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CONVERTING WASTE CHICKEN BONES INTO HETEROGENEOUS CATALYST FOR BIODIESEL SYNTHESIS FROM WASTE COOKING OIL

(Pertukaran Sisa Tulang Ayam kepada Mangkin Hetrogen Bagi Penghasilan Biodiesel dari Minyak Masak Terpakai)

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Abstract

Depletion of non-renewable energy sources such as petroleum has triggered researchers to design a green catalyst for the synthesis of biodiesel. Thus, in this study, chicken bones were subjected to calcination-hydration-dehydration treatment to obtain a catalyst of high activity for the production of biodiesel from waste cooking oil (WCO). The physicochemical properties of the synthesized catalysts were analyzed by Fourier Transform Infrared Spectroscopy (FTIR) and N_2 adsorption-desorption. The catalyst prepared via calcination-hydration-dehydration treatment (C900-600) exhibited improved surface area (SBET, 71.14 m^2g^{-1}) and BJH pore diameter of 31.03 nm, in comparison with chicken bone derived catalyst calcined at 900 ˚C (C900) and commercial CaO with 31.72 m^2g^{-1}, 29.35 nm and 2.21 m^2g^{-1}, 15.98 nm, respectively. After 45 min, the activity of 5 wt.% catalysts in the transesterification of WCO with 1:15 oil to methanol ratio at 65 °C, increased as the following trend: C900-600 (92.15%) > C900 (80.63%) > commercial CaO (73.30%). C900-600 was truly a stable and reused catalyst, sustaining its activity of 87.00% even after five consecutive cycles. This research undoubtedly promises a cheap utilization of waste chicken bones for the conversion of WCO into renewable energy sources.

Keywords: waste chicken bones, biodiesel, waste cooking oil, acid value, reusability

Abstrak

Kekurangan sumber tenaga yang tidak boleh diperbaharui seperti petroleum telah mendorong para penyelidik untuk menghasilkan mangkin hijau untuk sintesis biodiesel. Maka, dalam penyelidikan ini, sisa tulang ayam telah dirawat menggunakan kalsinasi-hidrasi-dehidrasi untuk menghasilkan mangkin yang aktif dalam penghasilan biodiesel dari minyak masak terpakai. Ciri fizikokimia mangkin telah dianalisis menggunakan Spektroskopi Inframerah Transformasi Fourier (FTIR) dan N_2 penjerapan-penyahjerapan. Mangkin yang disediakan menggunakan rawatan kalsinasi-hidrasi-dehidrasi (C900-600)menunjukkan peningkatan luas permukaan BET (SBET, 71.14 m^2g^{-1}) dan diameter liang BJH; 31.03 nm, dibandingkan dengan mangkin dari sisa tulang ayam yang dikalsinasi pada suhu 900 ˚C (C900) dan CaO komersial dengan masing-masing merekodkan 31.72 m^2g^{-1}, 29.35 nm dan 2.21 m^2g^{-1}, 15.98 nm. Selepas 45 min, aktiviti mangkin (5% berat) dalam transesterifikasi minyak masak terpakai dengan nisbah minyak dan metanol 1:15 pada 65 °C meningkat berdasarkan tren berikut: C900-600 (92.15%)> C900 (80.63%)> CaO komersial (73.30%). C900-600 merupakan mangkin yang stabil dan boleh digunapakai, mengekalkan aktivitinya sebanyak 87.00% walaupun setelah lima kekerapan dikitar semula. Penyelidikan ini pasti menjanjikan penggunaan sisa tulang ayam yang murah untuk penukaran minyak masak terpakai menjadi sumber tenaga yang boleh diperbaharui.

Kata kunci: sisa tulang ayam, biodiesel, minyak masak terpakai, nilai asid, kitar semula

Introduction

Growing depletion of non-renewable reserves, sickening environmental issues, and health concerns arising from petroleum-based diesel has positioned biodiesel as an alternative green energy fuel owing to its remarkable properties such as cleaner emission, non-hazard, and offers rapid biodegradability [1]. In biodiesel synthesis, feedstock contributes to 70% to 95% of the overall production cost [2]. This worrying situation draws the attention of global researches to utilise relatively cheap and easily available non-edible oil such as waste cooking oil (WCO). Asian countries, including Malaysia, produce nearly 40,000 tonnes of WCO per annum [3] and it is usually collected from households, fried food outlets, and restaurants [4]. Biodiesel price could be significantly reduced to 50% upon converting WCO into biodiesel [5] and this appears as the best practice to recycle WCO, which will otherwise end-up in palatable water and pose a substantial threat to the environment.

Nowadays, turning waste materials into a catalyst is the most viable way to address the production of biodiesel at a commercial scale. Solid base catalysts involving natural wastes, such as egg shell [6-9], chicken bone [2,10], fusion chicken and fish bone [11], ostrich bone [12], and scallop shell [13], are some of the potential CaO sources for transesterification reaction. For example, Borah et al. [9] have synthesised biodiesel from WCO using Zn doped egg shell derived CaO nanocatalyst that registered a maximum FAME yield of 96.74% after 4 h reaction. Nevertheless, great loss in the activity during the fifth recycle (64.49%) was reported, probably resulting from the leached active Zn species into the reaction medium. Similarly, a longer reaction duration of 5 h was required to reach 100% biodiesel yield over egg shell derived catalyst in the transesterification of WCO [6]. In another work, Dehkordi and Ghasemi [14] demonstrated excellent activity of ZrO_2 CaO in catalysing WCO into 92.1% biodiesel at only 2 h. However, the catalysis requires large catalyst dosage of 10 wt.% and 30 mole excess of methanol. Waste oyster shell employed as a sustainable base heterogeneous catalyst, yielded 87.3% of FAME after 3 h reaction in the presence of 9:1 methanol/WCO molar ratio and 6 wt.% catalyst [15]. However, no reusability study was reported in this work.

Malaysians consume nearly 50 kg of chicken per capita per year, making it rank first and third in Asia and in the world, respectively [16]. With this huge production, large numbers of chicken by-products such as chicken bone are generated and dumped in landfills, ending in poor solid waste management. Chicken bones are made up of organic and inorganic components with 65% (hydroxyapatite), 25 wt.% of protein, and the remaining % of water [17]. They have a high potential to be converted into a value-added product such as CaO based catalyst. However, very scarce studies were focused on chicken bone as a catalyst for biodiesel synthesis. For example, in 2015, Farooq et al. [2] discovered the efficiency of chicken bone as a base catalyst in the triglyceride conversion of WCO and registered a maximum biodiesel yield of 89.33% at optimised reaction conditions of 4 h, methanol to oil molar ratio of 15:1, reaction temperature of 65 °C, and 5 wt.% of catalyst loading.

The performance of Fe impregnated CaO derived chicken bone catalyst was documented by Hantoro Satraidi et al. [18]. The catalyst afforded a biodiesel yield of 22% after 3 h reaction time, with 6:1 of methanol:WCO. In a recent study by AlSharifi and Znad [10], lithium based chicken bone composite converted 96.6% of canola oil into biodiesel after 3 h. In this regard, the world’s biodiesel present demand has triggered the search for an active heterogeneous catalyst that offers high yield and excellent properties. As such, in this present work, waste chicken bone was subjected to the calcination-hydration-dehydration technique to enhance the surface area that apparently affects the biodiesel yield. Little literature has shown the importance of the calcination-hydration-dehydration route. For example, CaO derived waste clamshell prepared via calcination-hydration-dehydration treatment resulted in an enhanced surface area and basicity that greatly affected the biodiesel production from WCO [19]. Similarly, Niju and co-workers [7] reported an improved catalysis of egg shell based CaO catalyst synthesised by the calcination-hydration-dehydration route in the biodiesel synthesis (94.52%) from waste frying oil compared with commercial CaO that gave only 67.57%.

Hence, a lack of publication on the synthesis of CaO derived waste chicken bone catalyst by calcination-hydration-dehydration has encouraged us to prepare a preliminary characterisation of the catalyst using Fourier Transform Infrared Spectroscopy (FTIR) and N_2 sorption analysis. The catalysts were further evaluated in the synthesis of biodiesel from WCO and promised a green conversion of waste materials into value-added products.

Materials and Methods

Raw materials and reagents

The chicken bones and waste cooking oil (WCO) were collected from the cafeteria in Universiti Teknologi MARA (UiTM) Perlis, Malaysia. The chemicals used were CaO (HmbG), 99% anhydrous methanol (HmbG), 98% H_2SO_4 (QREC), sodium sulphate, potassium hydroxide (HmbG chemical), toluene (QREC Asia), phenolphthalein indicator (HmbG), and *n*-hexane (QREC). All the chemicals were used as procured.

Preparation of chicken bone

300 g of waste chicken bones were weighed and boiled in water for 1 h to remove adhering excess tissue and fats. Later, the boiled bones were thoroughly washed with copious amount of water to eliminate meat and other soft tissues [20], and allowed to dry at 110 ℃ for 2 h. The dried bones were crushed into small pieces and subsequently ground using a hammer mill into powder of < 2 mm particle size [21]. Finally, the sample was stored in a closed container to prevent moisture content changes.

Preparation of C900 and C900-600 catalyst

100 g of the powdered sample was placed in a crucible and heated at 900 ℃ for 4 h to obtain hydroxyapatites (HAP). The sample was designated as C900. To prepare C900-600, C900 was refluxed in water at 60 ℃ for 6 h and subsequently filtered and oven dried at 120 ℃ for 24 h [22]. Later, calcination was performed at 600 ℃ for 3 h. This stage allows the conversion of the hydroxide form into an oxide form [22]. For comparison, the commercial CaO catalyst was used.

Catalyst characterisation

The FTIR spectra of the catalysts were collected on a Thermo Electron Corporation Nicolet 380 spectrophotometer at wavenumber ranging from 4000 to 400 cm^{-1} with a resolution of 5 cm^{-1}. In prior analysis, the pellet was prepared with 1:10 w/w KBr:Sample. The surface area (S_{BET}) and BJH pore size distribution were obtained by means of N_2 adsorption-desorption (Micromeritics Instrument Corporation model ASAP 2000) after degassing for 12 h.

Treatment of WCO

The collected WCO was filtered to remove food residues and washed with hot distilled water. Later, it was treated with anhydrous Na_2SO_4 to remove any water content. The molecular weight of fatty acid in oil was determined by GC/MS (Agilent Technologies 6980N).

Acid value (AV) and free fatty acid (FFA) determination

Acid number refers to the quantity of KOH required to titrate 1 g of FFA in sample to its neutral point [23]. The acid number was evaluated according to standard testing procedure ASTM D664 as reported by Corro et al. [24] Briefly, 3 g of WCO was dissolved in 150 mL containing an equal volume of toluene and isopropanol. Then, 1to 2 drops of phenolphthalein were added into the prepared solution and titrated with 0.1 N KOH solution until the colour changed from light yellow to pink. The AV and FFA for feedstock and esterification product were determined as in equations (1) and (2), respectively.

$$Acid\ value\ (AV) = 56.1\ x\ \frac{M(A-B)}{W} \tag{1}$$

$$FFA\ (\%\ as\ palmitic) = \frac{A\ x\ M\ x\ 25.6}{W} \tag{2}$$

where M - KOH Concentration (N), A – Volume of KOH volume required to reach the neutral point (mL), B - the volume corresponding to the blank titration (mL), and W - Sample weight (g).

Catalytic transesterification and biodiesel production

A two-step transesterification reaction consisting of acid transesterification to reduce the FFA content followed by base transesterification was carried out. Acid transesterification was done following the method by Tan et al. [25], however, with slight modification. In brief, WCO was heated to 110 ℃ overnight to reduce the viscosity. In a 250 mL flask, 50 mL of methanol and 0.1 mL concentrated H_2SO_4 were stirred; and later, 100 mL WCO (preheated at 65 ℃) was transferred and the mixture was stirred at 65 ℃ for 1 h.

Base transesterification and FAME analysis

In a typical run, 5 wt.% of catalyst was added into a three-neck round bottom flask containing 1:15 Oil/MeOH and equipped with a condenser. The reaction was performed at 65 ℃ with continuous stirring (250 rpm) for 1 h. Afterwards, the pre-treated WCO was transferred into the round bottom flask and the reaction was continued for 45 min. Later, the catalyst was filtered off and the reaction mixture containing biodiesel and glycerol at the upper and lower layer, respectively, was allowed to stand in a separating funnel for 24 h, with prior washing using hot distilled water. The water content in biodiesel phase was removed with sodium sulphate. Product composition in biodiesel was identified by GC/MS (Agilent Technologies 6890N, carrier gas: He) with the following temperature program: initial oven temperature of 383 K; ramp of 283 K/min, holding time of 2 min; intermediate temperature of 473 K; final ramp of 278 K/min; and final temperature of 553 K for 9 min. The % conversion was calculated based on equation (3) [24].

$$\%\, Conversion = \frac{AV1-AV2}{AV1}\ x\ 100\% \tag{3}$$

where, *AV1*–acid value of raw materials, *AV2*–acid value of transesterification product

Catalyst reusability

Upon completion of each experiment, the spent catalyst was separated from the reaction mixture by centrifugation and washed with *n*-hexane and subsequently subjected to heating at 600 ˚C for 2 h [2]. The recovered catalyst was reused in the transesterification reaction at similar reaction conditions for five consecutive runs.

Results and Discussion

FTIR spectral studies

The FTIR spectra of uncalcined chicken bone, C900, and C900-600 are compared in Figure 1. Broad band at ~ 3419 to 3438 cm^{-1} for all the samples represents the stretching vibration of the hydroxyl group (OH) on the surface, which is often associated with the -OH group of $Ca(OH)_2$ [25].

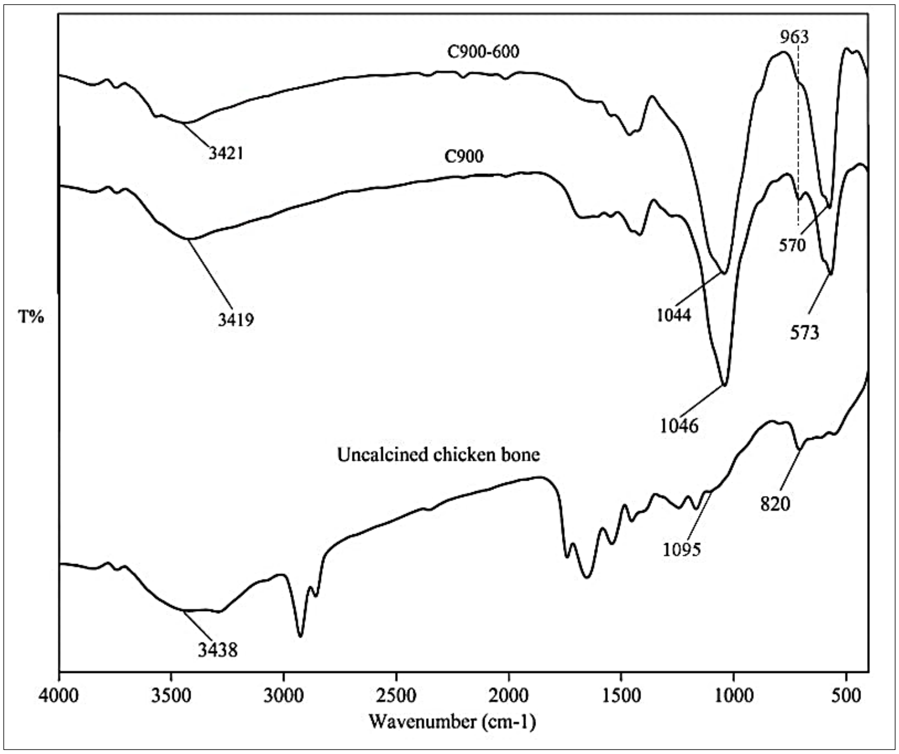

Figure 1. FTIR spectra of the samples

However, the interesting observation to be noted from Figure 1 is the existence of sharp peaks at 1044 to 1095 cm^{-1} and 570 to 573 cm^{-1} for both C900 and C900-600 that evidenced the presence of PO_4^{3-} group, and reflects the O-P-O bonds in calcium phosphate [21]. In addition, the shoulder peak at ~963 cm^{-1} for C900 and C900-600 belongs to PO_4^{3-} that resembles asymmetric stretching vibrations of P–O [24]. According to Corro et al. [24], the peaks at ~3400, 1036 to 1091, and 956 cm^{-1} evidenced the characteristic peaks of hydroxyapatite, which are undoubtedly shown by the calcined samples. Notably, the peaks are more intense for C900-600 compared with C900. This resembles the effectiveness of hydration-dehydration treatment on the chicken bone sample. On the contrary, the IR pattern of uncalcined chicken bone is almost similar to the spectra reported for uncalcined egg shell by Tan et al. [25]. In addition, the shoulder band detected at 820 cm^{-1} for the uncalcined chicken bone is due to CO_3^{2-} [21] and similar vibrations appeared at 1400 to 1500cm^{-1} for all the samples [26].

Textural properties

The S_{BET} and BJH pore diameter of C900-600, C900, and commercial CaO measured from N_2 adsorption-desorption analysis are summarised in Table 1.

Table 1. S_{BET} and BJH pore diameter of C900-600, C900, and commercial CaO

Sample	S_{BET} (m^2/g)	BJH pore diameter (nm)
C900-600	71.14	31.03
C900	31.72	29.35
Commercial CaO	2.21	15.98

According to IUPAC classification, the pore diameters of the synthesised catalysts are classified as mesopores (2 to 50 nm) and accordingly, mesoporous material is most suitable to be used as a catalyst since it allows the diffusion of larger molecules or reactants compared with micropores. It is worth reporting the higher S_{BET} and BJH pore diameter of C900-600 and C900 catalysts than the commercial CaO. To add, the synthesised catalysts in the present work showed greater S_{BET} than the lithium-based chicken bone catalyst prepared by AlSharfi and Znad [10]. The hydration-dehydration treatment significantly improved the surface area and pore diameter of the C900-600 catalyst as shown in Table 1. During dehydration, the removal of attached water molecules from the catalyst lattice might have led to a higher surface area by breaking the crystallite into smaller sizes.

N_2 adsorption-desorption isotherms of the catalysts are compared in Figure 2. Both C900 and C900-600 exhibited Type IV characteristics of mesoporous materials with H1 hysteresis loops, often associated with uniform pores. On contrary, commercial CaO showed a sigmoid type with almost no closure at $P/P_o = 0$ [27].

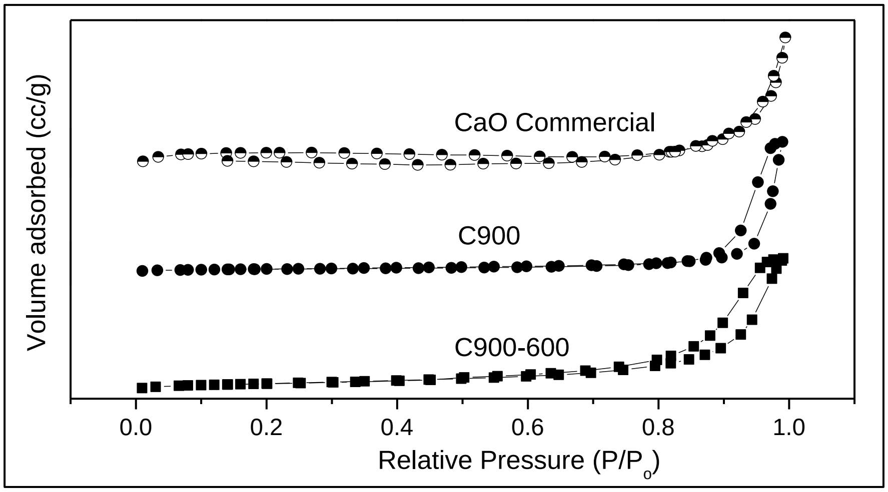

Figure 2. The N_2 adsorption-desorption isotherms of the catalysts

This characteristic is related with materials that have tendencies to swell [27]. Referring Figure 2, the broadening of the hysteresis loop of C900-600 compared to C900 is attributed to its larger pore diameter as evidenced in Table 1.

Production of biodiesel from WCO

The transesterification activity of the catalyst is compared in Table 2. C900-600 gave the maximum conversion of FFA to FAME (92.15%) compared with other catalysts.

Table 2. The activity of the catalysts in the transesterification of WCO

Sample	Acid Value (mg KOH/g)		Biodiesel Conversion (%)
	Initial	After	
C900-600	1.91	0.15	92.15
C900	1.91	0.37	80.63
Commercial CaO	1.91	0.51	73.30

Reaction conditions: Catalyst dosage: 5 wt.%; temperature: 65 °C; Methanol: WCO molar ratio: 15:1; time: 45 min

Higher catalysis of C900-600 can be inferred to its larger surface area and wider BJH pore diameter. Heterogeneous catalysis works on the principle of adsorption of reactants on surface. Thus, theoretically, larger surface area provides greater adsorption sites for the reactants to adsorb and react, which later influences the rate of reaction. In addition, the wider pore diameter of C900-600 eased the diffusion and accessibility of WCO and methanol deep into the active sites, actively participating in the reaction. Higher intensity of functional groups as evidenced through IR studies, have also aided the activity of C900-600 compared with C900. In addition, based on ASTM D-6751 and EN 14214 standards of biodiesel, the transesterification products registered using C900-600 and C900 catalysts were less than 0.5 mg KOH/g, which indicated the perfect fuel for automobiles [28].

Composition of biodiesel

The composition of FAME catalysed by C900-600 is shown in Figure 3 and summarised in Table 3.

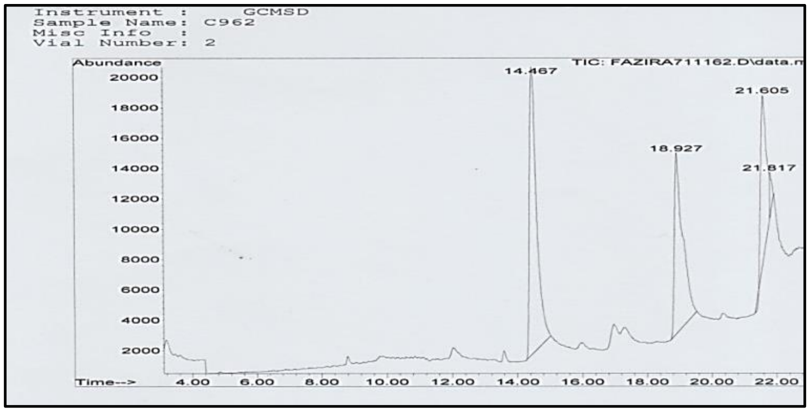

Figure 3. GC-MS profile of the biodiesel synthesised by C900-600

The major FAMEs identified in the synthesised biodiesel are as listed in Table 3. The findings are in accordance with Girish et al. [29], who evaluated the activity of clam shell catalyst in the conversion of waste frying oil.

Table 3. Methyl esters composition of the synthesised biodiesel

Retention Time (min)	Name of Compound	Molecular Formula	Molecular Weight (g/mol)
14.47	Dodecanoic acid methyl ester	$C_{13}H_{26}O_2$	214
18.93	10-Octadecadienoic acid methyl ester	$C_{19}H_{36}O_2$	296
21.61	Docosanoic acid methyl ester	$C_{23}H_{46}O_2$	354

Reusability of C900-600

The key characteristic of a heterogeneous catalyst is the ability to be reused with insignificant loss during the reusability. Figure 4 resembles the trend in the activity of C900-600 after five consecutive runs.

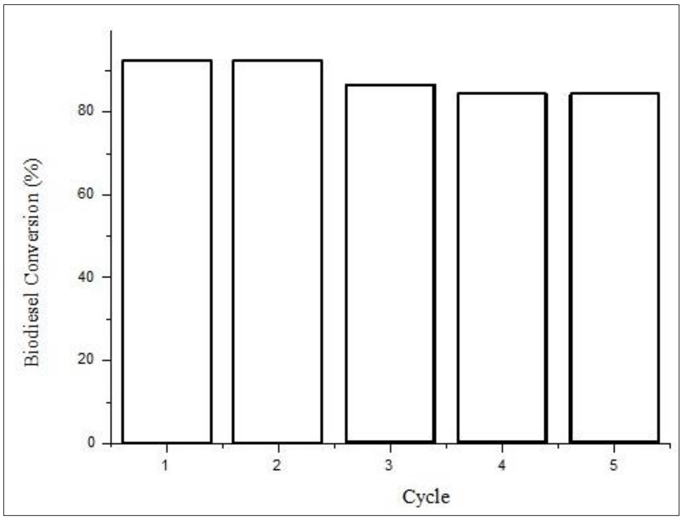

Figure 4. Regeneration activity of C900-600

C900-600 retained its activity up to the second cycle (92.12%), which indicated the high stability of the C900-600 catalyst. However, a slight reduction in the activity was experienced during the third (88.20%) and 87.00% for the subsequent runs. The lowering in the biodiesel conversion could be due to the attachment of unreacted reactants or products at the active sites that might have blocked the active sites and hindered the participation of the catalyst. The findings from this present work correlates well with several studies [7, 22] that demonstrated that the catalyst prepared from calcination-hydration-dehydration treatment can be reused for several runs compared with without treatment.

Conclusion

Heterogeneous CaO catalyst derived from waste chicken bone was synthesised using an improved hydration-dehydration method to cater to the drawbacks of lower surface area and poor activity of reported catalysts for biodiesel synthesis. Enhanced surface area and improved textural properties of C900-600 were the deciding factors for the better catalysis of C900-600 compared with C900. The preliminary characterisation proved the existence of hydroxyapatite, which are the active species for transesterification. However, the strength of basicity of C900-600 and C900 needs to be studied in the future for better insight into the effect of the hydration-dehydration treatment on the catalysis. To conclude, this research has successfully produced an active and recyclable catalyst for the sustainable production of biofuel within 45 min, employing 5 wt.% catalyst at 65 °C with insignificant loss of biodiesel after five regeneration studies, comparable to reported literature.

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