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RESEARCH PAPER

ZnO Nanoparticles Incorporated on Multi-Walled Carbon Nanotubes as A Robust Heterogeneous Nano-catalyst for Biodiesel Production from Oil

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ABSTRACT

Biodiesel is a sustainable alternative to fossil fuels, playing a crucial role in reducing greenhouse gas emissions and environmental impact. The development of efficient catalytic processes, particularly utilizing nanocatalysts, is of significant importance for enhancing biodiesel production. This study presents the synthesis and characterization of ZnO nanoparticles supported on multi-walled carbon nanotubes (MWCNTs). The physical and chemical properties of the synthesized nanocatalysts were analyzed through XRD, and SEM techniques, confirming successful deposition of ZnO on MWCNTs. The catalytic activity was evaluated for biodiesel production from soybean oil under different methanol-to-oil ratios and reaction times. Results demonstrated a maximum conversion efficiency of 99% at a methanol/oil ratio of 55:1 and a reaction time of 45 minutes. Reusability tests indicated that the catalyst maintained high performance over seven cycles with only slight activity loss. The proposed method offers notable advantages, including higher yields, cost-effectiveness, and environmental sustainability. Overall, this nanocatalyst system holds promise for industrial applications, contributing to more efficient and eco-friendly biodiesel manufacturing processes.

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INTRODUCTION

Biodiesel has emerged as a promising alternative to traditional fossil fuels, offering a renewable and sustainable source of energy derived from vegetable oils, animal fats, and other biomass resources [1-5]. Biodiesel consists of fatty acid methyl esters (FAME) and serves as a viable substitute for traditional fossil fuels [6, 7]. As a clean-burning fuel, biodiesel significantly reduces greenhouse gas emissions and has the potential to mitigate environmental pollution associated with conventional diesel [8]. The importance of biodiesel fuel lies in its eco-friendly nature, biodegradability, and compatibility with existing diesel engines, making it an attractive option for sustainable transportation [9, 10]. Recent advancements in biodiesel production techniques, including enzymatic transesterification [11], novel catalyst development [12-14], and feedstock diversification [15-17], have substantially improved yields and process efficiency. These developments are paving the way for wider commercial adoption and contributing to the global efforts toward energy security and environmental conservation.

The production of biodiesel primarily depends on the selection of suitable oils and fats as feedstocks, which serve as the starting materials for transesterification processes. These oils can be broadly categorized into edible and non-edible sources, including soybean oil [18-20], palm oil [21], rapeseed oil [22, 23], and sunflower oil [24], which are commonly utilized due to their high lipid content and availability. In addition, non-edible oils such as jatropha [25], castor [26], and pongamia have gained increasing attention as sustainable alternatives, especially in regions with limited edible oil resources. Waste cooking oils and animal fats also represent valuable feedstocks, contributing to waste valorization and cost reduction in biodiesel production. The choice of starting material significantly influences the physicochemical properties, economic viability, and environmental impact of the final biodiesel fuel, making the exploration of diverse feedstocks a critical aspect of ongoing research in this field [27].

Catalysts play a crucial role in the efficient production of biodiesel through transesterification, with both homogeneous and heterogeneous catalysts being extensively studied [28-30]. Homogeneous catalysts, such as sodium hydroxide (NaOH), potassium hydroxide (KOH), and sodium methoxide, are widely used due to their high catalytic activity and ease of operation [31, 32]. However, they often pose challenges related to soap formation, separation, and recyclability, which can impact the overall process economics. On the other hand, heterogeneous catalysts, such as calcium oxide (CaO) [33, 34], magnesium oxide (MgO) [35, 36], and various metal-supported solid catalysts, offer advantages including ease of separation, reusability, and reduced environmental impact. For instance, calcined waste eggshells and hydrotalcite-based catalysts have shown promising results as sustainable and cost-effective heterogeneous alternatives. The ongoing development and optimization of both catalyst types are vital for advancing biodiesel technology, aiming to improve reaction efficiency, product purity, and process sustainability [37, 38].

Carbon nanotubes (CNTs) are cylindrical nanostructures composed of single or multiple layers of carbon atoms arranged in a hexagonal lattice, forming single-walled (SWCNTs) or multi-walled (MWCNTs) configurations [39]. They are renowned for their extraordinary properties, including a high specific surface area, exceptional mechanical strength, excellent electrical and thermal conductivity, and chemical stability [40]. These features make CNTs highly attractive as catalytic supports or active catalysts in various organic reactions [41-43]. To enhance their reactivity and compatibility with different chemical processes, CNTs are often functionalized through chemical modifications such as oxidation, carboxylation, or grafting with various functional groups (e.g., amines, hydroxyl, or metal nanoparticles). Such functionalization improves their dispersibility, reactivity, and ability to anchor catalytic species, thereby enhancing catalytic efficiency. In the context of biodiesel production, functionalized CNTs have recently emerged as promising catalysts or catalyst supports for transesterification reactions involving vegetable oils and fats. Their high surface area allows for increased catalytic active sites, while the functional groups can facilitate the immobilization of metal catalysts or enzymes, leading to improved conversion rates, selectivity, and catalyst reusability. Recent developments have focused on incorporating metal nanoparticles such as Ca, Mg, or Cu onto CNTs, which act as heterogeneous catalysts with high activity under mild conditions. These advancements not only contribute to higher biodiesel yields but also promote greener processes through catalyst recyclability and reduced environmental impact. As research continues, functionalized CNTs are poised to play a significant role in making biodiesel production more sustainable, cost-effective, and scalable for industrial applications [44, 45] In this study, we report the synthesis and characterization of a novel ZnO nanoparticles supported on multi-walled carbon nanotubes (MWCNTs) and evaluate their performance as a robust heterogeneous nano-catalyst for efficient biodiesel production from soybean oil feedstocks.

MATERIALS AND METHODS

Chemicals and Instruments

The anhydrous solvents, chemicals, and reagents obtained from Merck and Fluka were utilized without any additional purification steps. MWCNTs was purchased from China, Shenzhhen, Co. Ltd. The catalysts' successful synthesis was confirmed through X-ray diffraction (XRD) analysis, performed on a Bruker D8 Advance device utilizing Cu Kα radiation (λ = 1.5406 Å) over a 2θ range of 25 ° to 80 °, revealing their crystalline structure. Additionally, Fourier Transform Infrared (FT-IR) spectra were recorded between 400 and 4000 cm^{-1} using a 6300D-JASCO spectrometer. Field emission scanning electron microscopy (FE-SEM) images were obtained with a MIRA3-TESCAN system. The acid value was measured via potentiometric titration employing a Metrohm 848 titrator (Switzerland), while the moisture content of the soybean oil was assessed using the Karl Fischer Aquamax KF method (UK). Fatty acid methyl esters (FAMEs) present in the biodiesel were analyzed using gas chromatography-mass spectrometry (GC-MS) on an Agilent GC 8800 system, coupled with an Agilent 5983 network mass detector. The analysis utilized an Agilent HP-8 MS capillary column with an internal diameter of 0.35 mm and a length of 40 m. The temperature program started at 90 ºC (held for 3 minutes) and increased to 310 ºC at a rate of 7 ºC per minute, with a final hold of 12 minutes, totaling a run time of 60 minutes. Helium was used as the carrier gas at a flow rate of 1.2 mL/min, and the sample injection volume was 0.6 µL.

Fabrication of ZnO Nanoparticles Incorporated on Multi-Walled Carbon Nanotubes

In a standard procedure, 1.5 grams of (zinc nitrate. hexa hydrate) $Zn(NO_3)_2{\cdot}6H_2O$ and 0.9 grams of NaOH (sodium hydroxide) were dissolved separately in 50 mL of deionized water. The $Zn(NO_3)_2$ solution was then added gradually drop wise to the NaOH solution, resulting in a white slurry. This slurry was maintained at 90 °C for a duration of 8 hours. The resulting white precipitate was collected by filtration, washed multiple times with deionized water and ethanol, and subsequently dried at 70 °C under vacuum for 12 hours. The multi-walled carbon nanotubes (MWNTs) were pretreated using acids such as HCl, HNO_3, and H_2SO_4. An appropriate quantity of synthesized ZnO nanoparticles and MWNTs was dispersed in a mixed solvent of water, ethanol, and methanol in a 1:1:1 ratio. The mass ratio of ZnO to MWNTs to solvent was set at 1:5:100. The mixture was subjected to ultrasonic agitation for 30 minutes after being stirred for 6 hours, resulting in a highly dispersed black colloidal solution [46-49].

ZnO Nanoparticles Incorporated on Multi-Walled Carbon Nanotubes for biodiesel production

The transesterification process was conducted in a small vial at ambient temperature using catalysts composed of ZnO nanoparticles supported on multi-walled carbon nanotubes. Methanol was combined with the catalyst (35 mg, corresponding to 3.4 wt%) and stirred vigorously. Next, soybean oil was added to this mixture, and the reaction was maintained with continuous stirring for 45 minutes at room temperature. The molar ratio of methanol to oil was set at 55:1. Upon completion of the reaction, n-hexane was introduced to the mixture, which was then centrifuged at 5000 rpm for 7 minutes to separate the desired product. Following centrifugation, two distinct liquid layers formed: the upper layer containing hexanes, biodiesel, and possibly residual oil, and the lower layer comprising methanol and glycerol. The upper layer was carefully decanted into a beaker and placed in a laboratory oven at 60 °C to evaporate the hexanes. The resulting crude biodiesel was then examined using ^1H NMR spectroscopy. The percentage conversion (CME) of soybean oil to biodiesel produced by the catalyst was determined based on the integration of the glyceridic and methyl ester proton signals in the ^1H NMR spectrum, calculated with Equation 1. I ME: Determine the integrated area of the methyl ester peak. I TAG: Evaluate the integrated area of the glyceridic peaks in the triacylglycerides (TAG) of the oil.

$$C\ ME = 100 \times \frac{5 \times I\ ME}{5\ \times I\ ME \times I\ TAG} \quad (1)$$

General procedure for the recoverability of ZnO Nanoparticles Incorporated on Multi-Walled Carbon Nanotubes

The recyclability of the nanocatalyst in the transesterification process of soybean oil with CH_3OH was assessed through a specific procedure. After each reaction cycle, the nanocatalyst was retrieved via centrifugation, then washed with n-hexane and CH_3OH to remove any residual materials. Subsequently, it was reactivated by heating in a muffle furnace at 650 °C for 5 hours before being used again with a new portion of the reactants. The level of Zn^{2+} leakage was determined for both the fresh catalyst and the recovered ones after the final cycle using ICP analysis.

RESULTS AND DISCUSSION

Characterization of the nanocatalyst

The morphology of ZnO nanoparticles combined with multi-walled carbon nanotubes was analyzed through scanning electron microscopy. As shown in Fig. 1, the samples are made up of uniform, solid ZnO nanostructures, with the nanosheet thickness ranging roughly from 35 to 55 nanometers.

Fig. 2 presents the X-ray diffraction (XRD) patterns for two samples: (a) pristine multi-walled carbon nanotubes (MWCNTs) and (b) ZnO nanoparticles incorporated onto MWCNTs. The XRD pattern of pure MWCNTs exhibits a broad peak around 26°, corresponding to the (002) diffraction plane, indicating their amorphous or partially crystalline nature. In contrast, the spectrum of the ZnO-decorated MWCNTs shows multiple sharp peaks at specific 2θ angles, which are indexed to the (100), (002), (101), (110), (103), (200), (112), and (201) planes of hexagonal wurtzite ZnO. These characteristic peaks confirm the successful synthesis and deposition of crystalline ZnO nanoparticles on the MWCNT surface. The presence of both peaks in the composite suggests a well-integrated hybrid material, which is promising for applications requiring synergistic properties of carbon nanotubes and ZnO nanostructures [50-

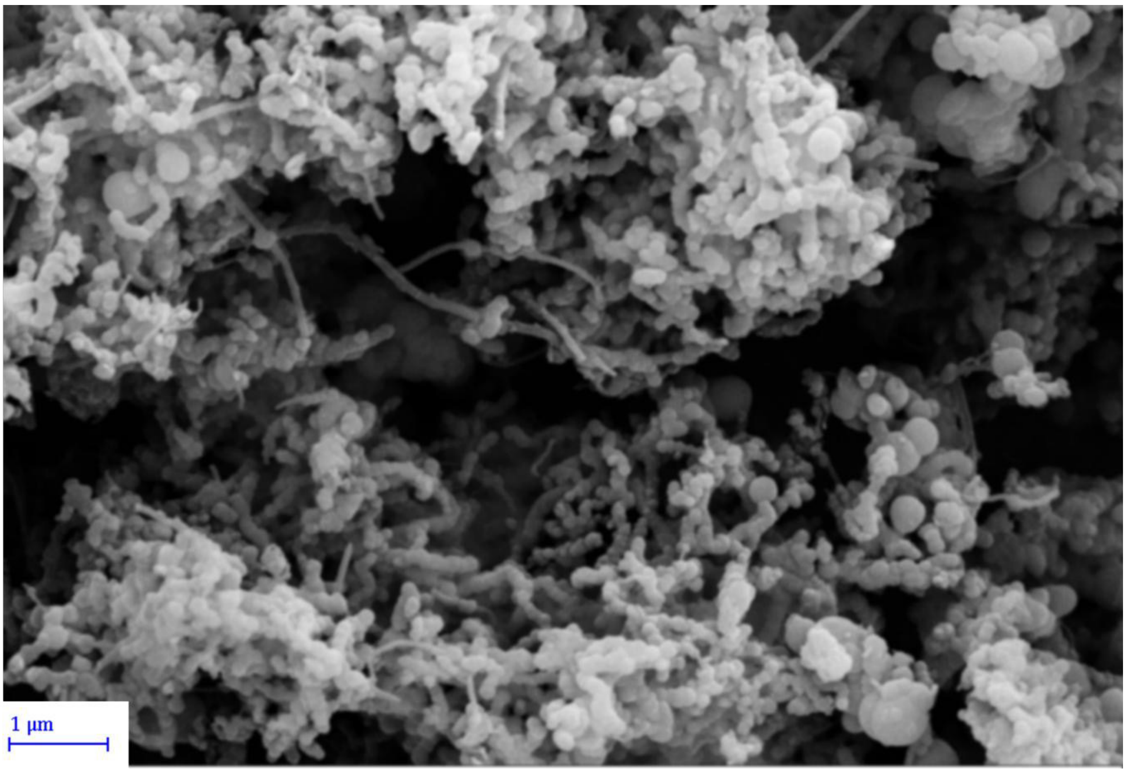

Fig. 1. SEM image of ZnO Nanoparticles Incorporated on Multi-Walled Carbon Nanotubes.

52].

Biodiesel fuels of soybean oil catalyzed by ZnO nanoparticles incorporated on multi-walled carbon nanotubes

In this study, we aimed to optimize the trans-esterification process by employing ZnO nanoparticles immobilized on multi-walled carbon nanotubes as a catalyst. To achieve the most efficient conversion, we systematically analyzed several key factors that influence the reaction. These included the molar ratio of methanol to soybean oil, which affects the availability of reactants; the reaction time needed to reach optimal conversion levels; and the amount of catalyst used, which impacts the catalytic activity and overall reaction rate. By carefully adjusting and evaluating these parameters, we sought to identify the optimal conditions that maximize biodiesel yield while ensuring process efficiency and catalyst stability (Fig. 3).

This study investigated how different methanol to soybean oil molar ratios specifically 15:1, 25:1, 35:1, 55:1, 65:1, and 75:1 affect the efficiency of biodiesel production. All experimental runs were carried out at ambient temperature, ensuring that temperature variations did not influence the reaction outcomes. A fixed amount of catalyst, 35 mg, was used consistently across all tests, and each reaction was maintained for a duration of 30 minutes to ensure comparability. The results revealed that the optimal biodiesel yield was achieved when the molar ratio of methanol to oil was set at 55:1, indicating that excess methanol can significantly improve conversion rates up to this point. As illustrated in Fig. 4, there was a marked increase in trans-esterification efficiency as the methanol to oil ratio was raised, demonstrating that higher alcohol concentrations promote more complete conversion of triglycerides into biodiesel. The findings emphasize the critical role of reactant proportions in enhancing process performance, while maintaining mild reaction conditions and a fixed catalyst amount, which simplifies the process and highlights the importance of balancing reactant ratios for maximum biodiesel yield.

In order to assess how reaction duration impacts biodiesel production, a series of five mixtures were prepared, each containing 35 mg of the catalyst, 0.37 grams of soybean oil, and 2.3

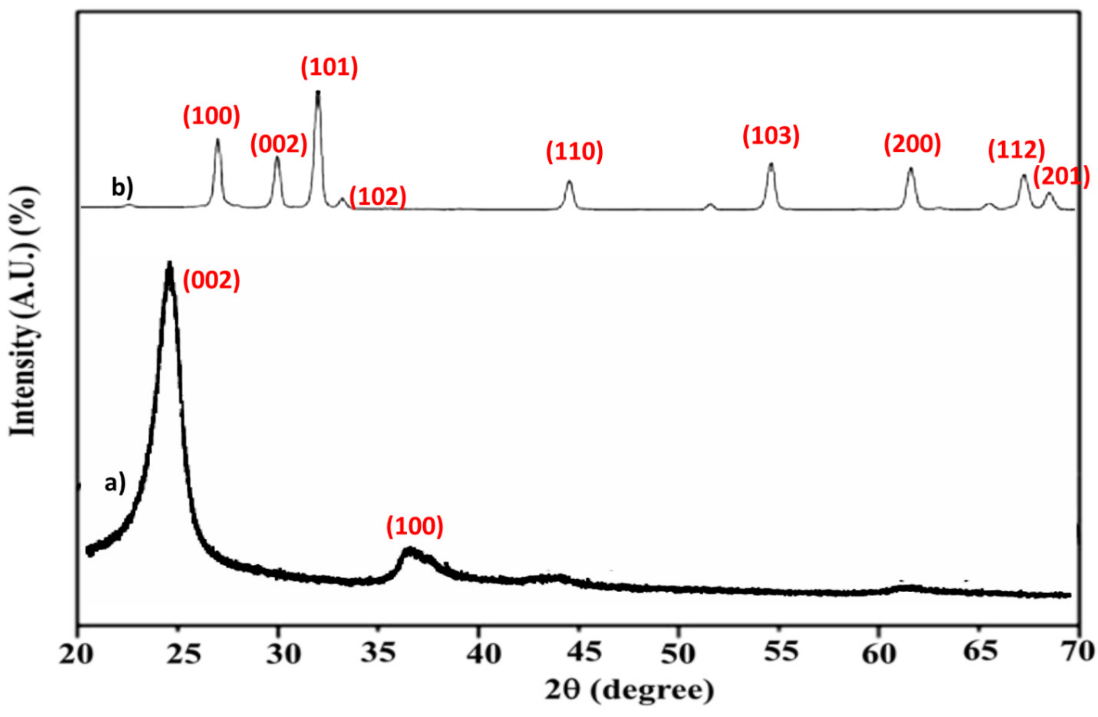

Fig. 2. XRD pattern of a) raw MWCNTs b) ZnO Nanoparticles Incorporated on Multi-Walled Carbon Nanotubes.

grams of methanol. These mixtures were stirred at ambient temperature for varying periods specifically 15, 25, 35, 45, and 55 minutes. After each reaction time, the amount of biodiesel generated was quantified using proton nuclear magnetic resonance (^{1}H NMR) spectroscopy, a technique known for its precision in measuring biodiesel yield. The data presented in Fig. 5 reveal a clear trend: as the reaction time increased, the conversion rate of triglycerides to biodiesel improved significantly. Starting from a lower efficiency at shorter durations, the conversion rate climbed steadily, reaching up to 99% at the 45 minute mark. Beyond this point, extending the reaction time yielded diminishing returns. Based on these findings, a reaction duration of 45 minutes was identified as the optimal period to maximize biodiesel yield while avoiding unnecessary prolongation of the process. This optimization underscores the importance of adequate

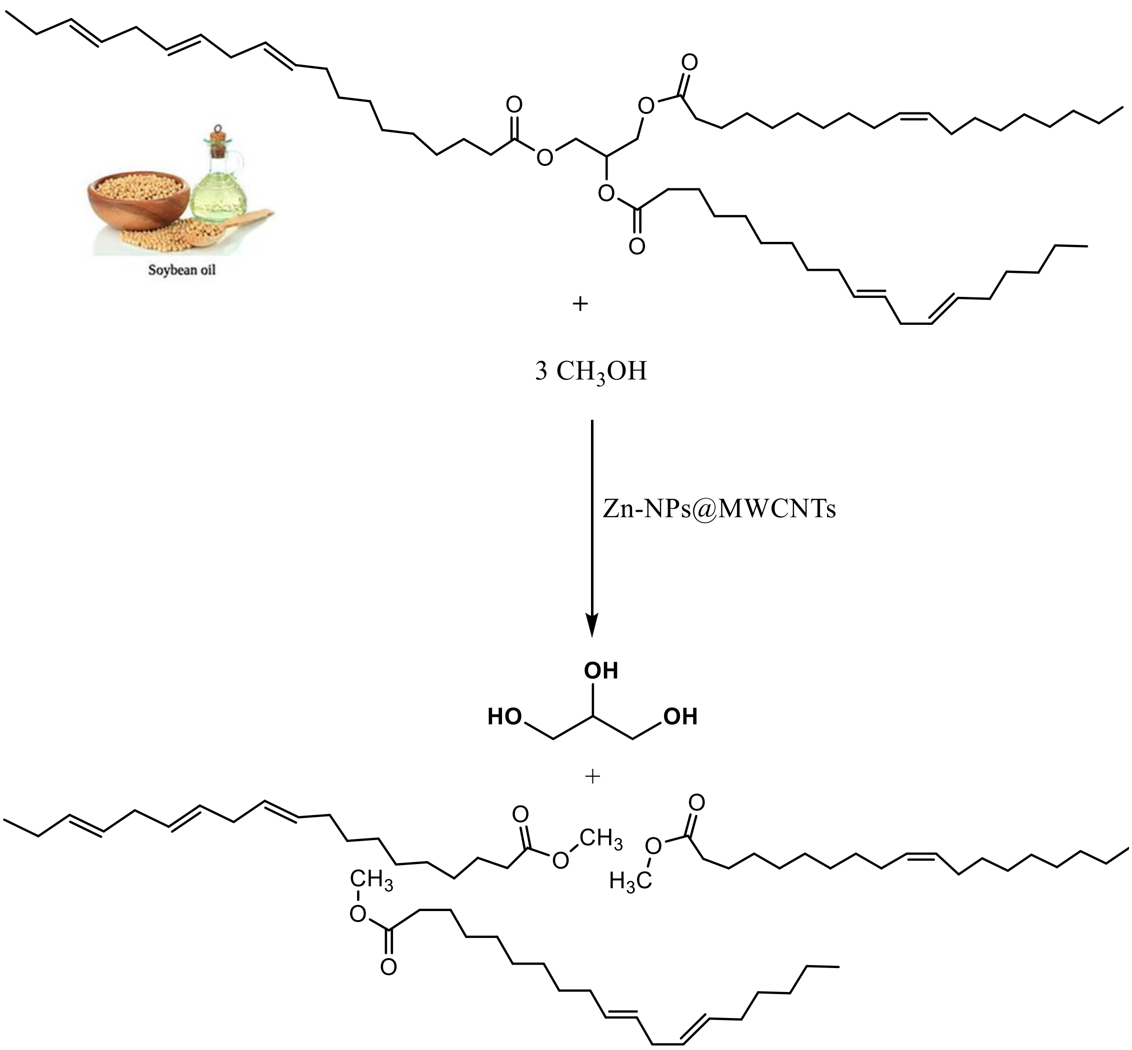

Fig. 3. Reaction of triglyceride to biodiesel fuels catalyzed by ZnO nanoparticles incorporated on multi-walled carbon nanotubes.

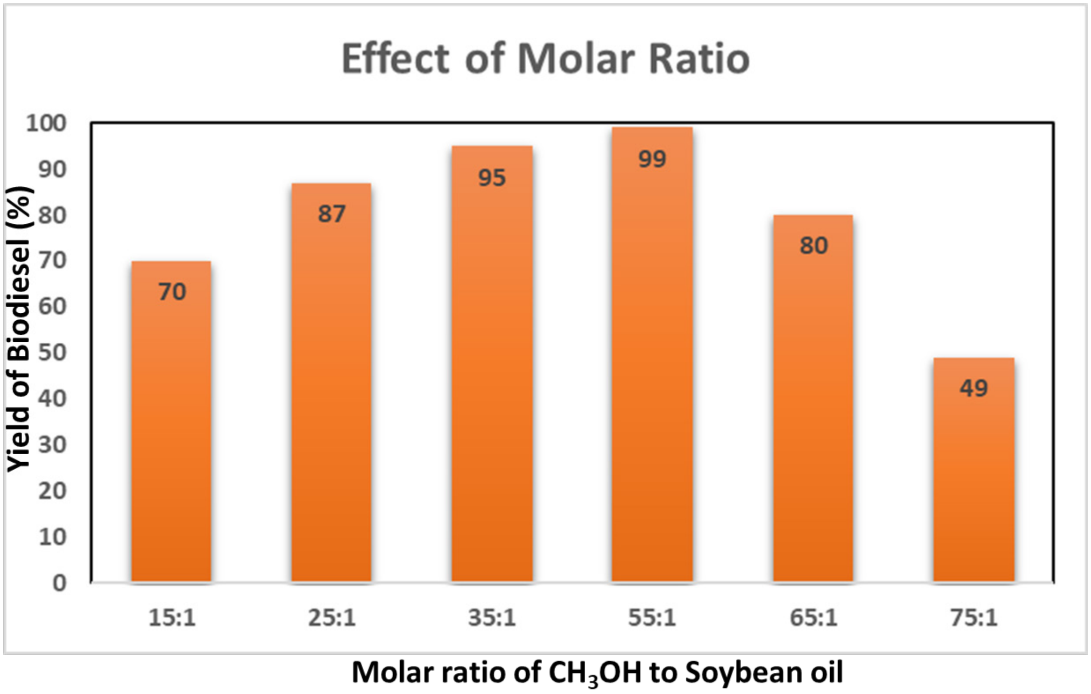

Fig. 4. The influence of the molar ratio of methanol to oil on biodiesel production. (reaction conditions: a mixture of methanol and oil; 35 mg nanocatalyst; T: room temperature; Time: 30 min).

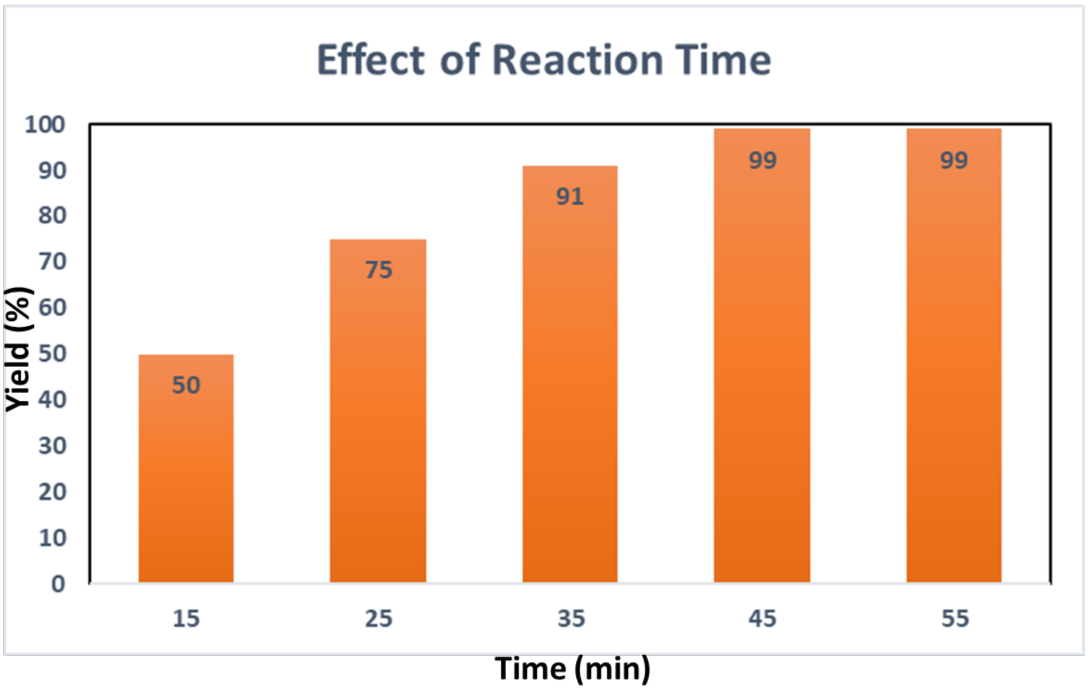

Fig. 5. The influence of reaction progress time of the yield of the biodiesel production (catalyst: 35 mg, T: room temperature, 0.37 grams of soybean oil, and 2.3 grams of methanol).

reaction time for achieving near-complete transesterification under mild conditions, contributing to the efficiency and practicality of the biodiesel production process.

The quantity of catalyst used in the transesterification process plays a crucial role in determining the overall efficiency of biodiesel production. To optimize catalyst loading, a series of experiments were conducted using different catalyst amounts specifically 5, 15, 25, 35, and 45 milligrams. Each of these quantities was evaluated for their impact on biodiesel yield, which was measured in terms of ester content. The results showed a progressive increase in ester production with increasing catalyst amounts: yields were approximately 65%, 71%, 84%, and 99% at 5, 15, 25, and 35 mg of catalyst, respectively, with a slight plateau observed at the highest tested quantity of 45 mg, which also achieved a 99% yield. Based on these findings, 35 mg of catalyst (equating to about 3.4 wt%) was identified as the most effective dose, providing near-maximum conversion efficiency without the unnecessary use of excess catalyst. Selecting this optimal amount not only improves the economic viability of the process but also minimizes catalyst waste, contributing to a more sustainable and cost-effective biodiesel production method.

A key benefit of using heterogeneous catalysts, as opposed to homogeneous ones, lies in their ability to be reused multiple times, which enhances the economic and environmental sustainability of the process. In our study, the recyclability of ZnO nanoparticles supported on multi-walled carbon nanotubes was systematically examined through successive biodiesel production cycles. After each cycle, the catalyst was recovered by centrifugation, then carefully washed with n-hexane and methanol to eliminate any residual reactants or products. To restore its catalytic activity, the recovered nanocatalyst was subjected to calcination at 650 °C for a duration of five hours in a muffle furnace. Following this regeneration step, the catalyst was reused with fresh amounts of reactants under the same reaction conditions described in the experimental section. The results, summarized in Fig. 6, reveal that the biodiesel yield gradually declined from 99% in the first cycle to approximately 83% after the seventh cycle. This decrease in catalytic efficiency can be attributed

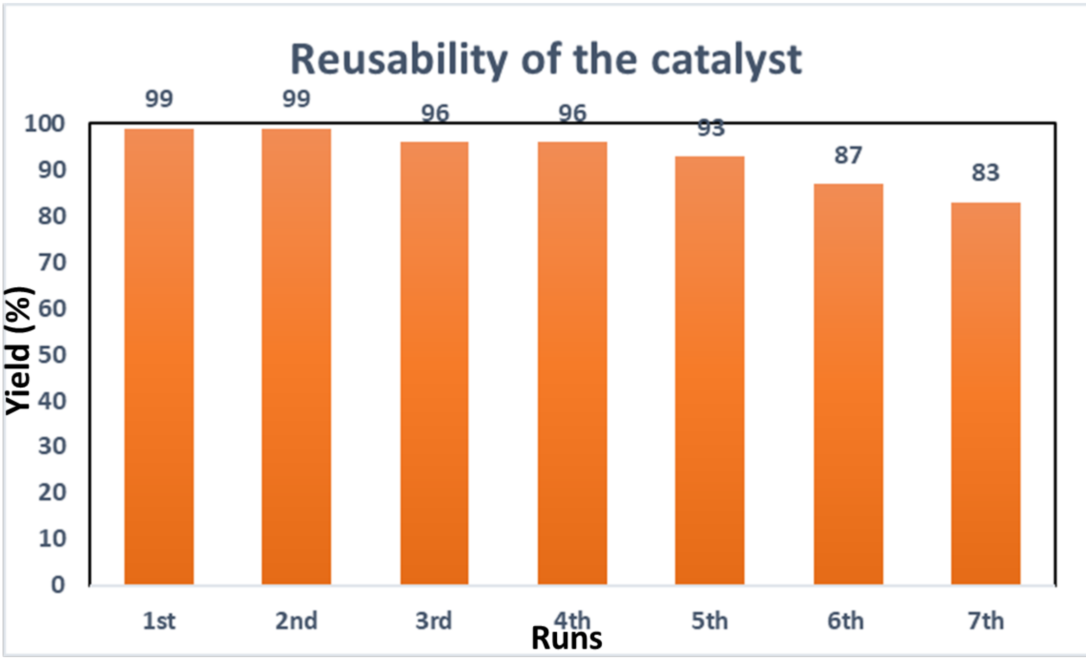

Fig. 6. The reusability of ZnO nanoparticles supported on multi-walled carbon nanotubes as a robust heterogeneous nano-catalyst for biodiesel production.

to several factors, including the possible hydration of ZnO by water molecules and its reaction with carbon dioxide from the ambient air during the separation process. Over multiple cycles, the active sites of the catalyst likely become partially blocked by byproducts or impurities, further reducing activity. The observed deactivation emphasizes the importance of understanding catalyst stability and potential mechanisms of deactivation, which are critical for optimizing long-term use and developing more durable materials for sustainable biodiesel production. Additionally, inductively coupled plasma (ICP) analysis showed that the amount of leached Zn^{2+} ions was minimal, with concentrations below 10 ppm, indicating excellent stability of the catalyst during repeated use and suggesting that ZnO leaching was not a significant factor in catalyst deactivation.

The utilization of ZnO nanoparticles supported on multi-walled carbon nanotubes as a robust heterogeneous nano-catalyst for biodiesel production represents a promising advancement in the field of renewable energy and catalysis. However, despite significant progress, several limitations and promising avenues for future research persist within this domain. One primary challenge is the stability and durability of nanocatalysts under industrially relevant conditions, where factors such as leaching of active metal species, deactivation due to fouling or poisoning, and structural degradation over multiple cycles can undermine long-term efficiency. Moreover, the synthesis and functionalization processes of such nanostructured catalysts often involve complex, time-consuming, and cost-intensive procedures, which hinder scalability and commercial viability. Additionally, there remains a need to optimize catalyst properties, such as surface area, active site accessibility, and electron transfer efficiency, to enhance catalytic activity and turnover numbers. Environmental and economic considerations also call for the development of greener synthesis routes and the use of non-toxic, abundant materials.

Future research directions could focus on designing multifunctional catalysts capable of simultaneously catalyzing multiple steps in biodiesel synthesis, such as esterification and transesterification, to streamline production processes. Investigating the synergistic effects of different nanomaterial supports, as well as exploring catalyst regeneration and recycling strategies, are also critical areas. Furthermore, studies on catalyst deactivation mechanisms at the molecular level, coupled with advanced characterization techniques like in situ spectroscopies and high-resolution microscopy, can provide insights necessary for the development of more resilient catalysts. Exploring the application of these catalysts in continuous-flow systems and scaling up laboratory findings to pilot and industrial levels are essential for practical implementation. Overall, addressing these limitations will be pivotal in advancing the field of heterogeneous catalysis for sustainable biodiesel production.

CONCLUSION

This study demonstrates the successful synthesis and application of ZnO nanoparticles supported on multi-walled carbon nanotubes (MWCNTs) as a highly effective heterogeneous nanocatalyst for biodiesel production from soybean oil. The characterization techniques, including SEM, XRD, and FT-IR, confirmed the uniform dispersion and crystalline nature of ZnO nanoparticles on MWCNT surfaces, promising enhanced catalytic properties. The experimental optimization revealed that the best biodiesel yield (up to 99%) was achieved at a methanol-to-oil molar ratio of 55:1, a reaction time of 45 minutes, and a catalyst amount of 35 mg, conducted at ambient temperature. Reusability assessments showed that the catalyst retained high activity over seven cycles, with a slight decline to about 83% yield, highlighting its potential for sustainable and cost-effective industrial applications. Minimal Zn^{2+} leaching further attests to its stability and environmental friendliness. These findings support the role of supported ZnO nanoparticles on CNTs as a robust, reusable, and efficient catalyst, aligning with the growing demand for greener biodiesel production methods. The tailored properties of this nanocatalyst make it a promising candidate for scaling-up in industrial biodiesel processes, offering significant advantages in yield, recyclability, and eco-compatibility. Future work should focus on enhancing catalyst durability and exploring continuous-flow systems to facilitate commercial viability.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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