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Full Length Research Paper

Assessment of saponification as a simple method for the preparation of glycerol

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Gycerol is very important industrially as it is used in the manufacturing of a wide array of commercially useful products such as confectionery, beverages, soaps and cosmetics, paints, lubricants, dynamite, etc. In this work, alkali-hydrolysis of groundnut oil (saponification) was employed to prepare glycerol which was then subsequently characterized. The results obtained show that the parameters of the glycerol compare favourably with pure glycerol in the literature. The method is recommended because it is simple, cheap and gives rise to product that can easily be separated out clean.

Keywords: Alkali-catalysed hydrolysis; Glycerol; Groundnut oil; Saponification.

INTRODUCTION

Glycerol is a clear, water-white, viscous, hygroscopic liquid with sweet taste at ordinary temperature. The term 'glycerine' normally used interchangeably with glycerol actually refers to the purified commercial product containing about 95 percent glycerol. Pure glycerol has so many important uses, which include applications such as in food products (to sucrage liqueurs), cosmetics (as moisturizing factor), textile industry, in pharmaceuticals, cellulosic industries. Other uses are in the preparation of nitrocellulose, as supplement in fodder for pigs, swine, and hogs, antifreeze in automobile radiators and lubricant in watches, among others (Pagliaro *et al*., 2007; Wang *et al*., 2001). Still, research is ongoing aimed at extending its uses and new derivatives (Johnson & Taconi, 2007; Behr *et al*., 2008; Pagliaro & Rossi, 2008).

There are several ways for preparation of glycerol. Glycerol could be prepared on a large scale synthetically from propylene, a by-product of crude oil refining. Since this synthetic method relies on crude oil, which is a finite and non-renewable resource, the method is losing popularity. Moreover, the petrochemical processes for obtaining the propylene from crude oil are tedious, expensive and fraught with myriad of pollution problems. Glycerol is also produced by microbial fermentation of lipids (Wang *et al*., 2001) but the process, apart from being slow, leads to yield that is quite small to meet with industrial demands. Glycerol is further obtained as a major by-product in the manufacture of biodiesel from fatty oils. This is usually termed raw or crude glycerol. Presently, the most often used biodiesel fuels are vegetable oil fatty acid methyl or ethyl esters produced by transesterification (Andre *et al*., 2010; Sendzikiene *et al*., 2007). For every three mol of ethyl esters, one mol of crude glycerol is produced, which is equivalent to approximately 10 wt. % of the total biodiesel production (Karinen and Krause, 2006; Pagliaro *et al*., 2009; Rahmat *et al*., 2010). However, glycerol produced through the biodiesel process is strictly limited because of presence of pollutant substances which

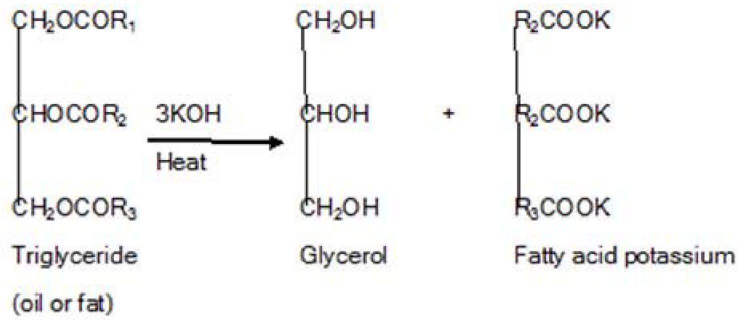

Figure 1. Potassium hydroxide catalyzed hydrolysis of oil

include spent catalysts, residual methanol, mineral salts, heavy metals, mono- and diacylglycerols, free fatty acids and soaps (Dasari, 2007). Efforts by biodiesel producers to purify this raw glycerol have involved complex installations, making use of processes of filteration, chemical steps, and vacuum distillation. Unfortunately, such installations are too expensive for small and medium production plants. They are, thus, out of reach of an investor with a small capital.

Due to its expanding applications, it is hoped that the demand for glycerol will continue to be on the increase, which makes the need to explore a simple, less costly, easily manageable and straight forward method for production of glycerol compelling. In this regards, the use of alkali catalysed hydrolysis of fats or oils (Figure 1), simply referred to as saponification, as a method of production of glycerol presents a win-win situation. The advantages of this method are two-fold: Firstly, the reactions are one-way rather than reversible. Secondly, the products are easier to separate. For example, the soap produced in this process can be easily salted out and separated from the lye (glycerol/water mixture). In this paper, the production of glycerol by saponification of groundnut oil is reported. Groundnut oil is easy to extract from the seeds and the plant which provides the source is cultivated in large quantities commercially in Nigeria.

MATERIALS AND METHODS

Materials: Crude groundnut oil used was purchased from a local market in Minna, Niger State, Nigeria. Analytical grade potassium hydroxide (KOH) was used for the saponification process.

Degumming of the crude oil: The essence of the degumming is to remove natural gums and phosphatides present in the oil. If left in the oil, gums give rise to substantial oil losses during alkali refining (Ibemesi, 1992). It was done by measuring some quantity (100 cm^3) of groundnut oil into a 500 cm^3 capacity separating funnel containing concentrated phosphoric acid (0.2 cm^3). After a swirling, the mixture was then washed with hot water several times. The gums and water separated out at the bottom of the separating funnel and is removed. The process of washing with hot water was continued until all the gums were removed and the effluent water tasted neutral to litmus as indication of absence of phosphoric acid.

Characterization of the oil: The degummed oil was characterized based on IUPAC standard methods to determine the following properties: density, refractive index, acid number, and saponification value, respectively.

Preparation and characterization of glycerol*:* Preparation of glycerol was carried out from alkali-hydrolysis of oil as described by Unilever (1964). The product was analysed to determine its yield, density, refractive index and boiling point.

RESULTS AND DISCUSSION

Table 1 presents the basic characteristics of the groundnut oil. The low-acid value of the oil indicates the low-level content of free fatty acids. The amount of free fatty acids generated in any vegetable oil usually depends on the extent of deterioration of the oil. Thus, free fatty acids content is an indicator of the freshness of oil. The saponification value obtained falls within the range of values (188-195) reported for similar oil in the literature (Gunstone and Norris, 1983; Ihekoronye and Ngoddy, 1985; Asiedu, 1989). The density value suggests characteristically that groundnut oil, like other fatty oils in

Table 1. Characteristics of the oil

Parameters	Values
Acid number	0.56 mgKOH/g oil
Saponification value	189.34 cm^3KOH/g oil
Density, 25^oC	0.91 g/cm^3
Refractive index	1.47

Table 2. Properties of the glycerol

Properties	Values
Density/gcm^{-3}	1.2969
Refractive index	1.45
Boiling point	260^oC
Yield of glycerol/100g oil	34.88 % (v/v)

general, is less dense than water (density, ca. 1 g/cm^3). The density can vary very easily according to temperature. For example, at 15^oC, density of groundnut oil is reportedly in the range 0.917-0.921, while at 20^oC, the parameter range from 0.910-0.915 (Ihekoronye and Ngoddy, 1985; Asiedu, 1989). Thus, the density varies inversely proportional with temperature. The obtained value of refractive index is also consistent with what is found in the literature (Gunstone and Norris, 1983).

Table 2 shows the properties of glycerol prepared from the saponification process. The density of pure glycerol commonly reported in literature is around 1.261, which is near to the determined value. Similarly, the refractive index (1.4500) of the glycerol is close to the literature value (1.4746). Only in the boiling point, that a significant difference is observed between what was determined (260^oC) and value (290 ^oC) in the literature (Lide, 1994). A number of factors might be attributed to this. They include differences in methods of determination, processing techniques employed in the preparation of the glycerol, age of the oil from which the glycerol is produced, to mention but a few. The yield of 34.88% of glycerol recorded is quite high and hence recommends this method as potentially viable for exploitation for production of glycerol in commercial quantity.

CONCLUSION

The saponification process provides a ready source of glycerol from vegetable oil. Glycerol produced by other large-scale sources is fraught with pollutants which cannot be removed except with use of expensive installation equipments. Although the latter methods result in glycerol glut, the cost implications for purification make the procedures unattractive. This impels the need to consider the saponification method for improved efficacy in production of glycerol.

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