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Study of Esterification between Oleic Acid and 2-Ethyl Hexanol by using the Microwave Method

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Abstract. Oleic acid is an unsaturated fatty acid, has a rancid odor and the main component of palm oil. To increase the value of oleic acid which has a C_{18} chain, it has the potential as a raw material for making long chain fatty acid esters. This fatty acid ester from vegetable oil is an emollient ester which is biodegradable which was synthesized with 2-ethyl hexanol using the Microwave method. In the last ten years, the microwave method has been widely used in organic synthesis. This esterification reaction was carried out using a modified home microwave by making a hole on top as a place for the ball cooler equipped with a stirrer, and using an acid catalyst. Characteristics of long chain fatty acid esters will be tested by FTIR and GC-MS. FTIR test results showed absorption of specific functional groups, while GC-MS was able to show the presence of long chain fatty acid ester compounds.

Keyword: Fatty Acid Esters, Microwave, Oleic Acid.

1 Introduction

Esters of carboxylic acids have become a topic of discussion among researchers because their usefulness is reported in various literature [1]. Oleic acid (9-octadecenoic acid) is an unsaturated fatty acid that is widely contained in vegetable oils. It is composed of 18 C atoms with one double bond between the 9th and 10th C atoms, with the chemical formula CH3(CH2)7CHCH(CH2)7COOH. This acid at room temperature is a viscous liquid with a pale yellow or brownish-yellow color, has a distinctive aroma, is insoluble in water, its melting point is 15.3 ° C and its boiling point is 360 ° C. Derivative products that can be produced from oleic acid include lubricants, emulsifiers, cosmetic creams, polymer additives and cleaning surfactants. Oleic acid has renewable and environmentally friendly properties, where there are not only single and double bonded carbon chains [2].

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The most common and best known method for making esters is Fischer esterification, which is esterification using reversible acid catalysts [3]. Esterification is used extensively in the synthesis of secondary metabolites, polymers, and pharmaceutical active ingredients with other biological ingredients [4]. Several researchers reported on ester esterification experiments using oleic acid in various literature such as: optimization of oleic acid esterification catalyzed by ionic liquid for the synthesis of green biodiesel [4], synthesis and physicochemical properties of new lauric acid made from oleic acid and its evaluation for bio-lubricating base materials [5], synthesis and kinetics of trimethylolpropane fatty acids from methyl esters of oleic acid as potential bio-lubricants[6], and bio-lubricating synthesis of oleic acid by oligomerization, polymerization and hydrogenation of petrochemicals [7].

In recent years, microwave-assisted reactions have received a lot of attention, as they are generally not only faster than regular conventional heating methods, but also potentially more efficient, cleaner, and safer [8]. Liu succeeded in conducting research on fatty acid ethyl esters produced by the presence of heterogeneous catalysts through microwaves and succeeded in shortening the duration of the reaction from 12 hours to 7 hours. Heterogeneous acid catalysts have become green chemistry-based catalysts, which use low waste in esterification processes. Several types of heterogeneous acid catalysts have been investigated, such as zeolite, zirconium oxide and ion exchange resin catalysts such as amberlyst [9]. It has successfully performed glycerol mono-palmitate synthesis with amberlyst catalyst [10], acetic acid esterification with ethanol using amberlyst catalyst [11], and esterification between gallic acid and glycerol using microwave radiation [12].

Microwave-assisted organic synthesis has been used in laboratory experiments for Knoevenagel condensation, Wolff−Kishner reduction, Wittig reaction, Diels−Alder reaction, Williamson ether synthesis, transesterification, esterification with base and acid catalysts, solventless aldol condensation, and ester hydrolysis reactions. Nowadays the use of microwaves is a technique commonly used in laboratories and is increasingly advanced so it is good to get a better understanding of how microwave radiation affects chemical reactions [13]. This study was used to study and determine the potential of oleic acid to be a raw material for long-chain fatty acid esters with alcohol using microwave radiation.

2 Method

Oleic acid was measured at 2 mL and put into a flask base. It was measured to add 2-ethyl hexanol as much as 9.0028 mL to the flask with the ratio of moles of oleic acid to 2-ethyl hexanol (1: 5). Then a 15% catalyst is added based on the percent weight to oleic acid. Then the flask base is put into the microwave, then assembled the tool and set the reaction time for 20 minutes, the power of the microwave power is 60P. The reaction results are titrated using 0.5 N NaOH solution to determine the acid number. The change in functional groups with the FTIR spectrophotometer and the obtained Ester is determined with GC-MS.

3 Results And Discussion

FTIR characterization is carried out to determine the changes in functional groups that occur before and after the esterification reaction. Changes in functional groups can be seen by the shift or emergence of new absorption peaks at certain wavenumbers [14]. FTIR spectra identify pure oleic acid as having a field area between 3500 and 2500 cm−1 [15].

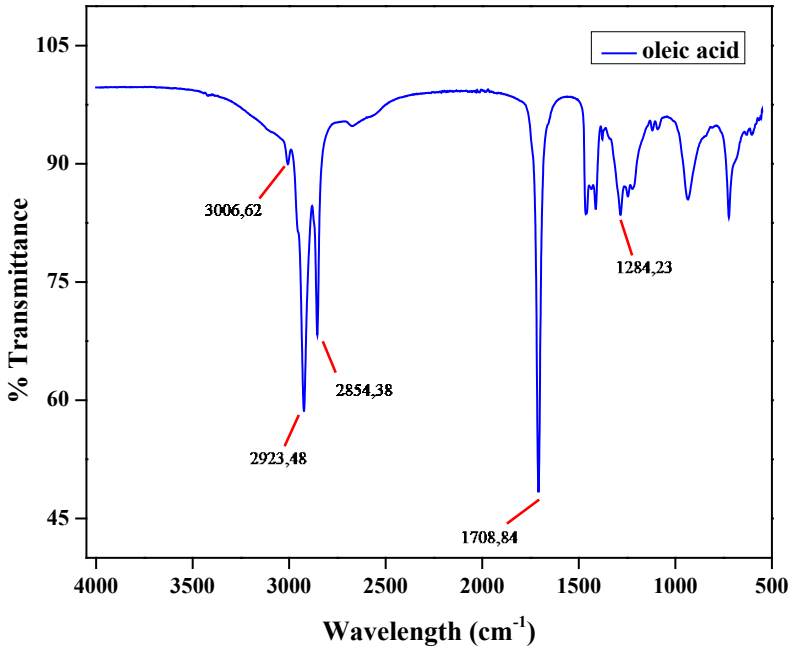

Figure 1 Oleic acid FTIR spectra

In figure 1 it can be seen that the FTIR spectrum of oleic acid shows an absorption peak at wavenumber 3006.62 cm-1 which shows the O-H stretch of the carboxylic acid and the double bond of the -CH=CH- group. The FTIR spectra of pure oleic acid show two bands at 2854.38 and 2923.48 cm−1 corresponding to symmetrical and asymmetric strain -CH2 [16]. The band read at 1708.84 cm−1 shows an asymmetrical -C=O stretch and the band at 1284.23 cm−1 shows the presence of C-O strain.

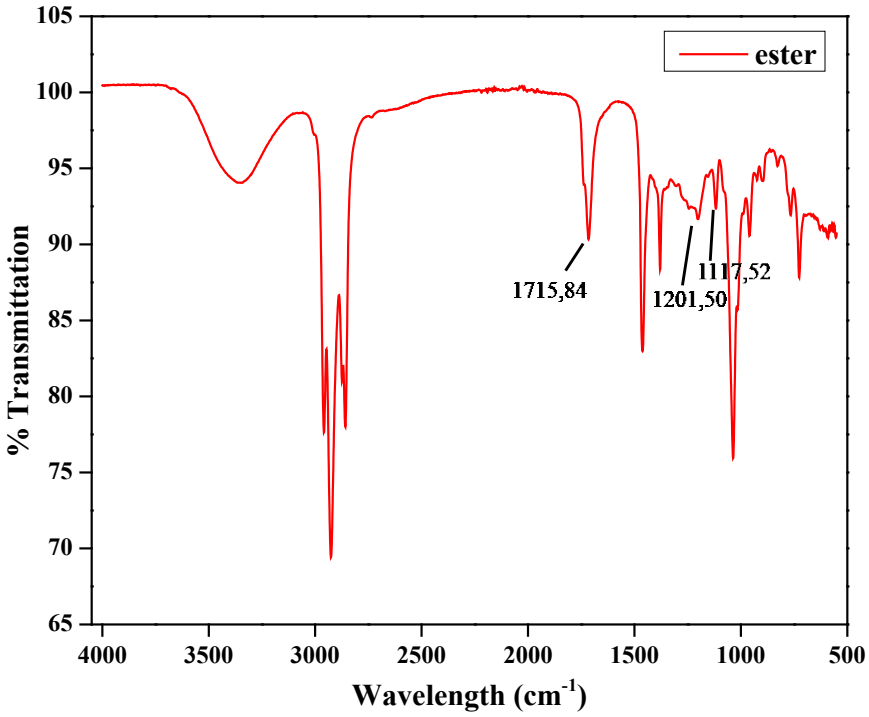

Figure 2 Esther FTIR spectra

The esters formed from the esterification process are confirmed by observing the FTIR spectra of the product and oleic acid. The FTIR spectra of esters are shown in Figure 2. Based on the comparison of FTIR spectra in Figures 1 and 2, it appears that the peak at wavenumber 1708.84 cm-1, namely for the C=O group of carboxylic acid in oleic acid moves to a wavenumber of 1715.84 cm-1, the C=O group in esters. The wavenumber shift in this band combined with two C-O stretch bands confirming ester formation at 1110-1300 cm-1 [17] in figure 2 shows one stronger and wider, occurring at 1117.52 and 1201.50 cm-1. The absorption of the carbonyl group of carboxylic acids at 1709.87 cm-1 has shifted and is no longer observed which confirms that an esterification reaction occurs.

4. Conclusion

Based on the results of research that has been done, it can be concluded that esters of oleic acid and 2-ethyl hexanol were successfully synthesized. The shift in the FTIR spectral wavelength of oleic acid proved the formation of esters in the final experimental results.

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