizing this cellular state, or by sintering polymer particles in a structure that contains a gas phase [4].

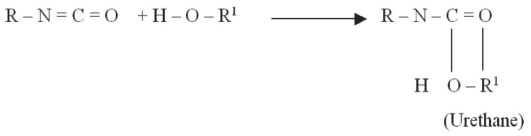
Foams are classified as open-cell or closed-cell. In closed-cell foams each cell (more or less spherical in shape) are completely enclosed by a thin wall or membrane of plastic (Figure 1 and Figure 2). The terms plastic foam, foamed plastic and cellular plastic are used interchangeably; they refer to foamed plastics regardless of cell structure (open or closed). Expanded plastic refers to closed-cell materials; "sponge" is sometimes used for open-cell foams (e.g, sponge rubber).

Plastic foams may be flexible, semi-flexible (or semi-rigid) and rigid, depending on chemical composition and the rigidity of the resin used as a matrix. Flexible foams have a glass transition (TG) below room temperature, whereas rigid foams have one above room temperature. The matrix is made up of the base resin and other compounding ingredients that may include plasticizers, stabilizers, surfactants, dyes and pigments, fire retardants and fillers. The composition of the plastic matrix plays an important role in determining foam properties such as chemical resistance, thermal stability, flammability, specific heat, transition temperature and rigidity [5].

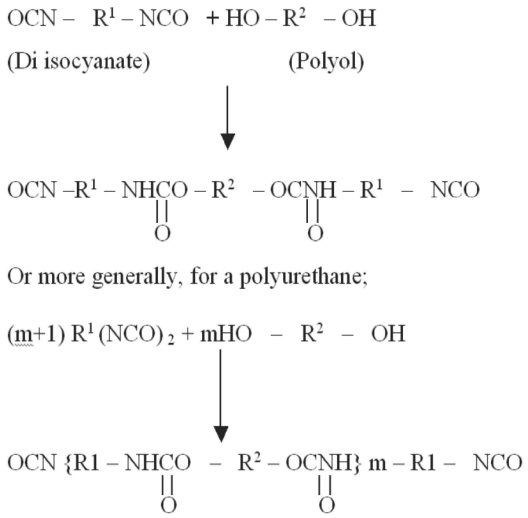
MATERIALS AND METHODS

Chemistry of Foam Making: A urethane is produced as a result of the reaction between an isocyanate group and a hydroxyl group.

This reaction can be extended if the simple isocyanate is replaced by a di isocyanate and the mono-alcohol by a polyol.



Or more generally, for a polyurethane;



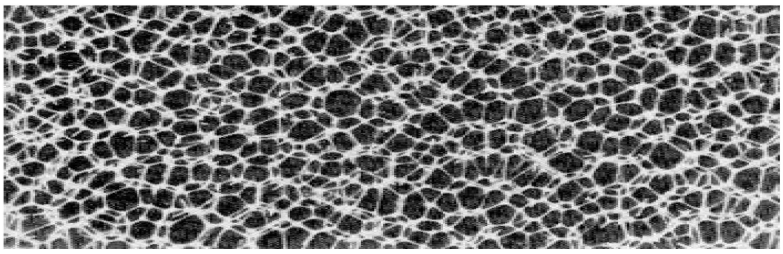


Fig. 1: Photomicrograph of cross-section rigid polyurethane foam

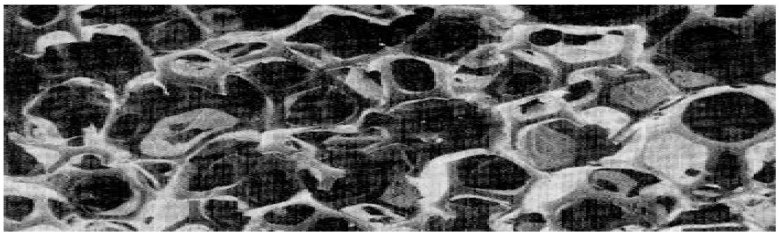
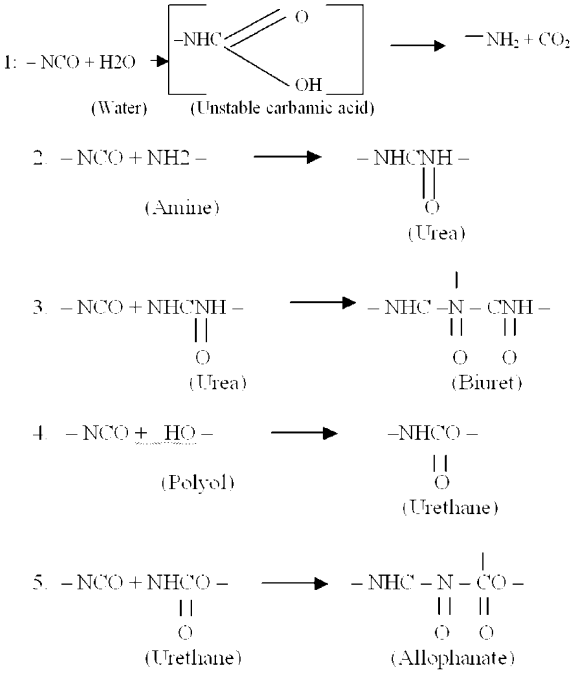


Fig. 2: Photomicrograph of cross-section of flexible polyurethane foam

Isocyanate reacts with materials having active hydrogen atoms and in the production of polyurethane foam a whole series of reactions of this type takes place. For simplicity, only one functional isocyanate group and the functional part of the reaction products are illustrated [6].



Reactions 2 – 5 inclusive result in the formation of crosslink's causing first, an increase in the viscosity of the reaction mixture and eventually the formation of a gelled polymer. Reaction 1 causes the mixture to foam due to the formation of carbon dioxide. The amine formed in this reaction takes part in reaction 2 and contributes to the formation of the polymer. Reaction 3 and 5 increase the number of crosslink's in the polymer and therefore influence the final properties of the foam.

MATERIALS AND MOTHODS

Materials used in the production of the foam are discussed in Table 1 below.

Other ingredients used in the production of the foam include:

- Water.
- Stannous octate.
- Silicone surfactant.
- Freon (TCFM 11)
- Fire retardant: Chlorine and inert filler (antimony).

Using the various materials, a standard formulation was designed as shown in Table 2 below:

Table 1: Properties of selected foam additives

S/NO	ADDITIVES	SOURCES	DISCRIPTION
1	Polyol.		Polyol are essentially propylene oxide and ethylene oxide copolymer with a trifunctional initiator and are therefore triols. It is a liquid of low viscosity blend especially designed for easy processing with minimum cycle time to give low density closed cell insulating foam. It has specific gravity of 1.080.
2.	Isocyanate.(TDI)		They are generally liquid mixture of poly-isocyanate with 4,4 di-isocyanate diphenyl methane as the main component
3.	Tertiary Amine.		Amines, compounds, often produced in the decomposition of organic matter, formed by replacing one or more of the hydrogen atoms in ammonia with organic groups. The number of organic groups attached to the nitrogen atom determines whether the molecule is classed as a primary, secondary, or tertiary amine.

Table 2: Typical formulation for flexible polyurethane foam production (F = formulation)

	F_1	F_2	F_3	F_4	F_5	F_6
Ingredients	Pbw	Pbw	Pbw	Pbw	Pbw	Pbw
Polyol	100	100	100	100	100	100
Water	4.80	4.80	4.80	12.00	4.80	4.80
Tertiary Amine	0.15	0.15	0.15	0.15	2.00	0.15
Stannous Octate	0.20	0.20	0.20	0.20	0.20	0.20
Silicone Surfactant	1.30	1.30	1.30	1.30	1.30	1.30
Freon 11	10.00	10.00	14.50	10.00	10.00	10.00
Di isocyanate (TDI)	58.4	70.00	58.4	58.4	58.4	58.4
Chlorine	-	-	-	-	-	0.15
Inert Filler (antimony)	-	-	-	-	-	2.00

The various ingredients were weighed into a suitable container according to the formulation above: they were mixed by means of an electrothermal stirrer. Good mixing is essential to produce homogeneous foam. The silicone surfactant further enhances the mixing operation since it serves as both an emulsifier and also reduces the surface tension of the polyol. Air bubbles were allowed into the liquid during the mixing operation. This air bubbles normally serves as initiation points for foam production. In other to increase the bubble formation, a small amount of air is normally introduced into the polyol prior to mixing. After about 12 seconds, the blowing gases viz: carbon dioxide and Freon 11 diffuses into these small air bubbles and enlarges them. As more blowing gas is generated, the bubbles expand and the foam begins to rise. The number of initiated bubble usually remains constant but its diameter enlarges with more blowing gas formation. The silicone surfactant thus help to stabilize the foam and prevent it from collapsing,as this happen, polymerization reaction also occur simultaneously in the liquid phase, thereby making the system to be more stable. With high level of the TCFM 11 blowing agent and knowing the functionality of the polyol, flexible polyurethane foam was produced. By varying the other additives, different samples of foam where produced as indicated in the formulations below:

F_1 : Standard flexible polyurethane foam formulation.
F_2 : Studying the effects of change in Isocyanate level.
F_3 : Studying the effects of change in the blowing agent level.
F_4 : Studying the effects of change in the water level.
F_5 : Studying the effects of change in the Amine level:
F_6 : Formulation aimed at reducing flammability of polyurethane foam.

Characterization of Foam Sambles: In other to ascertain the final properties of the foam samples produced, the samples were subjected to the following tests after conditioning.

- Compression test.
- Indentation load deflection.
- Density.
- Elongation.
- Flammability.
- Cell count.
- Blow index vs. Density analysis.

In conditioning, the test samples were kept inside a constant temperature oven at 23±1° and a relative humidity of 50±2%without any load on them for 12 hours prior to testing.

Compression as used with flexible polyurethane foam sample test is a measure of the degree of resilience of a flexible foam sample (i.e. the rebound properties of foam). A sample of FPF (Flexible Polyurethane Foam) was cut out at measured dimensions and it was subjected to the compressive force of a compression molding machine (Figure 3). The sample was left under compression for 15 minutes and the extent of return was measured as a function of the initial vs. original length expressed as a percentage. The compression was selected to 50% compression state.

Initial length of test sample (L_1) = 5.20
Compressed length C_1 = 50% compression = 2.60
Final length after release (L_2) = 4.98 (15 min)
Compression set = $(L_1 - L_2) / L_1 * 100$

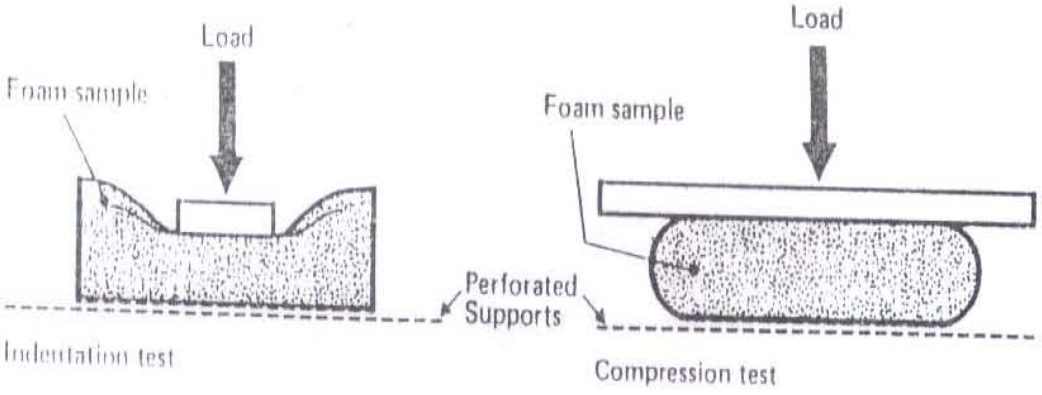


Fig. 3: Compression Test Module

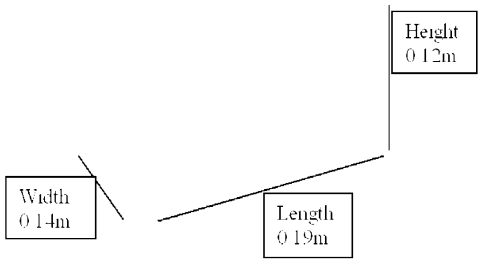


Fig. 4: Density Test Module

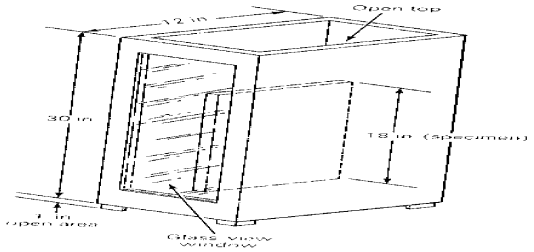


Fig. 5: Flammability Test Module

For indentation test, a sample of 380*380*20mm was used. The sample was placed on the support plate so that the indentor was over its center. The test area was indented twice by lowering the indentor to 75 – 80% of the sample thickness, the load was then removed and the sample was allowed to rest for 6±1 minutes before testing. The indentor was again brought into contact with the sample, a preload of 465g was applied and the thickness of the sample under this load was measured. The sample was then indented by 25% of this thickness at a rate of 5cm/min. and after 1.2min, the load was recorded. The deflection was then increased to 60% of the thickness under the preload and again after 1.2min, the load was recorded. This results expressed in Newton are the 25% and 60% ILD values [2].

For density test, a sample of FPF of specified dimension was cut out, it's mass was determined on an electronic weighing balance. Its volume was calculated mathematically from volume = L*B*H where L, B and H represent the length, breadth and height respectively. The density of the foam sample was obtained from the ratio of the mass to its volume (Figure 4).

Elongation test is a measure of the amount of extension of the FPF test sample before breaking. The procedure involves clamping the FPF test sample at both ends and a pulling force was applied until the sample breaks.

Initial length of test sample (L_1) = 5.20
Final length before break (L_2) = 10.2
Elongation (%) = ($L_2 - L_1$) / L1 * 100

Flammability test was carried out to asses the fire retarding tendency of halogenated compounds and inert fillers such as antimony. A tiny specimen of the FPF 0.05 inches thick and 18inches long was used. The sample was fixed vertically after marking a 12 inches gauge length, 3inches from each end. Timing begins when flame from a Bunsen burner reaches the lower gauge mark and ends when 12 in. of the strip was consumed; values were recorded in inches/minute (Figure 5).

The cell count test was done by cutting out a square sample of the FPF and counting the cell through the use of an electronic device capable of counting the number of cells in both rows and columns. Two or more rows and columns were run and at the end, the average number of cells in each rows and columns was gotten. The cell count was taken as the product of the average number of cells in each row and columns.

For the variability of density of FPF with its Blow Index, the blow index of FPF was first calculated using the simple mass balance equation of the form:

Pbw water per 100 polyol +
(Pbw TCFM 11 per 100 polyol / 10) (3.1)

Five different formulations were designed from formulation F1 above by varying the parts by weight (Pbw) of the secondary blowing agent (TCFM II) at constant water level of 4.8. The resulting blow index was calculated from the mathematical equation above and the density of each FPF samples were obtained from the density procedure outlined above.

RESULTS AND DISCUSSION

It can be deduce from the results of density versus blow index that with a constant level of water and a corresponding increase in the TCFM 11, the blow index of the foam increases with a corresponding decrease in the foam density. It means therefore that the most important factor controlling the foam density is the blow index which is a measure of the level of water and secondary blowing agent (TCFM 11) per 100 weight of polyol. It can be seen from the graph that for a density of 21kg/m^3, a blow index of 5.8 is required. The result of density in Table 3 shows that as the level of TCFM11 and water

Table 3: Typical formulation for the computation of blow index of FPF

	F_1	F_{1a}	F_{1b}	F_{1c}	F_{1d}	F_{1f}
Ingredients	Pbw	Pbw	Pbw	Pbw	Pbw	Pbw
Polyol	100	100	100	100	100	100
Water	4.80	4.80	4.80	4.80	4.80	4.80
Tertiary Amine	0.15	0.15	0.15	0.15	2.00	0.15
Stannous Octate	0.20	0.20	0.20	0.20	0.20	0.20
Silicone Surfactant	1.30	1.30	1.30	1.30	1.30	1.30
Freon 11	10.00	12.00	14.50	16.00	17.00	18.50
Di isocyanate TDI	58.4	70.00	58.4	58.4	58.4	58.4

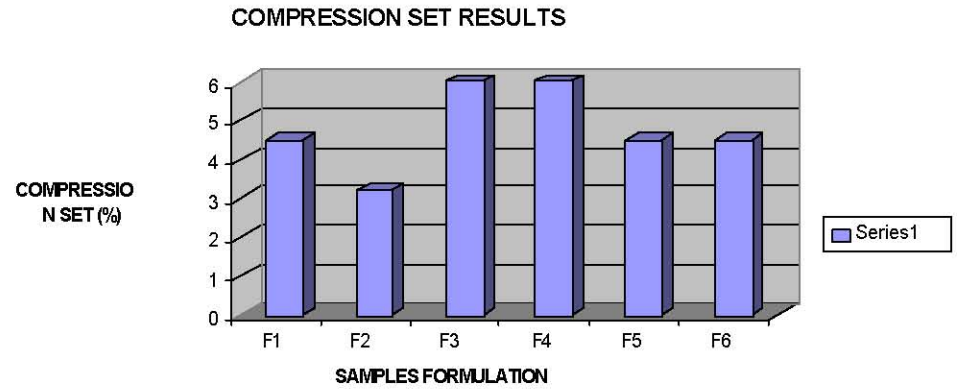


Fig. 6: Compression Test Result

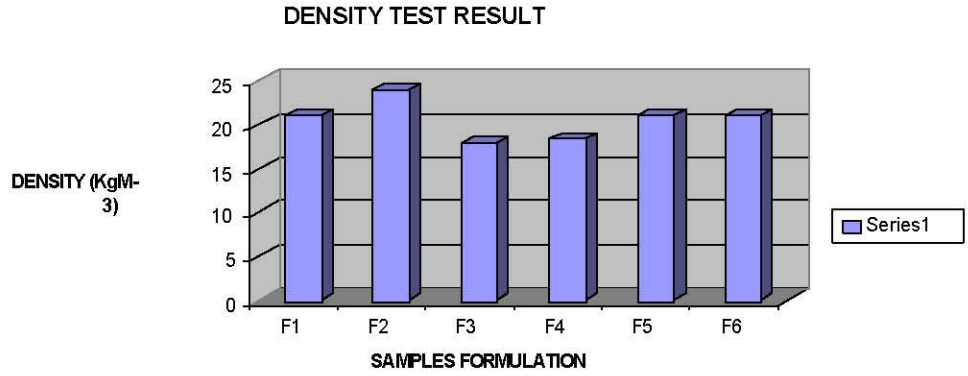


Fig. 7: Density Test Result

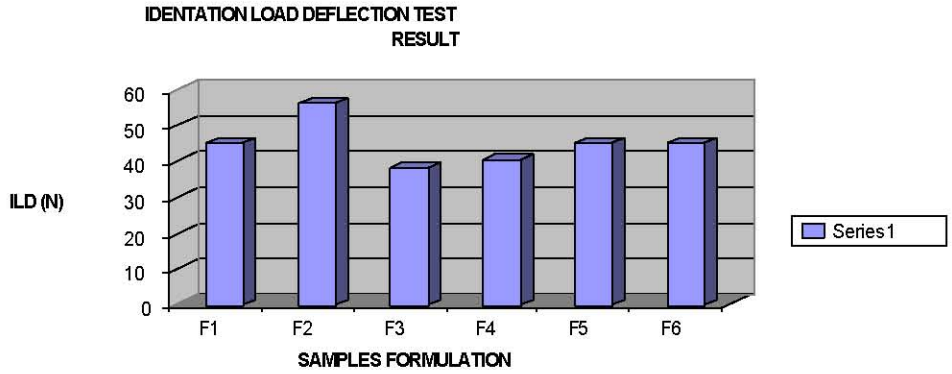


Fig 8: Indentation Load Deflection Test Result

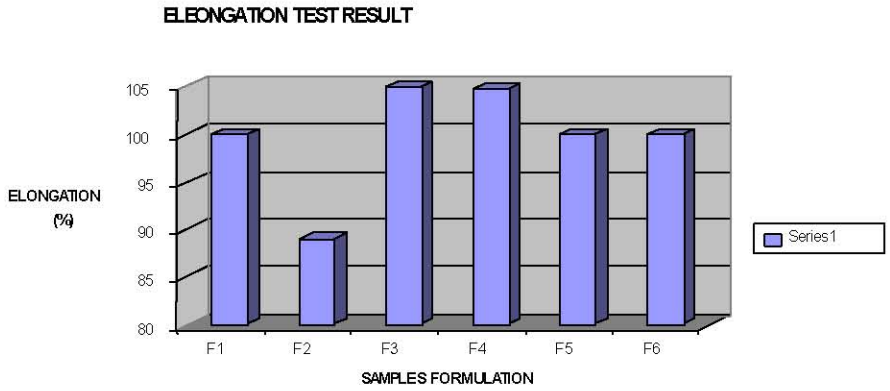


Fig 9: Elongation Test Result

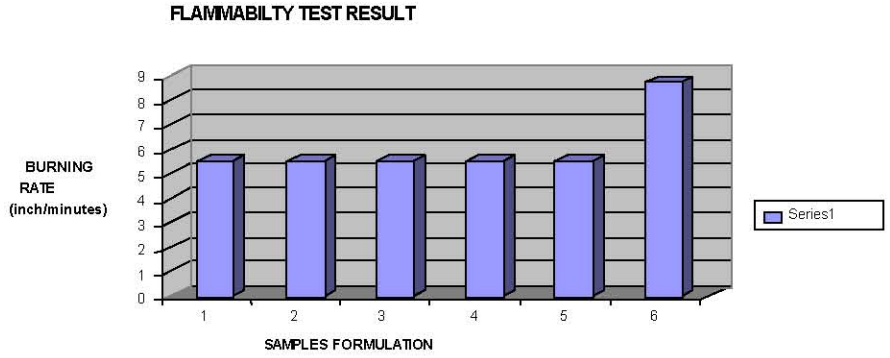


Fig 10: Flammability Test Result

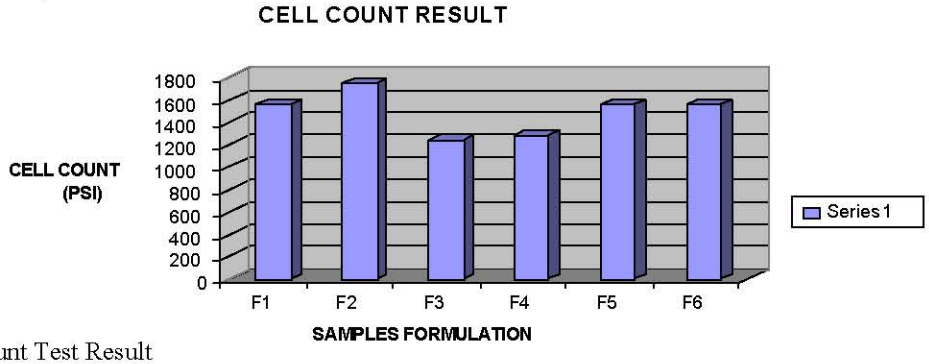


Fig 11: Cell Count Test Result

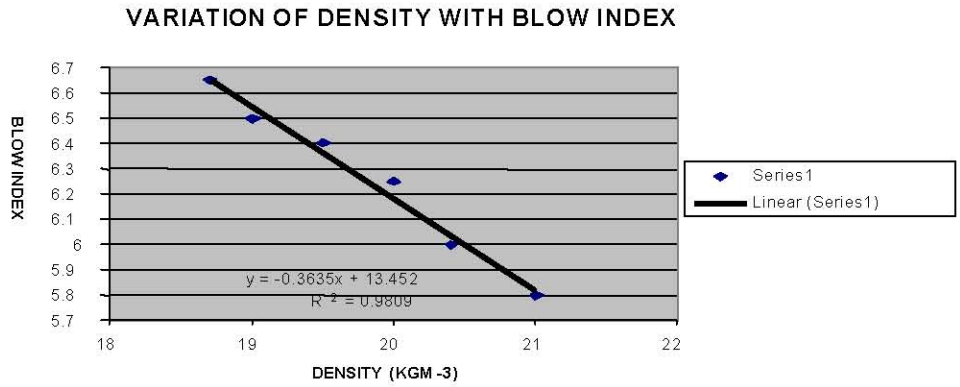


Fig 12: Variation of Density with Blow Index Test Result

increases, the density of the foam invariably decreases, where as an increase in the di isocyanate level invariably increases the density of the foam.

The results of compression, density, elongation and indentation test shows that as the isocyanate level increases, there is an increase in the density of the foam, i.e. its hardness, which invariably increases the number of cells (cell count), indentation load deflection value but decrease rate of compression and elongation.

Also it can be seen from the results that as the water level increases, the density of the resulting foam decreases, its cell count reduces from $1.74*10^3$ to $1.28*10^3$, indentation load deflection value falls, but there is an increase in the compression rate and the elongation rate.

The effects of TCFM 11 on the resulting properties of the foam is similar to that of water as it invariably reduces the density, indentation load deflection value, cell count but increases the compression and rate of elongation. The introduction of chlorine and inert filler (antimony) help reduces the flammability rate of the FPF, but resulted in little degree of scotch.

CONCLUSION

It can be concluded therefore, that an increase in the Isocyanate level results to an increase in the foam hardness, indentation load deflection, cell counts and lowers the compression and the elongation rate of the resulting FPF.

An increase in the water and TCFM 11 level brings about a decrease in the foam density, indentation load deflection value and cell counts but increases the compression and rate of elongation. The use of halogenated substances and inert fillers help reduce the flammability rate of the resulting foam, but care must be taken as it might result to scotching of the foam sample.

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