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Investigation of biodiesel production parameters by transesterification of watermelon waste oil using definitive screening design and produced biodiesel characterization

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ABSTRACT

In the present work, biodiesel was produced from watermelon waste oil by the transesterification process. A definitive screening design was used to investigate the effect of transesterification parameters such as catalyst concentration (4 – 10 %), methanol-to-oil ratio (2 – 10 *mol*/*mol*) and reaction temperature (46-60°C). The quadratic regression analysis was used to predict the yield of biodiesel. It is found that the optimum catalyst concentration, methanol-to-oil molar ratio and reaction temperature are 5.51%, 5.77:1 and 60°C, respectively, resulting in a 96.763% yield of methyl esters, which is in excellent confirmation with the predicted value. According to the results, all variables had a significant effect on the yield of biodiesel. The physicochemical property of the extracted watermelon waste oil and produced biodiesel using optimum process parameters was analyzed. The results revealed that the extracted oil and produced biodiesel met the requirements of ASTM and EN14214 (European committee for standardization) standard methods. Oleic acid and Linoleic acid were detected to be the dominant unsaturated fatty acids; while palmitic acid was found to be the highest amount of saturated fatty acid. It can be concluded that a definitive screening design for optimization of transesterification reaction parameters is an effective way to boost the yield of biodiesel. In addition, this method significantly reduces the number of experiments, which is important for a limited amount of raw materials. *In general,* the finding of this work proposes that the watermelon waste oils seem to be a potential and alternative feedstock for the production of biodiesel from waste materials.

1. Introduction

Today, fossil fuels are the main contributor to fulfilling the total global energy demand; and they remain the major source of the world energy supply scenario (Sun et al., 2019). Because of the industrial development in the last century, which enhanced energy demand, economic activity and consumption (Gustavsson et al., 2021), nonrenewable fuel was the main source of energy related to transportation (Griffiths et al., 2021). However, the requirement for fossil-based energy increased dramatically owing to a quick increase in human population and industrialization (Antar et al., 2021). This quick increase in oil usage increased care towards the accessibility and environmental issues because of the burning of fossil fuels-based energy. Therefore, sustainable resources and renewable energy such as biomass, energy can reduce some concerns associated with fossil fuels (Al-muhtaseb et al., 2018).

Biodiesel is becoming alternative renewable energy because of its environmentally eco-friend and non-toxicity (Anwar et al., 2019). Currently, biodiesel has attracted enhancing attention as a possible source of fossil fuel; it comprises long-chain alkyl esters produced through transesterification of animal fats or vegetable oil with alcohol (Al-muhtaseb et al., 2018). Mostly, biodiesel can be produced using edible and non-edible sources; however, owing to the consumable source, which is used as food (Abomohra et al., 2020), a new boulevard of producing biodiesel through non-edible sources as possible feedstock is an alternative for renewable energy production (Anwar et al., 2019). Low-cost oil from crop wastes is becoming an important source of energy to produce economical oils suitable for the production of biodiesel (Ewunie et al., 2021). One of the potential and alternative sources of feedstocks used for biodiesel production is watermelon waste generated from food processing industries (Devarajan et al., 2021; Fakayode et al.,

Abbreviations: ℃, Degree Celsius; Fig, Figure; g, Gram; h, Hour; I_2, Iodine; μL , Micro liter; mL, Mill liter; min, Minute; v, Volume; w, Weight.

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2021). Watermelon a popular vegetable plant consumed worldwide has been recently realized, as an alternative source of feedstock (Gómez-García et al., 2020), as a natural source of oil and antioxidants such as carotenoids and tocopherols used for human health and an excellent source of unsaturated fatty acids (Gómez-García et al., 2020). Due to its large production for consumable purposes, watermelon byproducts are commonly considered as agro-industrial wastes from the industries (Awasthi et al., 2021). The availability of watermelon during harvest and discharge in its processing industries leads a lot of wastes to the non-concerned watermelon wastes and thus, this could be a great option source of feedstocks for biodiesel production (Kassim et al., 2022). In this consideration, this feedstock could be considered as a sustainable source of materials for biodiesel production while upgrading watermelon wastes (Milano *et al.*, 2018; Macawile *et al.*, 2020).

Ethanol and methanol are the most often used alcohols in the transesterification reaction process for biodiesel production (Adepoju, 2020). Moreover, methanol is more suitable than ethanol, since it is low cost; can be obtained from biomass and eco-friendly reactant for biodiesel production (Mandari and Devarai, 2021). The selection of an appropriate catalyst is also a critical factor; because of the presence of impurities and high free fatty acid in the product (Milano *et al.*, 2018; Macawile *et al.*, 2020). Most investigators use the distinctive mechanism for the production of biodiesel as a basic homogeneous catalyst such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) (Júnior et al., 2020; Moazeni et al., 2019). The basic catalyst is acting the effective reaction to reduce free fatty acid up to 1% (Esonye et al., 2019; Mamtani et al., 2021).

Optimization of process variables is a critical step to increase the efficiency of biodiesel production. The definitive screening design (DSD) approach (of JMP profile Version 14 USA) was used to detect the main and interaction effects of the biodiesel production process variables (Ajala et al., 2021; Rha et al., 2021). By using a DSD, it is possible to estimate without confounding the main and quadratic effects of the variables with a relatively low number of experimental runs (Felix et al., 2019). The main independent variables used in this study are; catalyst concentration (NaOH), methanol-to-oil molar ratio and reaction temperature each having three levels.

To the best of the authors' knowledge, compared to *watermelon*, the research on *watermelon waste* is limited or no information about oil extraction from *watermelon waste*. In addition to this, no published literature report addresses the transesterification process variables such as catalyst concentration, methanol-to-oil molar ratio and reaction temperature using a definitive screening design to produce biodiesel from *watermelon wastes*. This motivated the present research, which aimed to investigate biodiesel production parameters and characterize biodiesel produced from watermelon wastes using a definitive screening design.

2. Material and methods

2.1. Materials and chemicals

Watermelon wastes were collected, from the Addis Ababa food processing industry and transported to the laboratory. All the solvents and chemicals [ethanol, potassium hydroxide, sodium hydroxide, ammonium sulfate ($\geq$ 98%), n-hexane ($\geq$ 99%), and ethanol ($\geq$ 96.5%), anhydrous sodium sulfate (Na_2SO_4; 99%), iodine (pellets), sodium methoxide (CH_3ONa)] analytical grade used throughout the experiments were purchased from chemical product suppers (Piasa, Ethiopia).

2.2. Methods

The collected watermelon wastes were first washed and cleaned with water to remove impurities such as dust and minerals. The watermelon wastes were then sun-dried for a week under room temperature (one atmospheric pressure). Watermelon wastes were powdered utilizing a mixer grinder and a sieved to obtain a similar particle size. The average particle size obtained using a micro sieve 45-mesh size was, followed by an electronic oven drying for 16 h at 105 °C to remove the moisture content. About 4.51 moisture content of the sample was obtained and the samples were stored for further analysis.

2.3. Oil extraction process

In this study, the watermelon waste oil was extracted according to the methodology suggested by Bakhshabadi *et al.* (2017) with some modifications. The watermelon waste powder (30g) was mixed with organic solvent 60 mL of n-hexane in conical flasks and placed in Soxhlet (Lab-line instruments, Inc., Melrose Park, Illinois, German), which was initially dipped into n-hexane so that the solvent diffused completely into the sample. After 15 min, the thimble was subjected to a system that contained n-hexane at 45°C for 4 h (Ravi et al., 2019). The experiments were done repeatedly and at the end of the extraction process, the oil was filtered using a vacuum filter and kept for 36 h to settle any suspended particles and the oils dissolved in n-hexane were recovered using a rotary evaporator at 40°C. Then the solvent-free oils were left in an airtight container and subjected to characterization as per the standard testing method to find their suitability for the production of biodiesel (Al-muhtaseb et al., 2018). The obtained oil percentage was calculated based on the weight of watermelon waste powder, and the recovery was expressed as percentages of the oil yield (Tan et al., 2019).

2.4. Physicochemical characteristics of watermelon waste oils

The physicochemical properties of the extracted oils (density, moisture content, kinematic viscosity acid value and caloric value) were determined according to American society for testing material (ASTM) standard methods.

2.5. Gas chromatograph-mass spectrometry (GC-MS) fatty acid composition analysis

The extracted oil and methyl esters were subjected to gas chromatograph-mass spectrometry (GC-MS) separately for their composition analysis using a model RQ2013 gas chromatography conjugated with a mass spectroscopy analyzer (monopoles, USA). A temperature was adjusted between 65 to 250°C and Helium was used as a carrier gas. The injection volume was fixed to 4 μL at 230°C with a flow rate of 1.50 mL/min. ACR scanner having 50–700 amu range at speed of 1500 was controlled for mass spectroscopy (monopoles, USA). NIST10 very spectral library (NIST10, 2013) was utilized as a standard to compare the recorded spectral data after the evaluation for both oil and methyl esters produced.

2.6. Biodiesel production: transesterification reaction process

Batch experiments were conducted to produce biodiesel from watermelon waste oil using NaOH basic catalyst. The biodiesel production from watermelon waste oil involved a two-step transesterification process accompanied by washing and drying. The watermelon waste oil was pre-heated at 55°C for 30 min to take off the moisture content in the oil. A known amount of NaOH was weighed and dissolved in the measured amount of methanol to produce a different percentage of catalyst concentration in the solution. Before contributing the sodium methoxide solution, preheated oil was cooled up to a selected temperature (55°C), and then sodium methoxide was mixed with oil. Hence, after the addition of a catalyst, the temperature of the reaction solution was manipulated to (46-60°C). The NaOH catalyst (4-10%) solution was combined with the pre-heated oil in a 250 ml of volumetric flask glass reactor and adjusted on a hot plate magnetic mixer with fixed stirring at a selected temperature (46-60°C). The mixtures were then allowed to react for 60 min (Nejad and Zahedi,

2018) of reaction time. The solution was then converted in a separating funnel and left overnight to obtain two layers (glycerol and methyl ester) (Antar et al., 2021) and to settle properly. The resulting biodiesel was collected from glycerol using the separating funnel and water washing was utilized for furthermore purification of biodiesel. The mixtures were agitated gently to remove foam formation and were kept overnight to settle into two phases: biodiesel and a water impurity (Sharma Dugala et al., 2021). The pure biodiesel fraction was then reheated for 60 min at 30 °C to evaporate the remaining water. The biodiesel fraction was titrated with (0.2N) sulfuric acid for the measurement of the methyl esters (Goh et al., 2020) and the percentage yield of watermelon waste biodiesel was calculated from the following Eq. (1).

$$\text{Biodiesel yield } (\%) = (\text{Amount of biodiesel})/(\text{Amount of oil used}) \quad (1)$$

2.6.1. Biodiesel characterization

The physicochemical property of the watermelon waste oil biodiesel was conducted at the optimum conditions depending on ASTM standard methods. Viscosity, density, acid value, saponification value and iodine value were determined according to the methods prescribed by ASTM standards (Dehghan et al., 2019; Zhao et al., 2021). The calorific value of the biodiesel samples was tested using a bomb calorimeter according to ASTM D240 and (Hirner et al., 2019), and the cetane number was detected according to ASTM D4737 and (Sani et al., 2018). While fatty acid compositions were determined using Gas chromatography-Mass spectrometry. The pour point (ASTM D97 method) and cloud point (ASTM D2709 method) were carried out using the method adopted by Eloka-Eboka et al. (2017) with some modifications.

2.7. Design of experiment

The selection of the specific experimental design methodology is utilized according to the aims of the study and the nature of the parameters. In common, the design of the experimental methodologies is categorized as process characterization, screening, matching target, robustness design and optimization that dominate in practical applications (Santos et al., 2019). Response surface methodology; central composite designs are widely used in practice for factor screening and building response surface models. However, star points are outside of the hypercube, so the numbers of levels that have to be adjusted for every factor are not clearly identified and sometimes it is not easy to achieve the adjusted values of factors (Yondo et al., 2018). Definite Screening design is an alternative type of experimental design for industrial experimentation. A definite Screening design is typically used in initial levels of experimentation to narrow down the long list of significant variables and interactions to only a few significant effects (Pereira et al., 2018). Definitive Screening design is concentrated on the recognition of the main variables involved in the phenomena, classifying the factors with a "critical impact on the process from several parameters. A definitive Screening design usually requires fewer experimental runs than other designs (Hundie and Akuma, 2022). The experiments are small and efficient, involving several variables. One significant property of a definitive screening design is that all linear effects are orthogonal to linear and quadratic effects so that the prediction of linear results is not biased by the existence of active second-order effects (Pereira et al., 2018). Three factors each with three levels were considered for this experiment, thirteen (13) total numbers of experiments were suggested through a definitive screening design matrix. The independent parameters chosen in this study were catalyst concentration (X_1: 4 – 10 %), the methanol-to-oil molar ratio (X_4: 2 – 10) and reaction temperature (X_5: 46-60°C). The biodiesel yield was selected as the dependent variable. The coded and uncoded levels of the independent parameters are shown in Table 1.

The experiment was carried out randomly to reduce the error. The second-order regression model of the result was performed to validate the biodiesel yield (Abomohra et al., 2020), according to Eq. (2).

Table 1.
Definitive screening design experimental parameters with their levels

Response	Goal		Lower Limit	Upper Limit
Biodiesel Yield %	Maximize		-	-
Parameters	Codes	Roles	Values	
Catalyst concentration (%)	X_1	Continuous	4	10
Methanol-to-oil Molar ratio	X_2	Continuous	2	10
Temperature (°C)	X_3	Continuous	46	60

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} X_i^2 + \sum_{i=1,\ i<j}^{n-1} \sum_{i=2}^{n} \beta_{ij} X_i X_j \quad (2)$$

Where, Y is the yield of biodiesel (predicted), β_0 β_{ii}, and β_{ij} are coefficients of regression analysis and X_i and X_j are independent variables.

3. Results and discussion

In the present study, the relationships between the yield of biodiesel as a response and three operating conditions, the catalyst concentration, methanol-to-oil molar ratio and reaction temperature were studied. Thirteen (13) experiments were conducted based on the definitive screening design (DSD) matrix and the results were analyzed (Table 2) and Transesterification reaction variables were optimized using DSD. The actual yields were evaluated to get a regression model. The anticipated values of biodiesel yield were determined utilizing the regression model and compared with the actual values. The large value of the coefficient of determination (Table 3: R^2=0.992) displays that the model adequately shows the experimental results (Abomohra et al., 2020). The biodiesel yields obtained in this study varied between 81.82% and 94.31 %. The anticipated theoretical maximum conversion efficiency of watermelon waste under optimal parameters is 94.37%. The maximum conversion efficiency of watermelon waste corresponding to optimal parameters of catalyst concentration (7%), methanol-to-oil ratio (6:1) and temperature (53°C) was found to be 94.31% yield of biodiesel (Table 2). The result of the analysis indicates that a conversion efficiency of 93.31% agrees closely with the predicted conversion efficiency of 94.37% of the maximum theoretical yield, meaning that the watermelon waste feedstocks can be successfully used for biodiesel production.

Devarajan *et al.* (2021), defined that methyl ester produced from watermelon oil at optimum operating conditions, methanol-to-oil ratio (20%), temperature (60°C), time (55 min), stirring speed (550 rpm), and catalyst (13 g) and 91% of yield and conclude that watermelon is a promising source of energy.

Table 2.
Experimental parameters and results

Run number	Catalyst concentration (%)	Methanol to oil ratio	Temperature°C	Actual Yield (%)	Predicted Yield (%)
1	10	6	46	88.2	88.19
2	10	10	46	81.82	80.95
3	4	10	53	86.51	86.62
4	10	2	53	84.5	83.64
5	4	2	60	90	90.29
6	4	6	60	91.6	91.8
7	7	6	53	94.31	94.37
8	4	10	46	85.1	84.43
9	4	2	46	85.6	85.91
10	7	10	60	89.1	88.83
11	7	2	46	87.04	86.91
12	10	2	60	85.01	84.85
13	10	10	60	83.03	83.37

Table 3.
Analysis of Variance and Summary of Fit

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	224.39	37.40	120.55
Error	6	1.86	0.31	Prob > F
C. Total	12	226.51		<0.0001*
Summary of Fit				
R-Squared			0.992	
R-Squared Adj.			0.984	
Root Mean Square Error			0.56	
Mean of Response			87.26	

3.1. Analysis of variance (ANOVA)

Analysis of variance depends on an approach in which the method uses variance to examine whether the means are differentiable (Ajala et al., 2021). It also evaluates the significance of contributing parameters by connecting the dependent variable to different levels. The coefficient of determination (R-squared = 0.992), value determines a good fit of the model and shows a better degree of correlation between the experimental and predicted values (Fig. 1). Fig. 1 indicates the relationship between the experimental and the predicted values of biodiesel production yield. The variation is quite close between the experimental and predicted values, which represents that the model used, is a perfect fit, and the development of correlation meets an important level.

The statistical importance of the demonstrated model was assessed by the F-ratio for investigation of ANOVA, which appeared that the model is statistically significant at a 95% of confidence level. The demonstrated model F-ratio of 120.55 and the low p-value (<0.0001; Table 3) represent that the model is highly significant in the yield of biodiesel produced. The coefficient of correlation(R = 0.992), proposes that more than 99.2 % of the variance is attributable to the factors and shows a greater significance of the model; meaning the model cannot describe 0.8 % of the total variance.

M*M; Methanol to oil ratio*Methanol to oil ratio, C*C; Catalyst concentration*Catalyst concentration. The P-value of the regression coefficients proposes that among the experimental parameters, methanol-to-oil molar ratio; X_2 (quadratic term), catalyst concentration; X_1 and temperature; X_3 (linear term) and catalyst concentration; X_1 (quadratic term), are highly significant respectively (Eq. (3)) and Fig. 2. Hence, statistical analysis of all the results indicated that self-interactive (methanol-to-oil ratio, catalyst concentration) and reaction temperature had a significant effect on the yield of biodiesel.

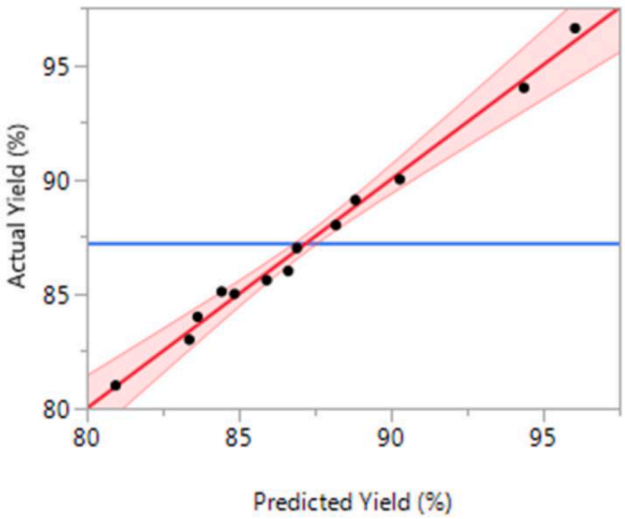

Fig. 1. Experimental value Vs predicted value of biodiesel yield

$$\text{Yield} = 94.4 - 0.649\,X_1 - 0.471\,X_2 + 0.418\,X_3 - 0.233X_1^2 - 0.846\,X_2^2 - 0.001X_1X_3 \quad (3)$$

Where, X_1, X_2, and X_3 are catalyst concentration, methanol-to-oil ratio, and temperature respectively.The significance of each coefficient of the process variables and those of their interactions were analyzed by F-ratio and p-value as indicated in Fig. 2. Fig. 2 indicates, that all contributing variables using the reference point, which represents that all variables cross the blue line, are highly significant with 95% confidence (Abbasi et al., 2021). The graph shows that the methanol-to-oil molar ratio (quadratic term), catalyst concentration and temperature (both linear term) and catalyst concentration (quadratic term) had a highly significant effect on the yield of biodiesel, while the methanol-to-oil ratio (linear term) had a lower effect. Fig. 2, also shows that the lower the p-value and the higher the log worth values, the more significant on the process (Miraculas et al., 2018).

3.2. Effect of process variables on biodiesel yield

Fig. 3 illustrates the conversion efficiency of watermelon waste oils to biodiesel with different transesterification parameters.

A set of tests were carried out by changing the amount of catalyst concentration from 4 to 10 % w/v (Fig. 3). The greater yield of methyl esters was attained during the transesterification of watermelon waste oil with the catalyst concentration of 7 % w/v. With the increase of catalyst concentration; beyond 7% w/v, the methyl esters yield reduced and the quality of methyl esters yield was based on the generation of soap and glycerol formation (Patel et al., 2019). The excess amount of catalyst concentration utilized contributed to the formation of emulsion which enhances the formation of gels and the viscosity (Tan et al., 2019). From this experiment, it was observed that, for an efficient transesterification reaction, 7% w/v of catalyst concentration was the optimum value. In common, the diminish within the biodiesel yield with an increment within the catalyst concentration is because of soap formation with excessive amounts of catalyst (Tan et al., 2019). According to K. Sandesh and P. Ujwa (Sandesh and Ujwal, 2021), the basic-catalyst transesterification reaction is very sensitive to water, whereby the existence of water may induce esters saponification in basic conditions. Hence, excessive amounts of catalyst concentration utilized will induce a shift of equilibrium of the reaction mixture towards a backward reaction, which will decrease the transformation of oil into biodiesel and glycerol (Milano et al., 2018a).

The effect of the methanol-to-oil ratio on the yield of biodiesel is shown in Fig. 3b. Finding a reasonable methanol-to-oil ratio is vital to decide the amount of methyl ester yield. If the methanol-to-oil ratio is not enough for transesterification reaction, this will decrease the biodiesel yield since the glycerides are not changed into fatty acid methyl ester (Narowska et al., 2019; Rozina et al., 2021). This may be because of the high mass ratio of reactants that maximize the contact between the alcohol and the oil molecules (Mueanmas et al., 2019). However, if the amount of methanol is high for a given solution then the polarity of the mixture will rise thereby enhancing the solubility of the glycerol back into the biodiesel phase ultimately decreasing the yield (Zhang et al., 2022). In common, excessive amounts of alcohol are not recommended, since this will diminish the methyl ester yield (Mandari and Devarai, 2021), inhibit the recuperation of glycerol, and raise the price of biodiesel production (Singh et al., 2021). In this study, the maximum yield of biodiesel was achieved at a methanol-to-oil ratio of 6:1 holding catalytic concentration and reaction temperature at their optimum values.

To attain maximum biodiesel yield, the reaction temperature is an important parameter that will affect the yield of production. The effect of temperature on the yield of methyl ester is shown in Fig. 3c. At higher temperatures (below the boiling point of methanol 64.5°C) the rate of reaction in the mixture is more since the rate of reaction is fast at higher

Source	Log-Worth	P-Value
M*M	5.34	0.00000
Catalyst concentration	4.83	0.00001
Temperature	4.15	0.00007
C*C	3.50	0.00032
Methanol to oil ratio	2.25	0.00568

M*M; Methanol to oil ratio*Methanol to oil ratio, C*C; Catalyst concentration*Catalyst concentration

Fig. 2. The interactive and individual effect of parameters on biodiesel yield

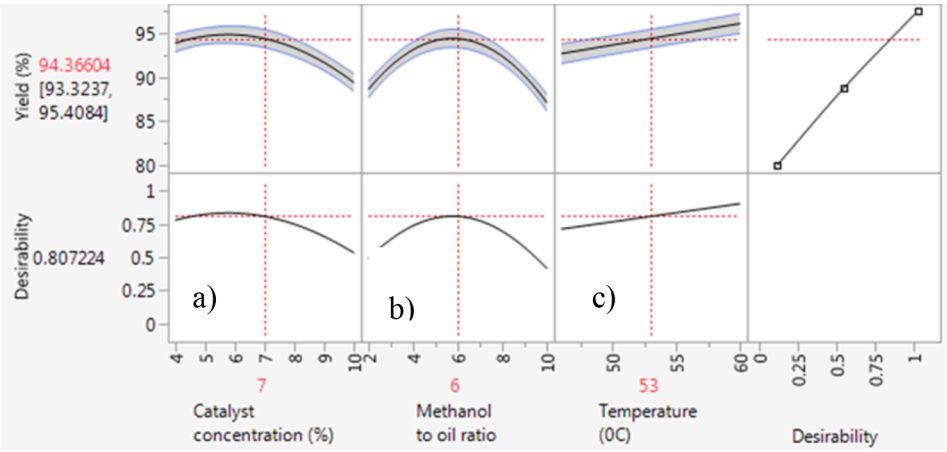

Fig. 3. Effects of catalyst concentration (a), methanol-to-oil ratio (b) and temperature (c) on the yield of biodiesel

temperatures and it reduces the time needed to overcome the process (Singh et al., 2020), leading to the yield of methyl esters to increase (Çelebi and Aydın, 2019). However, if the reaction temperature is beyond the boiling point of alcohol used, then evaporation of alcohol will take place thereby reducing the volume of the solution which will create a reduction in the methyl esters yield (Sharma et al., 2021). From Fig. 3c, a high yield of biodiesel was attained at a reaction temperature of 53°C, holding methanol-to-oil molar and catalytic concentration at their optimum values. Bhatti *et al.* (2008), conducted on biodiesel production from waste tallow and found that the optimum reaction temperature for methyl esters production changed from 50 and 60°C. In addition, the maximum yield of methyl esters was achieved at a reaction temperature of 60 °C by Olubunmi *et al.* (2020), for methyl esters produced from restaurant waste oil utilizing Fe-supported anthill catalyst.

3.3. Optimization of process parameters

3.3.1. Optimization of biodiesel production parameters and model validation

The aim of the present study was the production and Optimization of process parameters for biodiesel produced. Then the optimum values of the biodiesel yield were generated by using desirability maximization in a definitive screening design. The optimum predicted values of transesterification reaction for the yield of biodiesel were catalyst concentration (5.51 %), methanol-to-oil-ratio (5.77:1) and reaction temperature (60°C) corresponding to 96.76% of biodiesel yield. Using this predicted result, experiments were repeated and the attained results were 96.753%, 96.772% and 96.764%, with an average yield of 96.763% conversion. Fig. 4 elaborates on the obtained yields of biodiesel before and after parameters optimization. The result of the present work was in correspondence with the yield (97.06 %) of biodiesel obtained from Moringa oleifera Oil by Niju et al. (2019) at optimized conditions of 8.02 wt %, catalyst concentrations, 8.66:1 methanol to oil molar ratio and 130 min reaction time. However, in the present work, the values of all independent variables are below the result reported by S. Niju et al. The difference between the present yield and previous yield may be due to the differences in plant species, the time required for the plant maturation, variation in material composition or weather conditions (Ewunie et al., 2021).

Based on the results, it can be concluded that the definitive screening design is an effective method to predict the optimal parameters of the transesterification reaction to obtain a better yield of biodiesel.

3.4. Characterization of watermelon waste oils and Fatty acid methyl esters produced

3.4.1. Characterization of watermelon waste oil

The properties of watermelon waste oils were determined and indicated in Table 4. The watermelon waste oils have property values relatively confirmed to ASTM standards. The oil contents, kinematic viscosity, moisture content, and density acid value of the watermelon waste oil were evaluated by the standard method described by ASTMD11 and calorific value (ASTMD240) as indicated in Table 4.

The oil content of watermelon waste was found to be at 42.3± 11%

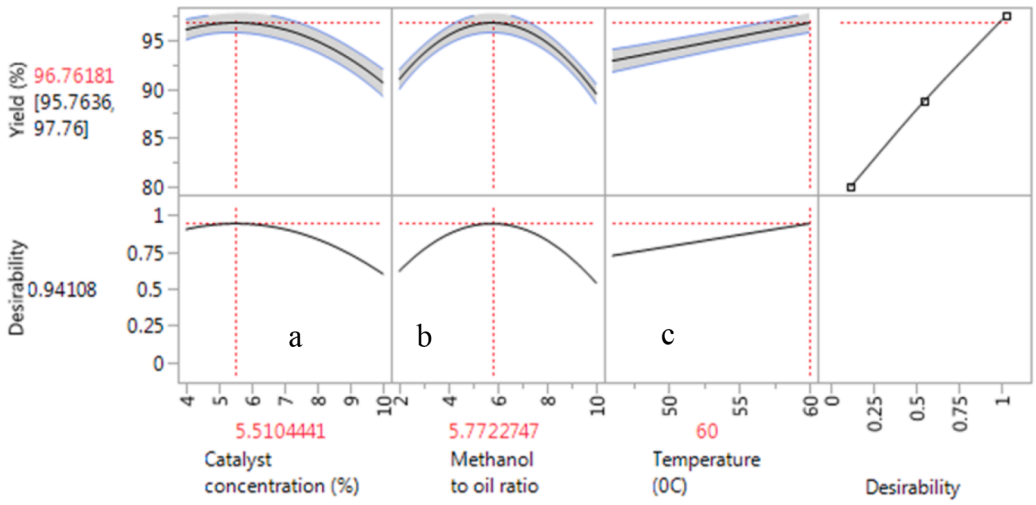

Fig. 4. Watermelon waste oil biodiesel yield (the lower part before optimization and the upper part after optimization).

Table 4.
Physicochemical properties of watermelon waste oil produced

Physicochemical properties	Value	ASTMD11 Method
Oil content (%)	42.3±0.11	-
Density (g/mL)	0.865	0.87
Colour (at room temperature)	light yellow	-
Kinematic viscosity at 40°C,mm^2/s	10.2	9.5
Moisture content (%w/w)	4.51	< 6%
Ash content	3.21	< 10
Acid value (mg KOH/g)	0.89	-
Calorific value ((MJ/Kg))	36.3	ASTMD240

by the soxhlet extraction method. From this result, taking into consideration that the amount of yield of watermelon waste can hold that watermelon waste could be considered a novel that is quite predicted as veritable tools in the quest to meet the demand for alternative fuel. In another report by Hoseini et al. (2018), the amount of oil yield was 38% for Ailanthus altissima (tree of heaven) seed oil. The result obtained in the present work was greater than the result reported by Hoseini et al. (2018) and below the result reported by Gan et al. (2019), 45.2% of oil extracted from Jatropha seed biomass for solid energy production. The variation in the oil content may be due to the particle size distribution and origin of the plant's materials (Páramos et al., 2020).

The proximate property of the watermelon waste oil is shown in Table 4. The experimental result proved that the moisture content of the watermelon waste oil was within the limit of the ASTM standard (Table 4). A high amount of moisture content than the recommended value induces self-generated hydrolysis, and deteriorates the oil (Sánchez-Arreola et al., 2019).

The amount of low ash content represents that the watermelon waste oil can be ignitable with a low quantity of ash (Rehan et al., 2018).

The watermelon waste oils display a lower degree of kinematic viscosity (10.2 mm^2/s) as compared to the viscosity of the majority of the oils currently utilized for biodiesel production, such as date pits oils (23.56 mm^2/s)(Jamil et al., 2016), castor seed oil (20.51 mm^2/s) and waste fish oil (23.20 mm^2/s) (Fadhil et al., 2017) and P. julifera seed oils (35.61 mm^2/s) (Hundie and Akuma, 2022). Usually, the high viscosity is contributed to an excess amount of saturated fatty acids (Jamil et al., 2016), while in the present case of watermelon waste oils, most of the oil contains unsaturated fatty acids hence resulting in low viscosity that can be regarded as an advantage due to low viscous oils are easily handled and have good cold flow properties.

As it can be observed from Table 4, the acid value of watermelon waste oil was found to be 0.89 mg KOH/g. The acid value shows the quantity of free fatty acid (FFA) present in oil or fat and it supplies information about how many formations of FFA have taken place (Keneni et al., 2021).

3.4.2. *Gas chromatography-Mass spectrometry (GC-MS) Analysis*

Different fatty acids were detected from the oil of watermelon wastes by chromatograph-mass spectrometry (Fig. 5). The Fatty acids were detected using their retention times and mass spectral fragmentation patterns by comparing their mass spectra with the NIST10; 2013 library of mass spectra. 9-Octadecenoic acid, 12-hydroxy-, methyl ester, [R-(Z)], cis-Methyl 11-eicosenoate, 9,12-Octadecadienoic acid (Z, Z)-, methyl ester and 6-Octadecenoic acid, methyl ester, (Z)- were some of the main fatty acid compositions detected from the watermelon waste oil (Fig. 5).

3.5. *Characterization of physicochemical properties of watermelon waste biodiesel*

The density of biodiesel altogether influences the motor engine performance and the fuel density is successful in breaking up the fuel splash from the injector (Tomić et al., 2019). In the present work, the density of biodiesel was found to be 0.884 g/mL, which was in the range of fuel property defined by ASTM standards.

The viscosity of the fuel determines the quality of fuel-air mixture combustion and drop establishment of biodiesel. The too low or too high viscosity of the fuel is unsuitable for the proper functioning of the engine (Aslam et al., 2018). Low viscosity contributes to low penetration which leads to the black smoke emission because of low combustion (Eloka-Eboka and Inambao, 2016). A high viscous fuel may penetrate to the reverse wall of the injector, which leads to a cold cylinder surface and results in the low combustion of fuel (Sharma Dugala et al., 2021). According to ASTM specifications, the limited amount of viscosity for biodiesel is between 1.9–6 mm^2/s (Igbum and Eloka-Eboka, A C Nwadinigwe, 2012; Fadhil et al., 2017). Hence, the viscosity of watermelon waste biodiesel was found to be 3.32 mm^2/s, which is in the range of fuel viscosity recommended by the ASTM standard as shown in Table 5.

Critical properties of the biodiesel pour and cloud point, essentially direct the usefulness of a diesel motor in a cold environment (Eloka-Eboka et al., 2014). Cloud point represents the temperature at which the formation of the crystals begins to make precipitate and is usually used to evaluate the low-temperature operability of the fuel. Low the cloud point, the crystals could drop to the lower part of the storage tank (Sahar et al., 2018). Hence far, to run the motor engine under the cloud point of the fuel, heating is a requirement to remove the waxing of the fuel (Abomohra et al., 2020). In the present study, the cloud point of

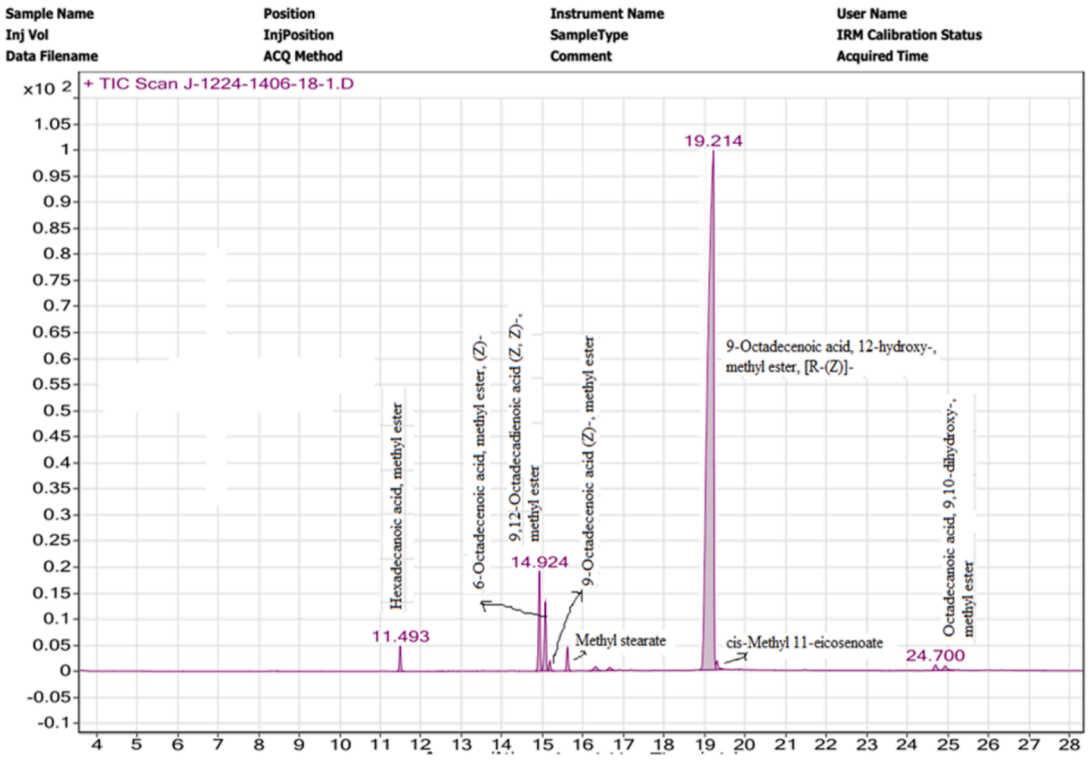

Fig. 5. Gas chromatogram of watermelon waste oil concerning its branded compounds

Table 5.
Physicochemical Properties of Watermelon Waste Oil Biodiesel

Physicochemical properties	Present study	Biodiesel standard	Test Method	
Viscosity at 40°C, mm^2/s	3.32	1.9 – 6.0	D240	
Density at 15°C,g/mL	0.88.4	0.86 – 0.90	D4052	
Acid value (mg KOH/g)	0.35	0.5	D445	
Cloud point (°C)	9.5	-3 to 12	ASTM	
Pour point (°C)	8.3	−30°C to 20°C)	ASTM D2709 ASTM D97	
Saponification value (mg KOH/g)	185.6	< 312	ASTM D 6751	
Iodine value (g I_2/100 g oil)	64.5 39.640	< 120 35	EN 14214 ASTM	
Caloric value (MJ/Kg)	53.5	47 min	D240 ASTM	
Cetane number			D4737	
Fatty acid composition (%)	Carbon Structure	Watermelon waste Biodiesel	Jatropha Biodiesel (Sharma Dugala et al., 2021)	Sandbox (HVO) (Eloka-Eboka et al., 2017)
Palmitic acid	16:0	15.4	14.6	18.66
Stearic acid	18:0	8.5	7.6	0.03
Oleic acid	18:1	43.4	44.6	6.14
Linoleic acid	18:2	31.6	31.9	18.13
Arachidic acid	20:0	0.31	0.3	-
Linolenic acid	18:3	0.59	0.3	9.24
Myristic acid	14:0	0.2	0.1	4.33
Saturated -	24.41	22.6	23.02	
Unsaturated -	75.59	76.8	33.51	

watermelon waste biodiesel was found to be 9.5°C, which is also within the proposed limits of the biodiesel standard (Table 5). Pour point is one more imperative property of biodiesel fuel. The pour point is the slightest temperature at which oil ceases to flow. According to the result in Table 5, watermelon waste biodiesel has a pour point of 8.3°C, which is following the ASTM standard value (Sani et al., 2018).

The acid value is used to check the percentage of free fatty acid in the biodiesel. The maximum degree of acid value for pure biodiesel, as recommended by AOCS and ASTM standards, is 0.5 g KOH 318/g (Tomić et al., 2019). If fats, oil, and biodiesel acid value are used incorrectly, there are some problems and hence acid value increases as the fatty acid break away into a short chain of acids (Goh et al., 2020), thus in this study, the acid value of the biodiesel was found to be 0.35 mg KOH/g, which is within the limited range of ASTM standard (Table 5). The purification process is very important to determine the acid value of biodiesel. It is seen that acid value becomes greater when hot distilled water is utilized for washing and purification purposes.

The saponification value is the quantity of KOH (mg) required to saponify a certain quantity of biodiesel. In addition, the saponification value is an index of the mean size and amount of fatty acids. The fatty acid methyl esters with carbon chain lengths between12 to 20 are taken as biodiesel (Sivalingam et al., 2019). The saponification value of watermelon waste biodiesel was determined to be 185 mg KOH/g which is in the standard range of biodiesel recommended by (Sahar et al., 2018) mgKOH/g). The saponification value in this work is confirmed by the result reported by (Hoseini et al., 2018); 184.5 mg KOH/g of saponification value. This saponification value proposes that the triglycerides in watermelon waste oil have a higher molecular weight of unsaturated and saturated fatty acids.

Iodine value determines the quantity of iodine in g assimilated by 100 g oil (Aslam et al., 2018). It is a measure of the level of unsaturation of biodiesel, thus important to determine the stability of the oil (Adepoju, 2020). A high level of unsaturation leads to the polymerization of fuel because of epoxide generation due to the addition of oxygen in double bonds (Goh et al., 2020). According to EN14214 (European committee for standardization), the iodine value must be below 120 g I_2/ 100 g sample for the oil to be suitable as feedstock for biodiesel production (Keneni et al., 2021). The iodine value of watermelon waste biodiesel in the present work was 64.5 g I_2/100 g oil, which was also

within the limit of the standard value of biodiesel.

The calorific value of a fuel is the heating energy discarded per unit amount of fuel when the fuel is completely burned and the results of combustion are chilled back to the original temperature of the combustible mixture (Depcik et al., 2020). It evaluates the energy capacity of fuel. This is an essential property of the biodiesel that influences the suitability of the feedstocks as an alternative to diesel fuels. Biodiesel has lower heating values relative to pure diesel due to oxygen content since the presence of oxygen in the biodiesel contributes to complete combustion in the I.C engine (Sani et al., 2018). The calorific value of the watermelon waste oil methyl esters determined using a bomb calorimeter following the ASTM D240 standard method is shown in Table 5.

The ignition properties of fuel to auto-ignite next to injection of fuel are known as cetane number. The cetane number is a very important property of biodiesel as it affects the engine directly. The cetane number refers to the quality of biodiesel in terms of ignition and depends on the composition of biodiesel (Jamil et al., 2016). It helps the researcher to select the feedstock and its fat acid alkyl esters for biodiesel production (Amini et al., 2017). An increase in the number of carbon atoms that exist in the chain of fatty acids will maximize the cetane number. The lower the time interval between the inflammation and the injection of fuel into the combustion chambers, the higher will be the cetane number (Al-muhtaseb et al., 2018). According to ASTM standard and European standard (EN14214), the minimum cetane number of biodiesel is 47 and 51 respectively (Azeem et al., 2016). In general, the cetane number for biodiesel is usually higher than the conventional diesel fuel (Rajesh et al., 2019). In the present work, the cetane number (53.5) of produced biodiesel from watermelon waste oils is indicated in Table 5.

3.5.1. Watermelon waste methyl esters Gas chromatography composition analysis

The physicochemical properties and fatty acid compositions of watermelon waste oil are indicated in Table 5. The fatty acid composition acts a significant role in methyl esters quality as it influences the oxidation stability, Cetane number (an indicator of ignition quality), calorific value, cold flow property and density of biodiesel (Singh et al., 2019). In the present work, seven fatty acid compositions were detected via Gas Chromatography-Mass Spectrometry. The percentage compositions of watermelon waste oil fatty acid increased from myristic acid, arachidic acid, stearic acid and palmitic acid for saturated fatty acid and linolenic acid, linoleic acid and oleic acid for unsaturated fatty acid (Table 5). It is concluded that watermelon waste oil methyl ester contains 24.411 % of saturated fatty acid and 75.59 % of unsaturated fatty acid. Oleic acid and Linoleic acid were determined to be the dominant unsaturated fatty acid; while palmitic acid was found to be a high amount of saturated fatty acids. This study was similar to the data reported by Sharma et al. (Sharma Dugala et al., 2021), for the evaluation of the physicochemical property of Jatropha dual biodiesel mixed with diesel (Table 5). Another researcher, (Devarajan et al., 2021) conducted the process parameter optimization to improve the conversion rate and waste minimization on bioconversion of Citrullus lanatus seed oil and obtained similar findings in fatty acid compositions.

4. Conclusion

Biodiesel Production from watermelon waste oil was carried out under different process parameters such as catalyst concentration, methanol-to-oil molar ratio and reaction and temperature using a definitive screening design. The following results were concluded from the experiments. The oil content of watermelon waste was found to be 42.3±0.11%. Using a definitive screening design, the significance of the parameters on the yield of biodiesel was investigated and the optimum parameters were selected and validated. The validity of the selected model was proved by conducting experiments at the anticipated optimum values and the results obtained were confirmed with the predicted yield. The optimum catalyst concentration, methanol-to-oil molar ratio and reaction temperature are 5.51%, 5.77:1 and 60°C, respectively, resulting in a 96.763% yield of methyl esters. According to the experimental results, catalyst concentration and temperature had the highest effect on the yield of biodiesel. The physicochemical property of the produced biodiesel revealed that the produced biodiesel is corresponding to the recommended values set for biodiesel property by ASTM standards. The extracted and biodiesel produced were subjected to gas chromatograph-mass spectrometry analysis to determine the fatty acid in the watermelon waste oil and the percentages composition of the methyl esters produced after transesterification of the oil. Oleic acid (43.4 %) and linoleic acid (31.6 %) were detected to be the dominant unsaturated fatty acids; while palmitic acid (15.4 %) was found to be the highest amount of saturated fatty acid. Based on the results, it was concluded that advanced optimization methods and definitive screening design could be used as effective optimization techniques for process parameters optimization. In general, the finding of this work proposes that the watermelon waste oils seem to be a potential and alternative feedstock for the production of biodiesel from waste materials to mitigate environmental pollution since it decreases the contamination created by the disposal of these materials in nature and produces renewable energy.

Data availability

The [Experimental design, definitive screening design, data analysis, Gas chromatography-mass spectrometry results] data used to support the findings of this study are included within the article.

Ethical approval

This article does not contain any studies with human participants or animals performed by any of the authors.

Supplementary Materials (Biodiesel production process flow chart and statistical data analysis Table and graph) are included

The [Experimental (Figures) and Tables (software information and statistical data analysis)] data used to support the findings of this study are included within the supplementary file(s).

Declaration of Competing Interest

The authors declare that no conflict of interest is associated with this study regarding to material as well as financial.

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Supplementary materials

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