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Waste ostrich- and chicken-eggshells as heterogeneous base catalyst for biodiesel production from used cooking oil: Catalyst characterization and biodiesel yield performance

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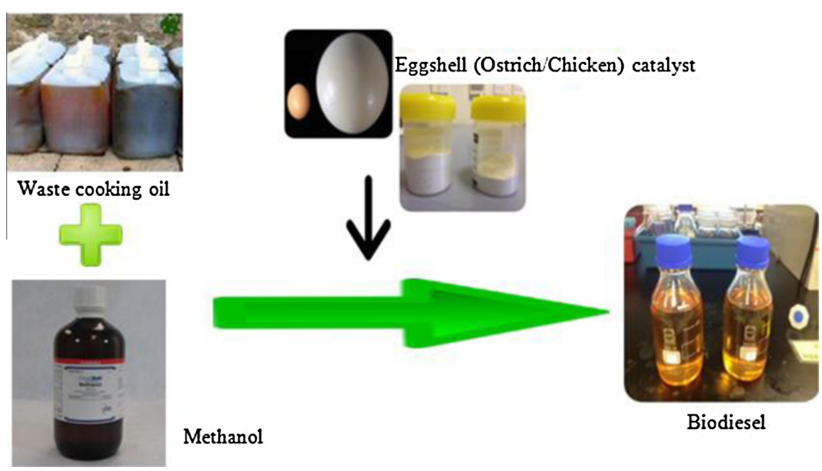
HIGHLIGHTS

- Ostrich-eggshell waste as the raw material to synthesize calcium oxide heterogeneous base catalyst.
- Biofuel, biodiesel is produced from waste cooking oil via two steps transesterification reaction.
- Biodiesel yield was 96% and 94% for calcined ostrich- and calcined chicken-eggshells under same optima reaction condition.
- Biodiesel produced from calcined ostrich-eggshell and calcined chicken-eggshell satisfied ASTM D6751.

GRAPHICAL ABSTRACT



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ABSTRACT

The primary goal of this paper is to investigate the catalyst characterization and biodiesel yield of a biodiesel converted from a used cooking oil source via heterogeneous catalysts derived from very rare type of eggshell: ostrich-eggshell (ostrich-eggshell derived CaO). It also aims to compare the performance of CaO catalyst derived from both waste ostrich-eggshell and the conventional chicken-eggshell, and to find the optimum conditions for biodiesel production. The prepared catalysts were then characterized by using XRD, FT-IR, BET, SEM, TGA and CO_2-TPD. The parametric effects on the biodiesel production, such as catalyst concentration, molar ratio of methanol to oil, reaction temperature, reaction time, speed and reusability of the catalyst were investigated. The experimental result showed that 1.5 wt.% catalyst, 12:1 M ratio of methanol to oil, 65 °C reaction temperature, 2 h reaction time with speed of 250 rpm gave the best results. It was found that the ostrich-eggshell derived CaO catalyst shows higher surface area, higher basicity and smaller particle size. The maximum biodiesel yield is 96% and 94% for calcined ostrich-eggshell and chicken-eggshell, respectively. The CaO catalyst derived from waste calcined ostrich and chicken-eggshell maintained a good catalytic activity even after being repeatedly used for 5 cycles with yield around 70%, which implies potential saving and affordable biodiesel production possibilities.
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Nomenclature

FAME	fatty acid methyl esters	FTIR	Fourier transform infrared spectroscopy
NaOH	sodium hydroxide	TGA	thermogravimetric analysis
KOH	potassium hydroxide	XRD	X-ray diffraction
$NaOCH_3$	sodium methoxide	CO_2-TPD	temperature-programmed desorption of CO_2
H_2SO_4	sulphuric acid	W	weight
HCl	hydrochloric acid	m_i	mass of internal standard added to the sample,
$RS(=O)_2—OH$	sulphonic acid	A_i	peak area of internal standard,
CaO	calcium oxide	m_b	mass of the biodiesel sample
$CaCO_3$	calcium carbonate	A_b	peak area of the biodiesel sample
CO_2	carbon dioxide	%w/v	percentage of weight over volume
SEM	scanning electron microscope	wt.%	weight percentage
BET	Brunauer–Emmett–Teller	rpm	revolutions per minute

1. Introduction

The traditional fossil fuels in particular petroleum, natural gas, and coal have been continuously used globally for the past few hundred years. The demand towards energy continues to increase year by year. As a result, biodiesel has drawn special attention from researchers as a potential alternate fuel for current petroleum-based fossil fuel due to the depletion of the oil reserves. Biodiesel is one kind of renewable fuel consists of fatty acid methyl esters (FAME). It is produced through transesterification of edible and non-edible vegetable oils, animal fat as well as waste cooking oil from the food industry. Apart from sustainability, biodiesel also presents some superior properties over fossil fuels: non-toxic, biodegradable, good combustion efficiency and environmentally friendly [1,2]. In the following paragraphs, a brief overview on the biodiesel production via various type of catalysts used is first mentioned leading to the use of heterogeneous catalyst including egg shells for biodiesel production.

Broadly for biodiesel production, the catalysts usually employed to catalyse transesterification reaction are homogeneous catalysts and heterogeneous catalysts. The common catalyst for the transesterification process of vegetable oils is via homogeneous catalysts. Generally, the catalysts reaction is fast and the process conditions are moderate [3]. Some catalysts include sodium hydroxide (NaOH), potassium hydroxide (KOH), and sodium methoxide ($NaOCH_3$) are widely used [4]. There are, however, some disadvantages associated with the using of the homogeneous catalyst. The presence of water in the homogeneous catalytic process could forms soap, the by-product via a process called saponification reaction. Soap formations are undesirable side-reactions, because soap formations consume the catalyst, as a result the process reduce the biodiesel yield as well as it causes difficult separation and purification steps [5]. All these adverse reactions result in the generation of huge amount of wastewater due to the washing process of catalyst residues and no reusability of the catalysts [6].

The acid catalysts used in homogeneous phases include sulphuric acid (H_2SO_4), hydrochloric acid (HCl) and sulphonic acid [$RS(=O)_2—OH$] where, R is an aryl group and $S(=O)_2—OH$ is sulfonyl hydroxide. Recently, Zhang et al. [7] reported that bagasse can be used as raw material to prepare carbon-based solid acid catalysts for the esterification of free fatty acid in waste cooking oil. The main advantage of the acid catalyst is that the free fatty acids content in the oil will not affect the reaction. However, the acid catalysts have not been used as widely as alkali catalysts due to it has several limitations, such as the demand of costly separation, slower reaction rate, high reaction temperature required, high molar ratio of alcohol to oil and purification steps, high corrosion and non-reusability [8–10].

Although transesterification catalyst-free in supercritical methanol can be an alternative method [11], this supercritical process requires very high temperatures and extremely high pressures conditions which can incurred high capital cost. Additional methods to produce biodiesel are the use of ultrasonic reactors and microwaves. Ultrasonic reactors produce good conversion yields; however, the use of a catalyst is still necessary apart from the high consumption of electricity to produce the biodiesel [12]. For the microwaves method, both heterogeneous and homogeneous catalysts can be employed to obtain lower reaction times than without the use of microwaves [13,14]. Such methods are not discussed in this paper.

Now, the heterogeneous catalytic process has potential to overcome the disadvantages of homogeneous catalytic process, in particular from the sustainability standpoints, some of which are quoted and listed as follows:

- The solid catalysts can be recovered easily and re-usable [15,16].
- The heterogeneous catalytic process can minimizes the purification process thus reduces energy and water consumption. This is made possible by eliminating the neutralization step of generating wastewater [17].
- Heterogeneous base catalyst can be obtained from a variety of waste sources such as bones, ashes, rocks and shells.

These waste sources have high potential as catalysts for the production of biodiesel [18,19]. The mechanism of heterogeneous catalyst is similar with the principle of homogeneous catalyst. In order to get the calcium oxide, CaO catalyst naturally, eggshell from waste is a good biomass resource [20,21]. The major component of eggshells is calcium carbonate, $CaCO_3$. The calcium carbonate component will convert to calcium oxide and carbon dioxide under high temperature.

In the synthesis of CaO, many studies show that CaO derived from bird eggs is a potential catalyst in transesterification process, e.g. Wei et al. [19], Viriya-empikul et al. [22], Jazie et al. [23], Cho and Seo [24], Sharma et al. [25], Niju et al. [26], and Chen et al. [27] illustrated in Table 1. The calcination of eggshell at high temperature of 600 °C up to 1000 °C had been used with biodiesel yield of 92–96% had been reported.

While many CaOs derived from bird eggs shells in particular chicken egg shells had been vastly reported in literature, in the present investigation however, a rare type of eggshell i.e. local ostrich-eggshell was employed to produce the CaO catalyst for the transesterification of waste cooking oil and biodiesel production.

The first commercial ostrich farm was first established in South Africa in about 1860. Ostrich farms began to spread gradually to

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