



# DEVELOPMENT OF BIO-BASED FOAM FROM *Ximenia americana* SEED OIL POLYOL: STUDIES ON THE EFFECT OF WATER LEVEL ON SOME PROPERTIES OF THE FOAM



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**Abstract:** In the formulation of biobased foam from *Ximenia americana* seed oil polyol, The tensile, mechanical and other physical properties of the biobased polyurethane (PU) foams with various concentrations of water from (0.04 to 1.8 ml) were characterized using physico-mechanical evaluations. These physical properties indicate that water level has various effect on the microstructures of biobased foam and affects the physical and mechanical properties of the biobased foam. These occurred because water is responsible for formation of gas in the isocyanate/polyol reaction it aids in the chain extension process of polyurethane resin. A decrease in the density and other mechanical properties occurred as water level increased from (0.04 to 1.8 ml), while the gel time cream time increased as the concentration of water increased. A low density biobased at (1.6 ml) concentration of water with good performance was prepared by optimizing the process.

**Keywords:** Biobased foam, polyol, density, mechanical properties

## Introduction

Bio-based renewable resources have gained little ground for use in the production of useful chemicals and new materials have grown so fast during the past few years. These potential advantages of these biobased materials motivated its use for the production of polyol (biobased) (Benecke *et al.*, 2008). This biobased polyol is environmentally compatible and economically feasible, as compared to the traditional petrochemical derivatives (Santibanez *et al.*, 2011). This polyhydroxyl compound called Polyol, is an important building block of polyurethanes and polyesters that are useful in wide range of applications such as construction, coatings agents, adhesives, sealants, elastomers, resins etc. Generally polyols are traditionally produced from petroleum. However, the production of polyols from petrochemicals is costly, requires a great deal of energy and also has adverse effects on the environment (Randall and Lee, 2002c). Recent investigations show that in recent years the focus from petrochemicals based polyols to alternative, non-petroleum based sources of polyols that are renewable, less costly and more ecofriendly (Chisti, 2007) became necessary. These biopolyols synthesized from vegetable oils proved to be an alternative for this purpose and has therefore drawn considerable current attention. Vegetable oils such as sunflower, canola, corn, olive, palm and soy oils are being explored for the synthesis of polyols. The vegetable oil molecules must however, be chemically transformed to introduce hydroxyl groups for formation of polyols (Ruppert *et al.*, 2008).

Isoocyanates and polyols are the two main parts of the chemical content in the synthesis of polyurethane foams. Blowing agents, catalysts, surfactant and other chemicals are also required to modify the processes and properties (Randall and Lee, 2002d). Water and methylene chloride- are sources of active hydrogen. These react with isocyanide to and cause the foam cells to rise by producing carbon dioxide (Randall and Lee, 2002a). The carbon dioxide produced from the water-isocyanate reaction acts as the principal source to blow the foam, other (physical and chemical) auxiliary blowing agents are also employed by some reaction involving (PU foams). These are (inert) low boiling solvents, are generally used to produce softer foams by reducing the foam density (Chia *et al.*, 2011). The forming reactions are exothermic with temperatures reaching up to 130°C approximately. This high temperature makes it possible to vaporize the low boiling solvents and provide supplementary gas to expand the foam. While maintaining water/isocyanate ratio, the physical blowing agent is added

which aid the formation of larger cells and opens the cells of the foam. All these processes also lead to an increase in the softness of the foam (Chen and Lu, 2009).

Until the early 1990's, the auxiliary blowing agent primarily used to were the liquids such as, ketones aliphatic, halogenated and aromatic hydrocarbons, alcohols, are volatile which all have low boiling points (Ramachandran *et al.*, 2011). But these fluids are chemically hazardous and are not environmentally friendly (Benecke *et al.*, 2008). However, water as a physical blowing agent for polymeric foams process can be very stable and produce foams with characteristics close to that of chemical blowing agents (Sow *et al.*, 2011).

$\text{RNCO} + \text{H}_2\text{O} \rightarrow \text{RNH}_2 + \text{CO}$  { reaction of isocyanate with water }

## Materials and Methods

### Materials

Toluene – diisocyanate, dimethyl ethyl amine, silicone oil, methylene chloride and stannous octate were obtained from Nima foam manufacturing company Yola, Nigeria, and were used as received. *X. americana* seed was obtained from Tongo (II), Song local government area of Adamawa State, Nigeria. Polyol was synthesized from *X. americana* seed oil. Materials used in the production of the foam are discussed in Table 1. Other ingredients used in the production of the foam include (Aniceto *et al.*, 2012):

- Water.
- Stannous octate.
- Silicone surfactant.
- TDI
- Biobased polyol
- Amine

With these materials, a standard formulation was designed as shown in Table 1.

**Table 1: Formulation for flexible polyurethane foam production (F stands for formulation)**

Materials	P <sub>1</sub>	P <sub>2</sub>	P <sub>3</sub>	P <sub>4</sub>	P <sub>5</sub>
TDI	2	4	6	10	12
Biobased polyol	8	10	12	14	15
Water	0.04	0.08	1.2	1.6	1.8
Silicon oil	0.01	0.02	0.03	0.04	0.05
Stannuous octate	0.01	0.02	0.06	0.09	1.0

The FTIR spectrum of polyol shows the broad band stretching at  $3402.54\text{ cm}^{-1}$  reveals the presence of  $\text{-OH}$  group,  $1718.63\text{ cm}^{-1}$  stretching is attributed to carbonyl units of ester linkage,  $2934.79\text{ cm}^{-1}$  corresponds to asymmetric stretching of  $\text{-CH}$  of adipic acid and hexanediol. These peaks are also present in the other seed oil-based polyol which confirms the authenticity of the vegetable polyol. The difference is the

presence of peak at  $2342.97\text{ cm}^{-1}$  which appears in the petroleum polyol spectrum; this indicates the presence of other  $\text{-CH}$  bonds in the commercial polyol (Fig. 1). Also more broad band in the *X. americana* polyol is probably due to the presence of  $\text{H}_2\text{O}$  in the sample (Asif *et al.*, 2004).

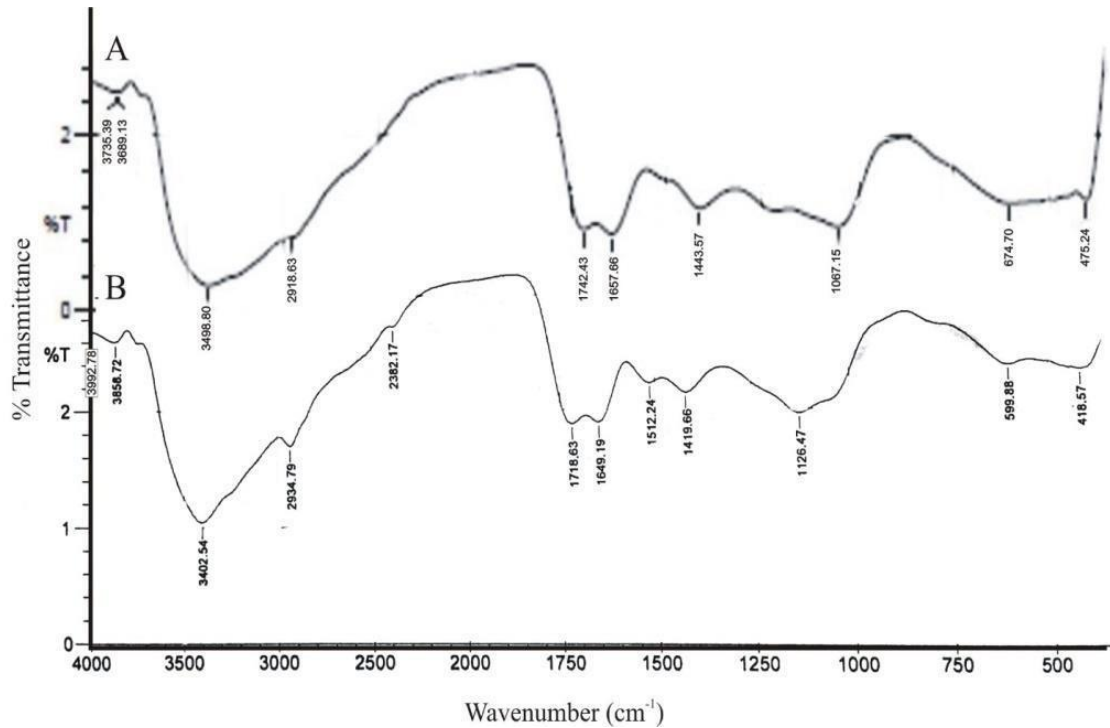


Fig. 1: FTIR spectra of (A) commercial polyester polyol and (B) polyol from *Ximenia americana* oil

**Effect of water concentration on the some physical properties of *X. americana* biobased Pu foam**

As can be seen from the Fig. 2, the density of the biobased foams increases initially with increase in water concentration (in-situ blowing agent). As more water is added, the foam density increases up to 8 ml of water inclusion after which it began to drop. This development can be explained as follows; at the initial stage of the graph, the cell size starts increasing normally but as more water is added there is a rapid increase in the cell size which due to the fast diffusion rate of  $\text{CO}_2$  -out of the foam (Aniceto *et al.*, 2012).

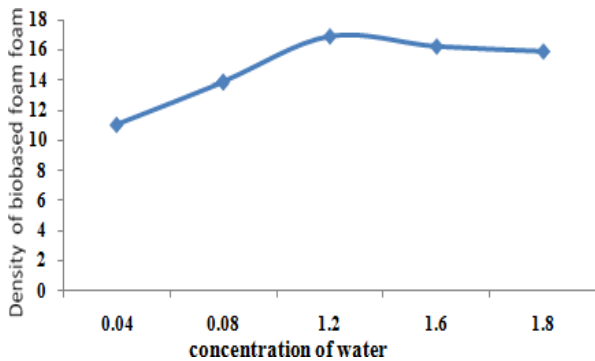


Fig 2: Effect of water amount on density of foam

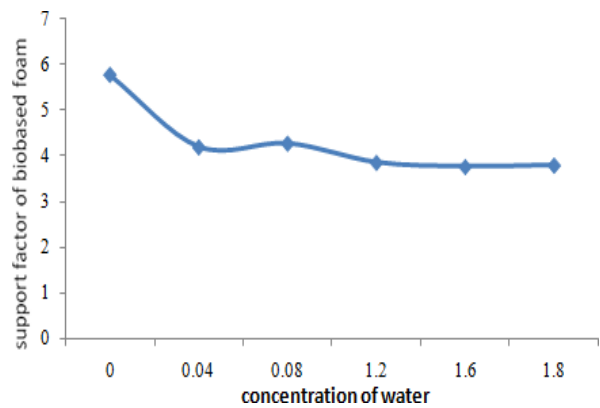


Fig. 3: Effect of water amount on support factor of foam

The Fig. 3 shows an initial rise in the support factor value followed by a decline in the value which remains seems to remain constant. These fluctuations may be caused by the rate at which water reacts with isocyanate creating a urea bond and a release of a lot of  $\text{CO}_2$  and heat being released in a chain reaction (Anand and Saxena, 2012). However, from the figure foam blocks with water concentration of 0.08 and 1.6 ml gave better results and fell within the ASTM D-3574 set standard range of 1-10% (NIS 295:1993) for compression tests of polyurethane foams, but the foam block with water concentration of 1.6 ml deformed after 72 h.

As can be seen from Fig. 4, the hardness of the biobased foams slightly increases with increase in the concentration of water. As more water is added, the foam hardness continues to

increase but drops slightly. This development can be explained as follows; at the initial stage of the graph, the bubble created during the reaction were firmer, giving the foam a denser nature with increasing hardness but with the latter increase in the concentration of water, the foam bubbles get weaker causing cracks and thereby leading to a decrease in its hardness. The increase in hardness noticed in the last area of the graph may be as a result of the increase in the molecular weight of the mixture (Alma and Shiraishi, 1998).

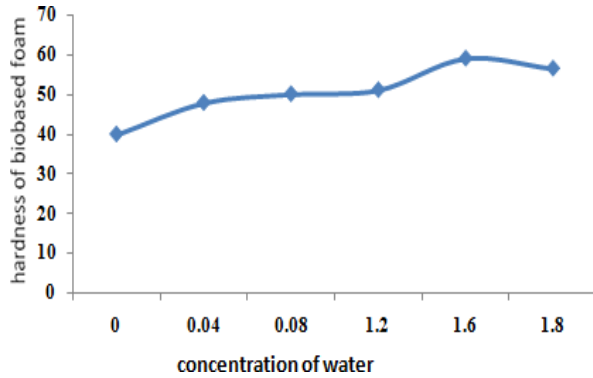


Fig. 4: Effect of water amount on hardness of foam

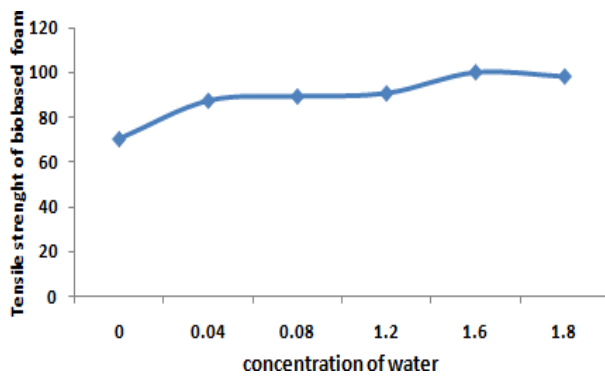


Fig. 5: Effect of water amount on tensile strength of foam

The Fig. 5 illustrates the tensile property of 6 different foam blocks. There was a little rise in the tensile strength then as the concentration of water increases a decrease in tensile property was recorded but with further increase of the concentration of water a little rise in the tensile strength is noticed. The initial rise may be due to the stirrer speed which creates turbulence leading to the creation of smaller size bubbles (giving rise to denser foam blocks) with good tensile properties. But as the concentration of water is increased and the stirrer speed remains constant, larger bubbles are created leading to a less dense foam production which automatically reduces the foam tensile property. With more increase in water concentration the tensile property increases due to TDI/water index ratio (Alhanash *et al.*, 2010).

As can be seen from the Fig. 6, the elongative property of the biobased foams increases initially with increase in water concentration (blowing agent). With the addition of more MC the density gradually decreases producing foam that has little or no cohesive properties. This initial behavior is due to the decreasing the gelling time of the mixture producing more cells per unit volume. The decrease in foam elongative property could be attributing to its curing time, the foam cured at a very slow rate creating very weak gas bubbles (Akiyama *et al.*, 2009).

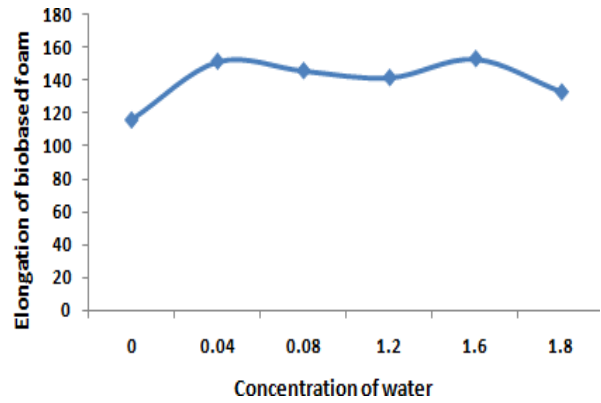


Fig. 6: Effect of water amount on elongation at break of foam

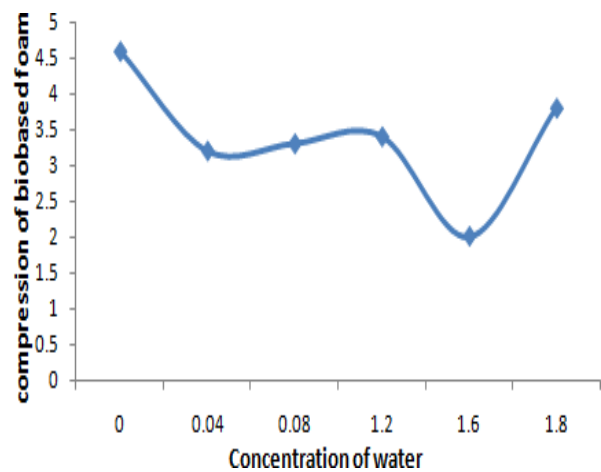


Fig. 7: Effect of water amount on the compression value of foam

The compressive value of the biobased foam increased slightly and then decreased to 0.08 ml with an increase in methylene chloride concentration (Fig. 7). The large cream time may be responsible since long cream times favor the growth of large bubbles, there by producing high compressive values. The drop of the compression value (Ahvazi *et al.*, 2011) may be due to a short cream time thereby making smaller bubbles which produces denser foams.

### Conclusion

Polyurethane foam was produced from X. Americana seed oil. Optimization of the production procedure was carried out by varying the water level. The foams produced showed water concentration of 1.6 mL with 14 ml of polyol reacting with 10 mL TDI gave a foam with optimum mechanical properties. Therefore these combination of (10 ml of TDI, 14 ml of biobased polyol, and 1.6 ml of water (H<sub>2</sub>O) respectively, may be most appropriate for the production of a flexible using polyurethane foam with optimum properties from X. Americana seed oil polyol. This study has therefore presented X. Americana seed oil as a feasible, cheap alternative source for the synthesis of polyol with good potentials as a feedstock in the foam industry. The study also presents this procedure that it may be adopted while using X. Americana seed oil polyol in the production of polyurethane foam.

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