Optimising Process Parameters for Bauhinia Monandra Biodiesel Production and Characterization

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Abstract: The objective of this study is to enhance the efficiency of biodiesel production from Bauhinia monandra seeds through the application of response surface methodology (RSM). The subsequent evaluation will focus on the fuel characterisation and properties measurement. The process was optimised by adjusting the methanol-oil molar ratio (MOR), reaction (RTe), and reaction time (RTm). The ASTM set the standards for conducting the property measurements, and the fuel characterisation was performed using Fourier transform infrared spectroscopy (FTIR). The optimisation analysis revealed that the highest yield of BMB was achieved by employing an MOR of 7.4:1, keeping a temperature of 80 °C, and allowing the reaction to occur for a duration of 64 minutes. In optimal circumstances, the yield rate of BPB is recorded at 89.3%. According to FTIR, the BMB consists of carbon-based components of superior quality, and the measured physicochemical properties of fuel meet the required standards.

Keywords: Waste to Energy, Bauhinia monandra, Optimization, FTIR, GC-MS.

1. Introduction

The utilisation of fossil fuels in transportation and electricity production has experienced a concurrent increase with the expansion of the global economy over the last forty years. Human activities are responsible for both the depletion of fossil fuel resources and the degradation of the ecosystem [1]. The utilisation of diesel power presents significant environmental and sustainability concerns, hence necessitating the prioritisation of renewable diesel fuel alternatives by engine manufacturers and scholars [2]. Biodiesel, an environmentally friendly fuel with potential benefits for engine performance, is being considered as a potential substitute for conventional diesel fuel. The implementation of the biodiesel diesel power concept has the potential to effectively mitigate climate change, ensure a stable energy supply, and improve the financial well-being of farmers [3].

Biofuels are categorised into three generations based on the feedstock used. First-generation biofuels encompass ethanol and biodiesel derived from agricultural crops. Maize and sugarcane are widely recognised as the predominant sources for bioethanol production [4]. Second-generation biofuels are derived from biomass that may be categorised into three types: homogeneous, quasi-homogeneous, and non-homogeneous. The distinction between the latter two types is based on factors such as the availability and cost of the feedstock [5]. Third-generation biofuels are frequently derived from algal biomass, which has a higher growth rate compared to lignocellulosic biomass. The lipid content of algae plays a crucial role in the production of biofuels, making water and geography important factors in this process [6]. Numerous scientists have successfully addressed the obstacles associated with biodiesel production by employing the conversion of agricultural waste into a viable energy source. The utilisation of Bauhinia monandra seed oil as a potential source of biodiesel within the category of solid biowastes has been demonstrated. Despite the recognised medical and cultural significance of Bauhinia tree leaves, farmers tend to disregard the potential benefits associated with the seeds [7, 8]. The utilisation of Bauhinia monandra seeds for the production of biodiesel is a promising approach to mitigating biowaste and enhancing agricultural profitability. The cultivation of Bauhinia monandra seeds sourced from semi-green woodlands and gardens in India is characterised by minimal effort [9, 10].

The majority of vegetable seeds undergo the process of expeller-pressing to extract bio-oil, which is subsequently transformed into biodiesel through transesterification using a catalyst. The conversion of high-viscosity triglycerides into the fatty acids methyl ester and glycerol is achieved with enhanced efficiency with the use of catalysts [11,12]. The utilisation of a heterogeneous catalyst such as sodium phosphate is suggested due to its ability to undergo recycling without the need for water treatment [13]. During the process of
transesterification, it is possible for the methanol-oil molar ratio (MOR), reaction (RTe), and reaction time (RTm), to undergo variations. The catalyst facilitates the hydrolysis of the strong base, resulting in the formation of ions, and enhances the rate of transesterification with methanol [14]. The conversion of crude oil triglycerides into diglycerides, monoglycerides, and methyl ester is a crucial step in the production of biodiesel. The duration of the transesterification process plays a significant role in determining the quantity of biodiesel obtained [15]. The reduction of biodiesel viscosity during esterification can be achieved by controlling the reaction temperature [16].

Perumal et al. [17] employed a two-step method consisting of esterification followed by transesterification in order to decrease the viscosity of biodiesel derived from seeds. The experimental methodology involved the utilisation of a mixture consisting of 0.3 to 0.35 volume/volume (v/v) of MOR and 1% v/v acid catalyst for a reaction duration of 60 minutes in the esterification process. Additionally, a combination of 0.25% v/v MOR and 0.7% v/v alkaline catalysts was utilised in the transesterification process to provide biodiesel of similar grade. The authors employed a comparable methodology to extract biodiesel from the seeds of Bauhinia variegata and documented the remarkable quality of the resulting biodiesel [18]. The identification of the optimal values for each input parameter is crucial for biodiesel producers in order to achieve maximum yield and minimum expenses [19]. The investigation on optimising the parameters of the transesterification process for pongamia oil revealed that a process temperature of 120 °C, a molar ratio of 16:1, a catalyst concentration of 0.75 wt%, and a reaction duration of 3 hours resulted in a significantly higher yield of pongamia methyl ester, around 90% [20]. In this study, response surface methodology (RSM) is employed to determine the ideal values for the controls.

Based on the above discussions, it is observed that the biodiesel derived from seeds belonging to the Bauhinia family have emerged as a potential substitute for conventional diesel. To the best of the authors' knowledge, there has been no prior research conducted to optimise the parameters of the transesterification process for the synthesis of BMB, and this work is the first to characterise the BMB prepared at optimum condition. The production of biodiesel from Bauhinia monandra seeds was achieved using the process of transesterification. The response surface approach, which is a multi-response optimisation technique, was employed to determine the ideal process parameters and validation degree. The utilisation of Fourier transform infrared spectroscopy (FTIR) facilitated the examination of the product, enabling the ascertainment of the composition of functional groups and the overall yield of the product.

2. Materials and Methods

2.1 BMB processing

The mechanical extraction method was employed to get crude oil from the seeds of Bauhinia monandra. In this experimental setup, a three-necked container was employed to house crude oil, which was subjected to mechanical agitation. A catalyst (sodium phosphate - Na3PO4), with a volume convergence of 2% and methanol (CH3OH), at varying concentrations, were mixed with crude oil at a temperature of 50 °C. The rotational speed of the transesterification cycle was set at 500 rpm. The fermentation process was conducted under optimal conditions in terms of temperature and duration. Following the completion of the reaction, the methyl ester, glycerine, and sodium phosphate catalysts were separated in a dedicated channel. Following the separation of the methyl ester from glycerine and the catalyst, the subsequent step involved the purification and desiccation of the compound. The transesterification flowchart is depicted in Figure 1.

![Figure 1. Transesterification flowchart](image-url)
Table 1. Input parameter and corresponding response

<table>
<thead>
<tr>
<th>Run No</th>
<th>MOR</th>
<th>RTe in °C</th>
<th>RTm in mins</th>
<th>BMB yield in %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.5</td>
<td>100</td>
<td>40</td>
<td>83.1</td>
</tr>
<tr>
<td>2</td>
<td>7.5</td>
<td>75</td>
<td>60</td>
<td>87.9</td>
</tr>
<tr>
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<td>7.5</td>
<td>50</td>
<td>80</td>
<td>83.3</td>
</tr>
<tr>
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<td>5</td>
<td>50</td>
<td>60</td>
<td>80.7</td>
</tr>
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<td>75</td>
<td>60</td>
<td>88.1</td>
</tr>
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<td>10</td>
<td>75</td>
<td>40</td>
<td>82.2</td>
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<td>75</td>
<td>80</td>
<td>81.9</td>
</tr>
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<td>5</td>
<td>75</td>
<td>40</td>
<td>79.9</td>
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<td>10</td>
<td>100</td>
<td>60</td>
<td>84</td>
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<td>7.5</td>
<td>100</td>
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<td>85.5</td>
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<td>75</td>
<td>60</td>
<td>87.8</td>
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</table>

Table 2. Analysis of variance

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F-value</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
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<td>14.44</td>
<td>293.83</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>A-MOR</td>
<td>8.05</td>
<td>7.34</td>
<td>148.02</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>B-RTe</td>
<td>8.23</td>
<td>7.53</td>
<td>151.85</td>
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</tr>
<tr>
<td>C-RTm</td>
<td>9.4</td>
<td>8.7</td>
<td>175.9</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>AB</td>
<td>1.9225</td>
<td>0.8334</td>
<td>15.24</td>
<td>0.0073</td>
</tr>
<tr>
<td>AC</td>
<td>1.2225</td>
<td>0.03456</td>
<td>0.6724</td>
<td>0.6233</td>
</tr>
<tr>
<td>BC</td>
<td>1.24</td>
<td>0.06</td>
<td>0.9245</td>
<td>0.4564</td>
</tr>
<tr>
<td>A²</td>
<td>46.09</td>
<td>45.39</td>
<td>930.05</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>B²</td>
<td>17.05</td>
<td>16.35</td>
<td>333.08</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>C²</td>
<td>32.82</td>
<td>32.11</td>
<td>657.16</td>
<td>&lt; 0.0001</td>
</tr>
<tr>
<td>Lack of Fit</td>
<td>0.3131</td>
<td>0.0998</td>
<td>6.42</td>
<td>0.0798</td>
</tr>
</tbody>
</table>

< 0.0001: significant; >0.05: not significant

Figure 2. (a) Residual vs Run; and (b) Predicted vs Actual

2.2 Process parameters selection

This study aimed to optimise the MOR, RTe, and RTm in order to enhance the BMB yield. Process parameter limits were established by selecting minimum and maximum values for MOR, RTe, and RTm. The MOR range was set between 5:1 and 10:1, while the temperature range for RTe was determined to be between 50°C and 100°C. Additionally, the time range
for was defined as 40 minutes to 80 minutes. The conversion of triglycerides into methyl esters requires the presence of alcohol to facilitate the reaction. Excessive methanol concentration leads to catalyst dilution, resulting in suboptimal mixing conditions. The dispersion rates of sodium phosphate are enhanced with an increase in process temperature, mostly attributed to improved mixing efficiency and reduced viscosity. The catalyst exhibits an enhanced yield of BMB as a result of an evenly distributed distribution [21]. The process of saponification is significantly enhanced when conducted at elevated temperatures. Therefore, the temperature range of the process was restricted to 50–100 degrees Celsius. The process of transesterification necessitates a sufficient duration for the conversion of triglycerides into methyl esters. The elevated hydrolysis rates at high temperatures enhance the potential for glycerine conversion [22]. Therefore, the lowest and maximum response times (RTs) were 40 and 80 minutes, respectively. The tuning of the three input parameters was conducted using the Box-Behnken design of RSM. Table 1 presents the input parameter and corresponding response.

2.3. Fuel characterisation

The chemical composition of BMB was determined through analysis of its FTIR spectrum. Infrared light induces distinct peaks in atomic bond frequencies, facilitating the identification of BPB functional groups. The wavelength peak of infrared light is contingent upon the specific functional group under investigation, thereby rendering it a distinctive characteristic. The identification of chemical bonds is facilitated by the analysis of their respective peak intensities and wavenumbers [23, 24]. The fatty acid profile of BMB was evaluated using gas chromatography (GC 6890N with flame ionisation detector). The physicochemical parameters were correctly quantified using ASTM standards. The density and viscosity of the test fuels were determined using ASTM D1298 and ASTM D445 methods, respectively. The measurements were conducted at 15 °C using a hydrometer and at 40 °C using a capillary viscometer. The test fuel's calorific value (CV) and flash point (FP) were assessed using an ASTM D240 bomb calorimeter and closed-cup flashpoint equipment, respectively. The cetane number (CN) of the test fuel was calculated using the ASTM D613 method.

3. Results and discussion

3.1 Analysis of variance

The quadratic model was suggested for output response analysis in the fit summary report based on the observation that the sequential p-value was above the threshold of 0.01. The data pertaining to the analysis of variance can be found in Table 2. The estimation of the influence of important process factors was conducted by calculating the mean square value of each parameter. The data illustrates that the variables MOR, RTe, and RTm have the greatest impact on the yield of BMB, with respective percentages of 30.9%, 31.9%, and 37.2%. The suggested model demonstrates statistical significance, as evidenced by a small margin of error of 0.01%. This is further supported by a model F-value of 293.83 and a P-value of 0.0001. The fit statistics report indicates that the corrected coefficient of determination (R²) of 0.99 aligns with the anticipated value of 0.96. Figures 2a and 2b display diagnostic graphs illustrating the relationship between residuals and run, as well as predicted and actual values, respectively. The residuals exhibit a random distribution within specified limits, and there is a tight correspondence between the projected and observed values, indicating the robustness of the proposed model. The determining factor that is critical in establishing the equation for the yield rate of BMB is:

$$\text{BMB yield (\%)} = 5.25 + 8.806 \times A + 0.5421 \times B + 0.868875 \times C + -0.0068 \times AB + -0.0015 \times AC + 0.0002 \times BC + -0.5224 \times A^2 + -0.003104 \times B^2 + -0.00685 \times C^2$$

3.2 Impact of input variables on BMB Yield

The optimisation process took into account the inputs of MOR, RTe, and RTm. Figure 3 illustrates the influence on biodiesel production. The interaction plot between MOR and RTe is depicted in Figure 3(a). According to the model, there is a positive correlation between the methanol molar ratio and the BMB yield rate, up to a specific threshold. The biodiesel production rate drops when there is an increase in molar ratios, which might be attributed to inadequate mixing of the catalytic effect [25]. The yield of BPB exhibits a consistent increase as the process temperature is raised under initial conditions. The yield of saponification significantly decreases beyond a particular threshold when conducted at elevated process temperatures [26]. Figure 3(b) displays the interaction plot between MOR and RTm in relation to the yield of BMB. Similar to the previous two inputs, the introduction of RTm results in an instantaneous rise in BMB production. The process of hydrolysis has been seen to result in a reduction in the slope of the yield rate for extended periods of reaction time [27]. The yield of the BMB is associated with the variables RTe and RTm, as depicted in Figure 3(c).

3.3. Optimum value and its confirmation

The outputs respond to that yielded the highest BMB following optimisation was referred to as the “maximum-the-best” response. The conditions that provide the highest BMB are characterised by a molar ratio of 7.4, a reaction temperature of 80 °C, and a reaction time of 64 minutes. In ideal circumstances, the
yield rate of BPB is recorded at 89.3%. Figure 4 displays the optimal input parameters and yield rate. An experimental confirmation was performed under ideal conditions (MOR of 7.4, RTe of 80 °C, and RTm of 64 min) to validate the data. The results of the confirmation experiment revealed a BMB yield of 89.3%. The marginal disparity seen between the experimental data and the anticipated values bestows upon the suggested design a confidence level surpassing 95%. The optimisation of process parameters for the production of Bauhinia parviflora biodiesel indicates that the highest yield rate of Bauhinia parviflora methyl ester (91.4%) is attained when the MOR is 9.2:1, the RTe is maintained at 76 °C, and the RTm is set at 67 min [1]. The optimisation of Carthamus lanatus (L.) Boiss. seeds biodiesel using the response surface technique reveals that a CC of 2.5%, a MOR of 11:1, a RTe of 115 °C, and a RTm of 140 min result in a methyl ester yield of around 95% [28]. The aforementioned results provided support for the current optimisation outcome in the field of optimising process parameters for Bauhinia monandra biodiesel.

3.4. FTIR characteristics of BMB and properties of test fuel

The results of a FTIR study performed on the carbon-based components of the synthesised BMB are depicted in Figure 5. The identification of a C=O bond in the test fuel can be achieved by observing absorption within the wavenumber range of 1700–1800 cm\(^{-1}\). Moreover, the elongation of the O–H bond in BMB may be ascertained through the examination of absorbance within the range of 2900–3000 cm\(^{-1}\). The presence of a C-H bond can be inferred from the absorbance of BPB in a wavenumber range of 1400–1500 cm\(^{-1}\), while the presence of a C-O bond can be suggested by the absorbance of the test fuel in a wavenumber range of 1100–1300 cm\(^{-1}\). The identification of a C=C bond can be inferred from the absorption detected within the wavenumber range of 700–750 cm\(^{-1}\) [23, 24]. The full assessment concluded that the proposed biodiesel demonstrates an adequate concentration of carbon-based components, such as ester, alkane, and unsaturated functional groups. This characteristic makes it a feasible alternative to traditional diesel fuel.

![Figure 3](image1.png)

**Figure 3.** Impact of input variables on BMB yield: (a) MOR vs RTe; (b) MOR vs RTm; and (c) RTe vs RTm

![Figure 4](image2.png)

**Figure 4.** Optimum value and its corresponding BMB yield
4. Conclusion

The present study examines strategies for enhancing the efficiency of response surface methodology in the context of BMB production from Bauhinia monandra seeds. The physicochemical qualities of biodiesel were assessed in accordance with the ASTM standard, while the fuel parameters were studied by FTIR. The results of the optimisation study indicated that the optimal parameters for BMB were a MOR of 7.4:1, a RTe of 80 °C, and a RTm of 64 minutes. Under ideal conditions, the recorded yield rate of BMB stands at 89.3%. The BMB is composed of elements that are obtained from hydrocarbons, which are formed by the conversion of fatty acids into methyl esters. The confirmation of this assertion is substantiated by the application of FTIR study. The physicochemical characteristics of BMB align with the prescribed criteria outlined in ASTM 6751 for biodiesel that is entirely composed of renewable sources.

References


M. Gurusamy, S. Vellaiyan, M., Kandasamy, Y. Devarajan, Optimization of process parameters to intensify the yield rate of biodiesel derived from waste and inedible Carthamus lanatus (L.) Boiss. seeds and examine the fuel properties with pre-heated water emulsion, Sustainable Chemistry and Pharmacy, 33, (2023) 101137. https://doi.org/10.1016/j.scp.2023.101137

Has this article screened for similarity? Yes

Conflict of Interest
The Author have no conflicts of interest on this article to declare.

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