TEST Engineering & Management

May-June 2020
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Epoxidation of Different Oils –A Review

M. Rajamehala*, M. Thasneem Banu[1], M. Lavanya[2], G. Gowsalya[3], K.Gilbert Ross Rex[4]

[1]M. Thasneem Banu, Department of Biotechnology, Vivekanandha College of Engineering for women, Namakkal, 637 205, Tamilnadu, India.

[2]M. Lavanya, Department of Biotechnology, Vivekanandha College of Engineering for women, Namakkal, 637 205, Tamilnadu, India.

[3]G. Gowsalya, Department of Biotechnology, Vivekanandha College of Engineering for women, Namakkal, 637 205, Tamilnadu, India.

[4]K.Gilbert Ross Rex, Department of Biotechnology, Vivekanandha College of Engineering for women, Namakkal, 637 205, Tamilnadu, India.

Corresponding author : rajamehala@gmail.com, thasneem0611@gmail.com,

Article Info
Volume 83
Page Number: 10714 - 10721
Publication Issue:
May-June 2020

Article History
Article Received*: 19 November 2019*
Revised*: 27 January 2020*
Accepted*: 24 February 2020*
Publication*: 19 May 2020*

Abstract:
Epoxidation of jatropha oil, castor oil and soybean oil was demonstrated by using various methods. These methods were carried out to form epoxidised oil containing epoxy ring. Chemical method was performed through in-situ epoxidation process forming peroxyformic acid by using aqueous hydrogen peroxide and formic acid. The enzymatic method was done in the existence of Lipase enzyme from candida antartica in neutral ph. Epoxidation by acid ion exchange resin was performed by in situ reaction with catalyst, and in or without toluene. These methods were evaluated on different parameters such as reaction temperature, molar ratio, and stirring speed. The presence of epoxy group can be determined by Oxirane Oxygen Content. The percent of oxygen absorbed by an unsaturated raw material during epoxidation was analyzed by these OOC test. Epoxidised oils were characterized by FTIR and HNMR spectroscopies.

Keywords: *Epoxidation, chemical, enzyme, catalyst, epoxy ring, OOC test, FTIR and HNMR.*

INTRODUCTION

Plastics are more useful than metals, papers, and other materials because of their properties such as lightness, cheaper, durability. They are being used in industrial field worldwide [1]. Nowadays plastics and polymers are part of our life, similar in many ways to natural resins found in trees and plants and organic polymers which can be processed in different ways [2]. The rapid increase in plastic waste can cause problems on dumping capacity site for landfills and it is not biodegradable. Recycling of proportion of plastics were very low and toxic emissions such as CO_2, methane, was produced because of incineration. Green house gas affects worldwide negatively[3]. The characteristics include resistance to chemical reactions and enzymatic reactions of plastics such as polyethylene terephthalate. Plastic debris affects wildlife, human health and environment and climate change and resource restraints, for fossils, have affected impact on society[4].Bioplastics were derived from renewable resources and starch, protein, and cellulose, lignin, and animals such as casein, and lipids [5]. The most known bio-based plastic was PolylacticAcid (PLA) and Polyhydroxyalkonates (PHA)[6]. They werenatural biopolymers manufactured and oxidized by various organisms.It requires oxygen for degradation, the biodegradable plastics which onlydegrades by microorganisms from their natural action[7]. Environmental and social challenges have enraged many researchers to replace petrochemical-based polymers with biodegradable one [8].Nowadays,

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bioplastics have become a necessity in many industrial applications like food packaging, agriculture and horticulture, composting bags, and hygiene [9].

To increase film flexibility, plasticizers were used, while increasing molecular space the internal hydrogen bonding between polymer chains were reduced due to their potential capability. The most commonly used plasticizers in starch films werepolyols, such as isorbitol, glycerol and polyethylene glycol[7].The plasticizers were added to help and provide mobility to the plastics with less molecular weight and minimal volatility which increases the intermolecular forces.Proteins structure consists of three-dimensional networks conserved by inter-chain interactions but do not provide the material to be enhanced with adding liquid/solid substance in order to increase the properties of plasticity. The plasticizer get added to help to minimize the glass transition temperature and provide mobility to polymeric chains[10].Polyethylene and PVC are two major plastics from ethylene gas obtained by cracked naptha. Natural gas, obtained from oil fields, it is the major source of raw material for plastics called formaldehyde [2].The use of petroleum phthalate plasticizers for the creation of bioplastics were now widely replaced by the bio based plasticizers[11].Natural based plasticizers are made from sustainable and environmentally friendly resource s which decreases the use of traditional plastic goods [12].It was characterized by low toxicity and low migration level.This group includes epoxidized triglyceride vegetable oils from soybean oil,linseed oil,castor oil, sunflower oil. Traditional polymers were used for biodegradable thermoplastics such as poly(3-hydroxy butrate)(PHB) is not possible and replacement of synthetic plasticizers by natural based plasticizers is possible for some areas [13].

Jatropha seed is a good source of oil where jatropha seed consist of 40-60% oil,75%unsaturated fatty acid, oleic acid and linoleic acid. Due to less iodine number and lofty cetane concentration of mature jatropha seeds oil has an renewable energy source[14]. The Jatropha Oil composition was much alike to the other commercially available edible oils. However, the existence of some anti-nutritional components includes toxic phorbol esters group in the oil makes inadmissible for cooking use. Thus, it is preferred as the commencement for epoxide oil production. One of the most commercially used vegetable oil was Soybean oil containing 26% oleic acid, 7% Linolenic, 4% Stearic, 11% Palmitic and 52% linoleic acid, similar composition of jatropha oil[15]. Physicochemical properties of vegetable oils and fatty acid configuration was similar to those of soybean oil. A large proportion of these renewable resources remain underexploited the overall purpose of developing value-added products from locally available vegetable oils that are presently not used in food [16]. Castor oil (CO) was retrieved from *Ricinus communis* ,a low-cost vegetable oil. The Castor oil has protracted shelf life, less toxicity, easy accessibility and its incomparable functionality makes it dominant above other vegetable oils.It consists of 12-hydroxy-cis-9-octadecenoic acid (ricinoleic acid) which represents 90% of its fatty acid content which is considered as hydroxyl fatty acids, castor oil is compared to that of other vegetable oils, shows higher tensile and flexural properties than epoxidized soybean oil (ESO) [17].

EPOXIDATION

Epoxidation is the chemical reaction which converts the carbon-carbon double bond into oxiranes using a variety of reagents including air oxidation, hypochlorous acid, H_2O_2, organic peracid. There are different methods can be accomplished for epoxidation process depending on feedstock, oxidation reagent, catalyst, and solvent. Epoxy compounds were involved in large number of reactions to form different types of

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products such as glycols, alcohols, carbonyl compounds, polymers and other derivatives via the ring opening process [14].Epoxidation of vegetable oils acts as a vital raw material and become important oleochemicals for commercialisation. Besides using polyol as a starting material epoxy groups are now widely used as an alternative to petrochemical based plasticizers [18]. Epoxidised jatropha oil has superior oxidative stability than jatropha oil. They can be used as an intermediates for the manufacturing of polymer derivatives due to their high reactivity of oxirane ring [19].

Various methods of epoxidation are as follows:

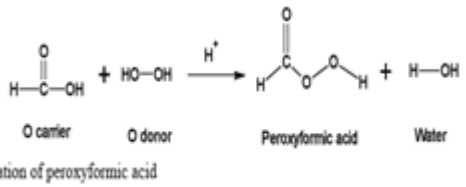
•Epoxidation by Conventional method

•Epoxidation using acid ion exchange resin (AIER)

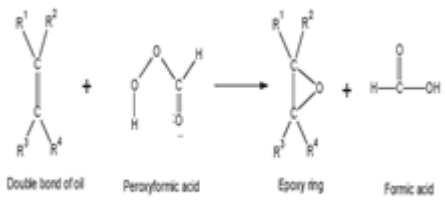
•Epoxidation using enzymes

•Epoxidation using metal catalyst

Epoxidised Jatropha Oil (EJO) consists of an epoxy ring which generated through an *in-situ* epoxidation process using H_2O_2 and HCOOH as an oxygen donor and oxygen carrier respectively. The formic acid acts as an oxygen carrier accepts the oxygen atoms from an oxygen donor, hydrogen peroxide which forms the peroxyformic acid. Then the erratic peroxyformic acid intervened the double bonds of the jatropha oil, leads to the ring formation, and the resulting epoxidised jatropha oil was produced [13].



Formation of peroxyformic acid



Formation of epoxy ring

Figure 1: Epoxidation process [13]

The organic peroxyacid was usually preferred for epoxidation because hydrogen peroxide has very limited solubility in oil. The organic peroxyacids has some handling problems since it is unstable and explosive. Therefore *insitu*peroxyacid is preferred. Various peroxyacids available in industry such as peroxyacetic acid, peroxyformic acid, peroxybenzoic acid, peroxyfluoroacetic acid, *m*–chloroperoxybenzoic acid and *m*–nitroperoxybenzoic acid. Among these peroxyacetic has been elected due to its easy availability, low price, and reasonable stability at high temperatures. Aqueous phase hydrogen peroxide was preferred for solubility in organic phase [14]. The another method for epoxidation was using acid ion exchange resin using homogeneous or heterogeneous catalyst. The acid ion exchange resin is an insoluble gel type catalyst in the form of tiny yellow type polymer beads. The peroxyacids interacts with the catalyst by entering through the pores. Amberlite IR-120 were used as a catalyst [20] and found to minimize side reactions which associated with con-ventional epoxidation [21].

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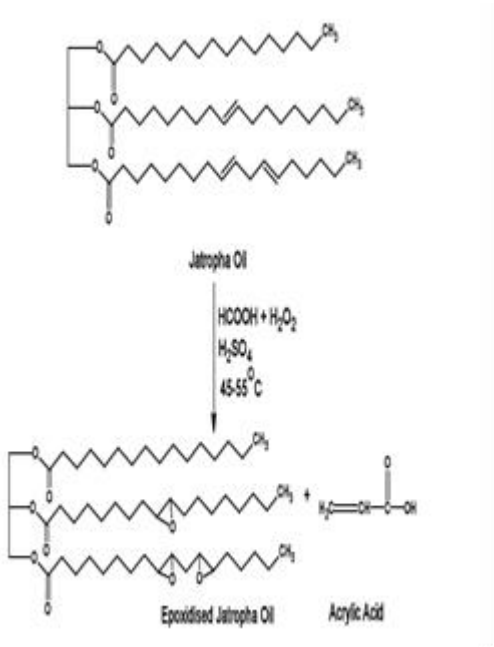

Figure 2: Production of Epoxidised Jatropha Oil [13]

Enzymatic method is a good alternative for chemical conventional method. Immobilized candida antartica lipase was used. Acid-freeand low temperature approach in enzymatic yield high oxiraneoxygen content in products and selectivity but require prolong reaction period [21]. It has certain limitations such as low solubility of lipase under the reaction conditions [20]. Enzymatic activity may also be lost due to elevated concentrations of hydrogen peroxide or to temperature effects [20]. In metal catalyst method various metals were used such as titanium, molybdenum, tungsten. Epoxidation of vegetable oil using methyl esters combined with the chemicals used in conventional method using a heterogeneous Ti/SiO2 catalyst [20].

OOC TEST

The significant properties in the characterisation epoxy oil is the determination of the oxirane oxygen content (OOC), in order to make sure the presence of epoxy compounds, epoxy resins, and epoxy plasticizers with increased speed and accuracy**.** Epoxy resins present in the oil with high OOC content are desired in the manufacturing of polymer. The OOC test was performed by using Hydrobromic acid solution in acetic acid and thetest was taken continuously once in an half an hour and the reaction was terminated after achieving a maximum value[13].

OOC (%) = (V-B) x N x 1.60 / W (1)
Where
V = volume of HBr solution in the sample
B = volume of HBr solution in blank
N = normality of HBr solution in acetic acid
W = weight of sample

METHODOLOGY

4.1. Epoxidation of Jatropha Oil

4.1.1. Chemical method:

Epoxidation of jatropha oil was carried out by conventional method. It was synthesised through insitu epoxidation reaction. The temperature was maintained at 55ºC,[13] at a stirring speed of 2500 rev/min to determine the kinetic control of the reaction[14]. EJO was prepared in a three-necked round bottom flask armed with thermometer, dropping funnel, and a mechanical stirrer with incessent stirring. The required amount of Jatropha oil was added into the flask and then pursued by calculated amount of formic acid at constant temperature with continuous stirring. Methyl ester can also be used as a catalyst with this mixture[22]. A calculated amount of hydrogen peroxide(30%) was addeddropwise into the reaction mixture until it reaches a dropping time of 30 minutes. The epoxidation process is exothermic. Precaution was taken to avoid overheating of the mixture.Further heating the mixture was cooled and filtered using anhydrous $MgSO_4$[13] [23]. Further chemicals are also used for filteration such as sodium bicarbonate[24], ethyl acetate[19], diethyl ether[14][25]. The epoxidation reaction was performed in a mechanically agitated contacter made of glass equipped with impeller and a reflux condenser.

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Keep the reactor in a water bath at (+1K). The above mentioned mixtures were placed in a reactor along with acid ion exchange resin called Amberlite IR-120.

4.1.2. Enzymatic method:

The enzymatic epoxidation was done in the existence of immobilized Lipase B from candida antartica. Epoxidation by lipase enzyme carried out in neutralph and has a high selectivity with no byproduct formation. Among all the lipases compared novozyme is the most excellent and effective enzyme for epoxidation. This method follows the same as the chemical method by using hydrogen peroxide but instead of formic acid lauric acid can be used and the immobilized lipase and toluene was also placed in the flask at 50^0C with rapid stirring of 900 rpm. The filteration was done by using sodium bicarbonate solution [26].

4.2. Epoxidation of castor oil

4.2.1. Chemical method:

Epoxidation of castor oil was carried out by using castor oil and formic acid (HCOOH) in a three necked round bottom flask[27]. Glacial acetic acid and H_2SO_4 have been used instead of formic acid[28]. Then stir the mixture by adding 30% hydrogen peroxide (H_2O_2) for 16 hours at room temperature, 30 to 40 mins at 55˚c [28] and 0 to 5˚c for 1hr[29]. Extract the crude product by using dichloromethane (DME). Similarly diethyl ether was used to extract the crude sample[28]. Wash the DCM extract by using separating funnel with distilled water and saturated sodium bicarbonate (Na_2Co_3) to remove the free acids. Then the organic layer was washed with sodium chloride solution and the DCM layer was dried over anhydrous sodium sulphate to evaporate the DCM. 11.2 gram of epoxidised castor oil containing 97% yield,[27] 89% yield containing OOC test 6.19% [28] and 10.38 gram containing 99.8% epoxidized castor oil [30].

4.3. Epoxidation of soybean oil

Epoxidation of soybean oil with H2O2 catalysed by heterogeneous catalyst using an amorphous Ti/SiO2 catalyst with the tert-butyl alcohol. The testedTi/SiO2 catalyst with dilute hydrogen peroxide (30%) of the solution .In first effect of temperature of soya bean oil reaction mole ratios of carbon double bonds C=C: H2O2 [31]. An excess amount of hydrogen peroxide was necessary in the reaction to achieve high reaction conversion and similarly 50 ºC± 2 and speed of 550 RPM with acetic acid and formic acid [32].An incorporation of Ti on an amorphous silica support produces oxidation catalysts that are highly effective in epoxidation reactions with hydrogen peroxide. The use of a H2O2 : C=C molar ratio of 1.1:1, slightly higher than the stoichiometric one, gives the highest reaction rate and yield [31].The epoxidation of soybean oil was prepared by chemo-enzymaticmethod was carried out by three necked round bottom flask placed in a water bath on top of a magnetic stirring hotplate with electric heating mantle. ThenNovozyme 435 Lipase B from Candida Antarctica added in the flask and stirred at 400 rpm with a polytetrafluoroethylene-coated magnetic stir bar. Lipase has high regio and stereoselectivity arrangement of constant hydroperoxides from fatty acid serious suppression of side reactions,and high conversion method [33].Hydrogen peroxide,oleic acid,acetic acid,anhydrous sodium sulfate identically and the rate of epoxide formation in soybean oil was greatly influenced by the concentration of lipase biocatalyst and their efficiency of the catalyst reduced sharply provided that the of catalyst was less than 4.0wt%. The temperature optimized by reaction temperature, molar ratio of H_2O_2 to ethylenic unsaturation, oleic acid concentration and solvent concentration [34].

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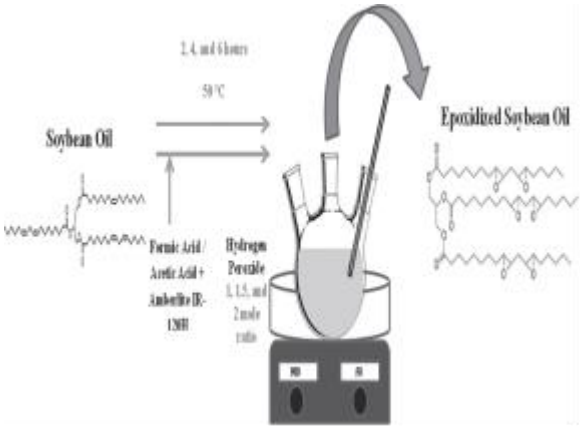

Figure 3: Schematic Representation for Synthesis of Epoxidised Soybean Oil [13]

CONCLUSION

The potential use of epoxidised oil has become the research hotspot and also be used in industrial applications. The most widely used epoxidised oil is soybean oil. But jatropha oil is a good option for the production of epoxide. The epoxy content were observed high on the jatropha oil with 4.99% produced by the conventional method. The epoxidation was confirmed through iodine value, FTIR analysis and NMR analysis.

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