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Synthesis of Granular Biochar-based Catalyst for Transesterification Reaction

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ABSTRACT

The use of biochar catalyst in the form of granules was proposed in biodiesel production. The biochar was prepared by fast pyrolysis of Leucaena wood. Mixtures of Leucaena-derived biochar, waste glass powder and sodium silicate, were extruded. The influence of biochar/glass ratio (30:70-70:30) and amount of sodium silicate as a binder (50 and 70%wt) on biodiesel conversion was studied. The granular catalyst prepared from 50:50 biochar/glass ratio with 70% wt binder exhibited the best catalytic performance, which corresponded to the highest biodiesel yield of 98%. The reusability of the catalyst was also determined. The granular catalyst could be reused for 8 times with minor degradation. In addition, it could be easily separated from the mixture by simple filtration. The produced biodiesel was tested in accordance with the ASTMD6751/ EN14214 procedure, showing that the tested biodiesel was usable for biodiesel fuel blend stock. When tested on a single cylinder 4-stroke engine, the obtained biodiesel showed a proof of a high-quality product. In summary, the granular catalyst prepared in this work showed good catalyst performance, which could be a potential alternative for feasibility study in large-scale production.

1. INTRODUCTION

The ever-increasing use of fossil fuels gives rise to the unprecedented global environmental problems such as climate change and energy security concerns [1], [2]. The demand for fossil fuels, especially petrodiesel, does not seem to fall in the last decade, leading to the search for replacement fuels. Biofuels, particularly biodiesel, are a viable option with the advantage of eco-friendly production using waste frying oils and emissions are lower than petrodiesel [3]. Biodiesel, as defined by the ASTM D6751, is a mixture of long chain monoalkylic esters from fatty acids. It can be produced from vegetable oils or animal fats and an alcohol, through a transesterification reaction. The most used oils are from rapeseed, soybean, and sunflower. For economic and environmental purposes, used cooking oils are also an excellent source of biodiesel feedstock [4].

During transesterification reaction, triglyceride will be broken down into a mixture of fatty acid methyl esters (FAME). Biodiesel is then obtained from the purification of FAME. In addition, a catalyst is required to accelerate the reaction. The catalysts used in transesterification reaction can be divided into three main types: homogeneous catalysts (alkaline or acid), heterogeneous catalysts, and enzymes. Alkaline catalysts are more favourable to biodiesel production because of their catalytic performance, chemical stability, and reaction rate. Among base catalysts used in biodiesel production, sodium hydroxide (NaOH) and potassium hydroxide (KOH) are more common. These catalysts have several advantages such as high conversion in a short period of time and availability at a low cost. However, homogeneous base catalysts possess some serious drawbacks. Purification and neutralization are required, leading to the use of large volumes of water and energy. Additionally, the catalyst cannot be regenerated. The presence of free fatty acids (FFAs) and water in the oil can contribute to soapstock and emulsions, complicating the separation process. Moreover, soap formation can reduce the amount of initial reactants and decrease the biodiesel yield [5]. To overcome these difficulties, solid heterogeneous catalysts with distinct properties have been introduced and studied. Those catalysts include alkaline earth metal oxides, hydroxides, metal alkoxides, nanocomposites (KOH-CaO-Al_2O_3), nanocatalysts and zeolite [6], [7]. Recently, biochar which is classified as a heterogeneous catalyst has been utilized. It is characterized as being fine-grained with high surface area and large porosity [8]. Biochar can be synthesized by the pyrolysis of various biomass sources such as agricultural residues, organic materials, and municipal solid wastes [9]. Biomass pyrolysis can result in biochar either as the main component or by-product, dependent on the

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pyrolysis rate. A high yield in the range of 30-50 wt% results from slow pyrolysis, while fast pyrolysis usually produces biochar as a by-product with a yield typically between 15-25 wt% [10]. Numerous studies have been conducted, reporting promising results regarding the use of biochar-based catalysts in biodiesel synthesis [11]. For instance, a biochar catalyst derived from giant Leucaena wood was able to produce 94%FAME [12]. Chicken manure-based biochar has also been utilized as a biodiesel catalyst, resulting in a 95%FAME [13]. A biochar catalyst was also prepared from citrus fruit peel, which resulted in a biodiesel yield as high as 96.3% [14]. Biochar is typically used in the form of powder, with a preference for very fine particles [15]-[19]. However, the use of fine-particle biochar has limitations which are the degradations of active sites during the separation, complex recovery, and recycling process [19]. As a result, the economic feasibility of using biochar as a catalyst for large-scale production is limited. To address this, extruded catalysts have been suggested as a potential solution [20]-[23]. These catalysts have good mechanical properties, controlled structure, and can be manufactured in different shapes and porosity levels, making them suitable for the recovery [19].

To the best of our knowledge, there has not been any work on the use of extruded biochar catalysts in biodiesel production. The current work is therefore the first report on the catalytic performance of extruded biochar catalyst. This study aims to investigate granular biochar catalyst extruded using Leucaena biochar derived through a fast pyrolysis process. The influence of catalyst loading, biochar/glass ratio, amount of binder and reaction time on the performance of the catalyst were determined. Finally, reusability of granular catalyst was also studied. The obtained biodiesel is for diesel engines, thus being tested according to ASTM D6751-12.

2. EXPERIMENTS

2.1 Materials and Chemical Agents

The synthesis-grade methanol and solvent used in the study were purchased from Sigma-Aldrich Merck Limited, with a minimum assay of 99% and maximum water content of 0.2%. The feedstock used was commercial palm oil, which consisted of 40.5% palmitic acid, 5.2% stearic acid, 38.7% oleic acid, and 10.7% linoleic acid with a free fatty acid content of approximately 0.3%. Biochar synthesis was carried out using *Leucaena leucocepphala* wood as the feedstock collected from a local sawmill. Its chemical composition was 49.2% cellulose, 21.1% hemicellulose, 28.7% lignin, and 1.0% ash on a dry basis, its elemental composition was 46.60% C, 6.11% H, 0.26% N, and 0.015% S.

2.2 Procedure for Preparing Extruded Granular Catalysts

Leucaena leucocephala biomass feedstock was first air-dried for 1 day and then ground to pass through a 2 mm screen being sieved into grain ranging in size from 0.2 to 0.6 mm. The dried grain was subsequently processed through fast pyrolysis to 600ºC in a fluidized-bed reactor under a nitrogen gas flow. Upon completion of the synthesis, bio-oil, gases, particularly biochar with a yield of 20% were obtained. Elemental analysis indicated that the biochar composed of 74.1% C, 2.9% H, 16.0% O, 0.18% S and 0.7% N. The BET surface area and iodine number of the biochar were 45.0 m2/g and 896.3 mg/g, respectively, with an average pore size of 2.1 nm. The biochar was then chemically activated by soaking it in 7M-KOH solution for 30 min under vigorous stirring at 500 rpm. Afterward, the activated sample was filtered and dried at 105ºC for 1 h. To fabricate the granular catalysts, glass powder derived from crushed borosilicate glass waste was required. The grass powder was passed through a 250-mesh sieve. To prepare catalyst mixtures, glass powder was mixed with activated carbon powder in varying proportions of 30-70wt%. Granular catalysts were then produced using an extrusion method, in which the catalyst mixture was combined with a sodium silicate binder at 50 and 70wt%. The resulting moist catalyst mixtures were extruded into spherical catalysts with a diameter of 5 mm, and then dried at 105ºC for 30 min. Figure 1 Shows the dried catalyst obtained from this process. The surface morphology of catalyst was analyzed using scanning electron microscopy (SEM) on a JEM-2010 instrument). Phase formation was determined using X-ray diffraction (XRD on a D2 phaser instrument. The specific surface area of the catalyst was determined using BET on a TriStar II 3020 instrument. The best biodiesel sample obtained under these conditions was analyzed using Gas Chromatography with Frame Ionization detection (GC-FID) on a Agilent (GC-Agro instrument from the United States of America.

Fig. 1. The granular catalyst.

2.3 Procedure for Transesterification Reaction

The transesterification process was performed in a 500 mL borosilicate glass beaker reactor, with a constant temperature of 65ºC and a vigorous stir at 750 rpm. The catalyst dosage was varied from 1 to 5%wt, and the reaction temperature was varied from 10 to 250 min. The methanol/oil molar ratio was fixed at 6:1. After the completion of the reaction, the reaction product was left for 1 h before and the obtained biodiesel was separated and washed using distilled water. Biodiesel yield was calculated according to mass ratio of biodiesel obtained and oil used. To test the reusability of the catalyst with the best catalytic performance, the catalyst used in the preliminary biodiesel preparation was filtered and washed thoroughly with methanol, dried for 24 hours, and then used in the same batch transesterification process at the optimized conditions.

2.4 Determination of Fuel Properties and Performance Tests of Diesel Engine with Biodiesel Blends

The produced biodiesel was characterized according to the procedure provided by ASTMD6751/ EN14214 standard. The biodiesel blends were also trialled on a single cylinder 4-stroke engine using commercial diesel fuel and the blends of biodiesel (B5, B10, and B20). The engine speed was varied from 1,200 rpm to 1,800 rpm in order to determine brake torque, brake power, and brake specific fuel consumption.

3. RESULTS AND DISCUSSION

3.1 Surface Analysis and XRD Pattern of the Granular Catalyst

Figure 2 shows the SEM analysis results. The finely-ground glass powder had irregular particle shapes, with sizes ranging from 2 to 20 µm. The Leucaena-derived biochar had a typical feature of wood-derived carbons, exhibiting a porous structure with aligned micro-channels. After KOH activation following fast pyrolysis, significant changes in micromorphology were observed. The surface morphology of the extruded catalysts was also investigated, revealing a strong bond on the mixtures and a homogeneous surface with well-distributed biochar particle.

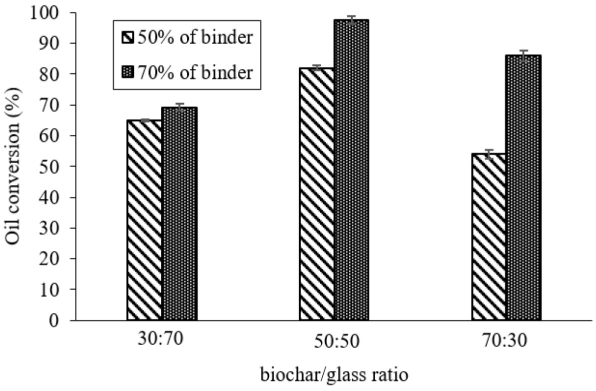

Fig. 5. Effect of binder content on biodiesel conversion (catalyst content= 3 %wt).

Figure 3 presents the XRD patterns of the granular catalysts. The broadened feature observed was associated with the amorphous nature of glass. As the proportion of biochar increased, the crystalline phases SiO_2 and $CaCO_3$ dominated.

3.2 The Effect of Catalyst Loading on Biodiesel Yield

Biodiesel conversion is strongly influenced by reaction variables particularly catalyst loading and reaction time. In this work, different amounts of the catalyst ranging from 1 to 5%wt were used. The transesterification reaction was carried out at 65ºC for 1 h using methanol/oil molar ratio of 6:1. Effect of the catalyst loading on biodiesel yield is presented in Figure 4. The biodiesel yield was found to steadily increase with the catalyst loading. It reached a maximum when the amount of the granular catalyst was 3%wt in all biochar/glass ratios. The highest biodiesel conversion was 97.57%, exclusively obtained on the 50:50 catalyst. The biodiesel yield, however, decreased when the catalyst loading was beyond 3wt%. The decrease in the yield of biodiesel could be attributed to an overdose of catalyst that was favorable for agglomeration on the wall of the reactor which was observed in the current work. Nevertheless, high power stirring was suggested for this issue [24].

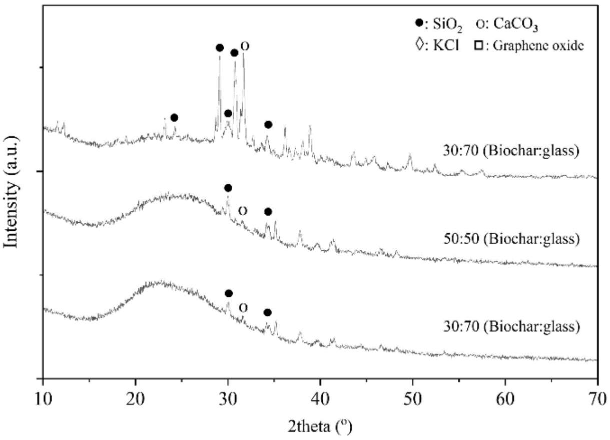

Fig. 3. XRD pattern of granular catalyst with different biochar/glass ratios at 50%wt of binder.

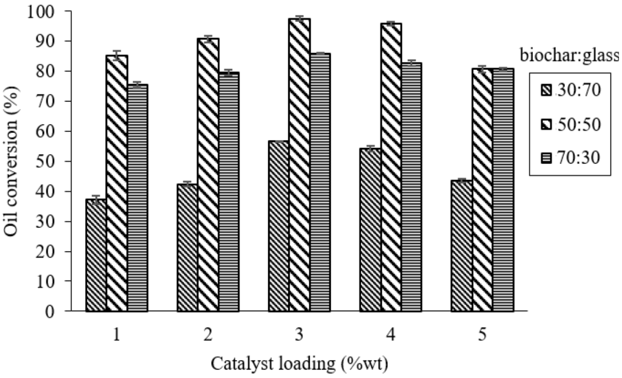

Fig. 4. Effect of catalyst loading and biochar/glass ratio with 70%wt of binder on biodiesel conversion.

3.3 The Effect of the Biochar/Glass Ratio on Biodiesel Yield

The influence of biochar/glass on biodiesel conversion was noticeable, as shown in Figure 4. The oil conversion increased and then decreased with increasing glass content. This trend was observed in all catalyst loadings. Regarding the effect of biochar/glass ratio, the catalysts containing 50% glass were observed to be more effective than any other. This could be attributable to an increase in specific surface area resulting from an optimized arrangement between biochar and glass particles. Compared to the 50:50 catalysts, however, the 70:30 catalysts generated a lower biodiesel yield, which is possibly due to an undesirable catalyst that adversely affected the mass transfer between the reactants.

3.4 The Effect of the Amount of Binder on Biodiesel Yield

As clearly seen in Figure 5, the biodiesel yield significantly increased with the amount of the binder on all the biochar/glass ratios, indicating that the sodium silicate binder has a positive effect on catalytic activity of the granular catalyst. This finding is consistent with the previous studies in which sodium silicate-derived catalysts were highly effective for transesterification reactions [25], [26].

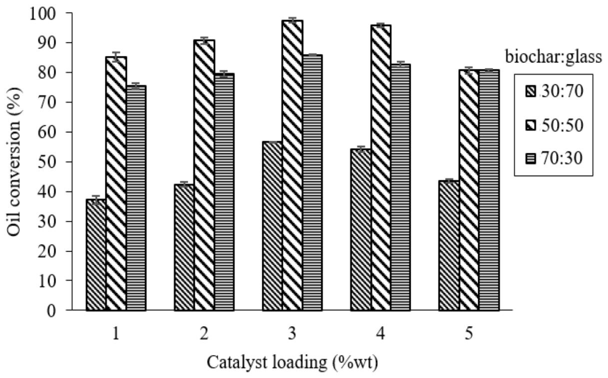

Fig. 4. Effect of catalyst loading and biochar/glass ratio with 70%wt of binder on biodiesel conversion.

3.5 The Effect of the Reaction Time on Biodiesel Yield

The effect of reaction time was investigated on the 50:50 biochar/glass catalyst with 70% binder. The transesterification reaction was carried out at a constant temperature of 65ºC using 3%wt catalyst. As observed in Figure 6, the mixture turned clear yellow within 30 min, generating a biodiesel yield of 93.23%, indicating a rapid reaction in the initial stage of transesterification. The yield reached the highest value 97.68% after a 40 min reaction, as seen in Figure 7. Subsequently, it slightly decreased when the reaction time was extended beyond 120 min.

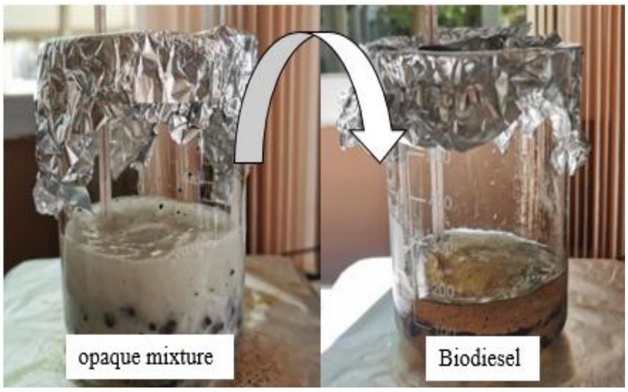

Fig. 6. Progression of the transesterification reaction using the 50:50 biochar/glass catalyst with 70% binder.

3.6 Reusability of Granular Catalyst

This indicates high stability of the granular catalysts prepared in this work. The biodiesel yield of the catalyst with the best catalytic performance under the optimum condition was also investigated for its reusability. As shown in Figure 8, a biodiesel yield of 97% was achieved using fresh catalyst. A slight decrease in yield was observed after the second and third runs, but the catalyst still maintained a high level of activity. However, the biodiesel yield dropped significantly to 91% after the seventh run and a substantial reduction was observed after the eighth run where the yield was lower than 90%. This indicates that the granular catalysts prepared in this work have high stability and can be effectively reused for several cycles before the catalytic activity begins to decline.

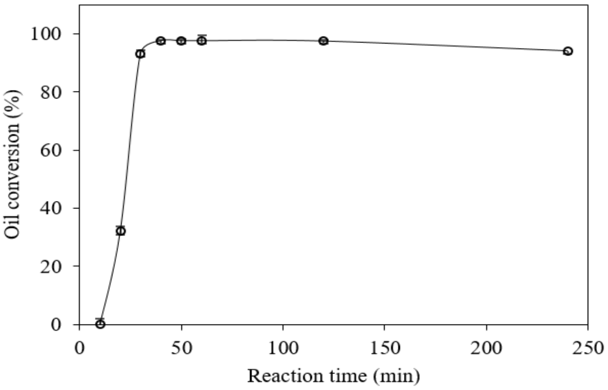

Fig. 7. Biodiesel conversion and reaction time (biochar/glass ratio of 50:50 with 70%wt binder).

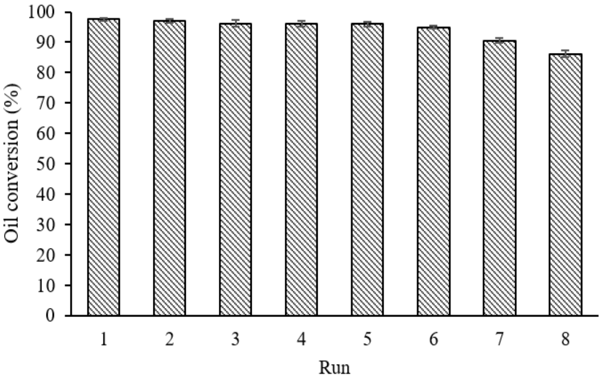

Fig. 8. Reusability of the biochar/glass=50:50 catalyst with 70% binder.

3.7 Comprehensive Analysis of Fuel Properties of the Synthesized Biodiesel

The best biodiesel prepared in this work was tested for all the biodiesel properties according to ASTMD6751/ EN14214. The results revealed that the density, viscosity at 40ºC and acid value were 890 kg/m^3, 4.51 mm^2/s and 0.42 mgKOH/g, respectively. In addition, the flash point and calorific value were 170ºC and 39.4 MJ/g. This shows that the synthesized biodiesel meets the standards suggested by ASTMD6751/ EN14214. Tested by GC-FID, the biodiesel obtained from the best condition had methyl ester as high as 99.7%. The fatty acid composition was analyzed, which is given in Figure 9. The biodiesel consisted primarily of palmitic acid (16:0) oleic acid (C18:1) linoleic acid steric acid (18:0) hepatodecaenoic acid (C17:0) arakidic acid (C20:0) and linoleic acid (C18:2).

3.8 Engine Performance

3.8.1 Brake torque (BT)

The variations of brake torque with engine speed for B5, B10, B20, diesel fuel and pure biodiesel fuel is shown in Figure 10. Regarding biodiesel blends, brake torque increased with an increase in proportion of biodiesel. Compared with the commercial diesel fuel, the obtained biodiesel exhibited higher BT. In addition, maximum brake torque (MBT) was found at the engine speed of 1600 rpm. BT was observed to reduce when the engine speed was increased to 1800 rpm. The reduction in BT was due to the low heating value of biodiesel [27].

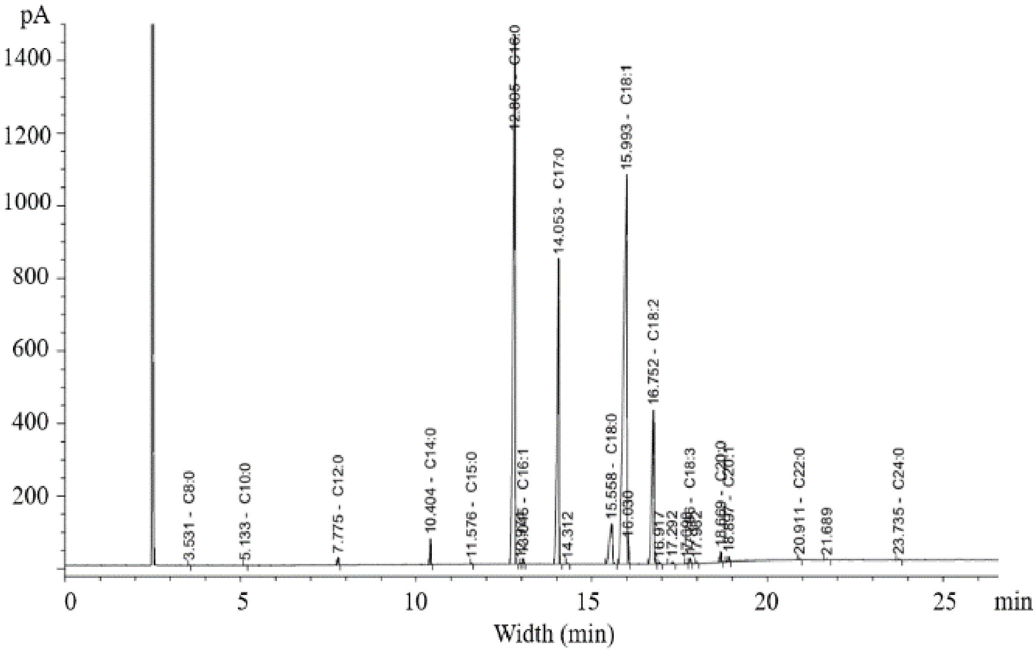

Fig. 9. GC chromatogram of the synthesized biodiesel.

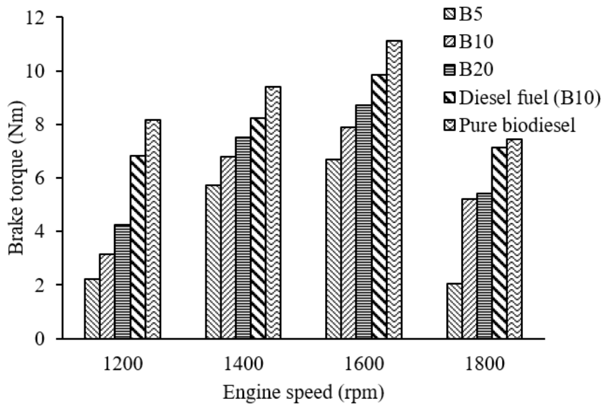

Fig. 10. Change of brake torque at different engine speeds and different fuels.

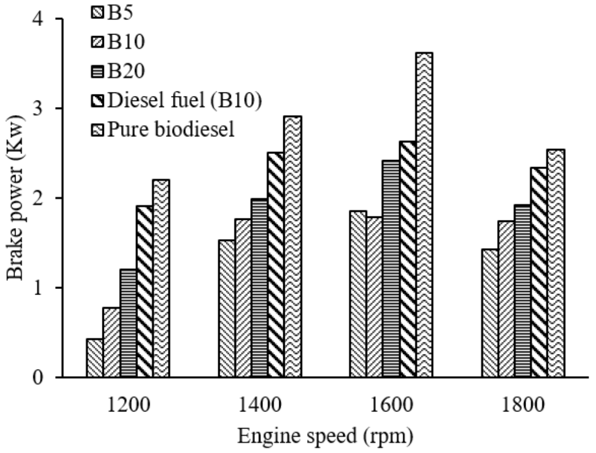

Fig. 11. Change of brake power at different engine speeds and different fuels.

3.8.2 Brake power (BP)

As shown in Figure 11, brake power increased with the percentage of biodiesel. Pure biodiesel exhibited higher brake power compared to commercial diesel. Similar to the brake thermal efficiency (BTE) results, brake power primarily increased with engine speed and reached its peak value at 1600 rpm before decreasing at 1800 rpm. This trend is consistent with the findings reported by Noorollahi *et al.* [24].

3.8.3 Brake specific fuel consumption (BSFC)

The variations of BSFC with engine load for B5, B10, B20, diesel fuel, and pure biodiesel are shown in Figure 12. It was found that BSFC increased with the percentage of biodiesel in the fuel blends [25].

Additionally, the biodiesel blends exhibited lower BSFC than diesel fuel and pure biodiesel in all engine speeds, indicating favorable use of biodiesel blends. Similar to BT and BP, a similar trend was observed in BSFC where it increased and reached the highest at 1600 rpm and then decreased with engine speed.

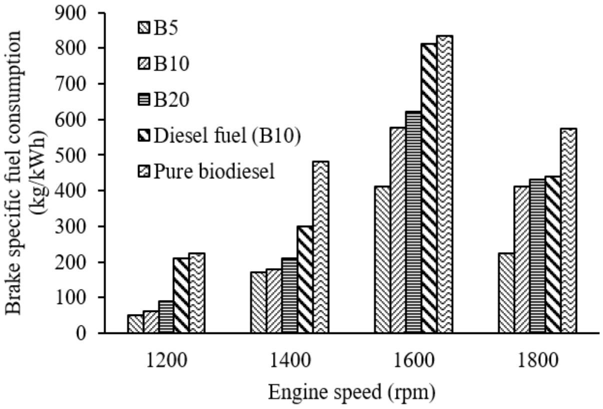

Fig. 12. Change of brake specific fuel consumption at different engine speeds and different fuels.

4. CONCLUSION

In summary, the study demonstrated the potential of using Leucaena biochar and waste glass as raw materials to produce granular catalysts for biodiesel production. The use of sodium silicate binder was found to have a positive effect on the catalytic activity of the granular catalysts. The optimum condition was found to be the use of a 50:50 biochar/glass ratio with 70%wt binder, which resulted in a biodiesel yield of 98%. The catalyst also showed good reusability, with minor degradation observed after eight cycles. In addition, it is noticed that the granular catalysts are advisable owing to their simple separation process via conventional filtration. The obtained biodiesel met the standard suggested by ASTMD6751/ EN14214. When tested on a single cylinder 4-stroke engine at 1600 rpm, the obtained biodiesel showed the maximum brake torque and the maximum brake power at 11.13 NM and 3.62 kW, respectively. This suggests that the granular catalyst prepared in this work showed good catalyst performance, which could be an economically viable approach for feasibility study in large-scale production.

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