Functional groups of bio-lubricants from crude catfish oil
(*Pangasius hypothalamus*)

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Abstract. Bio-lubricants have great potential in the production of lubricating in the future. Several studies have developed animal oils as lubricants, which come from a by-product of fish processing. Fish oil extracted from the material and processed into bio-lubricants reached the highest yield of 94%. This research aimed to study the bio-lubricants functional groups. The research steps extracted crude fish oil as raw material, hydrolysis using HCl catalyst, polymerization using benzoyl peroxide, and polyesterification using ethylene glycol. The extraction process used the wet rendering method with a ratio of catfish waste (viscera) to the water of 1:2 (w/v) at 70 °C for 30 minutes. The best bio-lubricants were analyzed for functional groups using an FT-IR instrument with a wave range of 4000-450 cm⁻¹. The results obtained were the absorption wavelength peak of 3472 cm⁻¹, indicating O-H bonds with the sloping peak and the weak bond. The absorption wave peak of 3006-2852 cm⁻¹ indicated a strong C-H bond (alkane). The absorption wave peaks of 1743 cm⁻¹ indicated the presence of a C=O double bond. The adsorption wave peaks at 1465 cm⁻¹ indicated carbon chain bonds between C-C, while the absorption wave peaks at 1115 and 1174 cm⁻¹ indicated C-O bonds. The three spectral indicated that the ester groups formed in bio-lubricants. In the polymerization reaction, there was no absorption wave of 1600-1500 cm⁻¹ which indicated that all C=C groups had been polymerized by benzoyl peroxide. Meanwhile, a sloping absorption wave of 3472 cm⁻¹ was found in the polyesterification reaction due to the weak O-H bond. The analysis obtained above showed the differences in wave peak between bio-lubricant and crude fish oil as raw material but had the same group shape.

1. Introduction
Lubricants are generally derived from mineral oil, vegetable oil, and synthetic oil. Mineral and synthetic lubricants are made from petroleum which contains aromatic and toxic compounds and is difficult to degrade in nature [1]. Along with the times, it is demanded to use materials that are environmentally friendly and easily degraded. Bio-lubricants are biodegradable from readily biodegradable materials around 98% in the soil, making them an environmentally friendly lubricant [2].

Bio-lubricants have considerable potential as lubricating oils in the future. Several studies have successfully produced lubricating oils from vegetable oils and have been applied to lubricate gas engines, gears, hydraulics, and motors. The vegetable oils used were castor oil with a yield of 98.6%, palm oil 98%, canola seed 99%, Karanja oil 95%, and sunflower seed oil 94% [3]. While animals oil,
one of which comes from a by-product from fish processing. Fish oil that was processed as a lubricant produces a yield of 94% [4].

The stages of production of bio-lubricants that are commonly carried out are hydrolysis, polymerization, and polyesterification. Hydrolysis is the process of breaking down triglycerides by water to form fatty acids and glycerol. Hydrolysis using alkali is widely carried out because the reaction time is relatively short, it can hydrolyze oil with high FFA content, take place at low temperatures, the reaction conditions are relatively soft and cheap [5]. Polymerization is an initiation reaction with free radicals in the double bonds of monomers, making it easier for monomers to bond with other monomers to form polymer compounds. The polymerization reaction generally used benzoyl peroxide [6]. Polyesterification was the process of combining two main groups, namely carboxyl and hydroxyl groups, to form esters [7] in which polyl ester chains (trimethylpropane, neopentyl glycol, ethylene glycol, and pentaerythritol) are joined with mono-esters (methyl esters or fatty acids). Polyesterification is a simple and inexpensive process by reacting methyl esters and fatty acids with methanol using a catalyst. This study aimed to analyze the functional group of the bio-lubricants produced to determine the ester functional group as indicated by the weakening of the OH group intensity. This weakening of the OH group intensity showed that the polyesterification process had been successful.

2. Materials and Methods

2.1. Materials
The raw material used in this study was crude catfish oil (*Pangasius hypophthalmus*). Other ingredients were HCl as a catalyst, benzoyl peroxide, and ethylene glycol.

2.2. Methods
Crude fish oil was obtained through the extraction process of a by-product from catfish filet processing (viscera) using the wet rendering method, the ratio of viscera and water was 1:2 (w/v) at 70°C for 30 minutes [8]. In Figure 1, we can see the step of bio-lubricants production from crude catfish oil.

![Figure 1. Production of bio-lubricants from crude catfish oil [9]; [10]; [8].](image-url)
The functional groups of bio-lubricants were analyzed using the Fourier Transform Infrared spectroscopy/FT-IR (Perkin Elmer Spectrophotometers) instrument at a wavelength between 4000-450 cm\(^{-1}\). Before the bio-lubricants were analyzed, the preparation carried out was that the bio-lubricants were dried using a concentrator (Scanvac Chermoscience) at 40°C for 24 hours with a rotation speed of 700 RPM and vacuum pressure of 1013 mbar.

3. Result and Discussion
The result of functional groups bio-lubricants analysis can be seen in Figure 2. The measured peaks were at wavelengths of 3472, 2923-2852, 1743, 1465, 1378-1174, 1054, and 720 cm\(^{-1}\). The absorption wavelength peak of 3472 cm\(^{-1}\) indicated O-H bonds with a sloping peak and a weak bond. The absorption wave peak of 3006 to 2852 cm\(^{-1}\) indicated strong C-H (alkane) bonds in the bio-lubricants. The absorption wave peak of 1743 cm\(^{-1}\) indicated C=O double bond but no C=C double bond. The peak of the absorption wave at 1465 cm\(^{-1}\) indicated carbon chain bonds C-C, while the peaks of the absorption wave at 1115 and 1174 cm\(^{-1}\) indicated C-O bonds. The three spectra showed the parameters of the ester group formation.

Wave peaks between crude oil and the produced bio-lubricants differed, but they still had the same group shape. The same group shape was because the crude catfish oil had polymerized and hydrolyzed naturally. FT-IR analysis showed that the polyesterification temperature did not affect the change in functional groups as seen from the graph’s peaks, which were the same.
The ester group present could decrease the pour point of the produced bio-lubricants, but branching in the bio-lubricants compound and long carbon chains that inhibit crystallization [11], the drop in pour point was not much. The success of the polymerization reaction in the bio-lubricants production process could be seen in the absorption wave of 1600-1500 cm\(^{-1}\). If the wave range was not found, it indicated that the entire C=C group had been polymerized by benzoyl peroxide [12]. The breaking of the C=C double bond affected the decrease in the flashpoint of the produced bio-lubricants [13]. Furthermore, the polyesterification reaction had successfully that the ethylene glycol reacted forming esters as shown from the FTIR spectra at 1743 cm\(^{-1}\) [14]. The bio-lubricants analyzed by their functional groups in this study had a relatively low flashpoint (127°C), and the pour point was relatively high (27°C) [8].

4. Conclusion
Polymerization reaction had successfully demonstrated that the entire C=C group had been polymerized by benzoyl peroxide. Polyesterification reaction had successfully that the ethylene glycol reacted forming esters. FT-IR analysis showed that the temperature of the polyesterification did not affect the change in functional groups. There was a difference in wave peaks between crude oil as raw material and the produced bio-lubricants, but it still had the same group shape.

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