

Foam Production Using Epoxidised Orange Seed Oil

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Abstract: The use of Epoxidised Orange Seed Oil (EOSO) as substitute for polyol in foam synthesis has been investigated. The presence of the tri-functional group, triol in orange seed oil presented a 3 dimensional structure which brought rigidity to the product. The result showed that the use of EOSO is not suitable for flexible foam production, but the use of 100% EOSO produced semi-rigid foam which can be used in other various applications. Also, it was discovered that formulation 2 which contained 25% of epoxidised orange seed oil was very close to the control formulation in terms of density ($20.59-24.70 \text{ Kg m}^{-3}$) and tensile stress (66.7 KN m^{-2}) compared to that for standard poly-urethane foam at (83.33 KN m^{-2}). However, its elongation at break point recorded as 144.44% is less than the standard value of 216.70%.

Key words: Epoxidised Orange Seed Oil (EOSO), Toluene-Diisocyanate (TDI), poly-urethane foam

INTRODUCTION

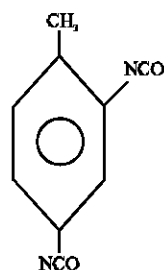
Production of flexible polyurethane foam in recent times has been characterized with importation of heavy and expensive chemicals from very distant and advanced countries of the world. This has posed a challenge to the industry and has made polyurethane foam expensive to afford. Over the years, seed has generally been regarded as a waste, except for a minute fraction that is used in Agriculture for planting. The natural existence of oil in seed and in varying proportion together with the application of the knowledge of seed oil extraction techniques suggests that vegetable oil could possibly be extracted and be used for industrial purposes (Olu-Arotiowa *et al.*, 2005; Beduacyk and Erickson, 1973).

Orange seed oil is obtained from the seed of a plant with the botanical name *Citrus cinensis*. The oil contains mainly esters of the type 12 hydroxyl 9 octadecanoic acid (Tsagli and Oldham, 1991). The presence of these hydroxyl groups makes the oil suitable for use in urethane type reactions, such as reactions between diisocyanates and hydroxyl-terminated compounds (Agra *et al.*, 1990). Also, the hydrogen bonding of these hydroxyl groups confer a high degree of viscosity on the oil (Oguniyi and Fakoyejo, 1998). Although this seed oil is not edible, it consists of 14% stearic acid, 20% oleic acid, 55% linoleic acid and 8% linolenic acid (Makanyjuola, 1999), the last three being unsaturated acids. Other seed oils such as soya oil and castor oil have been used in isocyanate

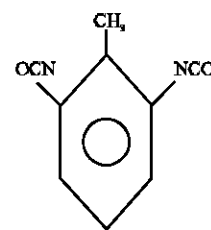
reactions to make polyurethane elastomers, millables, castables, adhesives and coatings (Aigbodia *et al.*, 1999). Also, the epoxidation of rubber seed oil at 200°C under reduced pressure has been used to obtain heptaldehyde and undecalonic acid (Vendenberg, 1975), while various treatments of the orange seed oil can be used to obtain sebacic acid and ω -amino undecanoic acid (Brenes *et al.*, 1992). This study investigated the suitability of using Epoxidised Orange Seed Oil (EOSO) as substitute for polyol in foam synthesis. Physical properties of some polyurethane foam prepared from Toluene Diisocyanate (TDI), a mixture of Epoxidised Orange Seed Oil (EOSO) and polyol were produced in various proportions and tested.

MATERIALS AND METHODS

Toluene diisocyanate, TDI (80:20 of 2,4 and 2,6 isomers, respectively),



2,4-Toluene di-isocyanate



2,6-Toluene di-isocyanate

Table 1: Properties of Epoxidised Orange Seed Oil (EOSO)

Properties	Experimental value	Literature data (cold pressed)	Literature data (Extracted)
Specific gravity (30°) (Kg m ⁻³)	0.86	0.96	0.96
Appearance	Clear	Clear	Clear
Refractive index (30°)	1.48	1.47	1.47
Colour	Yellow	Yellow	Yellow
Acid value	3.00	3.00	3.00
Hydroxy value	164.00	164.00	164.00
Iodine value	85.60	85.00	84.20
Water content %	0.56	0.48	0.47
Saponification value	180.00	182.00	177.00

Adapted from Oluarotowa *et al.* (2005) properties of EOSO

polyol, stannous octoate, dimethyl amino ethanol water in place of TCFM (Trichloro Fluoromethane) and silicone oil were supplied by Bode foam Nigeria limited Ibadan.

Preparation of orange seed oil: Orange seed was sun dried and the crushed seed oil was extracted with the use of cyclohexane at a temperature of 60-80°C in a Soxhlet extractor. The oil so obtained was then epoxidised and tested for colour, refractive index, specific iodine value, acid value, hydroxyl value, saponification value and water content as described previously (Olu-Arotiowa *et al.*, 2005; Ulrich and Reagan, 1978).

Foam preparation

Polyol/EOSO: Two hundred and eighty milliliter of water, silicone oil (a foaming agent), stannous octoate, dimethyl amino ethanol were mixed together. After which TDI was stirred in with an electric stirrer to ensure good dispersion of reagents and foam of good cell structure. The whole mixture was then poured into a mould. The foam samples produced were left to stand for 24 h before they were tested to ensure complete curing. The formulations used for preparing the foam were given in Table 1.

Mechanical properties: Polyurethane foam samples were tested for tensile strength and elongation at break using a J.J tensometer manufactured by New Brunswick Scientific Co., USA, at 30°C following the procedure of ASTM D 3489. A known volume of each sample was obtained and weighed to obtain its mass. Density was then calculated as mass per unit volume. The colour of each sample was observed by visual examination while naked flame was also applied to each sample to observe burning characteristic. Compression set measurement was obtained according to procedures of ASTM D 3574.

RESULTS AND DISCUSSION

The properties of Epoxidised Orange Seed Oil (EOSO) are shown in Table 2. The various polyurethane foam formulation and their respective properties are shown in Table 2 and 3.

Table 2: Categories of formulation used for Polyurethane foam synthesis

Chemicals	1	2	3	4	5
Polyol (mole%)	100	75	50	25	0
EOSO (mole %)	0	25	50	75	100
TDI (g)	46.2	50.6	56.4	61.0	66
Water (g)	3.7	3.7	3.7	3.7	3.7
Amine (mL)	0.25	0.25	0.25	0.25	0.25
Silicone oil (g)	1.0	1.0	1.0	1.0	1.0
Stannous octoate (mL)	0.25	0.25	0.25	0.25	0.25
Trichlorofluoro-methane (mL)	0.5	0.5	0.5	0.5	0.5

Table 3: Properties of synthesized foam samples

Properties	1	2	3	4	5
Density Kg m ⁻³	24.70	20.59	29.59	32.02	62.45
Tensile strength KN m ⁻²	83.33	66.67	88.24	100	152.78
Compression set %	5.20	24.0	30.2	38.5	47.0
Elongation at-break %	216.70	144.44	134.45	122.44	109.00
Colour (without figment)	White	Light yellow	Yellow	Deep yellow	Dark yellow

Formulation 1, a conventional polyurethane foam formulation made up of 100% polyol was used as the control. Formulation 5 contained 100% EOSO as a substitute for polyol while formulation 2-4 contained 25, 50 and 75% EOSO respectively. It was observed that in all the products obtained, the intensity of the yellow colour increased with increase in the quantity of EOSO used. However, in a separate experiment, where bleached EOSO was used, the foam produced was colourless.

The results as presented in Table 3 showed an increase in tensile strength with increase in EOSO quantity in foam formulation. In all the EOSO-containing formulation with the exception of formulation 2, the tensile strength were greater compared with the 100% polyol formulation.

The introduction of EOSO in formulations 2-5 led to a rapid increase in compression set. The increase in compression set observed in EOSO-formulated foam may be due to the cleavage of the rigid cross links formed between EOSO and Toluene Diisocyanate (TDI), which prevented substantial elastic recovery. This implies that foam formulations containing EOSO may not be suitable for cushioning purposes as recovery from elastic deformation will generally be poor. Among the five formulations, Formulation 2 had density that approached that of the control but the compression set of the control is far superior to all of the formulation tested. Considering that it was only Formulation 2 that has a density approaching that of the standard (i.e., 25 kg m⁻³ (9), 25% of EOSO may be used as polyol substitute in foam meant for cushioning.

The results showed a gradual decrease of the elongation-at-break with increase in quantity of EOSO used. This result may likewise be due to the rigid cross links produced between toluene diisocyanate and epoxidised orange seed oil. It is however, unlikely that the foams will be subjected to an elongation greater than

100% in any practical application. With the exception of formulation 2, the density of the foams showed an increase with increase in the quantity of EOSO.

It is to be noted that the properties of foam in formulation 5, where EOSO is 100%, compare reasonably with properties of semi-rigid foams (Ulrich and Reagan, 1978) which are types of foam used in thermal insulation, refrigeration, packaging and transportation. All the foam samples continued to burn when exposed to naked flame and they can be classified as flammable. However, a mono-hydroxyl compound can be used in place of polyol for further research work.

CONCLUSION

This investigation showed that foams synthesized from 100% EOSO and mixtures of EOSO and polyol do not compare favourably with the foam produced from 100% polyol, especially in percentage elongation at break-point. However, foams synthesized from 100% epoxidized orange seed oil compare favourably well with semi-rigid foams synthesized using polyol. More importantly, we can conclude that for semi-rigid foam synthesis, epoxidized orange seed oil can be used to substitute for polyol in polyurethane foam synthesis.

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