Bioresource Technology 374 (2023) 128769

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

# Investigations on evaluation of marine macroalgae *Dictyota bartayresiana* oil for industrial scale production of biodiesel through technoeconomic analysis

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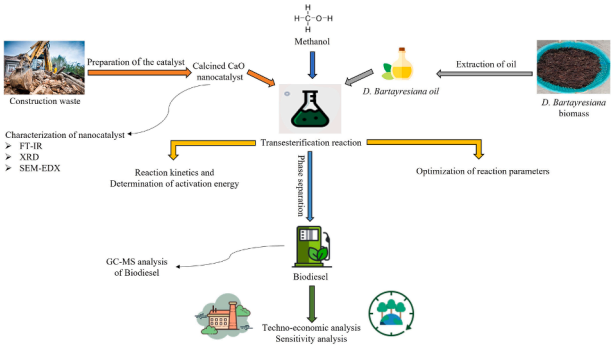
## HIGHLIGHTS

- Biodiesel production from marine macroalgae *Dictyota bartayresiana* oil was studied.
- CaO nanocatalyst was synthesized using waste collected from building demolition site.
- The reaction parameters for efficient production of biodiesel was optimized.
- Investigation on techno-economic aspects was studied for biodiesel production.
- Sensitivity analysis was used to study the uncertainty of biodiesel production process.

## GRAPHICAL ABSTRACT




## ARTICLE INFO

*Keywords:*
*Dictyota bartayresiana*
Marine macroalgae
Biodiesel
Response surface methodology
Process optimization
Techno-economic analysis

## ABSTRACT

The investigation on utilizing macroalgae for industrial scale biodiesel production is an imperative action needed for commercialization. In the present research work, the biooil from marine macroalgae *Dictyota bartayresiana* was used for biodiesel production using calcium oxide nanocatalyst synthesized using waste collected from building demolition site. The optimization results obtained were the calcination temperature 573 °C, concentration of catalyst 5.62%, methanol to oil molar ratio 14.36:1, temperature 55.7 °C and time 67.57 min for the transesterification with the biodiesel yield of 89.6%. The techno-economic aspects of biodiesel production were investigated for 20 MT/batch. The return on investment and internal rate of return from the biodiesel production plant was found to be 25.39% and 31.13% respectively. The plant payback period was about 3.94 years with a positive NPV value of about 14,053,000 $/yr. Thus, *Dictyota bartayresiana* biomass can be efficiently used for the sustainable production of biodiesel.

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https://doi.org/10.1016/j.biortech.2023.128769
Received 14 January 2023; Received in revised form 17 February 2023; Accepted 18 February 2023
Available online 24 February 2023

## 1. Introduction

The concern on the environment and the limited occurrence of the fossil-based fuels has created an essential need for the production of biofuels in order to reduce the emissions of carbon-di-oxide. Due to the increase in energy consumption, the need for the novel technologies for the sustainable production of biofuels from renewable resources gained an attention in recent decades. It is also predicted that the demand for the fossil fuels is to be increased up to 40% by the year 2040 (Vimali et al., 2022). The properties like non-toxicity, sustainability, low emissions, eco-friendly and biodegradability makes biomass the preferrable feedstock for the production of biofuels. The use of non-edible and waste cooking oils for producing biodiesel has been severally studied (Zul et al., 2021). But, their involvement in the food cycle paved the way for looking into alternative renewable source. Algal biomass considered to be the suitable feedstock and was investigated in recent years due to their lipid, carbohydrate and protein content for biofuels and various other by-products production (Firemichael et al., 2020). Marine macroalgae normally possess oil content between the range of 2–18%. In previous study, *D. bartayresiana* biomass showed oil yield of 8.86% and this was higher when compared with other macroalgal species like *Cladophora prolifera* (3.34%) and *Codium bursa* (1.46%) (Mona et al., 2020), *Nitella* sp. (7.86%) (Kanda et al., 2021), *Chara* sp. (1.06%) (Trifa et al., 2013), *Rhizoclonium* sp. (6.44%) (Saengsawang et al., 2020) and *Chara vulgaris* (3.60%) Siddiqua et al., 2015). However due to their low lipid content present in macroalgal species, the commercialization of macroalgal biodiesel is lagging at a stage where proper biorefinery approaches are to be involved. The residual biomass having higher content of carbohydrate can be efficiently used for bioethanol production and other value added biochemicals (Jeyakumar et al., 2022). The insights on the industrial production of biofuels by the utilization of marine macroalgae can be studied well for effectively involving them in biofuels production. Also because of their low production cost when compared to other feedstocks, these marine macrolage can be economically used for biodiesel production (Iyyappan et al., 2022). Several macroalgal species have been reported for biodiesel production like *Ulva lactuca*, *Caulerpa racemose*, *Codium tomentosum*, *Padina tetrastromatica* and *Sargassum swartzii* through transesterification reaction (Ashokkumar et al., 2017; Balu et al., 2020; Kalavathy & Baskar, 2019). The use of catalyst synthesized from waste materials like egg shells, carbon materials, plaster of paris (Abdala et al., 2020; Cherian et al., 2021; Baskar & Aiswarya, 2016) have been previously used and helped for economical and sustainable way of biodiesel production.

The optimization of process parameters like methanol to oil molar ratio, concentration of the catalyst, reaction temperature (℃) and time (min) are the determining factors of the transesterification reaction (Saengsawang et al., 2020). The study of response surface methodology (RSM) has been variously studied for the optimizing the process. The Minitab software was used here for conducting and planning the experiments for optimizing the process parameters. The highest regression ($R^2$) value assesses and provide the fitness of the model (Naveenkumar & Baskar, 2019). The research gap in macroalgal biodiesel production is the assessment of techno-economic analysis and lack of biorefinery approaches for production of value-added products. To provide proper technology transfer from lab to pilot or industrial scale, the investigation on the techno-economic aspects of the biodiesel production process using the experimental data plays a major role. The factors like the total investment caused to the project, revenue from the project, project payback time, internal rate of return, return on investment, net product value will be investigated and also simulating the economical and sustainable process flow provides the better information on the biodiesel production (Naveenkumar & Baskar, 2022). In biodiesel production from macroalgae, nearly 92.22% of the operation cost comes from the raw materials. The minimum selling price of marine macroalgal biomass was also ranges between 210 and 565 $/MT. The techno-economic analysis on the biodiesel production from *Codium tomentosum* biomass have been studied with the results of positive net present value of 1381 M$/year, return on investment of 11.64% and project payback time of 8.59 years (Gengiah et al., 2022). It is estimated that 111.07 ton/year of $CO_2$ was emitted for the algal biodiesel production process (Mondal et al., 2022). But the growth of macroalgae can consume $CO_2$ and this may reduce the atmospheric $CO_2$ to an extent. Due to this advantage, biomass has gained attention for biodiesel production than fossil derived biofuels. Among that macroalgae can be widely utilized for fixing $CO_2$ and also contributing towards reduction on the dependency of fossil fuels. The total greenhouse gas emissions were estimated to be 17.291 ton of $CO_2$ eq/ha (Tirkey et al., 2022). So, in order to conclude the net $CO_2$ will be lower after the production of macroalgal biodiesel. The environmental analysis of the macroalgal biodiesel production provides the positive results. The process of utilizing macroalgae for $CO_2$ fixation gained attention among the researchers in recent decades (Wu et al., 2023).

The present research work mainly focussed on the sustainable production of the biodiesel from marine macroalgae *D. bartayresiana* oil. The utilization of the *D. bartayresiana* oil for biodiesel production was not reported earlier in previous literature. It was reported for biomedical applications in most of the literature. The synthesis of CaO nanocatalyst from construction waste collected from building demolition site was examined. The process parameters for the transesterification reaction were optimized by OVAT and RSM methods for the efficient production of biodiesel. The synthesized CaO nanocatalyst and produced biodiesel were subjected through characterization. The investigation on the techno-economic aspects of overall production process flow was done by the use of Superpro simulation software. The sensitivity analysis was investigated to study the uncertainty of the biodiesel production process from *D. bartayresiana* oil. The investigation on techno-economic studies of biodiesel production process will promote the industrial scale production at technology readiness level. It also paves the way for reducing $CO_2$ emissions to the environment. The $CO_2$ sequestration by the growth of macroalgae also contributes to a circular bioeconomy.

## 2. Materials and methods

### 2.1. Materials

The marine macroalgae *D. bartayresiana* was collected from Mandapam area of Ramanathapuram district, Tamilnadu, India. The collected *D. bartayresiana* biomass was processed for algal oil extraction and the extracted algal oil was used for the transesterification reaction. The construction waste was locally collected from building demolition sites. The solvents such as hexane and methanol used were procured from SRL Private Limited, Mumbai, India. All of these chemicals were of analytical grade with 99% purity and thus used without further purification.

### 2.2. Preparation of catalyst from construction waste

The collected construction waste from demolition sites was grounded into fine particles using mortar and pestle. Later, the waste was washed with the use of distilled water for the removal of sand particles and impurities. Then the sample was allowed to dry at 80 °C in hot air oven for the removal of moisture content. The dried sample was then kept for calcination in muffle furnace at 600 °C for 3 hrs for the activation of the catalyst (Das et al., 2020).

### 2.3. Characterization of the catalyst

The prepared catalyst was subjected to characterization using FT-IR, SEM-EDAX and XRD to study their functional groups, morphological and crystalline structure of the catalyst respectively.

#### 2.3.1. Fourier transform- infrared spectroscopy

Fourier transform infrared spectroscopy with attenuated total reflectance (ATR FT-IR) was used to characterize the synthesized catalyst for studying the structure and investigating the functional groups present in the surface of synthesized catalyst. The FT-IR spectra measurements were recorded between the wave number region of 4000–500 $cm^{-1}$ acquired at room temperature and at a resolution of 1 $cm^{-1}$ using Perkin Elmer system spectrophotometer.

#### 2.3.2. Scanning electron microscope with EDX

The surface morphology and elemental analysis of the catalyst sample was characterized and studied using the Field emission - Scanning Electron Microscopy (FE-SEM) and Energy dispersive X-ray spectrophotometer (EDX) analysis. The analysis was conducted by FEI-Quanta FEG 200F system and the voltage and current were kept at 30 kV and 100nA during the operation and also at a resolution of 1.2 nm. The SEM images were acquired at different magnifications and studied further.

#### 2.3.3. X-ray diffraction

The study of crystalline structure and the identification of phase type and size of the crystalline was studied by X-ray diffraction (XRD). The analysis was performed on Bruker (D8 Advance) instrument using the radiation of Cu Kα at 40 kV and 30 mA with 2θ range scanned between 5° and 100° at the surrounding temperature and also with a step size of 0.02°.

### 2.4. Biodiesel production through transesterification process

The prepared CaO catalyst and the methanol solvent was utilized for the transesterification of the *D. bartayresiana* oil. The activation of the CaO nanocatalyst was achieved when mixing it with methanol solvent, which forms calcium methoxide that serves as the catalyst in the transesterification reaction (Kumar et al., 2008). The reaction was carried out in conical flask which was then heated and stirred using magnetic stirrer couple with hot plate. After the completion of reaction, the excess methanol was recovered by heating at 80 °C with the use of reflux condenser. Then the mixture containing glycerol and methyl ester was then transferred to the separating funnel and kept undisturbed to obtain the separation of layers. The top layer containing biodiesel and bottom layer with glycerol was separated and stored. The biodiesel produced was then washed with hot water for the removal of impurities. Atlast, the obtained pure biodiesel was collected and biodiesel yield (%) was estimated by using the formula shown in Eq. (1),

$$\text{Biodiesel Yield}\,(\%) = \frac{MW_{oil} \times W_{biodiesel}}{3 \text{ x } MW_{biodiesel} \text{ x } W_{oil}} \times 100 \quad (1)$$

Where $W_{biodiesel}$ and $W_{oil}$ is the weight of biodiesel obtained and oil used; $MW_{biodiesel}$ and $MW_{oil}$ is the average molecular weight of the obtained biodiesel and used oil respectively (Borghini et al., 2014).

### 2.5. Optimization of transesterification process using classical and statistical methods

#### 2.5.1. Optimization of process parameters through OVAT method

The reaction parameters necessary for the process of transesterification of *D. bartayresiana* oil such as the calcination temperature of the catalyst, concentration of catalyst % used in the reaction, methanol to oil molar ratio, temperature of the reaction and required time were optimized using one variable at a time (OVAT) method. The study of each parameter was done by keeping the remaining parameters constant for that reaction. Each optimized values were carried over to the further optimization of the reaction parameters followed (Veeranan et al., 2018).

#### 2.5.2. Optimization of the process parameters through RSM method

To improve the biodiesel yield, optimization of the parameters for transesterification reaction was studied using response surface methodology method. The model of central composite design was performed by using the Minitab software (Naveenkumar & Baskar, 2021). The five-factor levels were fixed based on the results obtained from OVAT method. The lower, middle, higher values of the factors (Calcination temperature – 500,600,700; catalyst concentration (%) – 4,5,6 %; Methanol to oil ratio – 9:1,12:1,15:1; Temperature – 50,55,60 °C; Time – 45,60,75 min) were used to plan the experiments for optimization of the transesterification reaction. The analysis on regression value using the obtained quadratic polynomial model is shown as Eq. (2),

$$\begin{aligned} Y = {} & \alpha_0 + \alpha_1 \cdot A + \alpha_2 \cdot B + \alpha_3 \cdot C + \alpha_4 \cdot D + \alpha_5 \cdot E + \alpha_{11} \cdot A^2 + \alpha_{22} \cdot B^2 + \alpha_{33} \cdot C^2 + \alpha_{44} \cdot D^2 + \alpha_{55} \cdot E^2 \\ & + \alpha_{12} \cdot A^* B + \alpha_{13} \cdot A^* C + \alpha_{14} \cdot A^* D + \alpha_{15} \cdot A^* E + \alpha_{23} \cdot B^* C + \alpha_{24} \cdot B^* D + \alpha_{25} \cdot B^* E + \alpha_{34} \cdot C^* D + \alpha_{35} \cdot C^* E + \alpha_{45} \cdot D^* E \end{aligned} \quad (2)$$

Where Y is the biodiesel yield in %, $\alpha_0$ the intercept form; $\alpha_1$, $\alpha_2$,$\alpha_3$, $\alpha_4$ and $\alpha_5$ were the linear co-efficients; $\alpha_{11}$, $\alpha_{22}$, $\alpha_{33}$, $\alpha_{44}$ and $\alpha_{55}$ were the quadratic co-efficients $\alpha_{12}$, $\alpha_{13}$, $\alpha_{14}$, $\alpha_{15}$, $\alpha_{23}$, $\alpha_{24}$, $\alpha_{25}$, $\alpha_{34}$, $\alpha_{35}$, and $\alpha_{45}$ were the interactive co-efficients; while A,B,C,D and E were the coded independent variables (A-Calcination temperature, B-Concentration of the catalyst, C-Methanol to oil molar ratio, D-Temperature and E-Time). The coefficients, nature of the model and $R^2$ value was assessed by F-test and T-test (Miriam et al., 2021).

#### 2.5.3. Reaction kinetics and determination of activation energy on biodiesel production process from D. bartayresiana oil

The investigation on the rate of biodiesel conversion was studied at different intervals of temperature and time. The rate of the reaction on biodiesel conversion is given by the following Eq. (3),

$$\frac{dY}{dt} = kY^n \quad (3)$$

Where Y denoted as biodiesel yield in %, n as order of the reaction, k as rate constant and t as the reaction time in min.

The determination of the activation energy was studied by relating the rate constant with the reaction temperature using the following Arrhenius Eq. (4),

$$k = Ae^{-Ea/RT} \quad (4)$$

Where denoted k and A as reaction rate and Arrhenius constant respectively, $E_a$ as activation energy in kJ/mol, R as universal gas constant and T as the reaction temperature in K (Baskar et al., 2018).

### 2.6. Gas chromatography-Mass spectrometry analysis of biodiesel

The obtained biodiesel was characterized for fatty acid methyl esters (FAME) present using Gas chromatography-Mass spectrometry (GC–MS). GC–MS was studied using Agilent 8890 GC system with Single quadrupole mass spectrophotometer analyser. The sample was injected and the column temperature initially maintained at 75 °C and then later raised to 300 °C for 5 min.

### 2.7. Techno-economic analysis on biodiesel production

#### 2.7.1. Process design

The analysis on the techno-economical aspects of the biodiesel production process from *D. bartayresiana* oil by the use of the synthesized nanocatalyst was studied using Superpro software. The results obtained from the laboratory scale experiments were used for simulating the process flow. The simulated model plant was created to produce biodiesel from *D. bartayresiana* oil (20MT/batch) with the construction period of 1 year, startup period of 0.4 year, 20 years of lifetime with annual operating time of 7920 hrs. The transesterification reaction process was conducted in CSTR with input of raw materials such as *D. bartayresiana* oil, methanol and CaO nanocatalyst. In addition to that, equipment like centrifugation, flash evaporator and the decanter was used for simulating the process. After the reaction in CSTR, the reaction mixture was then moved to centrifugation equipment to successfully recover the catalyst followed by flash distillation for the recovery of excess methanol. Then the reaction mixture was moved to decanter for separating the biodiesel and glycerol and finally washing the biodiesel by water to obtain the pure biodiesel (Rajendran et al., 2021).

#### 2.7.2. Economic analysis

The economic analysis of any process flow depends on the factors like the usage of raw materials and equipment, setting up, installation and maintenance of the equipment, costs due to consumption of power, labor and also on other utilities. The total cost of the plant involves both the capital investment and operating cost and this comprises the cost that are caused due to electricity consumption, labor and installation charges, equipment cost, research and development, laboratory utilities like QA/QC in addition to expenses for insurances and income taxes. The cost of the *D. bartayresiana* oil was estimated to be 0.7 $/Kg including the transportation and processing cost of oil extraction from *D. bartayresiana* biomass. Since, the catalyst was synthesized from construction waste material, the waste can be collected from demolition sites at minimal cost. The cost of transporting the material to the plant and processing them for using it as a catalyst for transesterification reaction makes the cost of the catalyst to be of 0.8 $/Kg (Naveenkumar & Baskar, 2020). The cost of the methanol solvent was assumed to be 0.24 $/Kg. The cost of the equipment, labor and other utilities were based on the rate of Indian market, data from biodiesel production industries and consultancies. The assumed biodiesel price is 0.84 $/Kg. The rate of currency exchange from US dollar to INR is 81.45 and income tax for Indian companies is 25% in the reference year 2022. The commercial viability of the process was analysed based on the total cost of investment and operating cost to the project, annual revenue from the plant, return on investment (ROI), internal rate of return (IRR), payback period and the net present value (NPV) (Naveenkumar & Baskar 2020).

## 3. Results and discussion

### 3.1. Characterization of the catalyst

#### 3.1.1. Fourier transform- infrared spectroscopy

The major phase of the spectral lines was found in the wave number region of 500 $cm^{-1}$ to 1500 $cm^{-1}$. Two large strong peaks were observed at 1420 $cm^{-1}$ and 981 $cm^{-1}$ and the peaks were assigned to O-H and C = C bending respectively. The carboxyl ions (C-H) with bending vibration were found to existing at the wave number region of 876 $cm^{-1}$ and 775 $cm^{-1}$. The bands at 644 $cm^{-1}$ were attributed to C-Br stretching vibrations and also acquired peaks at 577 $cm^{-1}$ and 533 $cm^{-1}$ denotes the presence of C-Cl and C-I stretching vibrations respectively. The calcined cement waste material showed weak signals attributed to the presence of $SO_4$, $CO_3$ and $OH^-$ and this may be due to calcination at higher temperatures. The peaks contributed highly to the presence of CaO with the observed FT-IR spectra. The obtained FT-IR peaks were compared with the results of calcined cementitious material. The comparison confirms the presence of relatable peaks observed with CaO nanocatalyst (Kumar et al., 2008).

#### 3.1.2. Scanning electron microscope with EDX

The morphology structure of the surface catalyst was analyzed by image obtained from scanning electron microscope (SEM) using the higher magnification of 80000x with 500 nm shows cluster of crystal-like particles and conveys that small particle were forming to an agglomerated dense structure. The size of the particles was measured and found to be in the range of 18–35 nm and the average size of the particles were calculated as 26 nm. The elemental analysis of the synthesized catalyst from construction waste by Energy dispersive X-ray spectrophotometer revealed the presence of calcium (41.25%) and oxygen (43.41%) with the major weight%. The discussion on the significant role of CaO in the transesterification process for converting the fatty acids to fatty acid methyl esters (FAME) was previously reported and thus shows the suitability of the CaO as catalyst in biodiesel production. Therefore, the occurrence of calcium and oxygen in CaO nanocatalyst shows better activity in the transesterification of oil to produce methyl esters. The role of CaO catalyst in the biodiesel conversion of soybean oil (Wang et al., 2012) and frying oil (Kataria et al., 2017) was studied and this highly supports the use of CaO nanocatalyst in the transesterification of *D. bartayresiana* oil.

#### 3.1.3. X-ray diffraction

The nanocatalyst synthesized from construction waste was analyzed using XRD instrument. The diffractogram of XRD has shown the intensity of peaks at 2θ angale at 20.8°, 26.6°, 27.4°, 28° of the synthesized catalyst. The XRD pattern obtained corresponds to the CaO (JCPDS Card No. 00–037-1497) and it thus proves the calcined catalyst consists of the pure form of CaO powder and can be effectively used for the transesterification reaction. The alteration of the Ca $(OH)_2$, $CaSO_4$ and $CO_3$ to CaO was observed due to subjecting the catalyst to higher temperatures and this supports the suitability of the catalyst in the production of biodiesel from *D. bartayresiana* oil (Kumar at al., 2018).

### 3.2. Optimization of biodiesel production process from D. Bartayresiana oil

#### 3.2.1. Optimization of process parameters using OVAT method

*3.2.1.1. Effect of the calcination temperature.* The effect of calcination temperature on transesterification of the *D. bartayresiana* oil was studied between 300 and 800 °C. The other reaction parameters like reaction temperature – 60 °C, methanol to oil molar ratio – 15:1, time – 90 min and catalyst concentration – 4% were kept as constant for the reaction. The results shows that the biodiesel yield increased on higher temperature of the calcined catalyst, the highest biodiesel yield of 73.5% was achieved at 600 °C and thus were shown in Fig. 1(a). After the calcination temperature of 600 °C, the biodiesel yield was found to be decreased and it may be due to the reduced activity of the synthesized nanocatalyst while calcinating at higher temperatures causing some changes in the catalytic activity of the catalyst. Hence, the optimized temperature of calcinating the catalyst was found to be at 600 °C which shows the higher yield of biodiesel from *D. bartayresiana* oil.

*3.2.1.2. Effect of the catalyst concentration.* The study on the effect of catalyst concentration on transesterification of the *D. bartayresiana* oil was optimized by varying the concentration of the catalyst from 2 to 7 % (w/v) by fixing other reaction parameters (Calcination temperature – 600 °C, Reaction temperature – 60 °C, Methanol to oil molar ratio – 15:1, Time – 90 min) as constant. From the results shown in Fig. 1(b), it was known that the maximum yield of the biodiesel 76% was achieved at the use of 5 % catalyst concentration in the reaction. Later, on increasing the concentration of the catalyst after 5 % showed no changes in the

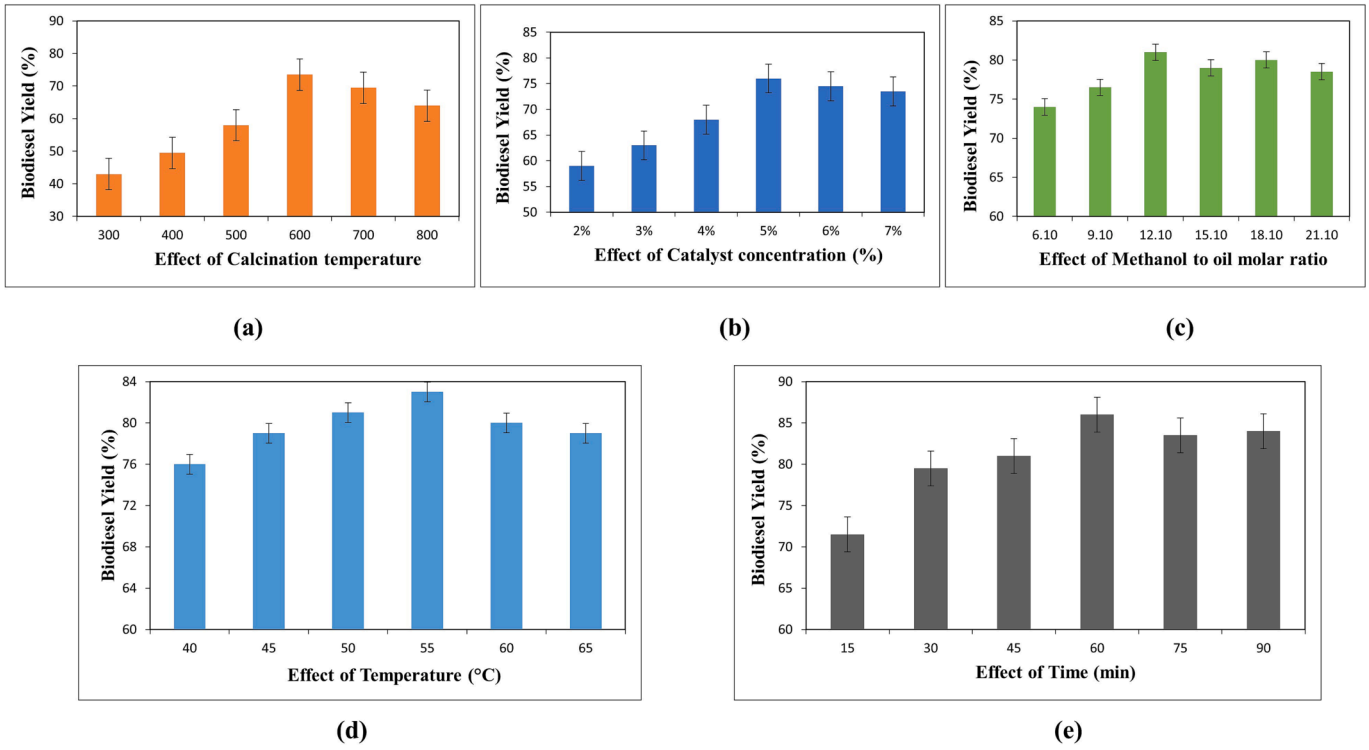


**Fig. 1.** Optimization of reaction parameters (a) Calcination temperature; (b) Calatalyst concentration (%); (c) methanol to oil molar ratio; (d) reaction temperature; (e) reaction time on biodiesel production from D. *bartayresiana* oil.

biodiesel yield and this might be due to the saturation in the availability of reaction sites for equilibration. This shows that after certain centration of catalyst, it has no major effect on biodiesel conversion. Thus, it is concluded that the catalyst concentration of 5 % is optimum for efficient for conversion *D. bartayresiana* oil into biodiesel.

*3.2.1.3. Effect of the methanol to oil ratio.* As methanol needed for the transesterification reaction, the effect of the methanol to oil ratio has a larger impact. The study on effect of methanol to oil ratio was varied from 6:1 to 21:1 and studied by fixing other parameters (Calcination temperature – 600 °C, Reaction temperature – 60 °C, Catalyst concentration – 5 %, Time – 90 min) as constant. The highest yield of biodiesel was acquired at methanol to oil molar ratio of 12:1 with yield of 81 %. Further, on increasing the ratio of methanol does not show any major changes in the biodiesel yield shown in Fig. 1(c) and this may be due to the accumulation of the methanol attained on the surface area of the catalyst during the transesterification reaction. The high amount of methanol does not contribute to higher conversion rate of biodiesel, this might be due to the saturation limit of catalyst. Thus, The methanol to oil ratio of 12:1 is found as optimum for transesterification of *D. bartayresiana* oil.

*3.2.1.4. Effect of the temperature.* The study on effect of temperature was studied by changing the reaction temperatures from 40 to 65 °C while keeping the other reaction parameters as constant (Calcination temperature – 600 °C, Catalyst concentration – 5 %, Methanol to oil molar ratio – 12:1, Time – 90 min). The yield of biodiesel was increased from 76 to 83 % on increasing the temperature of the reaction from 40 to 55 °C and later further increase in the reaction temperature found to be decreased in the biodiesel yield Fig. 1(d) and this may be due to the evaporation of methanol at higher temperature. Thus, the temperature of the reaction at 55 °C was found to be the optimal reaction temperature for the transesterification of *D. bartayresiana* oil.

*3.2.1.5. Effect of the time.* The time required for the transesterification reaction was optimized by varying the time from 15 to 90 min and fixing other parameters (Calcination temperature – 600 °C, Reaction temperature – 55 °C, Catalyst concentration – 5 %, Methanol to oil molar ratio – 12:1) as constant. The highest yield of biodiesel 86 % was obtained at 60 min and further there was no increase in the yield Fig. 1(e) and thus it shows that the optimum time for the reaction to be 60 min. The biodiesel yield showed no changes after 60 min and this may be due to the attaining of maximum conversion of fatty acids to fatty acid methyl esters. The increase in reaction time majorly affects the overall economic results. Hence, it is necessary to optimize the reaction time required for transesterification of *D. bartayresiana* oil.

### 3.2.2. Optimization of biodiesel production using RSM method

The 32 experimental trials were conducted and the biodiesel yield was compared with the predicted yields using Minitab software shown in Table 1. Based on the results obtained from statistical analysis, the quadratic polynomial equation was derived and the effect of the reaction parameters on biodiesel conversion was studied. The quadratic polynomial equation of biodiesel conversion was shown in Eq. (5),

$$Y = -323 + 0.2052 \cdot A + 0.58 \cdot B + 2.63 \cdot C + 10.30 \cdot D + 1.250 \cdot E - 0.000165 \cdot A^*A - 0.724 \cdot B^*B - 0.1429 C^*C - 0.0940 \cdot D^*D - 0.00938 \cdot E^*E - 0.00525 \cdot A^*B + 0.00233 \cdot A^*C - 0.000152 \cdot A^*D - 0.000167 \cdot A^*E + 0.188 \cdot B^*C + 0.1125 \cdot B^*D + 0.0092 \cdot B^*E - 0.0242 \cdot C^*D + 0.0064 \cdot C^*E - 0.00050 \cdot D^*E \quad (5)$$

The regression coefficients and the ANOVA analysis was tabulated in the Tables 2 and 3. From the results, it was found that the parameters such as calcination temperature, methanol to oil molar ratio and reaction time has higher significant effect on the rate of conversion of biodiesel. The ANOVA analysis results shows the linear and square terms with higher significance on the biodiesel conversion from *D. bartayresiana* oil. The determination of the regression coefficient ($R^2$) with the value of 92.67% shows the higher accuracy of the model. The interactive effects of the parameters on the biodiesel conversion were shown in Fig. 2, using the contour plots. By comparing the experimental

**Table 1**
Central Composite Design of RSM for optimizing the biodiesel conversion from *D. bartayresiana* oil.

| Order of the experiment | Calcination temperature (A), °C | Catalyst Concentration (B), % (w/w) | Methanol to oil molar ratio (C), v:v | Temperature (D) , °C | Time (E) | Biodiesel Yield, % | Predicted biodiesel Yield, % |
|---|---|---|---|---|---|---|---|
| 1. | 500 | 4 | 9 | 50 | 75 | 78.4 | 79.99 |
| 2. | 700 | 4 | 9 | 50 | 45 | 75.7 | 75.17 |
| 3. | 500 | 6 | 9 | 50 | 45 | 76 | 75.58 |
| 4. | 700 | 6 | 9 | 50 | 75 | 74.5 | 74.78 |
| 5. | 500 | 4 | 15 | 50 | 45 | 77.8 | 77.86 |
| 6. | 700 | 4 | 15 | 50 | 75 | 81.8 | 82.57 |
| 7. | 500 | 6 | 15 | 50 | 75 | 83.1 | 83.98 |
| 8. | 700 | 6 | 15 | 50 | 45 | 79.4 | 78.15 |
| 9. | 500 | 4 | 9 | 60 | 45 | 77.9 | 77.74 |
| 10. | 700 | 4 | 9 | 60 | 75 | 77.5 | 78.05 |
| 11. | 500 | 6 | 9 | 60 | 75 | 81.9 | 82.56 |
| 12. | 700 | 6 | 9 | 60 | 45 | 76.4 | 74.93 |
| 13. | 500 | 4 | 15 | 60 | 75 | 80.6 | 81.74 |
| 14. | 700 | 4 | 15 | 60 | 45 | 79.4 | 78.42 |
| 15. | 500 | 6 | 15 | 60 | 45 | 81.7 | 80.83 |
| 16. | 700 | 6 | 15 | 60 | 75 | 83.7 | 83.53 |
| 17. | 400 | 5 | 12 | 55 | 60 | 83.9 | 82.47 |
| 18. | 800 | 5 | 12 | 55 | 60 | 77.4 | 78.81 |
| 19. | 600 | 3 | 12 | 55 | 60 | 85.2 | 83.99 |
| 20. | 600 | 7 | 12 | 55 | 60 | 83.5 | 84.69 |
| 21. | 600 | 5 | 6 | 55 | 60 | 78.8 | 78.56 |
| 22. | 600 | 5 | 18 | 55 | 60 | 85.4 | 85.62 |
| 23. | 600 | 5 | 12 | 45 | 60 | 77.3 | 76.62 |
| 24. | 600 | 5 | 12 | 65 | 60 | 78.4 | 79.06 |
| 25. | 600 | 5 | 12 | 55 | 30 | 72.4 | 75.22 |
| 26. | 600 | 5 | 12 | 55 | 90 | 85.2 | 82.36 |
| 27. | 600 | 5 | 12 | 55 | 60 | 86 | 87.24 |
| 28. | 600 | 5 | 12 | 55 | 60 | 87.4 | 87.24 |
| 29. | 600 | 5 | 12 | 55 | 60 | 87 | 87.24 |
| 30. | 600 | 5 | 12 | 55 | 60 | 86.7 | 87.24 |
| 31. | 600 | 5 | 12 | 55 | 60 | 89.6 | 87.24 |
| 32. | 600 | 5 | 12 | 55 | 60 | 86.7 | 87.24 |

**Table 2**
Regression Co-efficient for optimizing the biodiesel conversion from *D. bartayresiana* oil.

| Terms | Coefficient estimate | Standard Error on the Coefficient | Value of *t*-test | Value of p-test | Significance of terms |
|---|---|---|---|---|---|
| $\alpha_0$ | –323 | 0.795 | 109.74 | <0.001 | Significant |
| $\alpha_1$ | 0.2052 | 0.407 | −2.25 | 0.046 | Significant |
| $\alpha_2$ | 1.58 | 0.407 | 0.43 | 0.675 | |
| $\alpha_3$ | 2.63 | 0.407 | 4.34 | <0.001 | Significant |
| $\alpha_4$ | 10.30 | 0.407 | 1.50 | 0.163 | |
| $\alpha_5$ | 1.250 | 0.407 | 4.38 | <0.001 | Significant |
| $\alpha_{11}$ | −0.000165 | 0.368 | −4.48 | <0.001 | Significant |
| $\alpha_{22}$ | −0.724 | 0.368 | −1.97 | 0.075 | |
| $\alpha_{33}$ | − 0.1429 | 0.368 | −3.50 | 0.005 | Significant |
| $\alpha_{44}$ | −0.0940 | 0.368 | −6.38 | <0.001 | Significant |
| $\alpha_{55}$ | −0.00938 | 0.368 | −5.74 | <0.001 | Significant |
| $\alpha_{12}$ | − 0.00525 | 0.498 | −1.05 | 0.315 | |
| $\alpha_{13}$ | +0.00233 | 0.498 | 1.40 | 0.188 | |
| $\alpha_{14}$ | −0.000150 | 0.498 | −0.15 | 0.883 | |
| $\alpha_{15}$ | −0.000167 | 0.498 | −0.50 | 0.626 | |
| $\alpha_{23}$ | 0.188 | 0.498 | 1.13 | 0.283 | |
| $\alpha_{24}$ | 0.1125 | 0.498 | 1.13 | 0.283 | |
| $\alpha_{25}$ | 0.0092 | 0.498 | 0.28 | 0.788 | |
| $\alpha_{34}$ | −0.0242 | 0.498 | −0.73 | 0.482 | |
| $\alpha_{35}$ | 0.0064 | 0.498 | 0.58 | 0.576 | |
| $\alpha_{45}$ | −0.00050 | 0.498 | −0.08 | 0.941 | |

and predicted yields of biodiesel, the RSM predicts the optimized conditions required for the transesterification reaction. The optimized conditions for the highest biodiesel conversion of 89.6% yield was found to be with calcination temperature 573 °C, 5.62% of catalyst concentration, methanol to oil molar ratio 14.36:1, reaction temperature 55.7 °C and the reaction time of 67.57 min.

#### *3.2.3. Reaction kinetics and determination of activation energy on biodiesel production from D. Bartayresiana oil*

The study on the reaction kinetics of the biodiesel production process at different reaction temperature and time was studied and it was concluded that the reaction follows the first-order kinetics model. The regression coefficient ($R^2$) value and rate constant of each temperature was obtained from the graph plotted between the values of ln[Y] versus ln[dY/dt]. The rate constant value was further used to determine the value of activation energy. The graph plotted between log k versus 1/T gives $R^2$ value of 0.97 which shows the exact fitness of the model. The activation energy was estimated and it was calculated to be of $E_a$ = 19.624 kJ/mol.

### *3.3. GC–MS analysis of biodiesel*

The occurrence of fatty acid methyl esters (FAME) in the obtained biodiesel was confirmed from the chromatogram obtained from GC–MS analysis. The peaks acquired in the chromatogram was compared with the NIST 17 GC–MS libraries. The methyl esters such as 9-Octadecenoic acid methyl ester and Hexadecenoic acid methyl ester along with methyl stearate with the retention time of 26.34, 30.67 and 30.91 min was found to be present in the biodiesel from *D. bartayresiana* oil. The occurrence of FAME of nearly 90% in the produced biodiesel supports that it can be efficiently used as a biofuel for various applications. In addition, this also contributes in the reduction of $CO_2$ emissions to the environment. Thus, the presence of FAME was analysed and studied using GC–MS analysis.

### *3.4. Techno-economic analysis on production process of biodiesel*

#### *3.4.1. Technical process*

The study of techno-economic analysis on biodiesel production from

**Table 3**
Analysis of Variance analysis for Optimization the biodiesel conversion from *D. bartayresiana* oil.

| Source | Degrees of Freedom | Adjusted Sum of Squares | Adjusted Mean Squares | Value of F-test | Value of P-test | Significance of terms |
|---|---|---|---|---|---|---|
| Model parts | 20 | 552.304 | 27.615 | 6.95 | <0.001 | Significant |
| Linear terms | 5 | 181.017 | 36.203 | 9.11 | <0.001 | Significant |
| Square terms | 5 | 344.073 | 68.815 | 17.32 | <0.001 | Significant |
| Interaction terms | 10 | 27.215 | 2.722 | 0.69 | 0.720 | |
| Error | 11 | 43.695 | 3.972 | | | |
| Lack-of-Fit | 6 | 35.922 | 5.987 | 3.85 | 0.080 | |
| Pure Error | 5 | 7.773 | 1.555 | | | |
| Total | 32 | 596 | | | | |

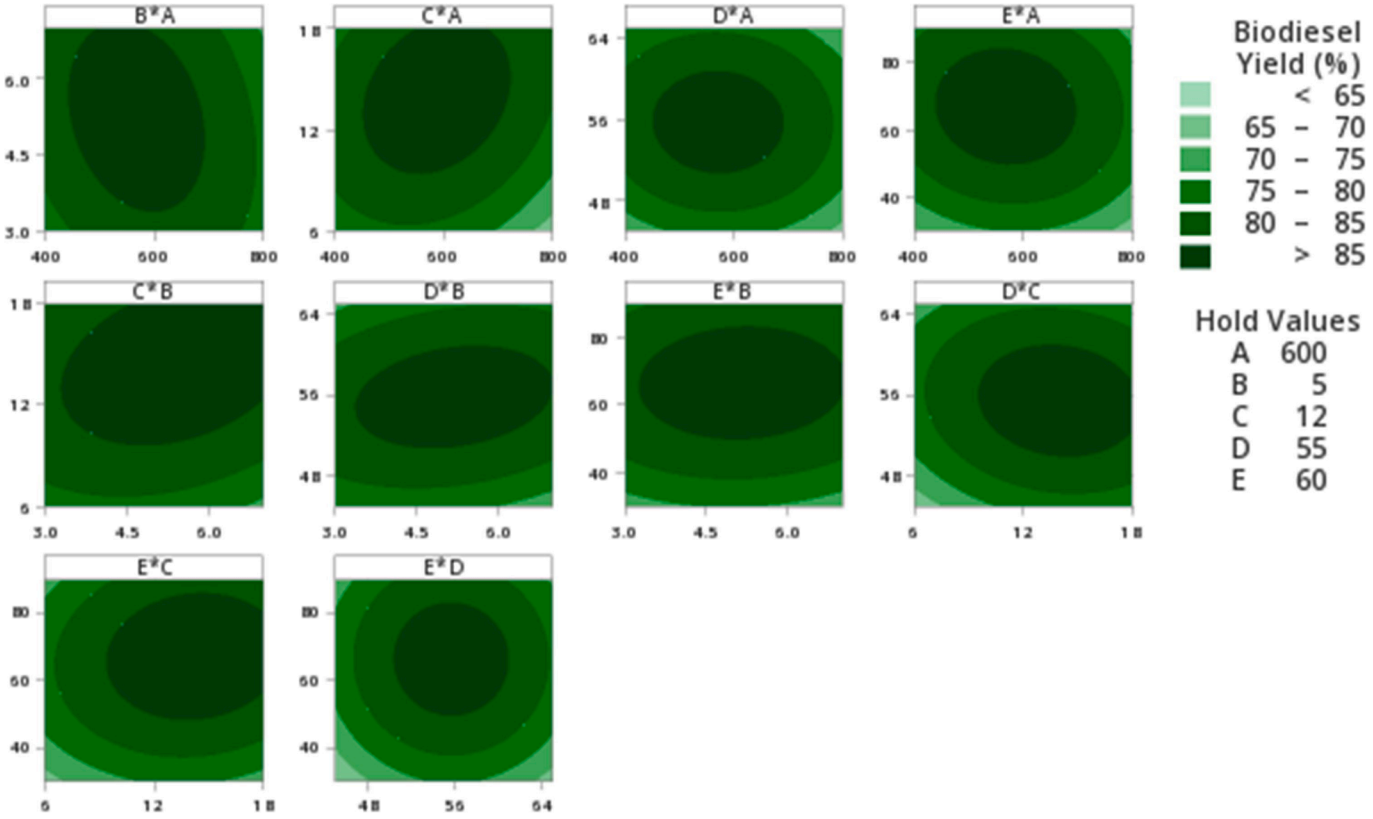


**Fig. 2.** Contour plots on biodiesel conversion from *D. bartayresiana* oil.

*D. bartayresiana* oil was done under the obtained optimum conditions from experimental data. In this process flow, 20 MT/batch of *D. bartayresiana* oil was processed for the production of biodiesel along with the addition of 782 kg/batch of CaO catalyst and 5645 kg/batch of methanol which was pumped into the reactor. After the reaction time, the transesterified reaction mixture was the moved into centrifuge for recovering the catalyst. Then the mixture is then again pumped into flash evaporate to recover the excess methanol. After the phase separation of the biodiesel and glycerol, the biodiesel is then washed with the water to gain pure biodiesel. Biodiesel was the main revenue from the simulated process with the annual production of 26,367,636 kg/yr. The overall process flow design of the production plant was simulated in Fig. 3.

### 3.4.2. Economic analysis

The detailed analysis on the total capital investment to the project,

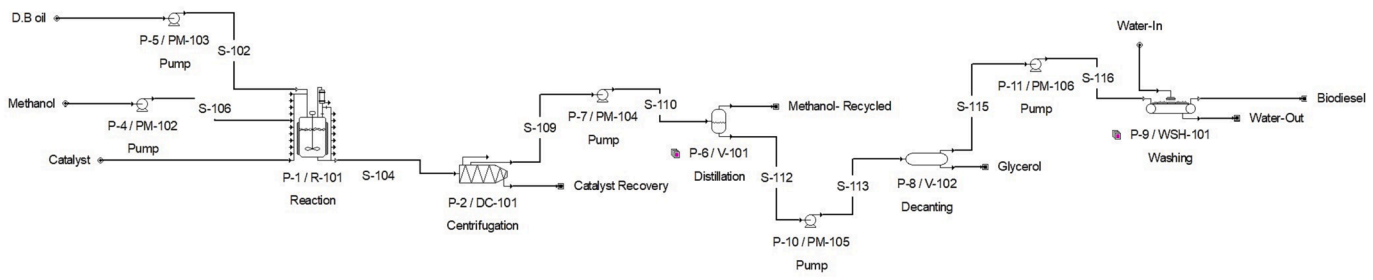


**Fig. 3.** Overall process flow of biodiesel production from *D. bartayresiana* oil.

**Table 4**
Overall data on economic analysis of biodiesel production from *D. bartayresiana* oil.

| Total Capital Investment | 11,954,000 $ |
|---|---|
| Capital Investment Charged to This Project | 11,954,000 $ |
| Operating Cost | 30,413,000 $/yr |
| Main Revenue | 22,149,000 $/yr |
| Other Revenues | 11,177,950 $/yr |
| Total Revenues | 33,327,000 $/yr |
| Batch Size | 14,981.61 kg MP |
| Cost Basis Annual Rate | 26,367,636 kg MP/yr |
| Unit Production Cost | 1.15 $/kg MP |
| Net Unit Production Cost | 1.15 $/kg MP |
| Unit Production Revenue | 1.26 $/kg MP |
| Gross Margin | 8.74 % |
| Return On Investment | 25.39 % |
| Payback Time | 3.94 years |
| IRR (After Taxes) | 31.13 % |
| NPV (at 10.0% Interest) | 14,053,000 $ |

operating and production cost, revenues, ROI, IRR, reimbursement period and NPV were assessed for studying the economic performance of the production process. The overall data on economic analysis was tabulated in Table 4. The cost of total capital investment for the process plant was estimated to be 11,954,000 $ with the annual operating cost of 30,413,000 $/yr where nearly 92.48% of it comes from the raw materials cost. The main revenue from the biodiesel was 23,731,000 $/yr. The return on investment for the plant period of 20 years is 35.32% with the payback period of 2.83 years. The IRR (after taxes of 25%) is 47.12%. The Net present value of the project (at 10% interest) is 23,709,000 $.

### 3.4.3. *Sensitivity analysis*

The sensitivity analysis on production of biodiesel from *D. bartayresiana* oil was studied to investigate the uncertainty in the output of the process flow. This involves on various factors such as the cost of input raw materials and output products. This also depends on the income tax and project life time. In this investigation, the factors like

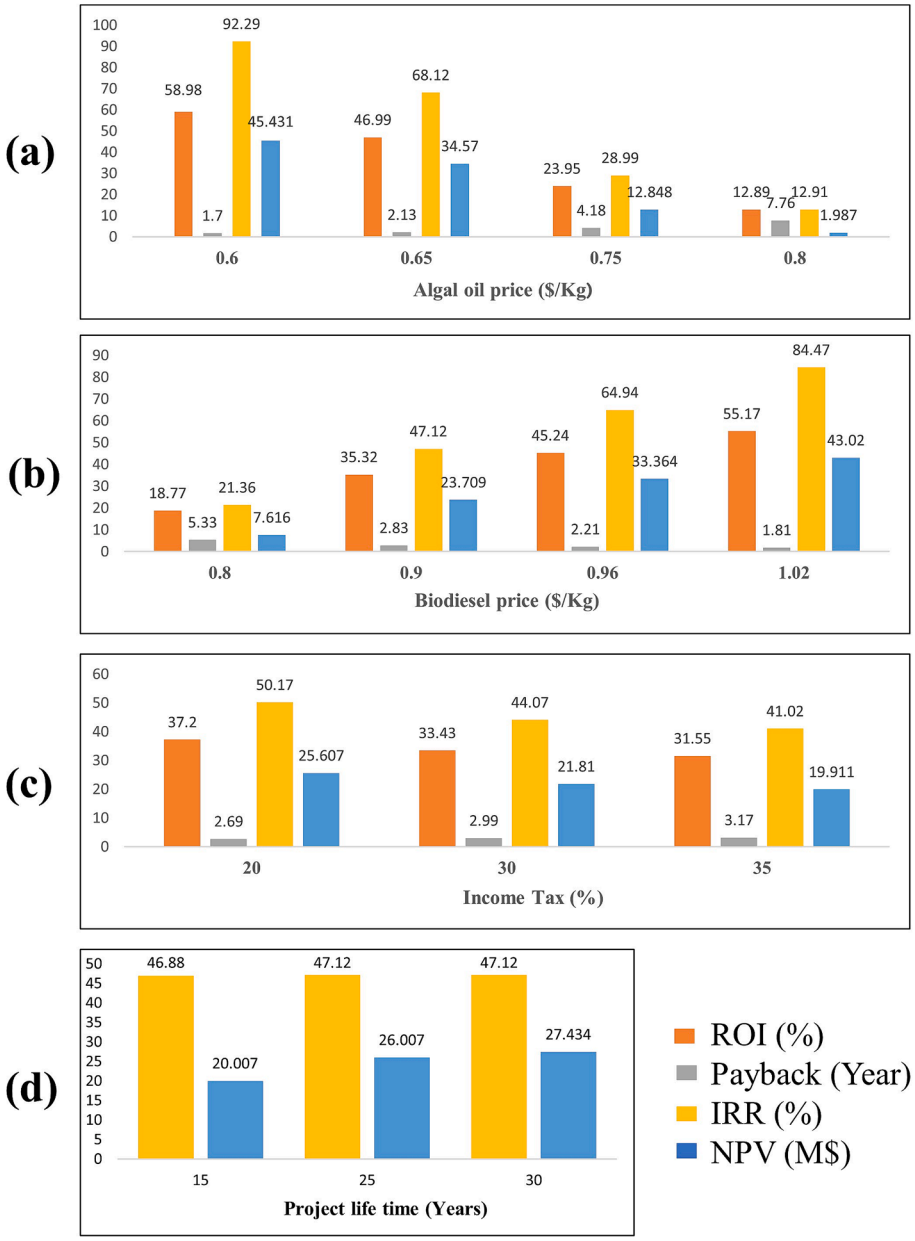


**Fig. 4.** Sensitivity analysis (a) Algal oil price; (b) Biodiesel price; (c) Income tax; (d) Project life time on biodiesel production from *D. bartayresiana* oil.

ROI, reimbursement period, IRR and Net present value were studied based on the price of *D. bartayresiana* oil (0.6, 0.65, 0.75, 0.8 $/Kg), biodiesel (0.8, 0.9, 0.96, 1.02 $/Kg), income tax percentage (20, 30, 35 %) and project lifetime (15, 25, 30 yrs). The effect of the cost variations on the economic factors in biodiesel production process was shown in Fig. 4. The results shows that when the price of algal oil increases, it affects all the economic factors and this indicates that algal oil price plays an important factor for the sustainability of biodiesel production. The highest selling price of biodiesel (1.02 $/Kg) and lowest purchasing price of algal oil (0.6 $/Kg) gives the highest NPV which is more essential for the sustainable production process.

## 4. Conclusions

The characterization results of the synthesized CaO nanocatalyst confirmed the better suitability and possibility of using as an efficient nanocatalyst for transesterification of *D. bartayresiana* oil. Also, the use of construction waste material as the catalyst makes it more cost effective for the overall process. The process optimization of biodiesel production from *D. bartayresiana* oil using the synthesized CaO nanocatalyst revealed the optimal conditions of calcination temperature 573 °C, catalyst concentration 5.62%, molar ratio of methanol to oil 14.36:1, reaction temperature 55.7 °C and required reaction time of 67.57 min with biodiesel yield of 89.6%. The investigation on the technoeconomic analysis of the process showed the sustainability of the biodiesel production from *D. bartayresiana* oil with the positive results. The sensitivity analysis provided the effect of the price of algal oil and biodiesel for sustainable production. This also confirmed the assumptions of the minimum buying and selling price of the *D. bartayresiana* oil and produced biodiesel.

## CRediT authorship contribution statement

**Pravin Ravichandran:** Conceptualization, Methodology, Investigation, Data curation, Formal analysis, Writing – original draft. **Naveenkumar Rajendran:** Investigation, Validation, Writing – original draft, Writing – review & editing. **Khalid A. Al-Ghanim:** Software, Writing – review & editing. **Marimuthu Govindarajan:** Resources, Writing – review & editing. **Baskar Gurunathan:** Conceptualization, Data curation, Formal analysis, Software, Supervision, Writing – review & editing, Project administration.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

No data was used for the research described in the article.

## Acknowledgements

Authors thank the Department of Biotechnology, St. Joseph's College of Engineering, Chennai, India for providing research support to carry out this research work. Authors express their sincere appreciation to the Researchers Supporting Project Number (RSP2023R48), King Saud University, Riyadh, Saudi Arabia.

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