Characterization of Fish Bone Catalyst for Biodiesel Production

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Abstract—In this study, fish bone waste was used as a new catalyst for biodiesel production. Instead of discarding the fish bone waste, it will be utilized as a source for catalyst that can provide significant benefit to the environment. Also, it can be substitute as a calcium oxide source instead of using eggshell, crab shell and snail shell. The XRD and SEM analysis proved that calcined fish bone contains calcium oxide, calcium phosphate and unburned hydrocarbon [1], [2].

Also, new alternative and renewable fuel (biodiesel, bioethanol) have received a considerable attention recently due to the occurrence of oil depletion, global warming and the greenhouse effect. Biodiesel is a liquid fuel similar to petroleum diesel in combustion properties, but essentially free of sulphur, making it a cleaner burning fuel than petroleum diesel in combustion properties, but essentially free of sulphur, making it a cleaner burning fuel than petroleum diesel. It derived from renewable energy sources, such as vegetables oils and animal fats. It has similar physical and chemical properties with petroleum diesel fuel.

Biodiesel is mainly and generally produce by transesterification of vegetable oils or animal fats using homogeneous basic catalyst (mainly sodium or potassium hydroxide dissolve in alcohol such as methanol). However there are some disadvantages of using homogenous catalysts. Homogeneous catalyst may lead to soap production. Furthermore, this type of catalyst is consumed during transesterification process thus reducing the catalytic efficiency and this will cause an increase in viscosity and the formation of gels. The removal of the catalyst after reaction is technically difficult and a large amount of wastewater is produced in order to separate and clean the products which increase the overall cost of the process.

Heterogeneous basic catalysts are hence preferred for biodiesel synthesis as they are noncorrosive, environment friendly and show fewer disposal problems. Besides, heterogeneous catalyst does not produce soaps through free fatty acid neutralization or triglyceride saponification. Unfortunately, the preparation of heterogeneous catalyst is quite expensive and complex [3]. Therefore, screenings on low cost solid heterogeneous catalysts from wastes are investigated to replace the homogenous catalyst.

I. INTRODUCTION

Biodiesel is becoming high of interest in the worldwide. In recent years, biodiesel has gained international attention as one of alternative fuel due to characteristic like high degradability, no toxicity and low emissions of carbon monoxide, particulate matters and unburned hydrocarbon [1], [2].

The use of heterogeneous catalyst is the key to overcome the problems caused by homogeneous catalyst. Successful ventures were reported on utilization of waste chicken egg shell, oyster shell, mud crab shell, golden apple snail and meretrix venus, and mollusk shell [3]-[6] as cheap resources of CaO for application as low cost heterogeneous catalyst for biodiesel synthesis. The heterogeneous catalytic process overcomes the homogeneous catalyst since solid catalyst such waste shell of mollusk and egg [3] can be easily recovered and therefore potentially be reusable. Besides, neutralization step with large amount of wastewaster production would be eliminated.

Fish bone is constituted by the remaining meat after removal of the fillet, bones and cartilages [7]. Fish bone consists of 60% to 70% of inorganic substances and is mainly comprised of calcium phosphate and hydroxyapatite. Also, [7] reported that in Nile Tilapia fish bone has high content of calcium. Edible fish bone contains a high amount of calcium. Fish bone ash consists of 34-36% calcium, particularly calcium phosphate. The waste fish bone will undergo calcinations process to get the catalyst called Calcium Oxide (CaO).

After calcinations process, the fish bone were analyzed using SEM to obtain the morphological structure of fish bone and the crystalline phases of calcined samples were analyzed by X-ray diffraction. CaO is an environmentally friendly material used as a basic oxide catalyst. Ca(NO3)2, CaCO3, CaPO4 and Ca(OH)2 are raw materials to produce CaO, but natural waste sources such as egg, shrimp, oyster and crab and cockle shells have also been employed [8]. Other bones that used as a catalyst in biodiesel production are animal bone [8], fish scale bone [5] and cuttle bone [9].

Researcher [8] used calcined waste animal bones from sheep and reported that the methyl ester conversion was 96.78%. Bone from waste Rohu fish (Labeo rohita) has been...
reported as another low-cost heterogeneous catalyst for the synthesis of biodiesel from soybean oil [5]. The analysis of TGA and XRD revealed that a significant portion of the main component of fish scale, hydroxyapatite, could be transformed into b-tri-calcium phosphate when calcined above 900°C for 2 h, with optimal conditions of MeOH/oil molar ratio, 6.27:1, catalyst at 1.01 wt.% for 5 h. This compound was able to yield 97.73% of methyl ester. In this study, the characterization of fish bone waste for biodiesel use has been investigated.

III. METHODOLOGY

Fish bone wastes were collected from cafeteria and canteen of International Islamic University Malaysia (IIUM). Fish bone wastes were soaking into boiling water for several hours and then rinsed with distilled water several times. Then, the fish bones were dried in the drying oven at 70°C for 24 hours to remove water and moisture. After that, the fish bone was milled and crushed until it become into powder form. After milling process, the crushed fish bone undergone calcinations process using muffle furnace.

A. Characterization of Catalyst

After calcinations (800-1000°C) procedure was completed, the crucible with the fish bones (catalyst) inside was cooled in dessicator. After it has been cooled at room temperature, the catalyst were weighted and transferred into petridish. Then, the catalyst was analyzed using SEM to determine the structure of the catalyst and it’s morphological. It also showed that the differences of its structure and its morphological for before and after calcinations took place. It showed that the structure of fish bone changed with optimizing of calcinations temperature calcinations time. Calcined fish bone also was undergone characterization process by using XRD machine to see the pattern of calcined fish bone and to check the presence of calcium oxide element in the calcined fish bone.

B. SEM analysis

After getting the catalyst from calcinations process, the catalyst in powdered form will undergo analysis on its structure and catalyst morphology using Scanning Electron Microscope (SEM).

The structure of catalyst will depend on the calcinations temperature from 800°C, 900°C, and 1000°C. The SEM images will indicate that the structure of fish bone changed with different calcinations temperature [4].

C. XRD analysis

This analysis will take place after calcinations process completed. The catalyst produced from calcinations process will be analyzed by X-ray Diffraction (XRD). The catalyst was pour onto the glass plate provided and putted in the space provided at the XRD machine. The sample will show result on composition of Calcium Oxide (CaO) from different calcined temperature. XRD pattern will show a crystalline of Calcium Oxide (CaO) [4].

IV. RESULTS AND DISCUSSION

A. Characterization of Fish Bone

Fish bone sample were tested using SEM machine for before and after calcinations process took place. The samples were analyzed on its structure and catalyst morphology. The result is as shown in Fig. 1. It showed that there is difference between before and after calcinations with equal magnification 1000x. The uncalcined fish bone showed bulky substances and the surface area is bigger compared to calcined fish bone from Fig. 1 (b).

![Fig. 1 SEM image for (a) uncalcined fish bone and (b) calcined fish bone](image)

**Fig. 1** SEM image for (a) uncalcined fish bone and (b) calcined fish bone

B. XRD Analysis

Fish bone sample were tested using XRD after calcinations process. There are differences of XRD results between uncalcined fish bone and calcined fish bone. The graph of uncalcined fish bone from Fig. 2 showed that the intensity of the graph lower than calcined fish bone (Fig. 3) narrow and high intense peaks of the calcined catalyst define the well-crystallized structure of the catalyst. Researcher [6] reported that the transformation of oyster shell composed of CaCO3 to CaO required the combustion temperature at 700°C or above. Further report from [4] also described that the temperature of eggshell transformation was above 800°C.

![Fig. 2 XRD pattern for uncalcined fish bone](image)

**Fig. 2** XRD pattern for uncalcined fish bone

As illustrated in Fig. 3, the catalyst sample showed that there are several matched peaks of calcium oxide (CaO) in the figure. Also, there were other elements in the catalyst such as hydroxyapatite and tri-calcium phosphate. All these three elements proved to catalyze the biodiesel production as stated by [4], [10].
This study concluded that highly active solid catalysts were obtained after calcination of the waste food materials. This calcined fish bone will be used for biodiesel production. Fish bone catalyst as a source of CaO has opened up new roads for employing heterogeneous basic catalyst in biodiesel production, providing a clean, almost glycerol free and soap free biodiesel production. Employment of waste resources in biodiesel production lowers the cost of production and environment friendly.

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