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Multi-Objective Optimization of Mustard Oil Biodiesel production parameters using Grey relational analysis with principal component analysis and Taguchi method

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Abstract

Biodiesel is a substitute fuel to fossil diesel or customary diesel. It can be delivered from various sources, for instance, straight vegetable oil, animal oil/fats, tallow and waste cooking oil. The way toward changing over oils into biodiesels is Transeaterification. Mustard is the regular name for various species in the Brassica family. The types of which a great many people know is vellow mustard, alluded to as white mustard in Europe, and is developed essentially for fixing mustard seed. Thus, it doesn't have as high oil content as the other generally known Indian mustard alluded reciprocally in commerce as brown or oriental mustard. This present mustard's organic name is Brassica juncea. Juncea varieties are developed for edible leaves or greens or for condiment mustard, and have been and are progressively developed as an oilseed crop. Mustard yields around 181.7 liters of biodiesel per acre. An attempt is made to optimize the process parameters of biodiesel produced using Indian Mustard oil (Brassica juncea). The identification of optimal processing parameters is fundamental in the transformation of Biodiesel from different feedstocks by prudence of the basic impact of such parameters on overall production cost and quality. Production of mustard seed oil ethyl esters (MSOEE) through alkali- catalyzed transesterification with ethanol utilizing potassium hydroxide as an catalyst. The impact of the process parameters, for example, catalyst concentration, ethanol to oil molar ratio, reaction temperature, reaction duration was researched in order to discover the optimal conditions for the transesterification process. This experimentation methodically builds up a hybrid optimization method for various quality attributes by integrating the Taguchi parameter design, grey relational analysis, and principal component analysis. To exhibit the efficiency and validity of the proposed hybrid optimization method in controlling all influential Biodiesel processing parameters amid its production are considered. To maximize the yield, minimize the viscosity; the ideal mix of various process parameters is resolved. Single step group transesterification process is done on Mustard oil with alcohols, within the sight of base catalyst potassium hydroxide (KOH) and methanol as solvent, to get biodiesel. The PCA is used to figure the weight of the responses while the optimization is done. Along these lines, the impacts of each of these input parameters are analyzed and exhibited. These results give the information that how to control the parameters keeping in mind the end goal to get the best yield and review viscosity as low. Yield of biodiesel and viscosity are optimized with consideration of performance characteristics namely reaction temperature, reaction time, Catalyst Concentration & Alcohol %. A grey relational grade obtained from the grey relational analysis is utilized to understand the biodiesel production with the various performance characteristics. Furthermore, the analysis of variance (ANOVA) is to be applied to distinguish the most significant factor. Finally, confirmation tests are performed to make a comparison between the experimental results and developed model. These values of this parameters are observed that optimal values for the input parameters for biodiesel production are Reaction temperature 60° C, Reaction time 20 min, Catalyst concentration 1.5 % wt, Alcohol % 10 the yield of Mustard oil biodiesel if increased to 97.12% and viscosity 4.10 cSt. are the desired optimal combination where the GRG value is maximum (0.9998) which are the best combination of this analysis. In this way, the Hybrid Integration of Taguchi Parametric Design, Gray Relational Analysis, and Principal Component Analysis Optimization ends up being extraordinary compared to other strategy for accomplishing parameters that suffice to the future needs of Biodiesel generation.

Key words: Mustard oil biodiesel, Taguchi method, Grey Relational Analysis, Principal Component Analysis, Optimization of process parameters.

1. Introduction

Biodiesel is a renewable source of energy that can help lessen greenhouse gas emissions and limit the "carbon footprint" of agriculture. Global warming can be reduced due to the fact that carbon in the fuel was expelled from the air by the plant feedstock. The global worry regarding the protection of environment and the conservation of non-renewable natural resources, has given rise to improvement of resources of energy as replacement for traditional fossil fuels. The predominant part of all energy consumed globally comes from fossil resources (petroleum, coal and natural gas). However, these assets are confined and might be exhausted in the close to destiny. Hence, searching out alternative sources of new and renewable energy which includes hydro, biomass (better sources of power), wind, solar, geothermal, hydrogen and nuclear is of vital significance. Anitha and Dawn in their publication mentioned that alternative new and renewable fuels have the capacity to solve the most of the current social problems and issues, from air pollution and global warming to other environmental enhancements and sustainability issues [1].

2. Production of biodiesel: Chemically, Vegetable oils are complex esters of fatty acids. These are the fats naturally existing in oil seeds, and known as tri-glycerides of fatty acids. These tri-glycerides in general have the molecular weight in the order of 800 kg/m³ or more. Due to their high weights these fats have high viscosity causing real issues in their utilization as fuels in CI engines. These molecules are to be divided into simpler molecules and hence they have viscosity and other properties similar to standard diesel oils. Adjusting the vegetable oils (to make them lighter) can be attained in many ways, including; Pyrolysis, Micro emulsification, Dilution and Transesterification. Among these, transesterification is the most often used commercial process to produce clean and naturally amicable light vegetable oil fuel i.e. biodiesel [2].

2.1 Biodiesel from Straight vegetable oils: Transesterification of sunflower oil utilizing alcohol and a catalyst delivered a fuel which could be utilized as a substitute for diesel fuel. Long-term operation of a direct injection diesel engine on ester fuel gave very good results. Atlantis Diesel Engines, the South African maker of Perkins engines, has no proclaimed its expectation to respect its ordinary manufacturing plant ensure on their direct injection Perkins engines when kept running on ethyl ester of sunflower oil. Pre-ignition type diesel engines don't appear to create a coking issue on degummed sunflower oil and trans-esterification does not appear to be essential for successful operation in these engines. Deutz 912W pre-combustion diesel engines presently carry the normal guarantee when worked on degummed sunflower oil. The expansion of plant assurances to two distinctively designed engines, demonstrates that sunflower oil, either degummed or trans-esterified, can be a fruitful diesel fuel substitute [3]. At the point when untreated cottonseed oil was explored in direct injection diesel engine, under naturally aspirated and supercharged conditions, the execution decayed with increased injection pressure. A decrease in BSFC of 15% was watched. By increasing the injection pressure in naturally aspirated condition, no development in performance was obtained. With increment in supercharging pressure, engine performance progressed. The investigation uncovered, a blend of supercharging at a prescribed fuel injection pressure, is the most ideal approach to utilize cotton seed oil as a fuel (or) by and large for every single vegetable oil as a fuel [4]. In the Experimental investigation by utilizing jojoba oil as a substitute to diesel fuel, different performance parameter of a naturally aspirated, single cylinder, diesel engine with direct injection, was measured. Fuels utilized were diesel and blends of diesel with jojoba oil. Chemical and physical properties of jojoba oil demonstrate that jojoba oil is an alternative option to Diesel motor fuel. High viscosity of jajoba oil was lessened by blending with Diesel. Measurements of engine performance parameters at various load conditions were done. Results appeared, when utilizing jajoba oil, and when contrasted with diesel, a slight increment in brake specific fuel consumption and negligible loss of engine power was observed. By changing injection timing and injection pressure, further improvement in the motor performance parameters is expected [5]. The performance and emissions characteristics of a direct injected, normally aspirated diesel engine working on 100 percent sunflower oil, 100 percent nut oil and 50 percent (by volume) blends of either sunflower oil or peanut oil with #2 diesel fuel were compared to baseline results come about utilizing #2 diesel fuel. Without recalibration of the rotary injection pump, the higher fuel densities and viscosities of peanut oil and sunflower oil caused fuel flow and energy delivery builds that yielded power and emissions increases. With the fuel flow changed in accordance with giving measure up to fuel energy input, engine power and thermal efficiency diminished somewhat, while emissions expanded marginally [6]. Potential of palm oil as a fuel substitute for automotive and industrial diesel engines was checked on and it was towards, palm oil utilization, its implications on ecological, economy, performance, organic, and social concerns. Performance and exhaust emissions utilizing palm oil fuel and its blends were similar to diesel fuel. Palm oil fuel is environmental friendly. Its use does not influence the engine and bearing components truly. Its use does not degrade lubricating oil[7]. Plausibility of utilizing rice bran oil in diesel engines, with no treatment, was broke down and Tests were led in a 4 stroke naturally aspirated direct injection (DI) diesel engine, with its power output as 4.4 kW. Pure rice bran oil and diesel blends of rice bran oil (25, 50, and 75%) were utilized. It was discovered that, at all loads, specific energy consumption of rice bran oil was higher, when compared with different fuels. For 25 % rice bran oil diesel blend, specific energy consumption was near that of diesel. Rice bran oil is an oxygenated fuel. Its utilization lessens emissions like CO and unburned hydrocarbon. The lessening of CO &HC is because of supply of extra oxygen from the fuel. At no-load condition, for rice bran oil and its blends, the delay period was marginally higher than for diesel. As the load increased, the delay periods were shorter when contrasted with diesel. Peak pressures occurred earlier, closer to TDC and were somewhat higher when contrasted with that of diesel. Normally, bran blends brought about the accompanying; viz., Combustion was better, the viscosity was lower and emissions were lesser, than unadulterated rice grain oil. 25 % rice wheat oil diesel mix, demonstrated preferred outcomes over pure rice bran oil and different blends of rice wheat oil [8]. Rape-seed oil and palm oil as a diesel fuel substitute was utilized for short duration of operation, exceptionally agreeable outcomes (motor execution and discharge levels) were acquired. Problems such as buildup of carbon deposits and piston rings sticking were observed after extended duration of vegetable oil operation. The useful arrangements proposed to conquer the previously mentioned issues are -(I)blending with vegetable oil with ethanol, 20% volume (ii) changing over the vegetable oils into methyl esters, (iii) expanding vegetable oil temperature more than 200 degrees Celsius and (iv) mixing 25 % volume diesel fuel in vegetable oil [9].

2.2 Transesterification of vegetable oils: The high value of soybean oil as a food product makes production of a cost-viable fuel extremely difficult. Nonetheless, there are a lot of low- cost oils and fats, for example, restaurant waste and animal fats that could be changed over to biodiesel. The issue with handling these low cost oils and fats is that they frequently contain a lot of free fatty acids (FFA) that can't be changed over to biodiesel utilizing alkaline catalyst. In this study, a technique is portrayed to decrease the free fatty acids content of these feedstocks utilizing an acid- catalyzed pretreatment to esterify the free fatty acids previously transesterifying the triglycerides with an alkaline catalyst to complete the reaction. Initial process development was performed with synthetic mixtures containing 20 % and 40 % free fatty acids, arranged utilizing palmitic acid. Process parameters, for example, molar ratio of alcohol, type of alcohol, acid catalyst amount, reaction time, and free fatty acids level were examined to decide the best system for changing over the free free fatty acids to usable esters. The work demonstrated that the acid level of the high ffree fatty acids feedstocks could be decreased to under 1 % with a 2- stage pretreatment reaction.

The reaction mixture was permitted to settle between steps so that the water- containing alcohol phase could be removed. The 2- stage pretreatment reaction was shown with actual feedstocks, incorporating yellow grease with 12 % free fatty acids and brown grease with 33 % free fatty acids. In the wake of diminishing the acid levels of these feedstocks to under 1%, the transesterification response was finished with an alkaline catalyst to fuel-grade biodiesel[10]. The economic feasibilities of four continuous processes to yield biodiesel. This incorporates alkali-and acid catalyzed processes. They utilized wastecooking oil and the standard procedure utilizing neat vegetable oil as the raw material [11]. Tests in a DI diesel engine utilizing olive oil methyl ester were conducted. The combustion efficiency and engine performance were observed to be same as that of diesel. The smoke emissions were observed to be lessened to around 25% [12]. Performance and emission of single cylinder DI diesel engine utilizing methyl esters of sunflower oil and castor oil was tried. Thermal efficiency of methyl ester of sunflower oil and castor oil, were equivalent with diesel. Esters of sunflower oil were superior to ester of castor oil, in terms of performance. Diminishment in smoke emission was observed to be 25% for both the methyl esters[13]. Experiments were carried out in a DI diesel engine utilizing Karanji oil ester. Thermal efficiency for Karanji was 29.6% contrasted with 31.5% for diesel. At all loads, the CO and HC emission for ester were lower than diesel. Smoke level was 3.0 Bosch Smoke Unit (BSU) for Karanji oil methyl ester. Maximum rate of pressure rise and peak pressure for Karanji oil methyl ester were similar to that of diesel [14]. Rice bran put away at room temperature demonstrated that most triacylglyceride was hydrolyzed and free fatty acid (FFA) content was brought up to 76% up in a half year. A two-step acid-catalyzed methanolysis process was utilized for the effective transformation of rice wheat oil into unsaturated fat methyl ester (FAME). The initial step was completed at 60 degrees C. Contingent upon the underlying FFA content of oil, 55-90% FAME content in the response product was gotten. Over 98% FFA and under 35% of TG were reacted in 2 h. The organic phase of the first step reaction product was utilized as the substrate for a second acid- catalyzed methanolysis at 100 degrees C. By this two-step methanolysis reaction, over 98% FAME in the item can be gotten in under 8 h. Distillation of reaction product gave 99.8% FAME (biodiesel) with recovery of over 96%. The residue contains enhanced nutraceuticals, for example, gamma-oryzanol (16-18%), mixture of phytosterol, tocol and steryl ester (19-21%) [15]. Usta et al. (2005) (b) tested a diesel engine utilizing methyl ester biodiesel produced from hazelnut cleanser stock/squander sunflower oil blend. The outcomes showed that, without modification of engine and preheating of the mix, methyl ester of hazelnut soap stock/waste sunflower oil can be partially substituted as diesel fuel [16]. Methyl ester of tobacco seed oil, was tried by Usta et al. (2005) (an), in a turbocharged indirect injection diesel engine. Engine demonstrated inferior performance with biodiesel of tobacco seed oil than that of diesel. CO emission was higher than diesel [17]. Investigations in a single cylinder, four stroke, and water cooled, direct injection diesel engine was done. Blends of Rubber-seed biodiesel and diesel were utilized. At full load condition, cylinder pressure data acquired, demonstrated marginally higher values for B20, and B60, when compared with diesel. Peak pressure of B100 was practically identical with diesel. It was finished up, higher peak pressures are because of dynamic injection advance[18]. The waste plastic oil was compared with the petroleum products and found that it can likewise be utilized as fuel in compression ignition engines.

The impact of injection timing on the performance, emission and combustion characteristics of a single cylinder, four stroke, direct injection diesel engine has been tentatively examined utilizing waste plastic oil as a fuel. Tests were performed at four injection timings $(23^{\circ}, 20^{\circ}, 17^{\circ} \text{ and } 14^{\circ} \text{ bTDC})$. At the point when contrasted with the standard injection timing of 23° BTDC the retarded injection timing of 14° bTDC brought about diminished oxides of nitrogen, carbon monoxide and unburned hydrocarbon while the brake thermal efficiency, carbon dioxide and smoke increased under all the test conditions[19]. The transesterification procedure with use of methanol and key uses of fatty acid methyl esters, was talked about. The general parts of this procedure and the relevance of various kinds of catalysts were displayed [20].

2.3 Optimization of Biodiesel from vegetable oils: A few researchers have effectively utilized the Taguchi technique as a helpful tool for optimization of biodiesel yield. The optimal process parameters for biodiesel production from Manilkara zapota (L.) seed were built up with the assistance of the Taguchi technique: 50 °C reaction temperature, 90 min reaction time, 6:1 M proportion of methanol to oil, and 1 wt% catalyst concentration [21]. Correspondingly, the utilization of Taguchi techniques were concentrated to look at the impacts of process parameters on the transesterification of soybean oil utilizing high-frequency ultrasound [22]. The authors decided the optimum conditions to get a biodiesel yield more than 92.5% and in under 30 min as takes after: 581 kHz ultrasound frequency, 143 W, and 0.75% (w/w) KOH loading at 1:6 oil/methanol molar proportion of 9:1, temperature of 60 °C, and agitation speed of 1250 rpm was achieved [23]. Fukuda et al. [24] detailed that methanol and ethanol are most much of the time utilized as a part of the production of biodiesel however methanol is more favored because of its less cost. Biodiesel has been produced through transesterification of edible oils [25]. By and by, over 95% of commercial biodiesel is produced from edible oil, for example, cotton seed, rapeseed, palm, sunflower and soybean oil [26].

3. Materials and methods

3.1 Materials:

Mustard seed or Brassica juncea oil was purchased from Neoglobe Enterprises, Bahadurpura, Hyderabad, India. All reagents such as methanol (GR grade, moisture < 0.02%) and analytical-grade catalyst potassium hydroxide (KOH) were obtained from a local chemical store.

The Mustard or Brassica juncea oil was sifted utilizing filter paper and was used for biodiesel production by the transesterification process (Fig. 1).

3.2 Preparation of Biodiesel from Mustard oil seeds:

Considering Orthogonal Array concept in Taguchi Analysis, the experimentation is carried out as per L27 array. All the samples are made and the procedure followed is as shown below in Table x.DOE of Mustard oil biodiesel preparation.

1. Mustard oil of 150gm is taken in a conical flask of 1 litre.

The oil is preheated at 45°C for 20 min in water bath shaker.
 Mix 0.5% of Potassium Hydroxide catalyst and 10% of methyl

Alcohol by weight of mustard oil in a separate flask. 4. This solution is added to preheated oil sample.

5. In a water bath shaker, the above sample is maintained at 45° C for twenty minutes by stirring constantly.

6. Then the samples are taken in a separating funnel, kept for twenty four hours so that at the bottom, glycerine settles down at the bottom and can be drained off.

7. The liquid remaining in the funnel is crude bio-diesel

as it contains alcohol (Methanol) and catalyst (KOH) in

it.It is also called as Fatty Acid Methyl Ester.

8. To get pure and moisture free biodiesel, ready to be used in an engine, boil the biodiesel.

Hence, all the above mentioned processes have to be repeated and the Yield and viscosity are obtained.

At molar ratio 6:1, transesterification is done and then allowed to settle for 24 hr in order to obtain maximum recovery of ester with lowest possible kinematic viscosity as reported by past researchers. Total 27 ester samples were prepared as shown in Table 1 to find optimal output parameters i.e. Yield ad Kinematic viscosity for the production of biodiesel. The water bath for transesterification is shown in Fig. .



Fig:2 a) Mustard seeds b) Mustard flowers c) Biodiesel separation from glycerine d) Biodiesel obtained from Mustard seeds

4. Taguchi/GRA/PCA Hybrid Method for the Optimization of biodiesel Process Parameters

1

4.1. Taguchi Method: The Taguchi technique is utilized in Design of Experiments in view of the Orthogonal Arrays (OAs). Taguchi OAs are very fractional orthogonal designs that can be utilized to contemplate the entire parameter space with few analyses. In analysing the results, the Taguchi technique utilizes a statistical measure of performance known as signal-to noise (S/N) ratio. The S/N proportion is a measure of performance to develop products or processes that are insensitive to noise factors in a controlled manner [27]. Noise factors are uncontrollable factors that influence product or process uncertainty. These factors include humidity and weather. Depending on the objective, three different methods can be used to calculate the S/N ratio in the Taguchi method:

(1) smaller-the-better quality characteristic:

$$\eta_{ij} = -10 \log \left(\frac{1}{n} \sum_{j=1}^{n} y_{ij}^2 \right),$$



Fig:1 Biodiesel preparation stages of Mustard oil

(2) bigger-the-better quality characteristic:

$$\eta_{ij} = -10 \log \left(\frac{1}{n} \sum_{j=1}^{n} \frac{1}{y_{ij}^2} \right), \dots 2$$

(3) nominal-the-better quality characteristic:

where y_{ij} is the *i*th test at the *j*th trial, *n* is the total number of tests, and *s* is the standard deviation.

4.2. *GRA*: In the analysis of the processing parameters of the biodiesel production, a suitable mathematical model is established to get the relationship between target values and the quality characteristics of the biodiesel obtained from the experiment. The real concern is to analyze the differences among the quality characteristics of biodiesel due to different preparing parameters and to comprehend the connection between quality characteristics and target values. GRA is a method that measures the correlation degree among factors based on the similarity or difference among factors. GRA is characterized by small data requirements and multifactor analysis. The procedure of the GRA is presented below.



Fig:3 Technical line of Hybrid Taguchi/GRA/PCA optimization method

Grey relational generation involves data pre-processing and calculation according to the quality characteristics. The computing method of the grey relational generation is as follows.

(1) The-larger-the-better (the higher the target value, the better):

$$x_i^*(k) = \frac{x_i^{(O)}(k) - \min x_i^{(O)}(k)}{\max x_i^{(O)}(k) - \min x_i^{(O)}(k)}.$$

(2) The-smaller-the-better (the smaller the target value, the better):

$$x_i^*(k) = \frac{\max x_i^{(O)}(k) - x_i^{(O)}(k)}{\max x_i^{(O)}(k) - \min x_i^{(O)}(k)}.$$

(3) The-nominal-the-better characteristic (if the target is a specific value, set the target value as OB):

 $x^{(0)}(k)$ is the measurement of the quality characteristic,

max $x^{(O)}{}_{i}(k)$ is the largest value of $x^{(O)}{}_{i}(k)$, and min $x^{(O)}{}_{i}(k)$ is the smallest value of $x^{(O)}{}_{i}(k)$. We determine the difference sequence $\Delta_{0i}(k)$ and the minimum value Δ_{\min} and maximum value Δ_{\max} in the difference sequence, including the parameter values required to set the reference sequence:

We employ the identification coefficient Z, which normally has a value of 0.5. After the data preprocessing, a grey relational coefficient is deliberated in accordance with specific the relationship among the ideal and true normalized empirical results. The grey relational coefficient can be expressed namely follows:

The average of the grey relational coefficient is then calculated to obtain the grey relational grade. The grey relational grade is defined as follows:

$$\gamma_i = \frac{1}{n} \sum_{k=1}^n \varepsilon_i(k) \,.$$

However, the effect of each factor on the system is not exactly the same in real applications. Thus, (9) can be modified as follows:

$$\gamma_i = \sum_{k=1}^n \omega_k \cdot \boldsymbol{\varepsilon}_i \left(k \right),$$

where w_k represents the normalized weighting value of factor k. Given the same weights, (9) and (10) are equal. In GRA, the grey relational grade is utilized to demonstrate the relationship among sequences. In the event that two sequences are indistinguishable, the value of the grey relational grade is equivalent to one. The grey relational grade moreover shows the degree of influence that the comparability sequence can apply over the reference sequence. Subsequently, if a specific comparability sequence is more important than the other comparability sequences to the reference sequence, the grey relational grade for that comparability sequence and reference sequence will be higher than other grey relational grades [28]. In this investigation, the relating weighting values w_k are gotten from PCA.

4.3. *PCA*: PCA was developed by Pearson [29] and Hotelling [30]. This approach describes the structure of variance covariance by the linear combinations of each quality characteristic. The procedures are described as follows.

(1) Original Multiple Quality Characteristics Array. Consider $x_i(j)$, i = 1, 2, ..., m, j = 1, 2, ..., n

$$x_{i} = \begin{bmatrix} x_{1}(1) & x_{1}(2) & x_{1}(3) & \cdots & \cdots & x_{1}(n) \\ x_{2}(1) & x_{2}(2) & x_{2}(3) & \cdots & \cdots & x_{2}(n) \\ \vdots & \vdots & \vdots & \cdots & \cdots & \vdots \\ x_{m}(1) & x_{m}(2) & x_{m}(3) & \cdots & \cdots & x_{m}(n) \end{bmatrix},$$

where *m* is the number of experiments and *n* is the number of quality characteristics. In this paper, *x* is the grey relational coefficient of each quality characteristic and m = 9 and n = 3.

(2) Correlation Coefficient Array. The correlation coefficient array is evaluated as follows:

where $\text{Cov}(x_i(j), x_i(l))$ are the covariance of sequences $x_i(j)$ and $x_i(l)$, respectively; $\sigma_{xi}(j)$ is the standard deviation of sequence $x_i(j)$; $\sigma_{xi}(l)$ is the standard deviation of sequence $x_i(l)$.

(3) Determining the Eigen values and Eigenvectors. The Eigen values and eigenvectors are determined from the correlation coefficient array

$$(R - \lambda_k I_m) V_{ik} = 0,$$

....13
$$\lambda_k = n, \text{ and } k = 1, 2, \dots, n;$$

 $V_{ik} = [a_{k1}, a_{k2}, \dots, a_{kn}]^T$ correspond to eigen value λ_k .

where λ_k is an eigen value.

(4) Principal Components. The uncorrelated principal component is formulated as follows:

$$Y_{mk} = \sum_{i=1}^{n} X_m(i) \cdot V_{ik},$$

where Y_{m1} is the first principal component, Y_{m2} is the second principal component, and so on. The entire technical line of the hybrid Taguchi/GRA/PCA process optimization method for plastic injection moulding is summarized and illustrated in Figure 3.

5. Implementation of the Proposed Hybrid Taguchi/GRA/PCA Optimization Procedures for Biodiesel production: 5.1. Most Important Variables That Influence the Transesterification Reaction

Transesterification of oil or fat to produce a high yield of biodiesel is typically investigated by optimizing the following reaction variables: alcohol/oil molar ratio, catalyst concentration, reaction temperature, and reaction time involved in the process [31].

i. Reaction temperature: From the literature, it was revealed that the rate of reaction is mainly influenced by the reaction temperature. But, the reaction is conducted near to the boiling point of methanol ($60-70^{\circ}$ C) atmospheric pressure for a given time. Such mild reaction conditions require the removal of free fatty acids from the oil by refining or pre-esterification. Therefore, degummed and deacidified oil is used as feedstock. Pre-treatment is not required if the reaction is carried out under high pressure (9000 kPa) and high temperature (240° C), where simultaneous esterification and transesterification take place with maximum yield obtained at temperatures ranging from 60 to 80° C at a molar ratio of 6:1[32].

ii. Ratio of alcohol to oil: Another vital variable is the molar ratio of alcohol to vegetable oil. As showed before, the transesterification reaction requires 3 mol of alcohol for each mole of triglyceride to give 3 mol of fatty esters and 1 mol of glycerol. With a specific end goal to move the response to one side, it is important to either utilize excess alcohol or expel one of the products from the reaction mixture. The second choice is typically favoured for the reaction to continue to completion. The reaction rate is observed to be most astounding when 100% excess methanol is utilized. A molar ratio of 6:1 is typically used in industrial processes to acquire methyl ester yields higher than 98% (w/w) [32].

iii. Catalysts: The concentration of the catalyst is an essential parameter of the transesterification reaction and a strong influence on the yield of the isolated methyl esters. Excess catalyst reacted with the oil, leading to the formation of soap, in this manner as the catalyst concentration increased; the detachment of esters became difficult [33]. The most suitable catalyst for this procedure ended up being potassium methoxide. The other essential catalyst, potassium hydroxide, achieved similar results however its methyl esters contents were somewhat lower. Then again, the acid catalysts examined, sulfuric and phosphoric acid, got yields in methyl esters are poor, even with higher catalyst concentrations. On account of phosphoric acid, its yield was immaterial. The optimum concentration of potassium methoxide catalyst is 1 wt % [35]. A catalyst is utilized to rush up the procedure and Sodium hydroxide (NaOH) and potassium hydroxide (KOH) are the common catalysts utilized as a part of the reaction process[34]. High concentrations of alkaline catalyst form soaps within the sight of large residues of fatty acids bringing about emulsion formation amongst soaps and water molecules, along these lines prompting low yields of methyl esters [33].

iv. Mixing intensity: Most literatures show that amid the transesterification reaction, the reactants at first frame a two-stage liquid system. The blending impact has been found to assume a critical part in the moderate rate of the reaction. As phase separation stops, blending winds up inconsequential. The impact of blending on the kinetics of the transesterification procedure shapes the reason for process scale-up and design [32].

v. Purity of reactants: Impurities in the oil influence the conversion level significantly. It is accounted for that around 65–84% conversion into esters utilizing crude vegetable oils has been gotten when contrasted with 94–97% yields refined oil under a similar reaction conditions. The free fatty acids in the crude oils have been found to meddle with the catalyst. This issue can be comprehended if the reaction carried out under high temperature and pressure conditions [32].

5.2. Determination of Quality Characteristics:

Table 1 shows the experimental data of chosen parameters for the preparation of biodiesel from brown Mustard oil. Four parameters Methanol-to-oil molar ratio, Catalyst concentration wt %, Reaction time min and Reaction temperature ${}^{0}C$ among the above prominent parameters were chosen as input parameters. The Yield (Y1) of biodiesel and Viscosity (Y2) at 40 ${}^{0}C$ in cSt were taken as output parameters or responses in the experiments. The mean response refers to the average value of the performance characteristic for each parameter at different levels. The average value of Yield of biodiesel for each parameter at levels -1, 0 and 1 are calculated. The main effects of the various process parameters when they change from the lower to higher levels can be visualized from the Fig. 1 that shows the response graphs of Yield of biodiesel from brown Mustard Oil.



Fig 4: Laboratory biodiesel preparation steps (a) methyl Alcohal (CH₃OH) and catalyst potassium hydroxide (KOH) at various wt% (weight percent) are mixed together.

(b) Homogeneous blend of methyl liquor and catalyst KOH along with oil reaction (c) Separation of biodiesel Table:1 Chosen parameters and their levels

		Le	vels	
Process parameters		-1	0	1
Reaction Temperature(°C)	А	45	55	60
Reaction time(min)	В	20	40	60
Catalyst Conc. % wt	C	0.5	1.0	1.5
Alcohol %	D	10	15	20



5.3. Selection of Taguchi OA:

Genichi Taguchi, a Japanese engineer, proposed a few ways to deal with experimental designs that are now and again called "Taguchi Methods." These techniques use two-, three-, and mixed level partial factorial designs. Twenty Seven experiments run utilizing the Taguchi technique was chosen by orthogonal array L27. The aim here is to make an product or process less variable (more robust) even with variety over which we have practically no control. The experimental design matrix and effect of factors on the performance parameters are shown in Tables 2 and 3.

Table:2	Taguchi's	Experimental	Design
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Expt. No.	Input parameters/levels			
1	1	1	1	1
2	1	1	2	2
3	1	1	3	3
4	1	2	1	1
5	1	2	2	2
6	1	2	3	3
7	1	3	1	1
8	1	3	2	2
9	1	3	3	3
10	2	1	1	2
11	2	1	2	3
12	2	1	3	1
13	2	2	1	2
14	2	2	2	3
15	2	2	3	1
16	2	3	1	2
17	2	3	2	3
18	2	3	3	1
19	3	1	1	3
20	3	1	2	1
21	3	1	3	2
22	3	2	1	3
23	3	2	2	1
24	3	2	3	2
25	3	3	1	3
26	3	3	2	1
27	3	3	3	2

	Input parameters/levels		Responses			
Expt. No	Reaction Temperature(°C) (A)	Reaction time(min)(B)	Catalyst Conc. % wt (C)	Alcohol %(D)	Yield %	Viscosity mm ² /sec or cSt.
1	45	20	0.5	10	94.6	5.64
2	45	20	0.5	10	96.25	5.24
3	45	20	0.5	10	94.85	4.15
4	45	40	1	15	90.89	4.78
5	45	40	1	15	95.04	4.65
6	45	40	1	15	88.46	4.26
7	45	60	1.5	20	90.16	4.38
8	45	60	1.5	20	94.25	4.2
9	45	60	1.5	20	88.18	4.18
10	55	20	1	20	90.75	5.54
11	55	20	1	20	91.08	5.6
12	55	20	1	20	85.56	5.83
13	55	40	1.5	10	90.12	4.02
14	55	40	1.5	10	89.42	5.58
15	55	40	1.5	10	85.65	5.84
16	55	60	0.5	15	83.49	4.34
17	55	60	0.5	15	87.84	4.06
18	55	60	0.5	15	79.48	4.49
19	60	20	1.5	15	95.18	5.47
20	60	20	1.5	15	93.89	5.92
21	60	20	1.5	15	91.25	5.93
22	60	40	0.5	20	95.76	4.24
23	60	40	0.5	20	94.49	5.52
24	60	40	0.5	20	87.25	5.81
25	60	60	1	10	91.58	4.12
26	60	60	1	10	92.45	5.52
27	60	60	1	10	88.26	5.76

Table:3 Test data summary for the responses

5.4. *S/N Analysis:* For constant processing parameters, the average value of three repeated results for yield percentage and viscosity are is calculated and is considered the final result. In processing optimization, all the results of yield percentage and viscosity are transformed into S/N ratio. The transformation of the outcomes into S/N ratio includes a series of computations of the mean squared deviation. For this case, the larger the-better category is utilized characterize the yield and the smaller-the-better category is utilized to describe the viscosity behaviour of Mustard oil biodiesel. The final measured results and S/N ratios for the two quality characteristics are shown in Table 4.

Table 4: Data for S/N ratios of performance characteristics

Experiment Number	S/N _{vield} (dB)	S/N _{viscosity} (dB)
1.	39.5178227	-15.0255821
2.	39.6680148	-14.3866257
3.	39.5407467	-12.3609619
4.	39.1703221	-13.5885579
5.	39.5581286	-13.3490591
6.	38.9349387	-12.588192
7.	39.1002781	-12.8294822
8.	39.4856272	-12.4649858
9.	38.9074019	-12.4235256
10.	39.1569327	-14.8701953
11.	39.1884604	-14.9637605
12.	38.6454155	-15.3133711
13.	39.0964237	-12.0845211
14.	39.0286933	-14.932684
15.	38.6545473	-15.3282569
16.	38.4326892	-12.7497946
17.	38.8738465	-12.1705207
18.	38.0051572	-13.0449268
19.	39.570914	-14.7597465
20.	39.4523868	-15.4464341
21.	39.2046575	-15.4610939
22.	39.6236827	-12.5473171
23.	39.507717	-14.8387816
24.	38.8153087	-15.2835226
25.	39.2360128	-12.2979443
26.	39.3181383	-14.8387816
27.	38.9152785	-15.2084497

5.5. Grey Relational Analysis:

Step1: Grey Generation of Raw Data. In the GRA, the experimental results for the S/N ratios of results of yield percentage and viscosity in Table 4 are first normalized according to the larger-the-better characteristic for yield and the-smaller-the better characteristic for the viscosity property of the sequence by using (4 and 5). The values of yield and viscosity are set as the reference sequence $x_0^{(O)}(k)$, k = 1,2,3 and the comparability sequences $x_i^{(O)}(k)$, i = 1, 2, 3, ..., 9, k = 1, 2, 3. Table 5 lists all the sequences after data preprocessing.

According to Deng (1989) [19], a larger value of the normalized results corresponds to better performance and the maximum normalized results that are equal to one indicate the best performance.

Normalization: Here the experimental data is to be normalized in the range of 0 to 1. As MRR is higher the better (HB) and SR is lower the better criterion is selected.

(1) Higher the better for Yield of Biodiesel

$$Xi(k) = \frac{Yi(k) - \min Yi(k)}{\max Yi(k) - \min Yi(k)}$$

(2) Lower the better for kinematic viscosity

$$Xi(k) = \frac{maxYi(k) - Yi(k)}{maxYi(k) - minYi(k)}$$

Expt. no.	Yield (Y ₁) of biodiesel	Viscosity (Y ₂) at 40 ^o C in cSt
Reference sequence	1.0000	1.0000
1.	0.90967835	0.871019577
2.	1	0.681787366
3.	0.92346425	0.081870254
4.	0.70070035	0.445432975
5.	0.93391725	0.3745034
6.	0.55914682	0.149166314
7.	0.65857768	0.220626414
8.	0.89031677	0.112677785
9.	0.54258688	0.100399012
10.	0.69264831	0.825000494
11.	0.7116083	0.852710616
12.	0.38503499	0.956250678
13.	0.65625974	0
14.	0.61552844	0.843507035
15.	0.39052663	0.960659245
16.	0.25710683	0.197026265
17.	0.52240756	0.025469497
18.	0	0.284432119
19.	0.94160609	0.792290177
20.	0.87032685	0.995658399
21.	0.72134878	1
22.	0.97333986	0.137060889
23.	0.90360102	0.815697055
24.	0.4872044	0.947410813
25.	0.74020507	0.063207066
26.	0.78959326	0.815697055
27.	0.54732365	0.925177328

Table 5: Normalization of S/N ratio of Yield and viscosity of Biodiesel

Step 2: Grey Relational Coefficient: It is used to find the Correlation between the ideal (best = 1) and normalized results.

$$\in (k) = \frac{\Delta min + \varphi \Delta max}{\Delta oi(k) + \varphi \Delta max}$$

Now, $\Delta_{0i}(k)$ is the deviation sequence of the reference sequence $x_0^*(k)$ and the comparability sequence $x_i^*(k)$, i.e.

$$\Delta_{0i}(k) = |x_0^*(k) - x_i^*(k)|$$

According to Table 5, the deviation sequences $\Delta_{0i}(k)$ can be calculated as follows:

 $\Delta_{01}(1) = |\mathbf{x}_0^*(1) - \mathbf{x}_1^*(1)| = 1.0000 - 0.90967835 = 0.090321646$

 $\Delta_{01}(2) = |x_0^{*}(2) - x_1^{*}(2)| = 1.0000 - 0.871019577 = 0.128980423$

The deviation sequence $\Delta_{0i}(k)$ can be calculated using above equation $\Delta_{max} = \Delta_{18}(1) = \Delta_{13}(2) = 1$;

 $\Delta_{\min} = \Delta_2(1) = \Delta_{21}(2) = 0;$

 Table 6: The deviation sequences

Deviation sequences	$\Delta_{0i}(1)$	$\Delta_{0i}(2)$
1.	0.090321646	0.128980423
2.	0	0.318212634
3.	0.076535754	0.918129746
4.	0.299299649	0.554567025
5.	0.066082755	0.6254966
6.	0.440853183	0.850833686
7.	0.341422323	0.779373586
8.	0.109683226	0.887322215
9.	0.457413116	0.899600988
10.	0.307351688	0.174999506
11.	0.288391698	0.147289384
12.	0.614965015	0.043749322
13.	0.34374026	1
14.	0.38447156	0.156492965
15.	0.609473367	0.039340755
16.	0.742893166	0.802973735
17.	0.477592443	0.974530503
18.	1	0.715567881
19.	0.058393909	0.207709823
20.	0.12967315	0.004341601
21.	0.278651222	0
22.	0.02666014	0.862939111
23.	0.096398985	0.184302945
24.	0.512795596	0.052589187
25.	0.259794934	0.936792934
26.	0.210406745	0.184302945
27.	0.452676347	0.074822672

Therefore, $\Delta_{01} = (0.090321646, 0.128980423).$

The same calculating method is performed for i = 1, ..., 27, and the results of all Δ_{0i} for i = 1, ..., 27 are listed in Table 6. By investigating the data presented in Table 5,

 $\Delta_{\max}(k)$ and $\Delta_{\min}(k)$ can be expressed as follows:

 $\Delta_{\max} = \Delta_{18}(1) = \Delta_{13}(2) = 1.0000,$

 $\Delta_{\min} = \Delta_2(1) = \Delta_{21}(2) = 0.0000.$

Step 3: Computation of the Grey Relational Coefficient of Response Variables. The grey relational coefficients for each quality characteristic have been calculated by substituting the distinguishing coefficient $\psi = 0.5$ by using (8). Examples on grey relational coefficient $\varepsilon_{1(k)}$ are provided as follows:

 $\epsilon_{1(1)} = 0.0000 + 0.5(1.0000) / \ 0.090321646 + 0.5(1.0000) = 0.846996$

 $\epsilon_{1(2)} = 0.0000 + 0.5(1.0000) / \ 0.128980423 + 0.5(1.0000) = 0.794937$

Thus, $\varepsilon_{1(k)} = (0.846996, 0.794937)$, $\Box = 1, 2, 3$. A similar procedure is applied for $\Box = 1, \ldots, 27$. Table 7 lists the grey relational coefficient for each trial of the L27 OA.

Expt. No.	Grey relational coefficient		
	Yield $\varepsilon_i(1)$	Viscosity $\varepsilon_i(2)$	
1.	0.846996	0.794937	
2.	1	0.611088	
3.	0.867249	0.352577	
4.	0.625548	0.474128	
5.	0.883263	0.444248	
6.	0.531433	0.370142	
7.	0.594232	0.390816	
8.	0.820098	0.360407	
9.	0.522241	0.357245	
10.	0.619309	0.740741	
11.	0.634203	0.772452	
12.	0.448445	0.919541	
13.	0.592599	0.333333	
14.	0.565309	0.761623	
15.	0.450664	0.927058	
16.	0.402287	0.383738	
17.	0.511461	0.339091	
18.	0.333333	0.41133	
19.	0.895425	0.706504	
20.	0.794063	0.991392	
21.	0.642136	1	
22.	0.949379	0.366854	
23.	0.838365	0.730671	
24.	0.493683	0.904831	
25.	0.658072	0.347997	
26.	0.703822	0.730671	
27.	0.524837	0.869833	

Table 7: Calculated grey relational coefficient for twenty seven comparability sequences.

Table 8: Eigen values and explained variation for principal components.

Principal component	Eigen value	Explained variation (%)
First	0.057793	64.0475
Second	0.032442	35.9527

Step 4: Computation of the Contribution of Respective Quality Characteristics by Using PCA: In optimizing a problem concerning multiple quality characteristics or performances, an engineering judgment or subjective estimation is needed to determine the weighting values for each quality characteristic. The use of the conventional method to determine the values for each quality characteristics is heavily reliant on experience and trial-and-error, thus resulting in an increase in uncertainty during the decision-making process. To reveal the relative importance for each quality characteristic in GRA objectively, the PCA is introduced. The PCA is adopted to determine the corresponding weighting values for each quality characteristic. The elements of the array for the multiple quality characteristics listed in Table represent the grey relational coefficient of each quality characteristic. These data are used to evaluate the correlation coefficient matrix and to determine the corresponding eigen values from equation(13) (Table 8). The eigenvector corresponding to each eigen value is listed in Table 9, and the square of the eigenvector can represent the contribution of the corresponding quality characteristic to the principal component. The contribution of yield and viscosity of Mustard oil biodiesel is shown in Table 10; these contributions are listed as 0.499849 and 0.499849 respectively. Moreover, the variance contribution for the first principal component characteristics is as high as 54. 4%. Therefore, for this study, the squares of the respective eigenvectors are selected as the weighting values of the related quality characteristic. Coefficients $\Box 1$ and $\Box 3$ in (10) are set as 0.499849, and 0.499849, respectively.

Table 9: Eigen vectors for principal components.

	Eigenvector		
Quality characteristic	First principal component PC1	Second principal component PC2	
Yield of Biodiesel	-0.867	-0.499	
Viscosity	0.499	-0.867	

Table 10: Contribution of each individual quality characteristic for the principal component.

Quality characteristic	Contribution
Yield of Biodiesel	0.751689
Viscosity	0.249001

Step 5: Computation Grey Relational Grades: On the basis of (10) and the data listed in Table 7, the grey relational grades are calculated as follows:

 $\Box_1 = (0.751689 \times 0.846996) + (0.249001 \times 0.794937) = 0.795674061$

Table 11: Grey relational grade and its order.

Trial	Grey relational grade	Order
1	0.795674061	7
2	0.613267743	14
3	0.355176899	24
4	0.475124252	15
5	0.446555558	16
6	0.371118568	20
7	0.392000723	18
8	0.362757048	22
9	0.358230537	23
10	0.740642002	10
11	0.772294777	8
12	0.917936818	4
13	0.33474018	27
14	0.761190284	9
15	0.92543334	3
16	0.384062815	19
17	0.340099614	26
18	0.411226393	17
19	0.707818682	13
20	0.991097726	2
21	0.998969237	1
22	0.369776757	21
23	0.731624454	11
24	0.903494778	5
25	0.349648194	25
26	0.731002328	12
27	0.868780985	6

By using the same procedure, the grey relational grade of the comparability sequence for $\Box = 1-27$ can also be obtained and is presented in Table 11. The processing parameters were optimized with respect to a single grey relational grade rather than complicated multiple quality characteristics.

Source	DF	Adj SS	Adj MS	F-Value	P-Value	Contribution
						%(ρ)
А	2	0.3446	0.17232	8.56	0.002	23.17
В	2	0.4073	0.20366	10.12	0.001	27.39
С	2	0.1484	0.07421	3.69	0.045	9.98
D	2	0.2247	0.11234	5.58	0.013	15.11
Error	18	0.3622	0.02012			24.35
Total	26	1.4872				100.00

Table 12: Analysis of Variance of Grey Relational Grade





Fig 5: Percentage contribution of Grey Relational Grade

6. Results and Discussion

6.1. Optimal Combination of biodiesel production process Parameters and Their Levels: To analyze the optimal combination of biodiesel production processing parameters for increasing yield and decreasing the viscosity, the average grey relational grade for each biodiesel parameter level is calculated by employing the main effect analysis of the Taguchi method. This process is performed by sorting the grey relational grades corresponding to the levels of the biodiesel production parameters in each column of the OA and then taking the average of parameters with the same levels. For instance, for factor A (Table 2), experiments 1, 2, 3...upto 9 are set to level 1.Therefore, by using the data listed in Table 11, the average grey relational grade for $\Box 1$ can be calculated as follows:

The average grey relational grade for $\Box 2$ and $\Box 3$ is calculated as follows:

 $\Box 2 = (0.597331 + 0.666895 + 0.662196 + 0.475089 + 0.629545 + 0.685129 + 0.437487 + 0.430211 + 0.42975) / 9 = 0.5571,$

 $\square 3 = (0.788194 + 0.876184 + 0.827791 + 0.647277 + 0.744165 + 0.705002 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.710623 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.708692 + 0.525187 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.708692 + 0.525187 + 0.708692 + 0.525187 + 0.708692) / 9 = 0.7260 + 0.525187 + 0.708692 + 0.525187 + 0.708692 + 0.525187 + 0.708692 + 0.525187 + 0.708692 + 0.525187 + 0.708692 + 0.525187 + 0.708692 + 0$

Table 13: Main effects table for grey relational grades.

Symbol	Process parameter	Level 1	Level 2	Level 3		
А	Reaction Temperature(°C)	0.463322821	0.620847358	0.739134793*		
В	Reaction time(min)	0.765875327*	0.591006463	0.466423182		
С	Catalyst Conc. % wt	0.50549863	0.638876615	0.678929728*		
D	Alcohol %	0.7650363*	0.6281411	0.487262		
Total mean value of the grey relational grade $=0.612529526$						
* Levels for optimum grey relational grade						



Figure 6: Effect of biodiesel production parameter levels on multiple quality characteristics.

By utilizing a similar method, calculations are performed for each biodiesel production parameter level, and the main effect analysis is built (Table 13 and Figure 5). Considering that the grey relational grade speaks to the level of correlation between the reference and comparability sequences, a larger grey relational grade demonstrates that comparability sequence exhibits a stronger correlation with the reference sequence. A larger grey relational grade results in better multiple quality characteristics. Figure 5 clearly shows that the Mustard oil biodiesel multiple quality characteristics are significantly affected by the adjustments of the processing parameters. The effect of Methanol-to- oil molar ratio on GRG decreases initially and gradually increases up to level 3 i.e. 8:1. The catalyst concentration increases, the GRG gradually decreases up to level 3 from level 1. The GRG at level 1 of Reaction time is less, gradually increased up to level 2 and then decreased at level 3. The effect of Reaction temperature on GRG at level 1 is more, decreased up to level 2 and then further, decreased up to level 3. These changes are shown in figure 5.

In this case, the best combination of processing parameters and levels can easily be obtained from the main effect analysis by selecting the level of each parameter with the highest grey relational grade. A3, B1, C2, and D1 show the largest value of grey relational grade for factors A, B, C, and D, respectively (Figure 5). Thus, the optimal parameter setting that statistically results in the maximum yield and minimum viscosity is predicted to be A3, B1, C2, and D1. Here in this case, the parameters according to Grey Relational Grade and statistical measure are the same. i.e. A3, B1, C2, and D1. The optimal parameters are at the following conditions Methanol-to-oil molar ratio 8:1, Catalyst concentration, 0.5 wt%, Reaction time, 75 min and the Reaction temperature 50° C.

6.2. Effects of Processing Parameters on Quality Characteristics:

To examine the extent in which biodiesel production parameters significantly influence the performance of yield and viscosity, ANOVA is performed on the Taguchi method for the grey relational grade of twenty seven comparability sequences (Table 11). The computed quantity of degrees of freedom (DOF), sum of square (\Box), Mean of Squares, and percentage contribution (ρ) are presented in Table 12. In this case, the percentage contribution of each processing parameter is directly calculated from S because the DOF for the error term is equal to zero [36] The significance of each processing parameter in the yield of biodiesel and viscosity of it, can be determined by the percentage contribution. Roy [37] suggested an alternative by using the 10% rule; that is, a parameter is considered insignificant when its influence is less than 10% of the highest parameter influence. From the results of ANOVA in Table 12, Methanol-to-oil molar ratio, Catalyst concentration, wt% appear to be the most decisive processing parameters in getting yield in production of biodiesel with the highest percentage contribution of 32.3899% and 35.3049% respectively thus outweighing the other process variables. The analysis also reveals that Methanol-to-oil molar ratio, Catalyst concentration, wt% are significant because their percentages are more than 10% of the highest parameter influence (3.53049). The Reaction time, min and Reaction temperature have percentage contributions 9.76112% and 9.34885% respectively which are also significantly more than 10% of the highest parameter influence is less. In this case, Reaction time, min and Reaction temperature are considered less significant in the yield and viscosity of Mustard oil biodiesel.

6.3. *Verification Test:* Once the optimal levels of the Mustard oil biodiesel process parameters are identified, the subsequent step is to verify the improvements in the quality characteristics by using this optimal combination. The verification test can be used to assess the accuracy of the proposed hybrid Taguchi/GRA/PCA optimization method. An experimental verification test is conducted by using the same procedures as previous runs under the optimal process conditions, namely, A3, B1, C2, and D1, to produce the Mustard oil biodiesel. Table 14 lists the results of five repetitions of the verification tests by using the optimal process conditions obtained by the proposed hybrid optimization method. After optimization, the maximum biodiesel yield and minimum viscosity, the Grey Relational Grade is improved from 0.6316 to 0.9041. The yield of biodiesel is also more compared to the initial parameters. The yield has improved and viscosity decreased with respect to those attained in the main experiment presented in Table 4.

7. Confirmation test

Confirmation test has been carried out to verify the improvement of performance characteristics in preparation of Mustard oil biodiesel. The optimum parameters are selected for the confirmation test as presented in Table 11. The estimated grey relational grade $\hat{\gamma}$ using the optimal level of the production parameters can be calculated using following equation

$$\hat{\gamma} = \gamma_{\rm m} + \sum_{i=1}^{\rm q} (\gamma_i - \gamma_{\rm m})$$

where γ_m is the total mean of the grey relational grade γ_i is the mean of the grey relational grade at the optimal level, and q is the number of the machining parameters that significantly affect multiple-performance characteristics.

Table 14: Improvements in grey relational grade with optimized biodiesel production parameters

Condition description	Optimal production parameters				
	Biodiesel production parameters	Biodiesel production parameters	Grey theory		
	In the initial trial of OA	in twentieth trial of OA	prediction design		
Level	A1B1C1D1	$A_3B_1C_3D2$	$A_3B_1C_3D_1$		
Yield (Y1) of biodiesel		91.25	-		
Viscosity (Y2) at 400C in		5.93	-		
cSt					
grey relational grade 0.7956		0.998969237	0.9998		
		0.6125(Mean GRG)			

With the Grey Relational Analysis, the improvement of Grey Relational grade is improved to 0.9989 from 0.6125. Hence the optimal values for the input parameters for biodiesel production are Methanol-to-oil molar ratio 4:1, Catalyst concentration 1.5 wt%, Reaction time, 90 min, Reaction temperature 55^oC, the yield of Mustard oil biodiesel if increased to 93.54% and viscosity 19.32cSt.

Table 15: Conformation results for Yield and viscosity of biodiesel

Reaction	Reaction time(min)(B)	Catalyst	Alcohol %(D)	Yield of	viscosity
Temperature(°C)(A)		Conc. % wt		biodiesel	
		(C)			
60^{0} C	20 min	1.5	10	97.12	4.10

8. Conclusion

In this investigation, a hybrid Taguchi/GRA/PCA optimization method for biodiesel production process parameters has been produced efficiently to defeat the weaknesses of individual strategies in multiple quality characteristics problems. The problem that emerges while computing the weighting value for every quality characteristic in GRA has been tended to by coordinating the PCA to decide the grey relational grades. In this manner, the conventional approach of utilizing building judgment or subjective estimation to decide the weighting values for every quality characteristics can be maintained a strategic distance from. Biodiesel production parameters Methanol-to-oil molar ratio, Catalyst concentration, wt%, Reaction time, min, Reaction temperature are taken as input parameters or variables, yield and viscosity as responses and validity of the proposed hybrid optimization method in controlling all influential biodiesel processing parameters during production. Through a series of investigations and optimizations of selected multiple quality characteristics for the case of biodiesel production, the yield and viscosity after optimization by the proposed hybrid optimization method is averaged as 93.54 %, and 19.32 cSt. separately, which are improved values than the values in the main experiment.

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