

Effects of Fly Ash Nano Particles on the Specific Heat Capacity of Transesterified Neem Oil

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Abstract

The impact of fly ash nanoparticles on the specific heat capacity of transesterified neem oil was explored in this study. The physical and chemical properties of the catalyst were examined through scanning electron microscopy (SEM) and X-ray fluorescence (XRF) characterizations, respectively. Crude neem oil underwent transesterification, with varying concentrations of fly ash (0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt%) serving as heterogeneous catalysts. The specific heat capacity (SHM) of transesterified neem oil with these different catalyst concentrations was determined. SEM analysis revealed dispersed particles with irregular structures, while the chemical composition analysis of fly ash indicated a predominance of SiO_2 at 73.23%. The results of specific heat capacity indicated that the sample containing 0.2 wt% fly ash end to be the best sample among the samples.

Keywords: Biodiesel, Fly Ash, Neem Oil, Specific Heat Capacity, Transesterification.

1. Introduction

Depletion of fossil fuels and escalating environmental concerns have underscored the urgency of seeking alternatives to mineral diesel, a cornerstone of the transportation sector worldwide. Biomass-based fuels, including vegetable oils and their derivatives, emerge as promising solutions to this pressing crisis on a global scale (Ismail et al., 2022). These renewable, environmentally friendly, and non-toxic resources could be produced at a small scale in rural areas, offering a decentralized and clean energy solution for rural sectors (Dhar et al., 2012). Biomass is recognized as a key renewable energy source for the future, owing to its vast potential, economic feasibility, and various social and environmental benefits. Despite its infrequent inclusion in official energy statistics, biomass already ranks as the fourth-largest energy source globally (Aidin et al., 2010). Given the escalating global energy demands and the imperative for cleaner alternative fuels and energy sources, biofuels have garnered significant attention (Bateni et al., 2016; Saidur et al., 2011; Bateni et al., 2017).

Biodiesel, derived from vegetable oils, animals, or fats, serves as a clean-burning diesel fuel composed of fatty acid alkyl esters (FAAE) (Muhammad et al., 2023). Compared to fossil diesel, biodiesel emits fewer toxic air pollutants, burns cleaner, and contains lower sulfur levels, thus reducing emissions (Nura et al., 2023). With its renewable origin, biodiesel is poised to increasingly compete with petroleum products in the future (Ismail et al., 2022). However, the International Energy Agency defines renewable energy as deriving from natural processes continually replenished by the sun or heat generated deep within the earth (Akorede et al., 2010).



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Biodiesel production involves the chemical reaction of vegetable oil or animal fats with methanol, typically facilitated by a base catalyst like sodium or potassium hydroxide, resulting in the production of a chemical compound known as methyl ester, or biodiesel. Biodiesel is a processed fuel derived from biological sources, akin to petro-diesel (Musa et al., 2022). It serves as a crucial plant-based fuel resource and is categorized as a Fatty Acid Methyl Ester (FAME) produced through various methods such as micro-emulsification. The transesterification process, catalytically reacting renewable feedstocks like vegetable oil or animal fats with methanol, is the most widely recognized method for biodiesel production, yielding glycerol as a by-product (Hook and Tang, 2013). Studies have shown that the incorporation of nanoparticles, either as catalysts or additives, enhances the transesterification process and improves certain properties of biodiesel (Ismail et al., 2022). Vegetable oils have gained increased attention in recent years due to their environmental advantages and renewable nature (Uba et al., 2024). They represent a renewable and potentially energy-rich source with an energetic content comparable to diesel fuel (Jamo et al., 2023). Oils derived from vegetables may eventually rival petroleum and coal tar products in importance. Recent declines in mineral oil prices, coupled with concerns over their availability, have reignited interest in vegetable oil fluids for industrial applications (Abas et al., 2015).

Neem, a tree from the Meliaceae family found in southern Asia and Africa, scientifically known as Azadirachta indica, is notable for its seeds, which contain approximately 40% oil, making it highly suitable for biodiesel production. Neem oil possesses higher molecular weight, viscosity, density, and flash point compared to diesel fuel (Dhar et al., 2012; Musa et al., 2022). Typically, light to dark brown in color, neem oil has a bitter taste and a distinctive odor reminiscent of peanuts and garlic (Thangaraj et al., 2019). Utilizing non-edible neem oil for biodiesel production through transesterification offers a potential solution to various issues. Additionally, the incorporation of nanoparticles into transesterified neem oil is expected to enhance its viscosity (Dhar et al., 2012).

Fly ash, a byproduct of coal utilization, remains an underutilized resource. Thermal power plants seek methods to exploit fly ash, with potential applications in the cement industry for concrete production and recycling materials for agriculture and engineering purposes (Ahmaruzzaman, 2011). Experimental findings have demonstrated the excellent activity and stability of fly ash as an additive in transesterified calabash oil (Nura et al., 2023). However, there is a lack of literature on the effect of fly ash on the specific heat capacity of transesterified neem oil. Therefore, this research aims to investigate the influence of fly ash on the specific heat capacity of transesterified neem oil.

2. Materials and Methods

2.1 Chemicals

The materials and reagents employed in conducting this research include neem oil, 8% sodium hydroxide (NaOH), 64% citric acid (C₆H₈O₇, purity: 99.7%), silicon reagent, activated carbon, fly ash, acetone, methanol, and distilled water.

2.2 Equipment's

The equipment utilized in conducting this study includes a magnetic stirrer with a thermostatically controlled rotary hot plate model (IKA C-MAG HS10), thermometer, measuring cylinder, Cheng Sang Vacuum oven model (MA 0 -30L), Digital weight balance (AND model GT2000 EC), beakers, conical flask, 24 cm filter paper, funnel, digital stopwatch, sampling bottles, spatula, calorimeter, electric heater, lighter, FTIR machine, and SEM machine.

2.3 Methodology

The methodologies employed during this research are; SEM characterization of fly ash, then sample purification, Transesterification, with addition of fly ash, as catalyst as well as Infrared spectral analysis.

2.3.1 SEM Characterization of Fly Ash



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The characterization of fly ash using Scanning Electron Microscopy (SEM) was conducted with a multipurpose Scanning Electron Microscope model PHENOM PRO X MVE01570775. One gram of fly ash was subjected to scanning with a focused electron beam emitted by the machine. These electrons interacted with atoms within the fly ash, generating diverse signals containing valuable information regarding its surface topography and composition. The electron beam was systematically scanned in a raster pattern, and the resulting image of the fly ash was formed by combining the beam's position with the intensity of the detected signal (Stokes, 2008).

2.3.2 XRF Characterization of Fly Ash

X-ray fluorescence characterization of fly ash was performed using the ARL QUANT'X EDXRF Analyzer model (S/N 9952120). During this process, incoming X-rays emitted by the XRF machine dislodge electrons from atoms within 1g of fly ash, leading to the excitation of these atoms and the emission of high-energy radiation, including photons, protons, and electrons. Subsequently, the emitted lines are detected and analyzed to determine their intensity levels. These intensity levels are then converted into elemental concentrations, which are displayed on the monitor (Haschke, 2014).

2.3.4 Purification of Neem Crude oil

The crude neem oil was subjected to purification through the following procedure: 200 ml of neem oil was measured using a measuring cylinder and then heated to 70°C using a hot magnetic stirrer equipped with a thermometer. Next, 0.5g of citric acid was measured and dissolved in 1.5 ml of distilled water, which was then added to the heated oil sample. The mixture was continuously heated and stirred for 15 minutes at 70°C. Subsequently, 4 ml of 8% NaOH solution (prepared by dissolving 8g of NaOH in 100 ml of distilled water) was added to the oil and continuously heated and stirred for another 15 minutes at 70°C.

The mixture was then transferred to a vacuum oven and heated at 85°C for 30 minutes. Upon completion, it was returned to the hot magnetic stirrer and reheated to 70°C. While being heated and stirred, 2g of silicone reagent was added, and the mixture was stirred for an additional 30 minutes. The temperature was then raised to 85°C, and 4g of activated carbon was added to each 100 ml of the oil sample. The mixture was heated and stirred for another 30 minutes. Finally, the mixture was separated using a separating funnel (Ismail et al., 2022).

2.3.5 Transesterification of Neem Oil without Catalyst

60g of neem oil was measured in a 250ml conical flask and then heated and stirred to a temperature of 60-65°C on a hot magnetic stirrer plate. 0.6g of NaOH was measured using an electronic weighing machine and dissolved in 21ml of methanol, then added to the mixture and allowed to heat for 60 minutes with the stirrer on the hot magnetic plate. After 60 minutes of uniform stirring and heating on the hot magnetic plate, maintaining a temperature of 65°C, the mixture was poured into a separating funnel through a glass funnel. The mixture was allowed to cool for about 40 minutes. Subsequently, it was observed to separate into two liquid layers, with the upper layer being biodiesel and the lower layer triglyceride fatty acid (Ismail et al., 2022).

2.3.6 Transesterification of neem oil using fly ash as Catalyst

60g of the Neem oil was measured in 250 ml of conical flask and then heated and stirred to a temperature of 60-65°C on a hot magnetic stirrer plate, 0.6g of NaOH and 0.1 wt% of fly ash has been measured using the electronic weight machine and allowed to dissolve in 21ml of methanol and then added to the mixture and allowed it to heat for 60 minutes with the stirrer on the hot magnetic plate. After 60 minutes of uniform stirring and heating on the hot magnetic plate maintaining a temperature of 65°C, then it has been poured into the separating funnel through a glass funnel. The mixture was allowed to cool for about 40 minutes. Afterwards, it has been observed to separate into two liquid layers. The upper layer is the biodiesel and the lower layer is triglycrol fatty acid. The same procedure has been applied to 0.2 wt%, 0.3 wt%, 0.4 wt% and 0.5 wt% fly ash (Ismail *et al.*,2022).

2.3.7 Measurement of Specific Heat Capacity

A certain quantity of crude neem oil, denoted as (m_l) (kg), was measured and poured into a calorimeter with a known mass (m_c) (kg) and specific heat capacity (c_c) (J/kg/K). An electric heater with a known power (P) (W) was employed to heat both the crude neem oil and the calorimeter for a specified duration (t) (seconds) until a change in temperature (θ, K) was achieved. Subsequently, the specific heat capacity of the crude neem oil, denoted as (C_l) (J/kg/K), was determined using the relationship described in Equation 1 (Ezebuiro, 2004).

$$C_{l} = \frac{Pt - m_{c}c_{c}\theta}{m_{l}\theta} \tag{1}$$

For the determination of specific heat capacity of purified, trans-esterified and trans-esterified with fly ash, the procedure above was adopted.

3. RESULTS AND DICUSSIONS

3.1 Scanning Electron Microscopic (SEM) of Fly Ash

Figure 1 depicts the SEM image of the fly ash, revealing the presence of dispersed particles with an irregular structure at a magnification of 1000X (80μm and 10KV). This finding aligns closely with results reported by Chindaprasirt et al. (2009) and Nura et al. (2022).

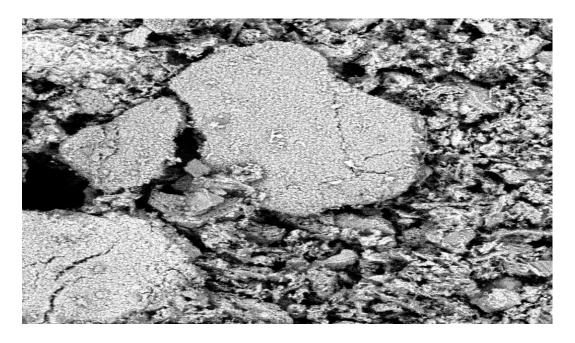


Figure 1: SEM of fly ash at a magnification of 1000X.

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3.2 Chemical Composition of Fly Ash

The XRF compound percentage concentration was presented in Table 1. In Table 1, the composition of fly ash is detailed, showing SiO₂ at 73.23%, CaO at 7.33%, K₂O at 5.45%, SrO at 4.66%, Al₂O₃ at 2.33%, MgO at 1.53%, P₂O₅ at 1.33%, SO₃ at 1.79%, Fe₂O₃ at 0.71%, with 1.30% accounted for by other elements, and 0.34% attributed to loss of ignition. Notably, SiO₂ constitutes the highest percentage, whereas Fe₂O₃ exhibits the lowest concentration. This suggests that fly ash primarily consists of silicon oxide, making it suitable for use as both an additive and a catalyst without environmental concerns. These findings closely align with those reported by Nura et al. (2022).

Table 1. Chemical composition of fly ash.

S/N	Compound	Percentage concentration (%)
1	${ m SiO_2}$	73.23
2	CaO	7.33
3	K_2O	5.45
4	SrO	4.66
5	Al_2O_3	2.33
6	MgO	1.53
7	P_2O_5	1.33
8	SO_3	1.79
9	Fe_2O_3	0.71
10	Lost of ignition	0.34
11	Others	1.30

3.3 Specific Heat Capacity of the Samples

Table 2 reveals the specific heat capacities of crude, purified, and transesterified neem oil with 0.1 wt%, 0.2 wt%, 0.3 wt%, 0.4 wt%, and 0.5 wt% of fly ash as catalysts. From Table 2, the specific heat capacity (SHC) for crude oil is 1.45 kJ/kg/K. After purification, the SHC increases to 1.74 kJ/kg/K, and upon transesterification, it further rises to 2.10 kJ/kg/K. When 0.1 wt% fly ash catalyst is added, the transesterified SHC increases to 2.11 kJ/kg/K, and with the addition of 0.2 wt% fly ash, the SHC rises to 2.13 kJ/kg/K. However, when transesterified with a 0.3 wt% catalyst, the SHC decreases to 2.09 kJ/kg/K.

Table 2. Specific heat capacity of transesterified neem oil with different concentrations of neem oil as catalyst.

S/N	Neem oil	Specific heat capacity (kJ/kg/K)
1	Crude oil	1.45
2	Purified	1.74
3	Trans-esterified	2.10
4	Trans-esterified (0.1wt% catalyst)	2.11
5	Trans-esterified (0.2wt% catalyst)	2.13
6	Trans-esterified (0.3wt% catalyst)	2.09
7	Trans-esterified (0.4wt% catalyst)	2.14
8	Trans-esterified (0.5wt% catalyst)	2.16
9	Biodiesel standard	2.13



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Conversely, transesterification with a 0.4 wt% catalyst results in an increase in SHC to 2.14 kJ/kg/K, and with a 0.5 wt% catalyst, it increases to 2.16 kJ/kg/K. Notably, the sample with 0.2 wt% fly ash appears to be the most favorable, closely resembling the biodiesel standard 2.13 kJ/kg/K. This trend suggests a rise and subsequent fall in SHC values with increasing catalyst concentrations. These findings are consistent with those reported by Balat et al. (2008).

4. Conclusion

In conclusion, biodiesel was successfully produced via transesterification using neem oil with the addition of fly ash as a catalyst. Scanning electron microscope analysis revealed the morphological characteristics of fly ash, illustrating dispersed particles and an irregular structure. Through X-ray Fluoroscopy, the chemical composition of fly ash was determined, with SiO_2 at 73.23% and CaO at 7.33% representing the highest elemental concentrations, followed by K_2O at 5.45% and SrO at 4.66%. These results indicate that SiO_2 , a component of calcite, is the predominant element in fly ash. This study aimed to investigate the influence of fly ash nanoparticles on the specific heat capacity of transesterified neem oil. The findings indicate that the sample containing 0.2 wt% fly ash exhibited the most favorable specific heat capacity.

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