

Application of Potassium Impregnated Calcium Oxide/Magnesium Oxide Catalyst for Transesterification of *Jatropha Curcas* Oil with Methanol



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Abstract

Biodiesel production using homogenous catalysts associates with high energy consumption and production cost due to the complicated separation and purification of the products. In this investigation, a solid base catalyst, 10%K-CaO/MgO was synthesized, analyzed and used to produce biodiesel from *Jatropha curcas* oil with methanol. The catalyst loading was 0.4 % mass of the oil while the transesterification times were 20, 30, 40, 50 and 60 minutes at 60°C. The methyl esters yields were 94.20, 92.00, 82.13, 82.81 and 82.09 % respectively. There was no glycerol in any of the five products.

Keywords: Application; Biodiesel; Production; Solid catalyst

Abbreviation: GHG: Greenhouse Gas; KNO₃: Solution of Potassium Nitrate; SSA: Specific Surface Area; CIE: Compression Ignition Engine; FFA: Free Fatty Acid

Introduction

Biodiesel is the name of a clean-burning, non-toxic alternative fuel made from vegetable oils and animal fats that can be used in compression-ignition engines. It refers to a diesel-equivalent, processed fuel derived from biological sources [1]. Biodiesel, which is mostly produced from vegetable oils, is mono-alkyl ester and is perceived to replace the diesel fuel [2]. Biodiesel fuel (B100) burns much cleaner than petroleum fuel due to its oxygen content. The exhaust emissions of sulfur oxides and sulfates, the major components of acid rain are essentially eliminated compared to diesel. Biodiesel is comprised of mid-carbon chains, which burn more completely than petroleum fuel. That is why there is a substantial reduction of unburned hydrocarbons, carbon monoxide, and particulate matter compared to emissions from diesel fuel. Biodiesel has exceptional lubricating qualities. Use of biodiesel or biodiesel blends have been shown to contribute significantly to the longevity and cleanliness of diesel engines. In new engines, biodiesel fuel consumption is similar to that of petro-diesel. In used engines, fuel economy substantially improves due to continuous cleaning effect of biodiesel.

The combustion of petroleum based fuels has created serious environmental concern over global warming effect due to

greenhouse gas (GHG) emissions [3]. Biodiesel is biodegradable and nontoxic when used in its pure form (B100). For the 20% and lower blends, the diesel fuel portion of the blend determines the toxicity and biodegradability. Biodiesel fuel and fuel blends smell better than conventional diesel. The fuel is especially promising as a marine engine fuel as a biodegradable, non-toxic fuel. Therefore, it can help prevent damage to marine environments such as wetlands, marshes, rivers, and oceans. It is a domestic, renewable resource that strengthens the domestic agricultural economy. The production of soy-based biodiesel has a positive energy balance (as much as 3:1), due to the high energy value of ester-based feedstock, the low-energy requirements of the conversion process, and the nitrogen-fixing characteristic of soybean.

Homogenous catalysts have been used overtime for conversion of triglycerides into fatty acid alkyl esters with high yield. However, the catalysts are not easily separated from the product streams [4]. At the end of the reactions, the homogenous catalysts are neutralized, washed with hot water, thereby producing waste water into the environment. The homogenous process associates with high energy

consumption and production cost, partly resulting from the complicated separation and purification of the products. The use of homogeneous base catalysts leads to soap production [5] which may terminate the reaction. The base homogenous catalysts react with free fatty acids in the feedstocks to produce soaps. Homogenous transesterification cannot be used in the continuous production process. Solid heterogeneous catalysts are suitable for continuous production process as the catalysts can easily be filtered from the product streams. The recovered catalysts can be reused; this provides another additional advantage over homogenous catalysts. In addition, the use of heterogeneous catalysts does not produce soap [6] as they do not react with free fatty acid in the feedstock.

In this investigation, biodiesel was produced from *Jatropha curcas* oil with methanol in the molar ratio of 1:3 using 10% potassium impregnated calcium oxide/magnesium oxide catalyst. The free fatty acid of the feedstock was reduced to 1.2mgKOH/g. The catalyst was prepared from Calcium oxide/magnesium catalyst by dissolving 20g of the mixture into 10% potassium nitrate solution and stirred for 30 minutes. This was heated to dryness on a Gallenkamp hot plate magnetic stirrer. The residue was calcined at 760°C for 30 minutes in a Gallenkamp muffle furnace.

Materials and Methods

20g each of CaO and MgO were mixed and dissolved in distilled water contained in 500ml beaker to incipient wetness. The slurry thus formed was dried in oven at 110°C to constant weight. The residue was calcined at 760°C for 30 minutes in Gallenkamp muffle furnace at 50% energy rate. 20g of this catalyst was dissolved in a 10% solution of potassium nitrate (KNO₃) in 500ml beaker and stirred for 30 minutes. The beaker and its content was placed on magnetic stirrer and heated until the water evaporated. The residue was transferred into a crucible and calcined in a muffle furnace at 760°C for 30 minutes [5]. 20g of the calcined catalyst was further dissolved in 350ml distilled water and stirred, then followed by 80g of alumina. The mixture was stirred on a hot plate magnetic stirrer to dryness.

The residue was pulverized and calcined in a muffle furnace at 760 °C for 30 minutes. 0.4g of this catalyst was used to produce biodiesel from 100g of *Jatropha* oil and 10.8 g of methanol making oil to methanol mole ratio 1:3. The transesterifications were carried out at 60°C for 20, 30, 40, 50 and 60 minutes.

Catalyst Characterization

The catalyst was characterized with SEM and BET machine. Sample of the catalyst was taken to the Chemical Engineering Department Laboratory of Ahmadu Bello University, Zaria for SEM analysis. The BET surface area of the catalyst sample was determined using a conventional flow apparatus by nitrogen adsorption. The Brunauer, Emmett and Teller (BET) theory describes how gas molecules accumulate in multiple layers over solid surfaces and pores [7]. The catalyst was degassed after heated to 300°C for 3 hours in a flowing gas mixture of 30% N₂ and 70% He to remove water vapour and then cooled. Still left exposed to N and He gas mixture, liquid N was applied and removed to obtain amount of N gas desorbed. When desorption is complete the machine calculate the Specific surface area (SSA) by dividing the desorption value by the catalyst weight [7]. This analysis was carried out with Surface Area and Porosimetry Analyzer V-Sorb 2800P in the Petrochemical Laboratory of NARICT, Zaria.

Ester content test

2ml of each sample was diluted with n-hexane. The resulting mixture was transferred into sample bottle and inserted into the GC-MS machine. The machine was run and the chemical components of the sample were analyzed. The methyl esters content was calculated from area% of the GC-MS analysis [8]. This analysis was performed on GCMS-QP@) PLUS SHIMADZU/JAPAN in the Quality Control Unit of NARICT, Zaria.

Result and Discussion

The catalyst had BET surface area of 34.27 m²/g and BET pore volume of 1.162 x10⁻²cm³/g. The catalyst average grain size as measured by SEM was 7.02µm and average pore area of 1.36µm². The detail SEM results of the catalyst are presented in Figure 1.

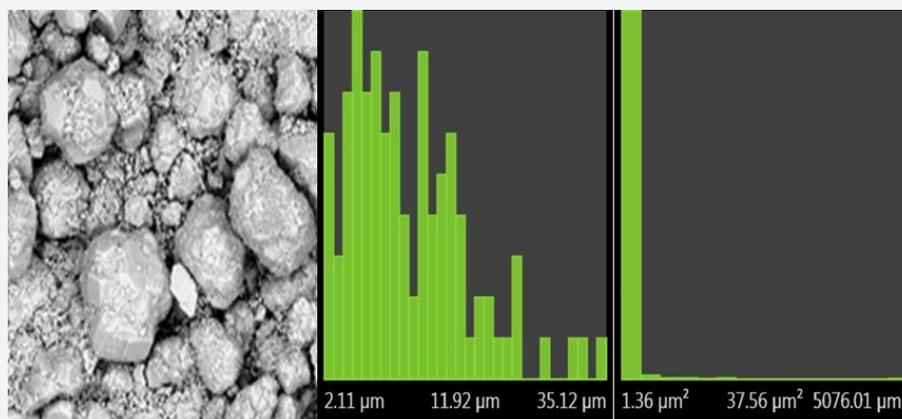


Figure 1: Results of SEM analysis of 10% K-CaO/MgO catalyst. a) Particle structures b) Particle size distribution c) Pore areas distribution

The methyl esters yields obtained by use this catalyst (10% potassium impregnated calcium oxide/magnesium oxide) were low compared to the minimum esters content required of acceptable biodiesel for use in Compression Ignition Engine (CIE). This could be due to high Free Fatty Acid (FFA) of the feedstock which was as high as 1.22mg KOH/g. This high acid content of the feedstock could have responsible for the formation of fatty acids, alcohols and carbonyl compounds found in the products referred to as other impurities. All the products had zero glycerol; hence one can achieve glycerol free biodiesel

production using this catalyst at 60°C reaction temperature. Glycerol free biodiesel production has been achieved by Ibrahim et al.[8] from *Jatropha curcas* oil using bulk calcium oxide catalyst. The highest methyl ester yield was that of 20 minutes run with 94 % and approximately 5.9 % other impurities. However, the highest ester yield obtained is less than the minimum require ester content 96.5% for biodiesel acceptable for use in CIE [9]. The other impurities increases with decrease methyl esters yield as shown in Figure 2.

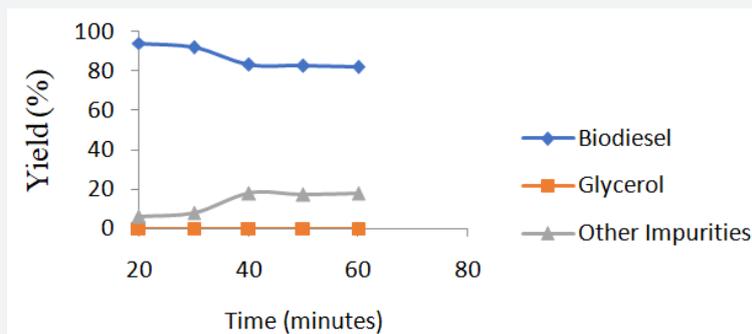


Figure 2: Efficiency of 10% K-CaO/MgO Catalyst for transesterification of *Jatropha*oil.

The biodiesel yield fell from 94-82 % esters for reaction time of 20 minutes to 40 minutes and then remains through to 60 minutes; likewise the other impurities rose from 20 to 40% and remained as shown in Figure 2. The glycerol line remains at zero throughout. 100% conversion has been achieved by Ibrahim et al.[8] from a *Jatropha curcas* oil feedstock with 0.44mgKOH/g FFA value, therefore, the acid value of 1.2 mgKOH/g for the feedstock used in this investigation was too high to obtain good conversion. According to Ibrahim et al.[10], solid base catalysts do not react with free fatty acid therefore; the free fatty acids in the feedstocks either appear in the product or convert to alcohols and carbonyl compounds such as alkanals and alkanones.

Conclusion

The catalyst, 10%K-CaO/MgO was very active in transesterification reaction. It suppressed the production of glycerol, the co-product of biodiesel. However, it could not convert free fatty acids in the feedstock into alkyl esters rather, some amount of the acids appeared in the products and some converted to alcohols and carbonyl compounds as found in the products. Feedstock with free fatty acids as high as 1.2mgKOH/g cannot be completely converted into alkyl esters using solid base catalyst. For complete conversion of feedstock into alkyl esters with solid base catalysts, a maximum of 0.5FFA is required.

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