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TRANS-ESTERIFICATION OF CASTOR OIL USING HETEROGENEOUS BIO-CATALYST OBTAINED FROM BOVINE BONE

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Abstract: Bones obtained from bovine species of animal was investigated in this study as heterogeneous bio-catalyst for trans-esterification process. The bones were washed, dried, comminuted and calcined at a temperature around 500-900°C. A solution of 10 wt% aqueous KOH was prepared to soak the comminuted calcined bones to enhance their surface chemical properties. The obtained bio-catalysts were characterized physico-chemically using x-ray diffraction (XRD) for phase identification of the synthesized biocatalyst, differential thermal analysis (DTA) for the measurement of the mass changes in materials as function of temperature, Fourier transform infra red spectroscopy (FTIR) for the identification of the functional groups and gas chromatography mass spectrometry to identify the fatty acid profile of the synthesized biodiesel. The biodiesel was synthesized using established optimum conditions of 60°C reaction temperature, methanol to oil molar ratio of 6:1, catalyst concentration of 1% weight of the oil, and at one hour reaction time and a maximum yield of 95.12% was obtained. After the 4th cycle of trans-esterification, a further yield of 78.40 % was obtained. The biodiesel produced was found to meet most of the ASTM specifications and more importantly, bovine bone derived catalyst has been identified as a promising and competing catalyst for the synthesis of green fuel through trans-esterification process.

Keywords: bovine, heterogeneous, bio-catalyst, trans-esterification, green fuel

INTRODUCTION

Trans-esterification reaction for the synthesis of biodiesel has gained mass and impressive attention of researchers particularly over the past two decades. For instance, researchers such as Folasegun et al., 2014; Yordanov et al., 2013; Kang and Rui, 2013; Owolabi and Osiyemi, 2013; Owolabi et al., 2011; Porte et al., 2010; Shu et al., 2010; Kasendo et al., 2009; Singh and Singh, 2009; Lapuerta et al., 2009; Canoira et al., 2008; Canoira et al., 2007; Kumar et al., 2007; Canoira et al., 2006; among others successfully trans-esterified various bio-sourced oil respectively, for biodiesel synthesis under varied catalyzed medium with each obtaining interesting results.

Generally, a catalyst offers safer and milder reactions, increases chemoselectivity and functional group tolerance, involves simple work procedure, reduces waste and also exhibits wide substrate scope (Gusev, 2013) still, over the years, only the use of homogeneous acids and bases as catalysts for trans-esterification reaction remained the common practise among researchers. The commonly used homogeneous catalyst is H₂SO₄ (Leung et al., 2010; Meher et al., 2006; Barnwal and Sharma 2005; Fukuda et al., 2001 and Ma et al., 1999) while commonly used homogeneous bases are lyes such as NaOH, KOH etc (Owolabi et al., 2012). Lye catalysts are corrosive to glass apparatus and also prone to saponification with a by product that poses separational issues both in terms of technicalities and cost (Wen et al., 2010 and Song et al., 2011). The homogeneous acid catalysts have also been reported to consume excess reactants coupled with its strong affinity for high methanol oil ratio (Canakci and Van

Gerpen, 1999). All these traits of homogeneous catalysts are not in favour of low cost biodiesel production especially at a scaled up level. Other recent studies have focused on some heterogeneous catalyst with some degree of successes. For instance, micro-crystalline cellulose was used by Ayodele and Dawodu, 2014; Beta-Zeolite (Sathyaselvabala et al., 2011); Ferric Sulfate (Patil et al., 2010); Calcium oxide (Zhang et al., 2010) and sulfonated Zirconia (Garcial et al., 2008) but with varied degree of successes. The last half decade has also witnessed synthesis of heterogeneous catalyst for trans-esterification process from agro-related materials such as palm trunk, sugar cane bagasse, coconut husk, coco pod husk and oyster shell waste (Ezebor et al., 2014a-b; Vadery et al., 2014; Ofori-Boateng and Lee, 2013; Nakatani et al., 2009) respectively. These forms of catalyst were reported by Gryglewicz, 1999; and Suppes et al., 2004 to facilitate post reaction mixtures separation but reaction progresses at relatively slower rate. Moreover, bones have been found to contain hydroxyapatite [Ca₁₀(PO₄)₆(OH)₂] which are porous with large surface area that facilitate catalytic actions (Nisar et al., 2017). Their report was based on the use of an unspecified but calcined waste animal bones as catalyst for biodiesel synthesis. They further ascertained the reasonable performance of the catalyst only at high proportion coupled with longer reaction time. These limitations require enhancement of the surface chemical properties for effective catalytic activity on the bone surface which this present study incorporates. It is therefore our focus in this study to synthesize, characterize and test run the functionality of the new set of catalysts from bovine animal bones for possibilities of improved catalytic

actions over the earlier catalysts. The study also focuses on the operational strength of the bio-catalyst in terms of its recyclability, overall biodiesel yield obtainable and quality.

SYNTHESIS AND ACTIVATION OF BIO-CATALYST

Bovine bones with particular focus on the leg were obtained fresh from an abattoir in Lagos state, Nigeria with 6.5244° N, 3.3792° E. They were washed with hot water to remove impurities and dried in day light for two weeks consecutively. The bones were then crushed, comminuted and pulverised using an industrial grinder while some were left as granules as shown in Figure 1. The bovine bones were calcined at a temperature around 500 - 900 °C in a furnace (Nisar et al., 2017; Madhu et al., 2016; Chopade et al., 2012). A solution of 10 wt% aqueous KOH was prepared to soak the calcined pulverised bones to enhance its surface chemical properties (Nisar et al., 2017). The admixture of the calcined pulverised bones and KOH were simultaneously heated and agitated at 500 rpm using a magnetic heater till cake formation and complete evaporation of water were observed (Alamu et al., 2007; Semwal, 2011.). The hard cake was left to cool and dry. Samples of the virgin pulverised bovine bones, calcined bones and KOH treated bones were kept for subsequent biodiesel production.



Figure 1: Calcined Bovine Bones

— Characterization of the Bio-Catalyst

The synthesized bio-catalyst derived from bovine bone was characterized physico-chemically using x-ray diffraction (XRD) for phase identification of the synthesized biocatalyst, differential thermal analysis or thermogravimetric analysis (DTA/TGA) for the measurement of the mass changes in materials as function of temperature, Fourier transform infra red spectroscopy (FTIR) for the identification of the functional groups and gas chromatography mass spectrometry which utilizes the combined features of both gas chromatography and mass spectrometry to identify the fatty acid profile of the synthesized biodiesel.

— Operation of Reactor

All reactions were performed in two neck batch reactor with reflux condenser, temperature sensor and controlled magnetic stirrer. The temperature was monitored by the digital display on the heating system. The reflux system was adopted to eliminate vapourization of methanol from the reacting system.

— Synthesis of Biodiesel (Green Fuel)

Castor oil for the synthesis of the green fuel was obtained from SIGMA ALDRICH with specification SKU-pack size 18722-1L and was used as obtained. The chemical composition of the castor oil in weight percent and other physico-chemical properties are contained in Tables 1 and 2. The biodiesel was initially synthesized using established optimum conditions of 60 °C reaction temperature, methanol to oil molar ratio of 6:1, catalyst concentration of 1% weight of the oil, and at one hour reaction time (Kang and Rui, 2013). The castor oil was initially pre-heated till desired temperature was reached. A prepared mixture of methanol and catalyst was introduced into the pre-heated oil for transesterification purpose and commencement of the reaction time (Banerjee et al., 2017; Kilic et al., 2013). Products were kept overnight for cooling and phases separation in a separating funnel. The desired upper phase consists of the castor oil biodiesel while the lower phase consists of glycerol, catalyst, unreacted reactants and other insignificant side reaction products. For the case of the synthesized bio-catalyst, the catalyst was separated and kept for further biodiesel production and yields were obtained using Eq.1

$$\text{Yield Biodiesel} = \frac{\text{Weight Fatty acid methy ester}}{\text{Weight of oil}} 100 \quad (1)$$

The recyclability of the synthesized bio-catalyst was examined for a number of experimental cycle under the same established optimum conditions. Yields of biodiesel obtained were calculated at the end of each cycle.

RESULTS AND DISCUSSION

— Characterization of the Castor Oil Feedstock

Table 1 and 2 contains the fatty acid profile and physico-chemical properties of the castor oil, respectively as directly obtained from the manufacturer.

Table 1. Fatty Acid Composition of Castor Oil

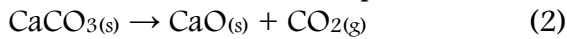
| S/N | Fatty Acid | % Composition |
|-------------|-----------------|---------------|
| 1 | Ricinoleic Acid | 87 |
| 2 | Oleic | 7 |
| 3 | Linoleic | 3 |
| 4 | Palmitic | 2 |
| 5 | Stearic | 1 |
| Merck, 2019 | | |

Table 2. Physico-Chemical Properties of Castor Oil

| S/N | Properties | Units | Value |
|---------------------------|------------|---------------------|-------------|
| Appearance (Light Yellow) | | | |
| Density | (20 °c) | g//ml | 0.952-0.965 |
| Viscosity | (25 °c) | Poise | 1.6-2.8 |
| Flash Point | | °C | 113 |
| Peroxide Value | | meq/g | ≤ 5 |
| Acid Value | | mgKOH/g | ≤ 5 |
| Hydroxyl Value | | mg/g | ≥ 160 |
| Iodine Value | | mgI ₂ /g | 82-90 |
| Saponification Value | | mgKOH/g | 176-187 |

— Activation and Characterization of the Synthesized Bio-Catalyst

Thermogravimetric analysis to monitor the influence of calcination temperature on weight loss of the cow bone is as depicted in Figure 2. The study was carried out over a temperature range of 32-900°C. A sharp drop in weight was recorded between temperature range of 32–500°C thereafter, it became approximately steady. Madhu et al., 2016 attributed the sharp drop in weight to the decomposition of calcium carbonate to calcium oxide coupled with effervescence of carbon (iv) oxide (Eq.2)



Steady weight loss was also observed between 500–900°C. Nisar et al., 2017 similarly observed this trend in their study and attributed such to the formation of gaseous fraction due to transformation of bone to hydroxyapatite (HAP). In Figure 2, no significant further loss in weight was observed. This is a confirmation of the high thermal stability of the calcined cow bone.

The scanning electron microscope of the synthesized biocatalyst (15,000 X and 10,000 X) are presented in Figure 3a-b and as specified are at different magnifications showing the surface morphology of the heterogeneous catalyst. The architecture observed seems porous which gives it a catalytic advantage.

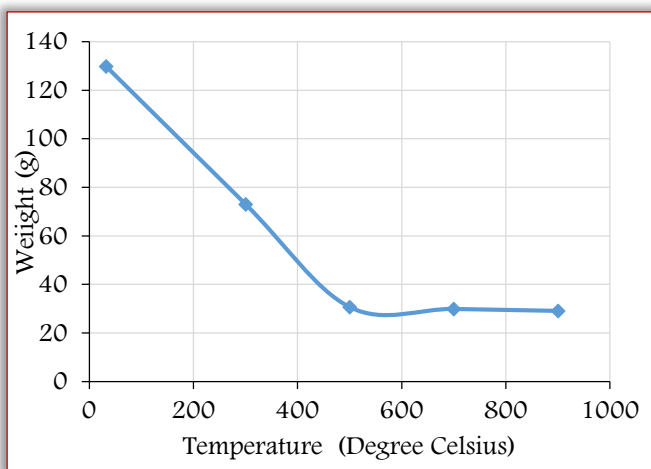


Figure 2: The TGA/DTA Profile of Cow Bone
The preparation of the bio-catalyst was supported with KOH(aq) which could be responsible for scattered particles on the catalyst surface. Though,

effects of the calcination temperature was not studied but reports (Goli and Sahu,2018) confirmed that at high calcination temperature, the catalyst particles becomes smaller.

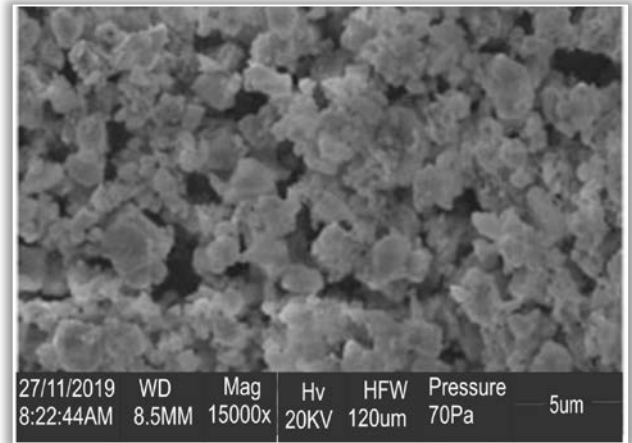


Figure 3 a : SEM Image of the Synthesized Biocatalyst (Magnification 15000x)

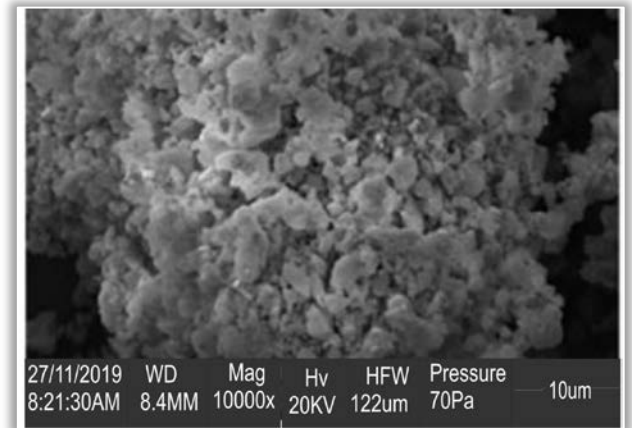


Figure 3b: SEM Image of the Synthesized Biocatalyst (Magnification 10000x)

In another study, the reduced particle were found to be proportional to the specific surface area and depends on the catalyst surface area (Khemtong et al., 2012; and Viriya- Empikul et al., 2012). From the biodiesel yield obtained in this study, the bio-catalyst particles exhibited remarkable catalytic acitivity. Figure 4 shows results from the X-RD where just few peaks were observed. Similar to the report of Tahvildari et al., 2014, modified materials which are crystalline in nature are apparent. This could be by virtue of the reaction between calcined bone and KOH (Tahvildari et al., 2014).

From the spectral analysis of the bio-catalyst, the major absorption bands include ,the C-Cl stretch that was detected at the wave numbers 557 and 597 cm⁻¹, both the meta and para C-H bend that were detected at 871 cm⁻¹ wave number, the C-H in plane bend that was also detected at the wavenumber 1388cm⁻¹.and finally, both the isolated and the conjugated C = C variable stretches were also detected.

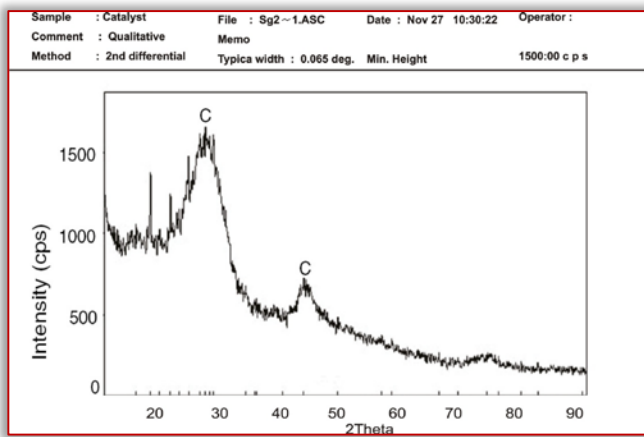


Figure 4: X-ray Diffraction of the Synthesized Bio-Catalyst

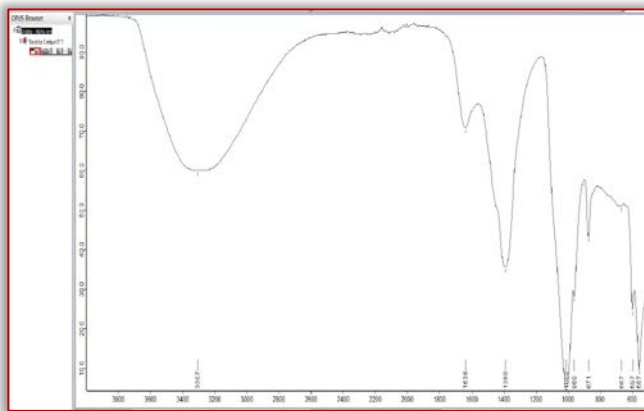


Figure 5: FTIR Spectrum of the Bio-Catalyst

— Bio - Catalyst Recyclability

The strength of the synthesized bio-catalyst was determined through the number of cycles it was used to catalyse trans-esterification reaction. The recyclability takes advantage of the heterogeneous nature of the catalyst which can be easily re-filtrated and reused. As shown in Figure 6, after 4 cycles of trans-esterification, the yield was still 78.40%.

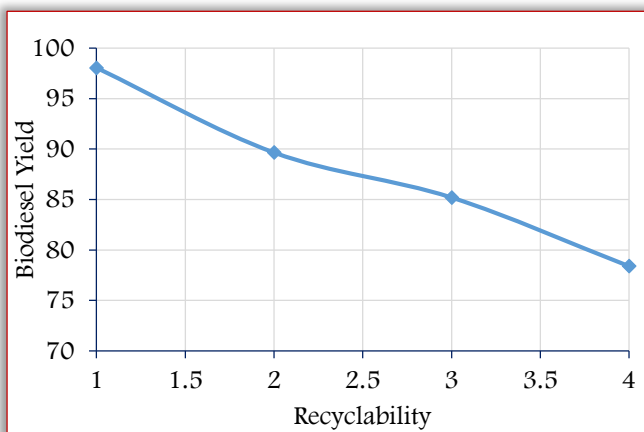


Figure 6: Recyclability profile of Bio-catalyst

The cycled reaction was carried out without further re-calcination of the used bio-catalyst. Obadiah et al., 2012 recorded 83.7% biodiesel yield after 5 cycles while Madhu et al., 2016, recalined the used bio-catalyst and obtained 84% biodiesel yield after 5

cycles and a closely 83% biodiesel yield without further recalcination after 5 cycles. It can be surmised that with re-calcination, catalytic sites are further activated with improved biodiesel yield. The decline in the yield of biodiesel as the catalyst was recovered is due to catalyst loss as well as reduced catalytic activity (Sani et al., 2014).

CONCLUSION

In this study, animal derived biomass specifically from bovine species was used as major feedstocks for transesterification heterogeneous catalyst development and was found to synthesize biodiesel with a maximum yield of 95.12 % which was found to be the optimum and a further yield of 78.40 % after the fourth trans-esterification cycle thereby, proving the catalytic activities of the biomass derived catalyst as further supported by the prior analysis the synthesized catalyst was subjected to. The green fuel (biodiesel) produced was found to meet most of the ASTM specifications. As an addition to knowledge, biomass derived catalyst from bovine species of animals has been identified as a promising and competing catalyst for the synthesis of green fuel through trans-esterification process.

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