

Study of Epoxidation of *Allanblackia floribunda* Seed Oil for Production of Epoxy Resin

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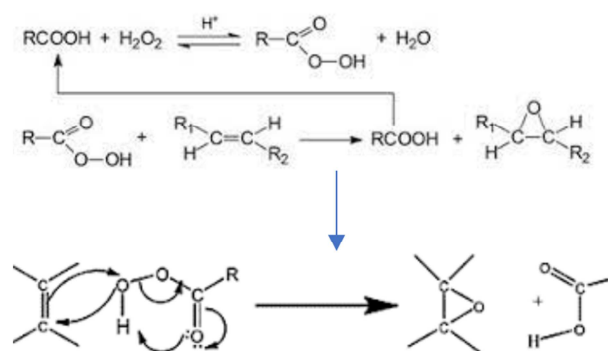
Abstract: Hot water floatation method was used to extract oil from *Allanblackia floribunda* seeds. The oil was epoxidized in-situ, using hydrogen peroxide and acetic acid; with sulphuric acid as a catalyst, to produce epoxy resin for plastic industries due to its high oxidative stability, so as to serve as an alternative to environmental toxic synthetic epoxy resins. The physiochemical parameters of the epoxidized oil (specific gravity (S. G), cloud point, pour point, density, iodine value, flash point) were measured using standard methods of analysis. The results obtained show that the epoxidized oil has a blackish brown colour, solid at room temperature, with density and viscosity of 0.7998 g/mL and 9.78 Cst, respectively, at 60°C. The flash point was above 100°C, iodine value was 16.2 gI₂/100 g, peroxide value was 0.65 Meq/g of sample, the acid value was 0.12 mg KOH of sample and the free fatty acid content was 0.06%. It was observed that at a minimal temperature of 60°C there was oxirane conversation. This was attributed to the low iodine, peroxide, free fatty acid values, and high flash point of the epoxidized oil compared to that of the un-epoxidized sample. This suggests that the oil can be used as stabilizer to reduce plastic degradation when exposed to sunlight and volatile compounds.

Keywords: Epoxidation, *Allanblackia Floribunda* Seed Oil, Epoxy Resins, Vegetable Oil, Oxirane, Plastic, Industry

1. Introduction

Availability, and the quest for ecofriendly materials have led researchers to seek for alternatives to petroleum-based products [1]. Renewable raw materials, such as: vegetable oils, sugar, starch, and cellulose are widely used in industries for various purposes. These materials have shown great potentials as alternatives to petroleum-based resins, and the interest in using them to produce bio-based resin keeps rising [2, 3]. Their use in bio-based applications is also attributed to their low cost, availability, low toxicity, and biodegradability [4].

The aim of this study was to produce epoxy resin from the oil extracted from the seeds of *Allanblackia floribunda*. Epoxidation is a process which converts the carbon-carbon double bond of oils into epoxides, otherwise known as oxiranes [5].



Scheme 1. Mechanism of Epoxidation of Oils [5].

The oxygen in the peroxide is electron deficient and is attacked by the electrons of the pi bond, through a concerted reaction between alkene and peroxyacid. The reaction is stereospecific: a cis-alkene produces a cis-epoxide and a trans alkene produces a trans-epoxide as seen in scheme 1

above. The peroxy-carboxylic acids formed are generally unstable. Epoxides are used as fumigants and antifreeze.

In Nigeria, many oil industries rely solely on importation of natural oils like castor oil and soybean oil (which are expensive) production of epoxides. Few readily available vegetable oils like coconut oil, groundnut oil and palm kernel oil are not enough because they are basically grown for consumption [6]. From research, oil from *Allanblackia floribunda* seed has little commercial worth in Nigeria presently and Nigeria has the ability of producing over 50 tons of *Allanblackia* oil [7]. The physicochemical properties of *Allanblackia* seed oil are similar to that of palm oil as presented by earlier researchers [8], hence the investigation of possible production of epoxides from this oil.

Allanblackia floribunda seed (also known as tallow tree seed), is of the family *Guttiferae* and genus *Allanblackia*. This tree grows in Nigeria, Ghana, Democratic Republic of Congo, Uganda and Tanzania in moist lowland and upland rainforest. It is an evergreen medium-sized tree, up to 30 metre tall. A matured tree produces about 100-150 fruits. The fruits contain 25-40% oil-rich brown seeds, with a seed containing 68-72% oil [9].

Table 1. Fatty acid profile for Refined *Allanblackia* Seed Oil.

| | |
|----------------------------------|-----------|
| Lauric acid (C12:0) | < 1% |
| Myristic acid (C14:0) | < 1% |
| Palmitic acid (C16:0) | < 2% |
| Palmitoleic acid (C16:1) | < 1% |
| Stearic acid (C18:0) | 45 – 58% |
| Oleic acid (C18:1) | 40 – 51% |
| Linoleic acid (C18:2) | < 1% |
| γ -Linolenic acid (C18:3) | < 1% |
| Arachidic acid (C20:0) | < 1% |
| Free fatty acids | max. 0.1% |

[10]

Table 2. Results of the physicochemical properties of *A. floribunda* seed oil.

| Characteristics | Values |
|--------------------------------------|---------------|
| State (25°C) | Solid |
| Colour | Golden yellow |
| Specific gravity | 0.805 |
| Density (60°C) in g/mL | 0.914 |
| Viscosity (60°C) in cSt | 32 |
| Cloud point (°C) | 25 |
| Flash point (°C) | 270 °C |
| Pour point (°C) | 25 |
| Iodine value (gI ₂ /100g) | 39 |
| Peroxide value (MEq/kg of sample) | 0.86 |
| Acid value (Mg KOH/g of sample) | 0.12 |
| Free fatty acid (%) | 1.15 |

[8]

2. Materials and Methods

The reagents used for this study were obtained from the Department of Chemistry, Rivers state University. They were all of analytical grade.

2.1. Sample Collection and Preparation

Allanblackia floribunda fruits were obtained from Okehi

in Etche local government area of Rivers state, Nigeria.

Extraction of oil

Hot water floatation method described by Rosenthal *et al.*, (1996) [11], followed with some modifications made by Alenyorege *et al.*, (2015) [12] and reported by Maduelosi *et al.*, (2019) [8] was used to extract oil from the seeds.

Sample purification

The sample was purified by washing the oil with hot water in the ratio of 1: 3, in a separatory funnel.

Epoxidation

Epoxidation of the oil was done by reacting the oil with peracetic formed by the reaction of acetic acid and hydrogen peroxide in ratio of 1: 4 according to the method given by Santacesaria *et al.* (2011) [13].

2.2. Characterization of Epoxidized

The epoxidized oil was characterized for density, specific gravity, flash point, cloud point, pour point, viscosity, iodine value, peroxide value, acid value and percentage free fatty acid according to AOCS (2012) official methods [14].

3. Results and Discussion

Table 3. Results of the physicochemical properties of the epoxidized oil.

| Characteristics | Values |
|--------------------------------------|----------------|
| State (25 °C) | Solid |
| Colour | Blackish brown |
| Specific gravity | 0.88 |
| Density (60 °C) in g/ml | 0.7998 |
| Viscosity (60 °C) in cSt | 36 |
| Cloud point (°C) | 42 |
| Flash point (°C) | Above 270 °C |
| Pour point (°C) | 34 |
| Iodine value (gI ₂ /100g) | 16.2 |
| Peroxide value (MEq/kg of sample) | 0.68 |
| Acid value (mg KOH/g) | 2.3 |
| Free fatty acid value (%) | 0.06 |

4. Discussion

The results of the analyzed physicochemical properties for both the un-epoxidized (crude) *Allanblackia* and the epoxidized oils (Colour, Specific gravity, Density, viscosity, Cloud point, Flash point, Pour point, iodine value, Peroxide value, total acid number, refractive index, acid value, and free fatty acid content) are presented in Tables 2 and 3. In terms of visual examination; the un-epoxidized sample had a clear liquid appearance, but the epoxidized oil formed a blackish brown semi-solid (slurry) product at room temperature. The change in colour could be due to the formation of oxirane in the oil. This is confirmed by the iodine value change. Similar report has been given by earlier researchers [15].

Specific gravity value of the epoxidised *Allanblackia* seed oil (ASO) was 0.88 as against 0.805 reported in the crude sample. Jalil *et al.* (2014) [16] in their studies performed on oleic acid-based palm oil reported that, as the epoxidation reaction progresses, the inclusion of oxygen in the fatty acid

chain increases the specific gravity value [10], indicating that a bulkier product has been formed.

The viscosity value of the epoxidized *Allanblackia* oil was higher than that of the un-epoxidized (from 32 Cst to 36 Cst). The change in viscosity is attributed to the change in the oil texture as the oil became semi solid. Viscosity increased as the molecular weight of the sample increased with the formation of epoxidized product.

Flash point of the epoxidized *Allanblackia* oil was higher than the crude sample (from 100 to 270°C). The high flash point of the epoxidized oil shows that the epoxidized *Allanblackia* seed oil can be used to make polymers for the storage of volatile compounds.

Result of iodine value of the epoxidized *Allanblackia* oil was lower than the value in the un-epoxidized sample; values were 39 and 16.2 gI₂/100g respectively. Iodine value tells degree of unsaturation of the fatty acids content of oil [16]. The lower iodine value of the epoxidized sample confirmed the formation of oxirane compound.

The acid value of the epoxidized oil was higher (2.3 mg KOH/g) than that of the un-epoxidized sample (0.12 mg KOH/g). This is attributed to the carboxylic group (peracid) added. Acid value is a chemical measurement that represents the acidity of the fatty acid. It is calculated from the mass of the base material used in neutralizing the acid. This is usually determined by the amount of potassium hydroxide (in mg) required to neutralize the acid in one gram of fatty acid as earlier reported [17].

The peroxide and free fatty acid values of the epoxidized oil were less than that of the crude sample as shown in Tables 2 and 3. The lower values suggest that the oil can be used in plastic industries as stabilizer to reduce plastic degradation when exposed to sunlight, because oil with low peroxide and free fatty acid values has high oxidative stability.

5. Conclusion

The values of physicochemical parameters of the epoxidized oil confirmed the production of epoxy resin. Underutilized seed oil, such as *Allanblackia* oil which is very cheap and readily available as compared with other edible vegetable oils should be employed in the industries to reduce cost.

Epoxidation of oils should be encouraged because the epoxide products obtained can be put into so many uses.

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