

Pre-Treatment of Used Cooking Oil Followed by Transesterification Reaction in the Production of Used Cooking Oil-Based Polyol

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Received: 6 June 2021

Accepted: 13 July 2021

Online First: 31 August 2021

ABSTRACT

Used cooking oil has been considered as an economical and sustainable material that can be used widely as a starting material in the production of polymer precursors such as polyol for polyurethane. Since the composition of fatty acids and glyceride in the structure of used cooking oil remain the same as virgin vegetable oil, used cooking oil can be synthesized using the same method. However, there are certain physicochemical modifications to the oil properties that arise during the process of oil fryings such as increases in viscosity, acid value, and color changes that will affect the conversion of used cooking oil into bio-based polyol. Thus, various pretreatment methods that can be applied to used cooking oil such as adsorption, chemical bleaching, and treatment with solvents will be reviewed in this paper. Transesterification of used cooking oil with alcohol in the presence of catalyst will produce used cooking oil-based polyol which will have two or more hydroxyl groups per molecule. The formation of polyol can be confirmed with the formation of O-H peak in the FTIR spectrum during the FTIR spectroscopy analysis. This paper will also discuss the type of alcohol and catalyst used in the transesterification reaction. Used cooking oil-based polyol obtained from transesterification reaction has been reported to be comparable to the commercial polyol.

Keywords: used cooking oil, transesterification, polyol, polyurethane, chemical modifications



INTRODUCTION

Polyols are materials that can be used in the production of polymer precursors such as polyurethane. Most of the polyols are made from petroleum that is known as non-renewable energy resource. The refining process of petroleum can give an impact to the atmosphere and it can be dangerous to human health and the ecosystem. Apart from petroleum, polyols can be derived from natural resources such as vegetable oils, animal fats, fish oils, and carbohydrates.

Vegetable oils are often used in the production of bio-based polyols. However, vegetable oils could not be used directly to yield polyol due to the absence of hydroxyl groups in the vegetable oil composition. With some chemical modification to the structure of vegetable oil, hydroxyl groups could be introduced in the triglyceride structure of vegetable oil. Double bond and ester bond in fatty acid chains act as an active site during the chemical modification. For instance, epoxidation and ring-opening reactions take place in the double bonds of the unsaturated fatty acid chain, while transesterification and transamidation make use of the ester functional groups in the fatty acid chains.

Used cooking oils have been found as an alternative to vegetable oils in the production of polyols since the price of vegetable oils is quite expensive, and most of them is edible oils where the synthesis of polyols will have to compete with the production of food [1]. Besides, used cooking oils can give bad impact to the environment if it is not handled properly. Moreover, the consumption of used cooking oils that have been deteriorated with each use of frying can affect humans negatively. However, some physicochemical properties of used cooking oils are affected, and it can lead to difficulty in further chemical modification. Thus, pre-treatment of used cooking oils is needed to overcome this problem.

This paper will review the composition of vegetable oils and used cooking oils. The properties of bio-based polyols and used cooking oil-based polyols from various synthesis methods will also be reviewed. Moreover, several pretreatment methods on used cooking oils will be reviewed along with the characterisation of polyols obtained from the transesterification reaction of used cooking oils.

COMPOSITION OF VEGETABLE OILS

Vegetable oils have been used extensively in the production of polymeric materials such as polyurethanes, epoxies, polyesters, and biofuel because of its wide availability. It is mainly extracted from the seeds of fruits of various types of mostly edible plants. In brief, vegetable oils contain a glycerol backbone that is esterified by three different types of fatty acids.

Pfister *et al.* [2] stated that the composition of fatty acid in the vegetable oil is used to differentiate the type of vegetable oils. Soybean oil contains mainly linoleic and oleic acids [3], linseed oil contains stearic, palmitic, linoleic, oleic, and linolenic acids [4], rapeseed oil contains linoleic, palmitic, oleic, stearic, and linolenic acids [5,6]. According to Kong *et al.* [7], canola and sunflower oils contain a similar compositions of fatty acids with different proportions which are oleic acid, linoleic acid, and linolenic acid.

Fatty acids in the triglycerides may be saturated such as palmitic and stearic acids or unsaturated such as oleic, linoleic, linolenic, and ricinoleic acids [4]. Determination of iodine value can give information on the degree of unsaturation contains in the structure of oil thus, the structure of vegetable oils can be determined.

Vegetable oils need to be chemically modified due to the lack of hydroxyl groups in the structure of vegetable oils [8]. The bio-based polyol can be obtained from vegetable oils through the insertion of hydroxyl groups in the active sited of ester bond and a double bond in the structure of the triglycerides of vegetable oils [9,10]. Vegetable oils must undergo several chemical modifications so that they will result in compounds that contain at least two hydroxyl groups in the backbone.

COMPOSITION OF USED COOKING OILS

Used cooking oils are mainly from edible vegetable oils such as corn oil, coconut oil, rapeseed oil, palm oil, and sunflower oil. Palm oil has a higher melting point than other cooking oils which make it suitable to be used for cooking and frying at high temperature. The structure of used cooking oils is consequently determine by the origin of the oils with the relevant physicochemical properties [11].

Kurańska *et al.* [12] has been analysed the chemical structure of a few samples of used cooking oils with virgin vegetable oil as a reference by using FTIR analysis and iodine value determination. The variation in the result of FTIR analysis between used cooking oils and virgin vegetable oil is low where all the tested samples show the presence of ester bond and carbon-carbon double bond at the peak of 1750 cm^{-1} and 3010 cm^{-1} respectively. Meanwhile, the iodine value of used cooking oils has been reported lower than the reference oil by 6-12% which indicates oxidization of the oils [12]. The triglyceride structure of used cooking oils is unaffected by the degradation due to the frying and cooking activity.

Furthermore, Rincón *et al.* [13] stated in the study that used cooking oils are highly heterogeneous where it indicates diverse physicochemical properties while having low variability in the density, iodine value, and saponification value. Low variability in the density, iodine value, and saponification value denote the equivalent structure of used cooking oils to the virgin vegetable oil. Moreno *et al.* [14] also reported low variability of iodine value and density between used cooking oil and virgin vegetable oil.

LIMITATIONS ON THE PROPERTIES OF USED COOKING OILS

The side effect of degradation on the vegetable oils from cooking and frying activities make it dangerous for a human to be consumed again. Hence, used cooking oils need to be disposed of due to the oxidation, hydrolysis, and polymerization that occurred [15]. However, the disposal of used cooking oils must be done properly to avoid further difficulties such as blockages in the piping system, corrosion of metal and concrete that can occur when the oils are solidified. Used cooking oils have been used widely in the production of polymeric materials to avoid such problems from occurring and reducing the environmental pollution.

Previous studies have proven that the chemical composition of used cooking oils remains unaffected with the degradation of the oils thus, the synthesis can be done in similar ways to yield hydroxyl group in the virgin vegetable oils. During the process of frying and cooking, the composition of fatty acid in the oils decomposed where the decomposition depends on

the temperature used during frying and cooking, types of oil, frying cycles, and times [16,17].

Furthermore, used cooking oils exhibit large variations in physicochemical and visual properties such as appearance, odor, and color that will eventually affect the conversion process of used cooking oils into polyols [18]. Rincón *et al.* [13] has characterised physicochemical analysis of used cooking oils by the measurement of acid value, density, iodine value, saponification value, volatile contents, peroxide value, photometric color index, and polar compound contents. According to Dijkstra [19], the acid value is related to the content of free fatty acids while saponification value is related to the content of free fatty acids and ester bonds in vegetable oils.

Used cooking oils have a higher acid value than reference oil [14]. The difference value of acid is caused by the usage of oils during frying and cooking activities and it also resulted from the inadequate storage or handling and low quality of oils. In addition, significant variance in the value of peroxide, color, volatile matter, and total polar compounds in the used cooking oils have been reported as well [13].

PRE-TREATMENT OF USED COOKING OILS

A few studies have been carried out the synthesis of the polyol from used cooking oils previously without further purification of the oils due to the high cost of pre-treatments while some simply remove contaminants in the oils by filtration. Kurańska *et al.* [20] stated in their study that neutralisation of used cooking oils is often necessary due to the free fatty acids contained in the molecular structure of oils that will lead to slow down the process of transesterification. This is due to the deactivation of catalysts used in the transesterification reaction when there is the presence of moisture, hydroxyl peroxides, and fatty acids in the used cooking oils [21]. Neutralisation of used cooking oils can be done by conducting pre-treatments such as adsorption, chemical bleaching, or treatment with solvent.

ADSORPTION WITH AGRICULTURAL WASTE ACTIVATED CARBON

The adsorption of used cooking oils with activated carbon can reduce the value of acid in the used cooking oils effectively. As stated by Cárdenas *et al.* [11], the adsorption of adsorption agents may take place on the surface of adsorbent material or stearic hindrance within the porous network of the molecular structure of material either by chemical or physical sorption. Activated carbon is one of the adsorption agents that is used widely apart from ion exchange resin, clays, silicates, and aluminum silicates.

It is necessary to purify used cooking oils before conducting synthesis to yield polyols thus, any contamination of residue can be avoided. The content of free fatty acids due to the degradation of oils during frying and cooking activity can also be reduced. Used cooking oils can be purified by using low-cost activated carbon, such as agricultural waste [22,23]. The example of agricultural waste activated carbon that can be used as the agent of adsorption is coconut husk, rice husk, and sugarcane bagasse. The organic structure of used cooking oils is not influenced by the adsorption of agricultural waste-activated carbon [23].

The viscosity value of adsorbed used cooking oils can be influenced by the types of activated carbon used. Based on the research, coconut husk-activated carbon has an adsorption ability of 8% [22] while, sugarcane-bagasse-activated carbon has the adsorption ability of 32% [23]. The processability of polyols can be increased with the decreasing value of viscosity [23]. Furthermore, the value of acidity in the adsorbed oils with coconut husk and sugarcane bagasse activated carbon is reduced by 68% and 83% respectively [22,23]. The adsorption of agricultural waste-activated carbon could reduce the viscosity and acid value of used cooking oils effectively.

CHEMICAL BLEACHING

Chemical bleaching may be required in the production of light-colored precursors from used cooking oils. Rinçon *et al.* [24] stated that the dark color of used cooking oils can be improved by the process of bleaching.

Hydrogen peroxide may be used in chemical bleaching and it is more efficient than bleaching earth treatment where the colour can be improved with a short processing time and low loads of hydrogen peroxide [24]. The cost of this treatment is slightly higher, but it is acceptable when simultaneous reactions such as transesterification and epoxidation is carried out later [11]. Cárdenas *et al.* [11] mentioned that adsorption and bleaching treatments can remove larger polar molecules present in the used cooking oils effectively. Polar compounds with high molecular weight are the result of extensive use of cooking oils at high temperatures which will affect in odour changes of used cooking oils [11].

TREATMENT WITH SOLVENT

According to the method stated by Rinón *et al.* [24], low molecular weight alcohol may be used in the liquid-liquid extraction so that it is possible to reduce the incompatibility between oils and solvent. Concerning the prevention of solvent loss and high cost of recovery, it is essential to ensure the mutual immiscibility between the solvent and used cooking oils [11]. Cárdenas *et al.* [11] stated that the variation in solute solubility between used cooking oils and solvent influences the separation thus, lead to the neutralisation of used cooking oils. The example of low molecular weight alcohols that may be used in this treatment is methanol, ethanol, and isopropanol. Due to the non-polar behaviour of the colour composition in the used cooking oils, this treatment is only effective in removing polar compounds and reducing the value of acid.

Aside from the low molecular weight of alcohol, water can also be used in the treatment of used cooking oils with solvent due to the immiscibility of water to oils [16]. This treatment is also known as degumming where the soluble contaminants contained in the oils are being carried out by hydrating it with the water [24]. Previous research has been conducted the degumming treatment with variations in the temperature and proportions of the water and it can be observed that the use of high temperature of water at high loading will result in greater reduction of viscosity in the used cooking oils which indicates that soluble contaminants are successfully removed from used cooking oils.

SYNTHESIS OF BIO-BASED POLYOL

Variou methods have been proposed by researchers in the production of polyols from vegetable oils. For example, transesterification, epoxidation and ring-opening, hydroxylation, and ozonolysis [25]. Based on previous studies, the synthesis of bio-based polyols can be done through a single step, two steps, and three steps reactions. For instance, research from Yusuf *et al.* [26] practice a single step reaction where polyol has been yielded from castor oil through glycerolysis while, two-step reactions where polyol is obtained from soy oil through epoxidation followed by hydroxylation have been reported [27]. Three steps reaction has been reported by Kong *et al.* [7] where epoxidation, ring-opening, and transesterification reaction have been conducted in the synthesis of five different types of vegetable oils. The types of reaction used to yield polyol from used cooking oils could influence the structure and properties of the resulting polyols. A suitable reaction needs to be chosen carefully to produce polyol with excellent properties.

BIO-BASED POLYOL

Previous studies have successfully obtained bio-based polyol from various types of vegetable oils where some of the vegetable oils are not edible, such as camelina oil. Non-edible oils have to compete for less with the consumption of food than edible oils in the production of polymer precursors.

The Hydroxyl number of polyols is measured to determine the functionality and molecular weight of the polyol. The properties of bio-based polyols based on previous studies are listed in Table 1. It can be found that different approaches in synthesizing rapeseed oil will result in the bio-based polyol with different properties. As mentioned earlier, Kong *et al.* [7] has reported similar composition of fatty acids from the structure of sunflower oil and canola oil but with different proportion. However, polyol obtained from sunflower oil has a higher hydroxyl value than polyol obtained from canola oil despite having an equivalent degree of unsaturation and undergo similar processing conditions. The proportion of fatty acids in the structure of the triglycerides can influence the properties of bio-based polyol produced.

Table 1: The Properties of Bio-Based Polyol

Type of oils	Type of synthesis	Hydroxyl value (mg KOH/g)	Viscosity (mPa-s)	References
Rapeseed oil	Transesterification	363	160	[28]
		307	154	[29]
	Transamidation	387	1035	
	Epoxidation and ring-opening	230	5340	
	Epoxidation and ring-opening with Diethanolamine	471	22 000	[9]
	Epoxidation and ring-opening with Triethanolamine	430	1800	
	Epoxidation and ring-opening with Diethyleneglycol	242	6500	
Palm oil	Transesterification	140	>245	[30]
Soybean oil	Epoxidation and ring-opening	358.51	1920	[27]
Canola oil	Epoxidation, ring-opening, and transesterification	259	2400	[7]
Sunflower oil		286	3300	
Camelina oil		272	4700	

Kirpluks *et al.* [9] stated that rapeseed oil polyol produced from the epoxidation and ring-opening reaction with further addition of diethanolamine to carry out transesterification has the highest viscosity with the range of 22 000 to 23 400 mPa-s but it is still in the standard polyol range that can be used in the industry especially to produce polyols with high functionality and branched structure. Hydrogen bonding of polar amide groups present in the chemical structure of polyol has led to the increasing

value of viscosity [6]. Despite the acceptable range value of viscosity, the ease of polyol synthesis will be depending on the viscosity value of the precursor where low viscosity of polyol will yield polymer with high molecular weight and crosslinked network [28].

Researchers are developing polyols from natural raw materials to replace petroleum which are non-renewable resources either partially or entirely. When a high amount of bio-based polyol is used, it will result in low reactivity during the production of polyurethane foam due to the existence of less reactive secondary hydroxyl groups in the structure resulting from the epoxidation and ring-opening reaction [31,32]. However, Marcovich *et al.* [32] stated that the increasing amount of palm oil-based polyol in the modified semi-rigid foams of polyurethane provides excellent dimensional stability. In addition, research from Mizera and Ryszkowska [3] stated that the replacement of petroleum-based polyol with soy-based polyol that varies in the chemical constitution resulted in a material with properties that can be controlled. It can be concluded that polyol can be derived from natural raw materials successfully and it can replace the use of petroleum polyol in the production of polyurethane.

USED COOKING OIL-BASED POLYOL

Recent studies have successfully developed polyols from used cooking oil via the process of transesterification, epoxidation, and epoxidation followed by oxirane ring-opening. Kurańska *et al.* [20] stated that vegetable oils can be synthesis directly into polyol since the iodine value of used cooking oils and virgin vegetable oils are similar. Iodine value is used in the determination of double bonds contains in the structure of vegetable oils. In addition, Mohd Tahir *et al.* [23] stated that used cooking oil-based polyol can be synthesized in similar ways as bio-based polyol due to the same organic structure between used cooking oils and vegetable oils. The properties of used cooking oil-based polyols based on previous studies are listed in Table 2.

Table 2: The Properties of Used Cooking Oil-Based Polyol

Type of synthesis	Hydroxyl value (mg KOH/g)	Viscosity (mPa-s)	References
Transesterification	148.79	162.5	[23]
	46.3	155	[22]
Transesterification with Triethanolamine	348	182	[8]
Transesterification with Diethyleneglycol	277	56	
Epoxidation with Sulfuric Acid	174.5	193 100	[20]
Epoxidation with Phosphoric Acid	4.9	100	
Epoxidation with Amberlite resin	1.6	149	
Epoxidation and ring-opening	295	15 306	[34]
Epoxidation and ring-opening with Fluoroboric Acid	139.6	3275	[35]
Epoxidation and ring-opening with Sulfuric Acid	159.2	961	

Used cooking oil-based polyols obtained from epoxidation and ring-opening reaction yield a higher value of viscosity with a lower hydroxyl value. Epoxidation and ring-opening reaction modified double bonds in the fatty acid chain while, transesterification reaction modified ester bonds in the triglycerides structure where sometimes, the glycerol backbone is fully removed from the structure thus, reduce its viscosity [36]. The properties of used cooking oil can be influenced by the type of reaction and materials used to yield hydroxyl group.

TRANSESTERIFICATION REACTION

Transesterification is also called alcoholysis due to the de-esterifying act on one of the fatty acid chains of the glycerol with alcohol along with acids, bases, or enzymes as catalysts. Transesterification reactions take place in the ester moieties and sometimes, the glycerol backbone can be removed during the reaction. The removal of the glycerol backbone will yield polyol with low molecular weight and viscosity [36]. The lower viscosity of polyols is necessary to ease the production of polymeric material based on polyols.

Alcohol is used in the transesterification reaction to modify the ester chain by introducing a hydroxyl group in the backbone of triglyceride. Glycerol is often used in the transesterification reaction besides ethanol and methanol. The high amount of monoglyceride can be obtained from the transesterification reaction of glycerol with triglycerides [37]. Diethanolamine and Diethyleneglycol have been used as transesterification agent [8]. Another compound such as a combination of Diethanolamine and Monoethyleneglycol along with Potassium Acetate catalyst in the transesterification reaction has been reported [22,23].

CHARACTERISATION OF POLYOLS

The synthesis of used cooking oil-based polyol via transesterification can be considered successful based on the O-H peaks that can be observed from Fourier-transform infrared spectroscopy analysis (FTIR). Enderus and Tahir [22] has been reported that polyester polyol is successfully derived from transesterification reaction of used cooking oils based on the hydroxyl group peak at a wavenumber of 3303.13 cm^{-1} and carbonyl stretching of ester group at 1743.81 cm^{-1} obtained from the FTIR spectroscopy analysis. Kuranska *et al.* [8] also reported a broad peak of the hydroxyl group at wavenumber ranges from 3340 cm^{-1} to 3390 cm^{-1} from the spectrum of FTIR analysis which indicates the presence of hydroxyl groups in the molecular structure.

The formation of polyols can also be observed through the Gel Permeation Chromatography analysis (GPC). Kuranska *et al.* [8] has reported the changes of GPC chromatogram of used cooking oil after being transesterified based on the peaks of diglycerides and monoglycerides at the

minute of 27 and 29. Triglycerides are generally converted into diglycerides and monoglycerides after undergoing a transesterification reaction.

CONCLUSION

In summary, polyols with low viscosity and high hydroxyl value can be obtained successfully through the transesterification reaction that takes place in the ester linkage of fatty acids in the triglycerides structure. Although the structure of used cooking oils remains the same as its origin, the physicochemical properties of used cooking oils such as acid value and viscosity have been affected by the activity of frying and cooking. It is important to reduce the high acid value in the used cooking oils as it could influence the reactivity of transesterification reaction by retarding the catalyst efficiency. Thus, pre-treatment of used cooking oils will be necessary to reduce the acid value and the viscosity for the ease of further reaction in the production of polymeric materials especially polyurethane. The polyol obtained from the transesterification reaction can be observed by FTIR analysis and also GPC analysis. O-H peaks at wavenumber in the range of 3300 cm^{-1} to 3390 cm^{-1} indicate that polyol is successfully derived from the used cooking oils.

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