



BARIUM FERRITE ($\text{BaFe}_{12}\text{O}_{19}$) NANOPARTICLES FOR EFFICIENT BIODIESEL SYNTHESIS USING NON-EDIBLE TARAMIRA OIL

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ABSTRACT

This study examines the potential of nanoparticles to investigate the transesterification reaction of non-edible taramira oil (TMO) for efficient biodiesel synthesis. The barium ferrite nanoparticles ($\text{BaFe}_{12}\text{O}_{19}$) were synthesized and calcined at 700 °C. X-ray diffraction (XRD) analysis revealed a cubic spinel nanostructure, indicating the pure-phase barium ferrite nanoparticles with an average size of 44 nm. Fourier transform infrared (FTIR) spectroscopy confirmed the presence of metal-oxide bonds, as evidenced by bands at 540–615 cm^{-1} . The SEM images showed the grain size of barium hexaferrite nanorods between 40 nm to 60 nm. The synthesized barium ferrite nanoparticle-based taramira oil biodiesel (BTMBD) was confirmed by FTIR spectroscopy through characteristic C=O stretching peaks at 1738.24 cm^{-1} . The maximum yield of 91.5 % of BTMBD (biodiesel) was achieved at (TMO) to methanol of 1:12 molar ratio, 2 % barium ferrite nanoparticles concentration, 60 °C of reaction temperature, and 120 minutes of reaction time. In conclusion, the current barium ferrite nanoparticles have the potential to aid as a potent nanocatalyst to produce biodiesel, demonstrating their role in the current energy crisis solution, and could be utilized for large-scale biodiesel production.

KEYWORDS: Nanocatalyst, Non-Edible Oil, Transesterification, Biodiesel, FTIR

Introduction

The intense need to decrease the emission of greenhouse gases and to expand the liquid-fuel supply has led to research into renewable biodiesel as an alternative to non-renewable fuels. Biodiesel, known as fatty acid alkyl esters, is obtained from triglyceride feedstocks via the transesterification reaction. Due to the limitations of the homogeneous base catalysts (KOH, NaOH), there is a motivation to improve heterogeneous catalyst systems for industrial-scale biodiesel production¹. The heterogeneous nano catalyst, in comparison to the bulk catalyst, provides active site availability with a high surface area, which leads to faster biodiesel synthesis at mild conditions. Recent research highlights rapid progress in metal oxides, functionalized magnetic particles, mixed oxides nanocatalysts, and establishes improved stability, recyclability, and catalytic activity during transesterification reactions².

Iron-based oxides, such as magnetic ferrites, have been explored as active catalysts and supports for organic transformations and biodiesel production. The ferrite nanocatalyst (MFe_2O_4 , where M = Co,

Zn, Ni, Ba, Fe) was functionalized and revealed permanent chemical robustness and magnetism, providing useful platforms for improving catalyst handling and conversion³. Barium ferrite (Ba–Fe oxide), due to its chemical stability and magnetic properties, gained attraction as a magnetically recoverable catalyst. Ba-ferrite nanocatalysts are easily recoverable from the reaction mixture, and their synthesis method can influence their separation and catalytic activity³.

The selection of feedstock strongly influences biodiesel sustainability and process design. In the synthesis of biodiesel, the choice of feedstock solution plays a critical role in sustainability. Taramira oil is an efficient feedstock for the effective synthesis of biodiesel. The non-edible taramira (*Eruca sativa*) is an annual herb and is mostly grown in the Middle East, Pakistan, and India. Research has explored the conversion of taramira oil using a base catalyst with optimized reaction parameters⁴. The *E. sativa* seed oil has a pungent odor and flavor, making it unsuitable for food. Due to the ability to gain oil content to 35 % by weight, the *E. sativa* seed oil is well-suited for large-scale production of biodiesel⁵.

In the present study, the barium ferrite nanoparticles were prepared via a co-precipitation route. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were used to analyze the synthesized nanocatalyst. The performance of characterized heterogeneous nanocatalysts was evaluated in taramira oils transesterification. The synthesized biodiesel was characterized by using FTIR analysis, which confirms ester formation with functional group changes from the parent oil. The study aimed to reveal a practice able, eco-friendly, and sustainable biodiesel production using nanocatalysts and non-edible seed oils.

Materials and Methods

Synthesis of barium ferrite nanoparticles (BaFe₁₂O₁₉)

Barium ferrite nanoparticles were synthesized by using the methodology with slight modifications and were obtained via the co-precipitation route from a solution of barium and ferric nitrates⁶. The aqueous solution of iron nitrate and barium nitrate with a Fe³⁺/Ba²⁺ molar ratio of 8 was prepared. This aqueous solution was co-precipitated via sodium hydroxide (NaOH) solution (0.5 M). The solution was stirred at 200 rpm for 2 hours until it reached a pH of 9 to achieve good homogeneity. The resulting precipitation was washed 3 to 5 times with distilled water, dried for hours at 90 °C, and calcinated for 3 hours at 700 °C. The final calcinated barium ferrite nanoparticles were placed in a glass vial and labeled for further use in characterization and biodiesel synthesis.

Characterization of nano-catalysts

The prepared nano catalyst Barium ferrite was characterized according to composition phase, crystalline size, containing functional group, and structural morphology using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscopy (SEM), respectively.

X-ray diffraction (XRD)

The X-ray diffraction pattern of the current nanoparticles was measured by using an X-ray diffractometer. The diffraction angle range of $2\theta = 10^\circ$ to 80° , scanning speed of $2^\circ/\text{min}$, and $\lambda = 0.15406 \text{ nm}$ was used to generate crystallite size and phase composition analysis of synthesized nanoparticles⁷. The crystalline structure and phase of the synthesized nanoparticles using the coprecipitation method were identified using XRD analysis. The Debye-Scherrer formula (equation 1) was applied to calculate the average crystalline size and expressed as.

$$D = (0.9 \lambda) / (\beta \cos \theta) \quad \text{Equation 1}$$

Where,

D = average crystalline size for the sample

λ = wavelength of the CuK α radiation source

β = Full width at half maximum

θ = Bragg angle

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a useful approach for evaluating nanomaterial conformational changes linked through chemical interventions. The functional group of nano catalysts was studied using the (FT-IR) Spectrophotometry with Bruker-Tensor 27. In the wavelength region of 4000 – 500, spectra were collected⁸.

Scanning electron microscopy (SEM)

Tuscan VEGA 3 (SEM) was used, and the morphological characteristics of the synthesized nano catalyst were studied⁹. The nanoscale surface morphology and distribution of the synthesized barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) were analyzed via SEM. The images of SEM were noted at 30,000x ($5\mu\text{m}$) and 60,000x ($0.2\mu\text{m}$) to understand the surface topographies and size characteristics of the nanoparticles. For enhancing electrical conductivity as well as to get better resolution of the images, all the samples were coated via a sputter coater with a thin layer of gold.

Screening of non-edible taramira oil (TMO) for biodiesel synthesis

Numerous functional groups of feedstock oils and produced biodiesels were identified utilizing FTIR spectroscopy¹⁰. The non-edible TMO was used as feedstock for biodiesel production using barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) nanoparticles. The feedstock was analyzed for FTIR analysis, and the collected spectra were analyzed for their functional groups.

Transesterification reaction via nano catalyst for biodiesel synthesis

The transesterification catalyzed by barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$) was performed in a round-bottom (250 ml) flask with two necks related to a reflux condenser and a temperature-controlled hot plate. A 40 ml known quantity of (TMO) was utilized for the transesterification reaction. After the transesterification reaction, the transesterified mixture was centrifuged at 4000 rpm to remove the barium ferrite catalyst. The mixture was then aseptically transferred to a separating funnel, resulting in distinct layers of biodiesel and glycerol. The synthesized biodiesel was purified by washing five times with distilled water, and equation (2) was used to estimate the total biodiesel yield¹¹.

Percentage yield

$$= \frac{\text{Gram of biodiesel produced}}{\text{Gram of oil used}} \times 100 \quad \text{Equation 2}$$

Characterization of synthesized biodiesel

The prepared biodiesel was characterized by using Fourier-transformed infrared spectroscopy (FTIR). To study the structural arrangement and functional group of prepared biodiesels, the Bruker-Tensor 27 FTIR was utilized, and the spectrum was achieved in the range of 4000 cm^{-1} - 500 cm^{-1} ¹².

Optimization of synthesized biodiesel

Various parameters, like (TMO) to methanol molar ratio, concentration of the catalyst, temperature of reaction, and time for the reaction, were evaluated to obtain the maximum yield of biodiesel.

TMO to methanol molar ratio

The feedstock (TMO) to alcohol ratio serves as an important variable and can directly affect the biodiesel yield. The effect of different TMO-to-methanol ratios ranging from (1:3 to 1:15) on biodiesel yield was evaluated. The remaining transesterification reaction parameters, like catalyst concentration (1.5 w/v %), 90 minutes of reaction time, 60°C reaction temperature, and stirring speed with 600 rpm, remained fixed during TMO to methanol molar ratio optimization. A suitable oil-to-methanol molar ratio of 1:3 is required for the transesterification reaction to produce triglyceride (3 moles) and glycerol (1 mol).

Catalyst concentration

In the transesterification reaction of TMO into biodiesel, the effect of barium ferrite nano-catalyst concentration (0.5 to 2.5 w/v %) on biodiesel yield was evaluated. The other transesterification reaction parameters, TMO to methanol molar ratio (1:12), temperature of 60 °C, reaction time of 90 minutes, and 600 rpm, remained fixed during the catalyst optimization experiment.

Reaction temperature

The different reaction temperature ranges from 50 °C to 90 °C were employed during the present study to examine the influence of temperature on the production yield of biodiesel. The previously optimized TMO to methanol ratio (1:12), 2 % catalyst concentration, 90 minutes of reaction time, and 600 rpm stirring speed remained fixed during the study of the reaction temperature.

Reaction time

The effect of different reaction times, ranging from 60 minutes to 180 minutes, was evaluated on the production yield of synthesized biodiesel. The previously optimized TMO to methanol ratio of 1:12, 2 % w/v nano catalyst concentration, 60 °C temperature, and 600 rpm stirring speed were fixed during the optimization of the reaction time experiment.

Results and Discussion

Synthesis and characterization of ($\text{BaFe}_{12}\text{O}_{19}$) nanoparticles

The barium ferrite nanoparticles were synthesized via the co-precipitation route and analyzed using XRD, FTIR, and SEM analysis.

X-ray diffraction analysis of ($\text{BaFe}_{12}\text{O}_{19}$)

The XRD pattern of the barium ferrite nanoparticles is presented in Fig.1, which reveals 2θ values peaks at 30.52 °, 32.21 °, 34.12 °, 37.31 °, 56.21 °, and 62.42 ° of the (114), (203), (205), (206), (220), and (304). These peaks are referred to as the cubic spinel structure, thus indicating that the barium ferrite nanoparticles synthesized here are pure-phase barium ferrite. Like the above results, the XRD data also matched well with the standard reference for $\text{BaFe}_{12}\text{O}_{19}$ (JCPDS card No. 00-43-0002)¹³.

Fourier transform infrared spectroscopy (FTIR) analysis of ($\text{BaFe}_{12}\text{O}_{19}$)

The barium ferrite nanoparticles were analyzed via FTIR analysis in the functional group region of 500–4000 cm^{-1} as depicted in Fig.2. The FTIR spectrum shows evident peaks with a strong absorption band at 862.36 cm^{-1} , indicating the Ba-O bond as metallic stretching vibration^{14,15}. The band at about 1450 cm^{-1} is also assigned to the stretching of the –OH group. Moreover, a generally diffused band in the region 3200–3500 cm^{-1} refers to O-H stretching vibrations of free and hydrogen-bonded hydroxyls¹⁴. The structure contains both tetrahedral (A) and octahedral (B) iron sites. The metal-oxygen stretching vibrations (Fe-O) in these distinct sites produce two main absorption bands in the FTIR spectrum, which are crucial indicators for the single-phase formation of ferrite structure. The bands at 540–615 cm^{-1} indicate metal-oxygen (Fe-O) bond stretching and are evident in hexaferrite structures with both tetrahedral and octahedral sites in the formation of single-phase ferrite nanoparticles¹⁶.

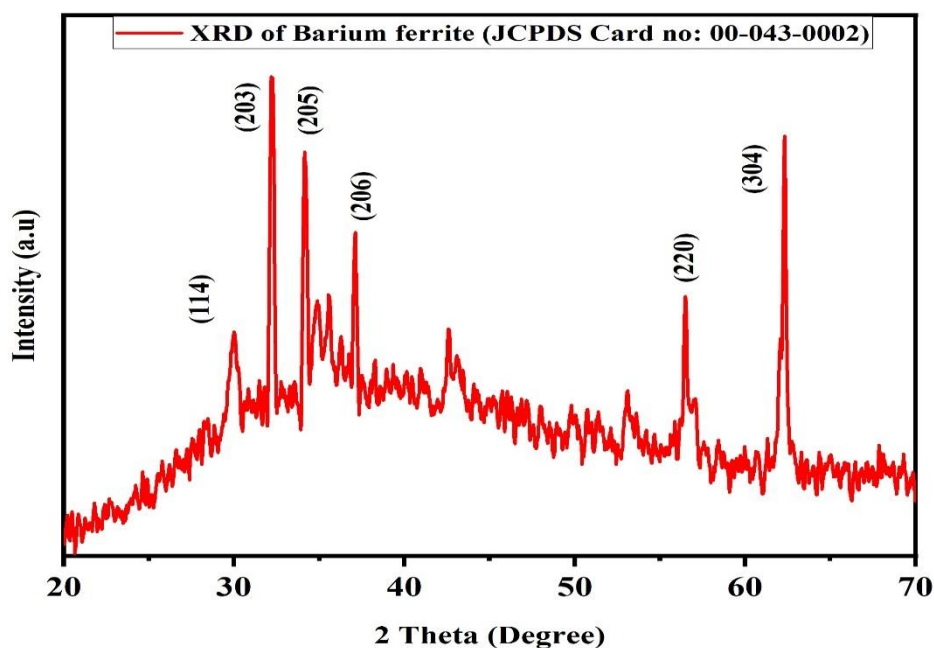


Figure 1: X-rays diffraction pattern of barium ferrite nanoparticles

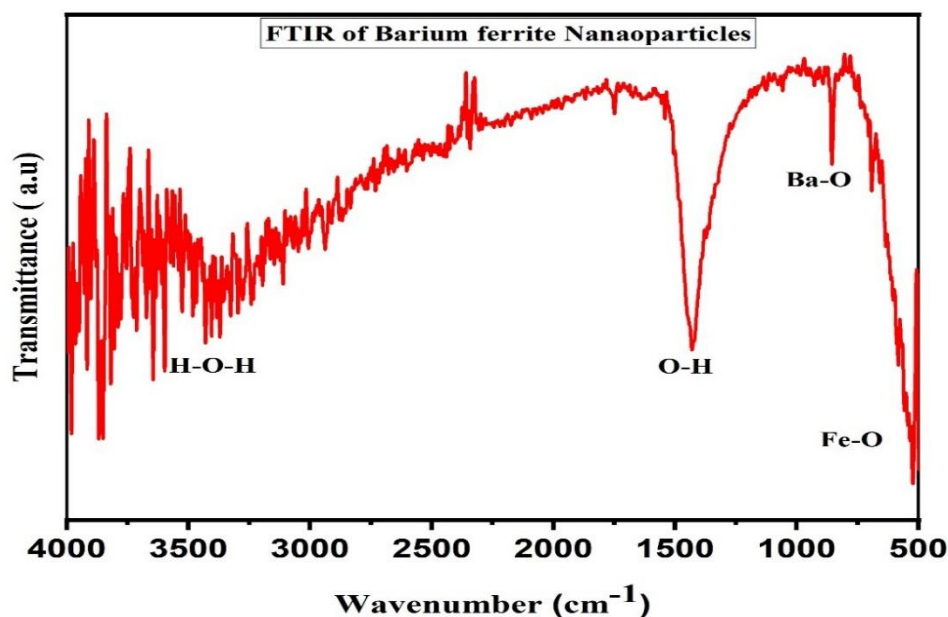


Figure 2: FTIR spectra of barium ferrite nanoparticles

Scanning electron microscopy (SEM) of ($\text{BaFe}_{12}\text{O}_{19}$)

The SEM images depicted in Fig. 3A and 3B show a nice spherical cubic shape and sharp corners, which state that the synthesized material is more crystalline in nature. At slow magnification, the particles are uniformly distributed and of smooth spherical surface. The particle sizes were between 40 and 60 nm, with about 44 nm of average size, reasonably close to the values documented in other studies of barium ferrite nanoparticles prepared using co-precipitation techniques¹³. Similarly, the crystalline size of barium hexaferrite nanocatalyst depends on heat treatment and synthesis and ranges from ~25–100 nm¹⁷. These findings, therefore, validate the success of synthesizing nanoparticles

with a narrow size distribution and different morphology, where most of the differences may have been caused by differences in synthesis methods.

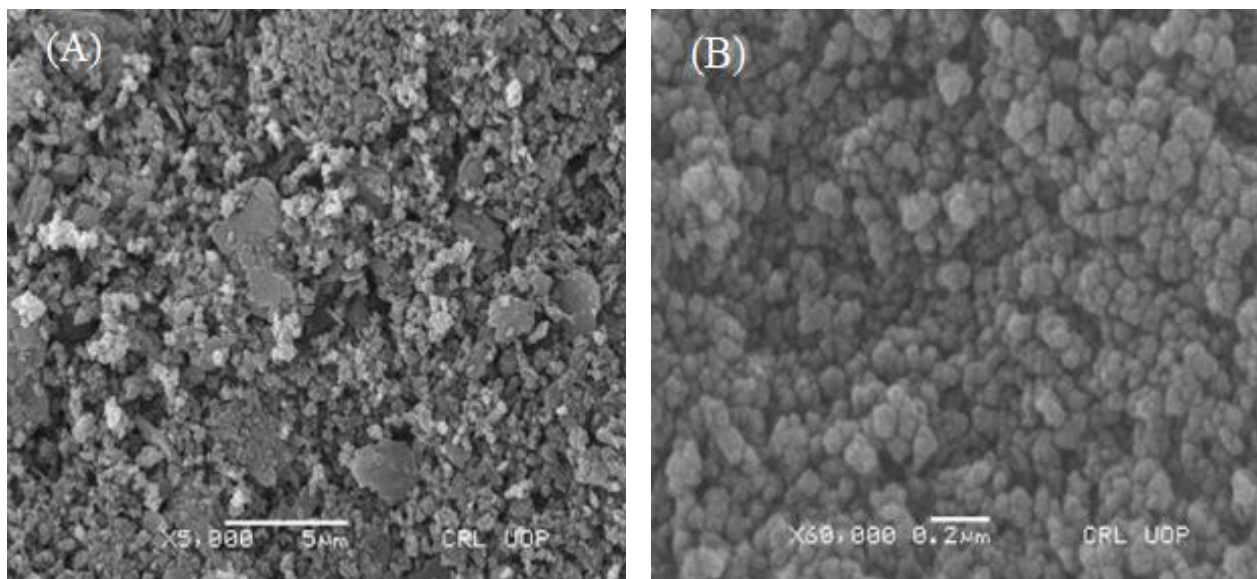


Figure 3: (A): Barium ferrite nanoparticles SEM image with 30,000 (5μm) resolution

(B): Barium ferrite nanoparticles SEM image with 60,000 (0.2μm) resolution

FTIR analysis of non-edible TMO for biodiesel

The collected FTIR spectrum of non-edible taramira oil (TMO) was noted around 3005.96 cm^{-1} , 2919.66 cm^{-1} , 2855.91 cm^{-1} , 1460.86 cm^{-1} , 1376.62 cm^{-1} , and 721.18 cm^{-1} , representing the alkanes group (C-H) stretching. The robust peak at 1744.40 cm^{-1} indicated the ester group (C=O) stretching. The peak at 1238.95 cm^{-1} indicates the group of alkyl halide. The peak with medium size at 1000.24 cm^{-1} indicates the aliphatic amine group. Fig.4 shows the collected FTIR spectrum of non-edible taramira oil (TMO).

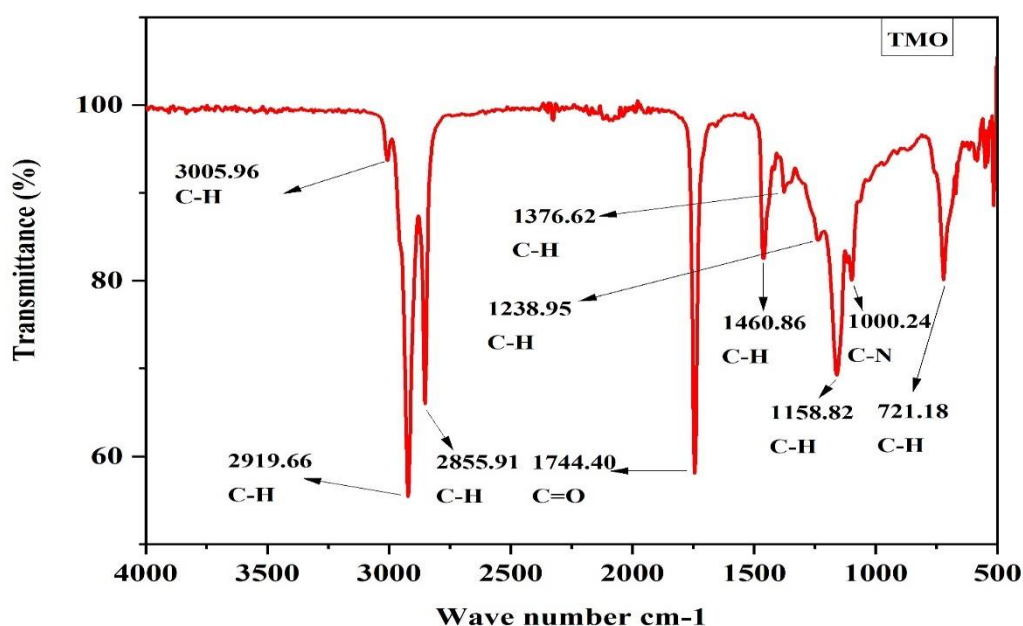


Figure 4: FT-IR spectrum of non-edible taramira oil (TMO)

FTIR analysis of barium ferrite nanoparticles based on taramira oil biodiesel (BTMBD)

The functional group peaks of BTMBD (biodiesel) appeared around 3010.07 , 2925.80 , 2849.80 , 1462.91 , 1454.15 , and 754.05 cm^{-1} , indicating the presence of alkane groups (C-H stretching) in the synthesized biodiesel. The characteristic and strong peak in FTIR spectra was observed at 1738.24 cm^{-1} in BTMBD (biodiesel), which denotes the C=O carbonyl group in the synthesized biodiesel. The presence of the carbonyl group (C=O) at 1738 cm^{-1} in the FTIR spectrum of nanoparticle-based biodiesel confirms the production of methyl ester¹⁸. Peak at 1360.18 cm^{-1} denoted the methyl group, while the alkyl halide group was noticed at 1167.04 cm^{-1} . The medium-sized peak at 1051.94 cm^{-1} identifies the aliphatic amines group (C-N stretching) in BTMBD (biodiesel). Fig. 5 shows the collected FTIR spectrum of BTMBD (biodiesel).

Both the TMO and BTMBD show minor changes in biochemical and display analogous spectra, while a few variations in absorption frequency and band strength were recorded for both spectra. The confirmative stretching (C=O) was observed in BTMBD (biodiesel) by a peak at 1738.24 cm^{-1} with an IR shift. The IR shift in the C=O carbonyl group confirms the successful conversion of free fatty acids in TMO (oil) into their comparable methyl esters in (BTMBD) biodiesel¹⁹. In comparison to the TMO spectrum, the BTMBD (biodiesel) spectrum was observed with an extra peak at 1462.91 cm^{-1} representing additional (C-H stretching) in BTMBD (biodiesel). The peak position at 721.18 cm^{-1} in the TMO spectrum was observed, while the same peak position at 754.05 cm^{-1} was observed in the BTMBD (biodiesel) IR spectra. The TMO FTIR spectrum had two peaks for the alkyl halides group, while the BTMBD (biodiesel) spectrum was observed with only one peak for the alkyl halides group.

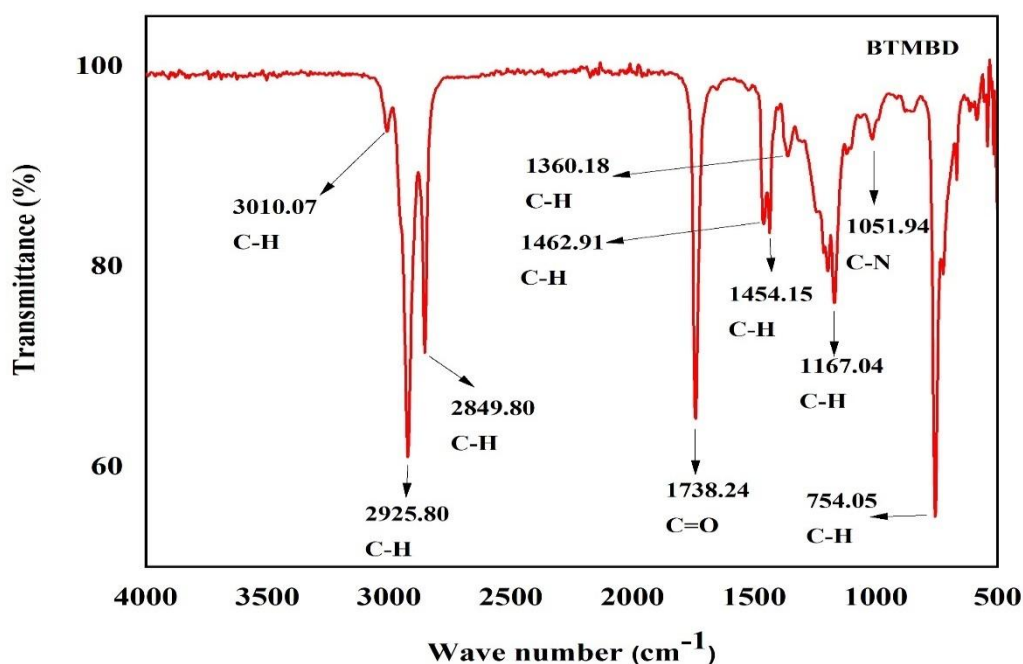


Figure 5: FT-IR spectrum of BTMBD using $\text{BaFe}_{12}\text{O}_{19}$ nanocatalyst

Optimization of BTMBD (biodiesel)

TMO to methanol molar ratio

The current study results revealed that the BTMBD (biodiesel) yield increases from 62 % to 88 % by increasing the TMO to methanol molar ratio from 1:3 to 1:12, and the optimum (BTMBD) biodiesel yield of 88 % was achieved at 1:12 TMO to methanol molar ratio. An excessive volume of oil to methanol molar ratio is required to make methoxy species, as a result catalyst surface accelerates and moves the transesterification reaction in the forward direction²⁰. Furthermore, increasing the feedstock (oil) to methanol molar ratio to 1:15 led to a decreased BTMBD (biodiesel) yield of 79 %

(Fig. 6A). The reduction of biodiesel yield occurred due to an increase in the methanol ratio, which led to catalyst deactivation and caused trouble in biodiesel separation from glycerol²¹.

Catalyst concentration

The barium ferrite nanocatalyst at 0.5 % showed no significant effect and resulted in a 64 % yield of BTMBD (biodiesel). However, the yield of BTMBD (biodiesel) improved from 64 % to 90 % by increasing the nano catalyst concentration from 0.5 to 2 w/v %. It was obtained that the barium ferrite nano catalyst at a concentration of 2 w/v % yielded 90 % of BTMBD (biodiesel) yield (Fig. 6 B). The metal-oxide nanocatalysts enhance reaction kinetics, facilitating mass transfer by lowering the activation energy, resulting in improved yield of biodiesel²². A significant decline was observed in biodiesel yield when the catalyst amount exceeded 2 w/v % and 85 % yield of BTMBD (biodiesel) was observed at 2.5 w/v % concentration of barium ferrite nano catalyst. The exceeding of nano catalyst concentration from their optimum point can lead to viscous bodies synthesis, which disturbs sufficient stirring during the transesterification reaction, resulting in decreased yield of biodiesel²³.

Reaction temperature

The lower yield of 78 % of BTMBD (biodiesel) was obtained at 50 °C temperature, while increasing the temperature gradually to 60 °C, the optimum yield of 90.5 % of BTMBD (biodiesel) was obtained. The temperature at a particular range significantly increases the rate of transesterification reaction, and at this point, the solubility of methanol increases, resulting in sufficient biodiesel yield²⁴. The yield of biodiesel declined by exceeding the reaction temperature beyond 60 °C, and the lowest yield of 63 % of BTMBD (biodiesel) was obtained at 90 °C (Fig. 6C). The overall result indicated that the optimum (90.5 %) of biodiesel yield was achieved at 60 °C temperature and marked as an optimal temperature during transesterification.

Reaction time

The (Fig. 6D) shows the result and indicates that the yield of BTMBD (biodiesel) increased by increasing the time of reaction from 90 to 120 minutes. Initially, a 60-minute duration of the transesterification reaction yields 77 % of BTMBD (biodiesel). The optimum yield of BTMBD (91.5 %) was achieved at 120 minutes. The yield of biodiesel declined with increasing reaction time, from 120 minutes, and at 180 minutes, 78 % of BTMBD (biodiesel) yield was obtained. This is evidenced by the present study result that the optimum yield of biodiesel (88 %) was obtained with an optimum reaction time of 120 minutes²⁵. The overall result for BTMBD (biodiesel) optimization revealed that a maximum yield of 91.5 % of BTMBD (biodiesel) was achieved at a TMO to methanol molar ratio of 1:12, 2 w/v % barium ferrite nano catalyst concentration, 60 °C reaction temperature, and 120 minutes of reaction time.

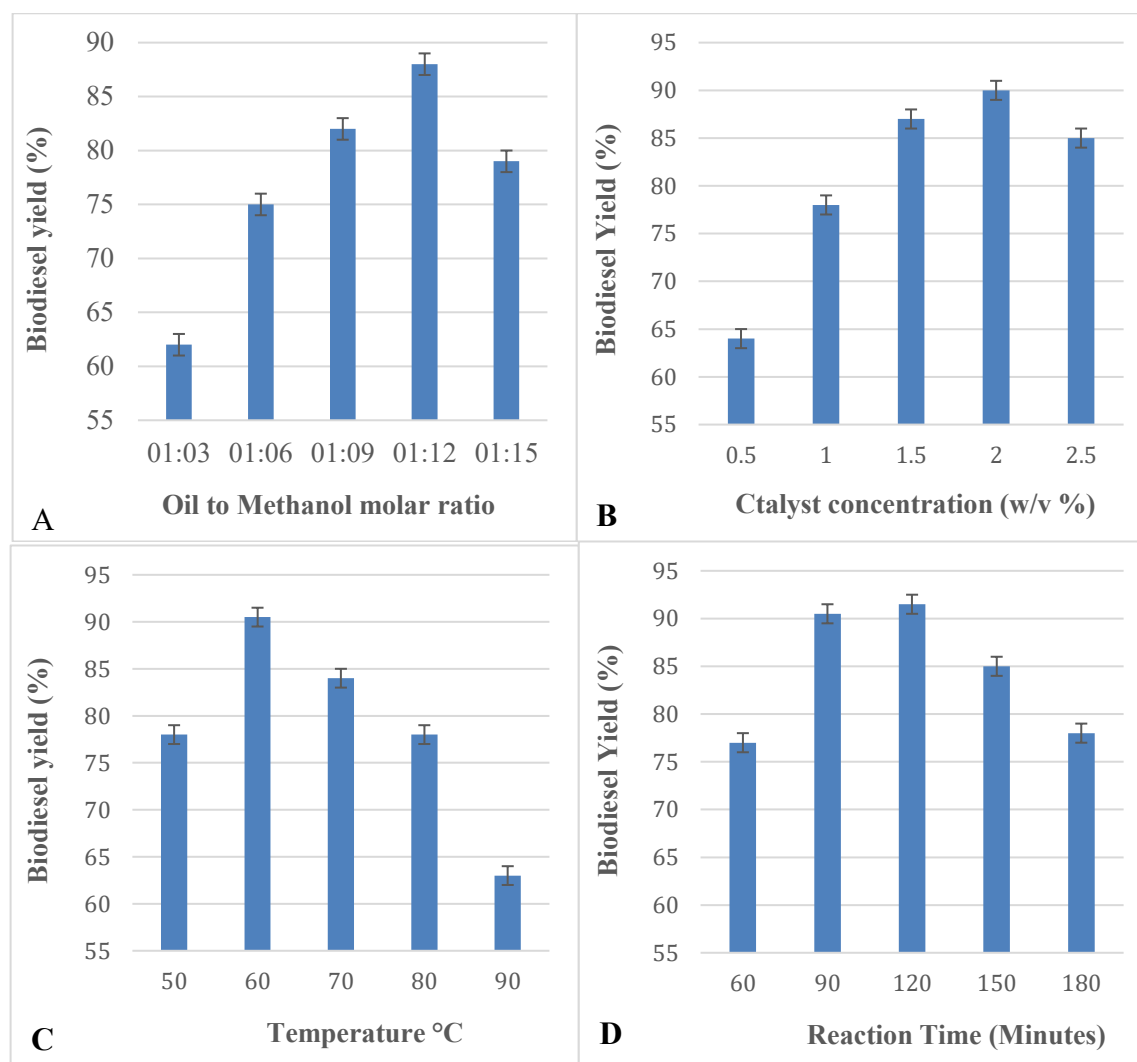


Figure 6: Effect of (A) TMO to methanol molar ratio (B) Catalyst concentration. (C) Temperature (D) Reaction time on BTMBD (biodiesel)

Conclusion

The current study reveals that barium ferrite nanoparticles might be a decent source for the large-scale synthesis of biodiesel. XRD, FTIR, and SEM analysis of barium ferrite nanoparticles reveals a cubic spinel nanostructure, indicating that the nanoparticles are pure-phase barium ferrite. The current study was limited to pilot-scale trials, and further research is needed to evaluate the barium ferrite nano catalyst stability and reusability with the feasibility of a large-scale biodiesel production process. Similarly, expanding the scope of other non-edible and waste oil substrates can improve sustainability. Likewise, the environmental and economic assessment will support industrial-scale production of biodiesel. The study provides insight into cleaner biodiesel synthesis by using plant-based raw materials and barium ferrite nano catalysts. However, further research is needed, which would facilitate the design and cost assessment of biodiesel efficiency achieved through large-scale operation.

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Conflicts of Interest

The authors declare no conflict of interest

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