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PROCESS OPTIMIZATION, KINETIC MODELLING AND CHARACTERIZATION OF BIODIESEL PRODUCED FROM MORINGA OLEIFERA OIL: A REVIEW

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Abstract

The rise in world population has resulted in subsequent increase in demand for energy which led to insufficient energy supply. Fossil fuel reserves such as coal, crude oil, and natural oil has been utilized as fuel energy and has been continuously used in large-scale and would get exhausted. Hence development, adoption and diffusion of several alternative technologies such as biomass, wind, solar, ocean thermal, hydrogen, and geothermal energy. Biodiesel has garnered increasing attention because it is renewable and eco-friendly because of its non-emission of CO_2 compared to the conventional diesel. Optimization and kinetic modelling are receiving more importance in characterization of biodiesel production. This paper is an attempt to review recent development and application of different optimization and kinetic modelling processes for the optimum production of biodiesel. Optimization of different reaction parameters such as reaction temperature, time, solvent/solid ratio, catalyst concentration, catalyst amount, particle size, stirring speed, etc., optimization software's such as response surface methodology, different statistical tools (factorial design, ANOVA etc.) were reviewed. Also, thermodynamic and kinetic studies and modeling has been studied. Among these optimization parameters studied, it has been observed that temperature and time has more effect on the biodiesel production yield. Advanced optimization and modelling software's such as Artificial Neural Network (ANN), Laplacian Harris Hawk Optimization (LHHO), and adaptive neuro-fuzzy inference system (ANFIS) were observed to be efficient in the production of high yield (91.45 %, 96.8199 %, 99.8 %) biodiesel.

Keywords: *Biodiesel, Moringa Oleifera, Optimization, Characterization, Kinetic Modelling*

1. Introduction

The rise in world population has resulted in subsequent increase in demand for energy which led to insufficiency in energy supply [1]. Fossil fuel reserves such as coal, crude oil, and natural oil has been utilized as fuel energy has been continuously used in large-scale and would get exhausted [2]. Hence development, adoption and diffusion of renewable energy technologies as alternative sources of energy such as biomass, wind, solar, ocean thermal, hydrogen, and geothermal energy has been increased to reduce the exploitation of fossil fuel reserves. Biodiesel is one of the alternatives of such renewable sources of energy that can be used directly in commercial diesel engines without any major modifications [3-5]. This paper is an attempt to review recent development and application of different optimization and kinetic modelling processes for the optimum production of biodiesel.

2. Biodiesel Overview

Biodiesel is made from renewable energy sources such as vegetable oils and animal fats including sesame oil, sunflower oil, olive oil, etc., agricultural wastes, and small algae [6, 7].

The vast availability of raw materials, renewability, biodegradability and lower $CO_2(g)$ emissions make biodiesel superior to other energy sources [8]. Biodiesel a biofuel derived from the transesterification of bio-oil, and its energy content is comparable to diesel derived from fossil fuels [9-11]. Biodiesel, a combination of methyl esters, has been reported to be of similar quality to fossil diesel [12]. There are four possible ways to use oils and fats for diesel fuel. These includes direct blending, (pyrolysis (thermal cracking), emulsification (microemulsions), and transesterification [13]. Direct use of vegetable oils and fats as fuels in diesel engines has not been satisfactory, because of their high viscosity, low volatility, polyunsaturated compound content, high content of free fatty acids, resins and rubber (formed by oxidation or spontaneous polymerization), and cinder deposits [14]. Pyrolysis and microemulsion methods of biodiesel production is related to an incomplete combustion which is due to a low cetane number [13]. Transesterification remains popular at this moment and the most convenient method for biodiesel production from vegetable oils and fats [13]. In transesterification, vegetable oil (triglycerides) is made to react with primary alcohol in the presence of a catalyst to produce biodiesel (fatty acid alkyl esters) [15]. In this process glycerol emerges as a by-product. Transesterification consists of sequent reversible steps. The first step is the conversion of triglycerides to diglycerides, followed by the conversion of diglycerides to monoglycerides and glycerol, producing one methyl ester molecule at each step [16]. Transesterification reaction is primarily aided by an external catalytic system. Catalysts used in the transesterification of vegetable oils and animal fats can be classified into homogeneous, heterogeneous and enzymatic catalysts [17]. When the catalyst remains in the same phase (usually liquid) with the reactants during alcoholysis, then it is homogeneous catalyst. But when the catalyst is in a different phase (usually non-liquid) with reactants, then it is the heterogeneous catalyst [18]. The homogeneous catalysts used to produce biodiesel are categorized into two types: homogeneous alkaline catalysts (Sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium methoxide ($NaOCH_3$), potassium methoxide ($KOCH_3$), sodium ethoxide ($NaOC_2H_5$), sodium peroxide (Na_2O_2) and sodium butoxide (C_4H_9NaO)) and homogeneous acidic catalysts (sulfuric acid (H_2SO_4), sulfonic acid (HSO_3R) and hydrochloric acid (HCl)) [19]. One major setback of homogeneous catalysts is that the separation of the catalysts from the medium is intricate and often non-economical; as such, reuse of these is often impossible. Also, several washing steps associated with the catalyst removal from the product results in the consumption of water, often deionized, and significant generation of wastewater [20]. Heterogeneous base catalysts are materials like hydrotalcites and zeolites, which contain alkaline oxides or alkaline earth metals supported over large surface area [21]. Utilization of heterogeneous catalyst for biodiesel production has offered some relief to biodiesel producers by improving their ability to process alternative and cheaper feedstock with simplified processes and cheaper manufacturing processes with prolonged catalyst lifetime. The use of heterogeneous catalysts for biodiesel synthesis is considered an eco-friendly process [17]. The heterogeneous catalytic process minimizes the biodiesel filtration process and, thus, it reduces energy and water consumption [22].

3. Moringa Oleifera

Moringa oleifera is an essential oil seed and perennial crop and is widely cultivated in many tropical and subtropical countries [23]. It grows up to 12 m in height and 30 cm in diameter with a deciduous tree shrub and open umbrella-shaped crown [24]. Its pods are triangular in cross-section (300-500 mm long) and legume-like in appearance [25]. All the parts of *Moringa oleifera* tree have been found to be medicinal particularly in African and South Asia [26-28]. The Moringa seeds yield 38–45 % edible oil (called Ben oil, from the high concentration of Behenic acid contained in the oil) that can be used in cooking, cosmetics and it has been reportedly used for machine lubrication, biofuel (biodiesel), [29]. It is also used as a fodder for livestock because of its high biomass yield of 24 tons ha/yr of total dry matter (DM) yield and crude protein (CP) content in fresh leaves varying from 193-264 g/kg DM [30]. It is a rich oil seed belonging to the monogeneric family that can be cultivated on marginal land with high temperature and low water availability or where cultivation of other crops is difficult. Furthermore, *Moringa oleifera* can be cultivated under tropical dry forest conditions when phosphorus and potassium are available in the soil [31]. The oil extracted from *Moringa oleifera* seed (known as Ben oil), has reasonable percentage oil yield of about 45 %, which is reach in oleic acid around 73 % with a low amount of polyunsaturated fatty acids (< 1 %), resulting in a high tendency to resist oxidation [32]. The seeds kernel also contains about 30-50 % oil [33]. Oleic acid from *Moringa oleifera* seed oil (MSO) is an 18-carbon long monounsaturated fatty acid (MUFA).

4. Extraction and Characterization of Moringa Oleifera

Various extraction methods can be used in the extraction of oil and the method is normally dependent on what type of botanical material is been used. These methods include mechanical, traditional and solvent extraction [34]. Soxhlet extraction has been a standard technique for extraction for over a century [35]. The ground material is placed in a thimble filled with solvent for extraction purposes in this protocol. When the liquid reaches the overflow level, a siphon aspirates it from the thimble and unloads it back into the distillation flask with the extracted phytochemical. As this process is continuous, the method will be continued until the extraction is complete. Furthermore, when the sample is continually brought into contact with fresh sections of the extractants, the mass transfer equilibrium is displaced [36]. Solvent extraction is of major commercial importance to the chemical and biochemical industries, as it is often the most efficient method of separation of valuable products from complex feed stocks or reaction products. Some extraction techniques involve partition between two immiscible liquids; others involve either continuous extractions or batch extractions. Because of environmental concerns, many common liquid/liquid processes have been modified to either utilize benign solvents or move to more frugal processes such as solid phase extraction. The solvent can be a vapour, supercritical fluid, or liquid, and the sample can be a gas, liquid or solid [37]. The yield using this process is usually higher than that of mechanical method; and the residue usually contains less than 2 % oil. Common solvents used are hexane and benzene (hydrocarbons) both of which are petroleum derivatives. Solvent extraction plants are becoming more as processing industries now aim to produce meals with minimum oil contact for commercially acceptable production levels without impairing oil or meal quality [36]. The characterization of essential oils through chemical analysis is a mandatory step in the production chain, to be carried out by both researchers and quality control laboratories. Essential oils are primarily composed of terpenes and their oxygenated derivatives [34]. Conventionally, gas chromatography methods are used to perform overall analytical cycle of the essential oil due to its high performance and high level of accuracy. The colour, viscosity, solubility and smell of the extract also can be tested to ascertain the quality of an optimum grade of the extract [35].

Barakat and Ghazal [38] carried out physicochemical properties of moringa seeds and their extracted oil that cultivated at different regions in Egypt. Their results revealed that acid value, iodine value, un-saponifiable matter, peroxide value, refractive index, saponification value of cold pressed moringa seeds oil found to be 0.29 - 0.37 $mg \cdot g^{-1}$, 65.7 - 67.5, 0.60 to 0.74 g 100 g^{-1}, 1.67 - 2.47 mEq/Kg, 1.4607 - 1.4613, 171.7 to 178.3 mg KOH g^{-1}, respectively.

Recently Ameh *et al.*, [39] studied performance evaluation of Moringa oleifera biodiesel synthesized from cow bone catalyst and its blends in diesel engine. They compared the performance of petroleum-based diesel fuel and directly cracked triglycerides (biodiesel) oleifera oil obtained from northern Nigeria. The performance evaluation was conducted using BD100, BD80, BD60, BD40 and BD20, this is to evaluate the performance of the diesel engine in terms of brake thermal efficiency (BTE), brake specific fuel consumption efficiency (BSFCE), carbon monoxide (CO), oxides of nitrogen (NOx) and oxides of sulphur (SOx) emissions. BD blends recorded higher brake thermal efficiencies relative to petroleum-based diesel fuel. BD100 and all the blends under study recorded 16.67 % BSFCE lower than that of diesel fuel at optimum engine load. For all the sample fuels under study, decrease in the amount of CO is directly proportional to the engine load. The result of their study shows that the NOx emissions increase with corresponding increase in engine loads. For all the sample fuels under study, negligible increase in the amount of SOx was directly proportional to the engine load and vice versa.

Francisco *et al.*, [37], in their study on the Turbidity and Acidity as Monitoring Parameters in the Purification of Moringa oleifera Oil for Biodiesel Production, extracted crude oil from the *Moringa oleifera* Lamarck seeds by pressing and by hexane extraction. The crude (by pressing, by solvent extraction and mixed) and purified oils were characterized in saponification index, iodine value, acidity index, peroxide index, water content, turbidity, specific mass and kinematic viscosity. They concluded that the turbidity, had a great influence on clarification of 31.1 NTU for the mixed crude oil and of 2.1 NTU for purified oil, which correspond to the removal of 93.16 % of colloids. The acidity index of 18.1 mgKOH/g for mixed crude oil was mitigated to 0.2 mgKOH/g for the purified oil, with the removal of 98.62 % of the acid species.

Shumi and Bach [40] investigated the physicochemical characteristics of Moringa stenopetala seeds' oil extracted using solvent extraction with food-grade hexane as a solvent and Analysis of Variance (ANOVA) of the extraction parameters. Their study reveals that the seed oil exhibits an average moisture, ash, fiber, protein, and oil contents of 6.3, 4.2, 2.3, 27.5, and 40.2 %, respectively. The physiochemical characteristics of refractive index (40 °C), 1.4625; viscosity 49.4 Cp; density at 25 °C, 0.9317 g/cm^{-3}; saponification value, 191.4; peroxide value, 11.52 millieq O_2/kg; iodine value of 89.21; and Acid Value (AV) 2.21 mg KOH/g. They concluded that the Moringa stenopetala seeds' oil could be used as a substitute for other oil-bearing seeds sources, such as soybean, sunflower, and groundnut.

Adedunni *et al.*, [41] used Oils of Zyziphus spina (Z. spina) and Moringa oleifera (M. oleifera) seeds and analyzed their physicochemical, phytochemical and lipid composition. Refractive index, density and acid value (mg KOH g^{-1}) of M. oleifera oil were 1.471, 0.81, and 1.16. Similarly, Iodine (Wijs), Saponification (mg KOH g-1) and Peroxide (meq kg-^{1}) values obtained for M. oleifera oil were 46.50, 166.77 and 2.16 respectively.

Oyinade and Damilare [42] extracted moringa oleifera oil on a laboratory scale using a Soxhlet extractor. The Soxhlet extractor was connected to a reflux condenser via a heating jacket at 60 °C. After acid treatment, the oil is transesterified using methanol, with potassium hydroxide as a catalyst. The synthesized biodiesel is also characterized by flash point, cloud point, pour point, kinematic viscosity and amount of acid from biodiesel, which are 135 °C, 18 °C, 17 °C, 4.85 mm^{2}s^{-1} and 0.26 mg KOH/g respectively.

Bombo *et al.*, [43] produced biodiesel from Moringa Oleifera oil and Jatropha Curcas oil with the sole aim of assessing the feasibility of the feedstocks as viable sources of biodiesel in Botswana. Oil was extracted from seeds through a soxhlet extraction method using the solvent, n-hexane. The extracted oil was then trans-esterified at 60 °C using a methanol/oil ratio of 12:1 at a stirring rate of 350 rpm, 3 wt.% catalyst loading and 120 min reaction time. Zinc Oxide modified with fly ash was used as heterogeneous catalyst for the process.

Ojewumi *et al.*, [44] in their study extracted oil from Moringa oleifera seed using a 250 ml Soxhlet extractor with Hexane and Ethanol as solvent. The Moringa oleifera seed powder was packed inside a muslin cloth placed in a thimble of the Soxhlet extractor. The extraction was carried out at 60 ºC using thermostatic heating mantle. The solvent in the extracted oil was evaporated and the resulting oil further dried to constant weight in the oven. They deduced that Moringa oleifera oil can be extracted from its seed using ethanol and acetone as extraction solvent. The optimum process variables for both solvent (ethanol and acetone) was determined at sample weight of 40 g, particle size of 325 μm and extraction time of 8 hours.

Efeovbokhan *et al.*, [45] investigated the suitability of three solvents-hexane, Isopropyl Alcohol (IPA) and Petroleum Ether (PE) in the extraction of moringa seeds from northern and southern parts of Nigeria using a soxhlet extractor. The percentage yield of oil from the two samples was found to be dependent on the solvent used, the residence time and the source of the seed sample. Petroleum ether gave the highest yield of 49.38 and 37.57 %, next was hexane with 44.94 and 34.71 % while isopropyl alcohol gave 36.39 and 28.43 %, for samples 1 and 2, respectively. For all solvents, sample 1 produced higher oil yield. The percentage oil yield increased with time reaching an optimum at between 8-10 h. The results obtained from their investigation showed that alternative solvents (IPA and PE) considered could potentially substitute n-hexane in Moringa oilseed extraction.

Moorthi *et al.*, [46] studied the transesterification of Moringa oleifera seed oil by Sodium Silicate Catalyst using different co-solvents. Solvent extraction was carried out for 6 to 8 hours at 40 ˚C to 60 ˚C. The mixture of oil and solvent were separated by means of water bath at 85 ˚C to remove excess solvent from the extracted oil. The biodiesel produced exhibits high yield using diethyl-ether as co-solvent with 60 ˚C as the reaction temperature and 1 hour as the reaction time. Furthermore, the optimum ratio of methanol to oil is 7:1 and the amount of catalyst required to produce highest yield is 0.30 g.

5. Optimization and Kinetic Modelling

Optimization is used to determine the most appropriate value of variables under given conditions. The primary focus of using optimization techniques is to measure the maximum or minimum value of a function depending on the circumstances [47]. It has been reported that recent development in chemical and process engineering industry has undergone significant changes during the past few years due to the increased cost of energy, increasingly stringent environmental regulations, and global competition in product pricing and quality [48]. One of the most important engineering tools for addressing these issues is optimization of the technique involved. Several optimization studies have been reported in the production of biodiesel.

Monica and Sharma, [49] carried out study on the Oil Extraction Optimization and Kinetics from Moringa Oleifera (PKM1) Seeds. Different designed conditions using Central composite face centered design of Response surface methodology was used in their study. The kinetics of extraction was investigated and its parameters were determined based on a second order model. They found that the optimum conditions were at 3.6 h reaction time, temperature, 67.84 °C and solvent to solid ratio of 9:1. The maximum extraction was found to be 33.30 %. The characterization revealed that the oil had Iodine value 67.23, saponification value184.65, density 0.907 and refractive index 1.436 as respectively. They concluded that among three process variables studied, solvent to solid ratio had the most significant effect on the oil percent followed by extraction temperature and time.

Chisa *et al.,* [50] transesterified oil extracted from Moringa seed oil into biodiesel by using factorial design of experiment of 2^4 to obtain different combination factors at different level of reaction temperature, catalyst amount, reaction time and alcohol to oil ratio, that give rise to 48 experimental runs. The oil sample was transesterified in 48 experimental runs, in each case the biodiesel yield was recorded in percentage. Factorial design model was developed using Design Expert 7.0, the model generated R of 0.987 and Mean Square Error (MSE) of 5.0453 and was used to predict and optimize biodiesel yield. Artificial Neural Network (ANN) model from MATLAB R2016a was developed using 4 input variables and 30 runs, the remaining 18 runs were tested with the ANN model to predict and compare the biodiesel yield with the experimental biodiesel yield, the model generated R value of 0.99687 and MSE of 3.50804. It was found that optimum biodiesel yield of 91.45 % was obtained at 5:1 alcohol/ oil molar ratio, 18.89 wt.% catalyst amounts, 45 minutes reaction time and at 45 ℃ reaction temperature. The experimental validation yielded 88.33 % biodiesel. The ANN model adequately predicted the remaining 18 runs with R2 value of 0.99649 and MSE of 4.914243. Both models proved adequate enough to predict biodiesel yield but ANN model proved more adequate.

Aviara *et al.,* [50] studied the Effect of processing conditions on oil point pressure of moringa oleifera seed. The result of Analysis of Variance (ANOVA) showed that all the processing variables and their interactions had significant effect on the oil point pressure of moringa oleifera seed at 1 % level of significance. This was further demonstrated using Response Surface Methodology (RSM). Tukey's test and Duncan's Multiple Range Analysis successfully separated the means and a multiple regression equation was used to express the relationship existing between the oil point pressure of moringa oleifera seed and its moisture content, processing temperature, heating time and their interactions. The model yielded coefficients that enabled the oil point pressure of the seed to be predicted with very high coefficient of determination. The result of multiple regression analysis carried out to express the oil point pressure (P) as a function of the processing parameters of moisture content (M), heating temperature (T), heating time (t) were presented. From the result, the analysis yielded coefficients with which the function that was used to predict the oil point pressure of moringa oleifera seed on the basis of processing parameters established. The model (Eq. 1) is expressed as follows:

$$
\begin{aligned}
P = -0.348 + 0.151M + 0.009T + 0.024t - 0.002MT - 0.007Mt + 6.444 \times 10^{-5}MTt + 0.009M^2 \\
+ 2.038 \times 10^{-5}T^2 R^2 = 0.95 \qquad (1)
\end{aligned}
$$

Theresa *et al.*, [51] performed lipid extraction from non-edible seeds of Indigofera colutea using Soxhlet technique with ultrasonic pre-treatment to evaluate maximum yield of oil. Different parameters such as solvent, particle size, temperature, time, and solvent-to-solid ratio were examined. The high yield of oil extraction was obtained at 6 % moisture content, 0.15 mm particle size, 65 °C temperature, 210 min, and 6:1 solvent-to-solid ratio using n-hexane as solvent.

Jessica *et al.,* 2012, studied Extraction and Characterization of Oil from *Moringa oleifera* Using Supercritical CO_2 and Traditional Solvents. The result of the kinetic run performed for 7 hours using experimental conditions that showed the best yield after 30 min of Sc-CO_2 extraction (2 % yield, at P = 403 bar, T = 40 °C) gave a maximum yield of only 2.8.

Gangadhara and Prasad, [52] studied optimization of transesterification of certain oils to produce biodiesel. In their work, optimization of transesterification process and analysis of biodiesel from non-edible oil was done; based on optimized protocol for biodiesel production from non-edible oilseeds of Neem and Pongamia converted into fatty acid methyl esters (FAME) through base catalyzed transesterification using an optimum ratio of 1:6 (Oil: Methanol) at 60 ^{o}C. Based on qualitative and quantitative analysis of biodiesel, it is concluded that the biodiesel from these species can be feasible, cost effective and environment friendly.

Christopher and Hilary, [53] carried out a study on the Optimization and Modeling of Process Variables of Biodiesel Production from Marula Oil using Response Surface Methodology. The study was carried out using a central composite design of experiments under response surface methodology. A mathematical model was developed to correlate the transesterification process variables to biodiesel yield. The transesterification reaction variables were methanol to oil ratio, (10-50 wt.%), reaction time, (30-90 min), reaction temperature, (30-90 °C) stirring speed, (100-400 rpm) and amount of catalyst, (0.5-1.5 g). The optimum conditions for the production of the biodiesel were found to be: methanol to oil ratio (29.43 wt.%), reaction time (59.17 minutes), reaction temperature (58.80 °C), stirring speed (325 rpm) and amount of catalyst (1.02 g). The optimum yield of biodiesel that can be produced was 95 %. The results revealed that the crucial fuel properties of the biodiesel produced at the optimum conditions met the ASTM biodiesel specifications.

Shumi and Bacha, [54] investigated the physicochemical characteristics of Moringa stenopetala seeds' oil extracted using solvent extraction with food-grade hexane as a solvent and Analysis of Variance (ANOVA) of the extraction parameters. Temperature, extraction time, and particle size had a significant ($p < 0.05$) effect on the oil yield. According to the Experimental Design Response

Surface Methodology (RSM) and ANOVA analysis, the optimum process parameters' combination to find the highest oil yield was particle size of 0.83 mm, the temperature at 78.82 °C, and extraction time of 5.13 hours. The model predicted oil yield was 39.7 ± 0.32 %.

Danane *et al.*, [55] carried out optimization for the production of biodiesel through three factors: Face-Centered-Composite Design (FCCD), building a mathematical model, and statistical analysis. The effects of three variables, ethanol–oil molar ratio, catalyst concentration and stirring speed on biodiesel yield were studied. The obtained experimental data were evaluated by ANOVA and fitted to a second order polynomial equation by multiple regression analysis using JMP Pro 14 software. The optimal conditions of catalyst concentration (1.62 wt.%), stirring speed (200 rpm) and molar ratio of ethanol to oil (12.9:1) were obtained, resulting in a biodiesel efficiency of 89.75 %. The model was also experimentally validated, achieving about 90 % biodiesel yield.

Dagne *et al.*, [56] used Response surface for the optimization of biodiesel production from leather fleshing waste. The oil yield of the goat, hide, and sheep delimed feshing wastes were 23.08 %, 12.05 %, and 26.7 %, respectively. The conversion to biodiesel by KOH-catalyzed transesterifcation was achieved above 96 % under optimum conditions: a methanol-to-oil molar ratio of 6:1, catalyst amount of 1 % w/w, and reaction temperature of 60 ˚C for an hour reaction time.

Tulashie and Kotoka [57] investigated the kinetics and thermodynamics of the viables of moringa oleifera oil extract for biodiesel production. The biodiesel kinematic viscosity (3.75 ± 0.04 mm^2 /s), cetane number (67.12), oxidative stability (15.2 ± 0.5), acid value (0.012 mg/KOH), pour point (-9 ˚C) and carbon residue (0.020 6 0.001) satisfies the American Society for Testing and Materials (ASTM) limits. The highly negative activation entropy (-214.11 6 0.16 J mol^{-1} K^{-1}) and greater activation enthalpy (30.39 6 0.05 kJ/mol) indicate a slower extraction rate due to the higher energy requirement and stiffer transition of the extraction, respectively. The slower extraction rate agrees with the lower mass transfer coefficients (0.0119–0.0210 min^{-1}). The equilibrium constant (K) is positive whilst the Gibbs free energy (DG) is negative, indicating a forward and spontaneous process. This investigation of kinetics, thermodynamics and transesterification essentially provides in-depth knowledge on oil extraction and biodiesel production.

Kundu *et al.,* [58] carried out a kinetic study on acid catalyzed esterification of free fatty acids in ricinus communis oil for the production of biodiesel. The study emphasizes the optimization of acid esterification of non-edible castor (Ricinus communis) oil towards preparation of biodiesel under the influence of four process parameters viz. reaction time (0 to 4 h), reaction temperature (40 to 80 ˚C), catalyst concentration (0.25 to 3.25 w/w) and molar ratio of methanol to oil (1:1 to 20:1). The effect of each parameter on the conversion of fatty acids into FAME has been discussed. Optimal conditions during which the FFA was reduced from 4.038 to 1.076 % were: 1 % w/w conc. H_2SO_4, 15:1 molar ratio of methanol to oil, reaction temperature 50 °C and reaction time 2.09 h. Among all the process variables, reaction temperature exhibited the most significant effect. Kinetic study of the optimized process parameters was performed. The experimental results were found to fit pseudo first order kinetics (Figure 1- 2). These results were further fitted into the Arrhenius equation to determine the effect of temperature on rate constants. Activation energy and frequency factor were estimated as 22.148 KJ/mol and A = 2.710 min-1 respectively. They concluded that the optimum conditions found for acid catalyzed esterification of castor oil can be commercially practiced in the oil sector in future.

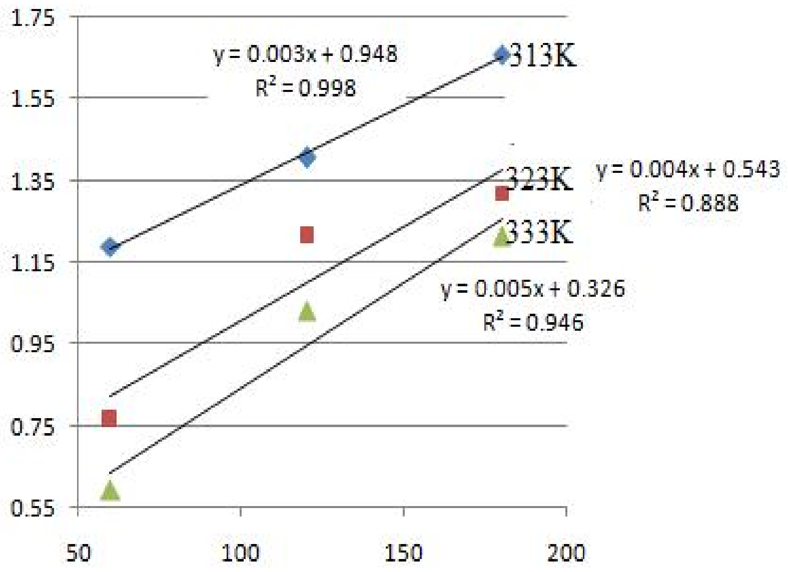

Figure 1: First order kinetic fit for the esterification of FFA at various temperatures: 313 K, 323 K and 333 K

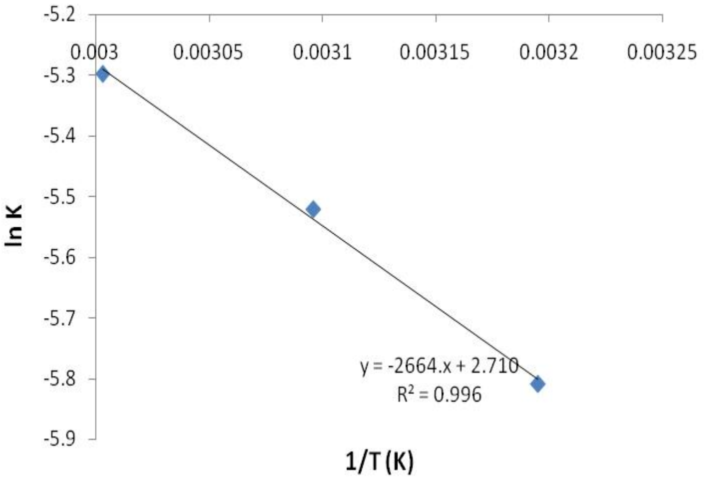

Figure 2: Arrhenius plot of pseudo first order kinetics

Bharti *et al*., [59] synthesized calcium oxide nanocatalysts through the sol–gel method and prepared the particle size of ranged up to 8 nm. Soybean oil was used as the raw material for the synthesis of biodiesel. Nano-CaO catalyst was used to synthesize biodiesel and optimize the reaction variables through optimization processes to achieve a high yield of biodiesel. The reaction variables that were optimized were catalyst amount, oil to methanol molar ratio and reaction temperature. Upon optimization, the conversion of biodiesel was found to be 97.61 %. The optimized value of the reaction variables was: catalyst amount of 3.675 wt.% with respect to oil, molar ratio (alcohol to oil) of 11:1, and reaction temperature of 60 °C for 2 h.

Kale and Ragit, [60] studied optimization of babassu (Orbignya sp) biodiesel production from babassu oil by Taguchi technique and fuel characterization. In the study, signal-to-noise ratios (S/N ratio) with analysis of variance (ANOVA) were employed to optimize the variables and prediction of maximum yield. The various factors influencing the methanolysis of BBO were considered. The temperature of reaction was observed to be most valuable in BBO conversion. It was noted that for low-cost purpose, the most efficient conditions for overall process are 50 °C process temperature, 1 wt.% of catalyst concentration with 6:1 methanol/oil molar ratio for 1 h. With these optimum combinations up to 99.42 % babassu oil methyl esters (BBOME) can be achieved. They concluded that BBOME could be used as a highly beneficial substitute for diesel fuel as its properties are within the acceptable limit and comparable to the biodiesel standard.

This research carried out by Hazrat *et al.*, [61] focuses on the transesterification process for biodiesel production because of its higher output efficiency, reactivity with feedstock, techno-economic feasibility in terms of FFA content, and environmental sustainability. Response surface method with the Box–Behnken model was used to optimize the process. Analysis of variance (ANOVA) was also performed to investigate the effectiveness of the regression model. The optimal process conditions were found to be 5.89 M methanol, 0.5 % (w/w) KOH, 60 °C and 120 min. The predicted yield was 99.5 % for a 95 % confidence interval (99.1, 99.9). The experimental yield was 99.6 % for these conditions. Two different kinetic models were also developed in their study. The activation energy was 16.9 % higher for the pseudo-first-order irreversible reaction than for the pseudo-homogenous irreversible reaction. They concluded that their comprehensive analysis will assist stakeholders in evaluating the technology for industrial development in biodiesel fuel commercialization.

Venkataramana *et al.*, [62] reported a cost-effective approach for biodiesel conversion from Niger seed (NS) oil by employing the transesterification process, Box–Behnken design (BBD), and artificial intelligence (AI) tools. The performances of biodiesel yield are reliant on transesterification variables (methanol-to-oil molar ratio M:O, reaction time Rt, catalyst concentration CC, and reaction temperature RT). BBD matrices representing the transesterification parameters were utilized for experiment reductions, analyzing factor (individual and interaction) effects, deriving empirical equations, and evaluating prediction accuracy. M:O showed a dominant effect, followed by CC, Rt, and RT, respectively. All two-factor interaction effects are significant, excluding the two interactions (Rt with RT and M:O with RT). The model showed a good correlation or regression coefficient with a value equal to 0.9869. Furthermore, the model produced the best fit, corresponding to the experimental and predicted yield of biodiesel. Three AI algorithms were applied (the big-bang big-crunch algorithm (BB-BC), firefly algorithm (FA), and grey wolf optimization (GWO)) to search for the best transesterification conditions that could maximize biodiesel yield. GWO and FA produced better fitness (biodiesel yield) values compared to BB-BC. GWO and FA experimental conditions resulted in a maximum biodiesel yield equal to 95.3 ± 0.5 %. The computation time incurred in optimizing the biodiesel yield was found to be equal to 0.8 s for BB-BC, 1.66 s for GWO, and 15.06 s for FA. GWO determined that the optimized condition is recommended for better solution accuracy with a slight compromise in computation time.

Ameh *et al.*, [63] developed heterogeneous catalyst via dealumination of Ukpor clay and calcined snail shells for biodiesel production from Moringa oliefera seed oil. Optimization of seed oil biodiesel production was carried out via Central Composite Rotatable Design matrix (CCRD) and Response Surface Methodology (RMS). They investigated variables such as temperature, time, catalyst concentration and agitation speed. At a combination of 240 min, 300 °C, 4.0 wt.%, and 300 rpm of time, temperature, catalyst concentration and agitation speed, the maximum yield of 45.50 % were obtained. The statistical model developed for the effects and percentage contributions of the optimization variables is in the form of; $\text{Yield} = +25.85 + 5.88*A + 3.19*B - 2.60*C + 0.71*D + 2.29 *A*B + 1.67 *A*C - 0.069*A*D+2.54*B*C + 0.66*B*D - 2.27*C*D + 0.14*A^2 + 2.12 *B^2 + 1.49*C^2 - 0.78*D^2$ while the reaction obeys first order kinetics, the reaction proceeds faster at elevated temperatures. The calculated activation (Ea) recorded is 2.94 kJmol-1K -1.

The aim of the presented by Díaz *et al.*, [64] was to obtain a phenomenological model for the Moringa oleifera oil extraction process using Soxhlet. Effective diffusivity for Moringa oil through the kernels is obtained, using the kinetics of the extraction process (experimentally determined) and the Fick's diffusion second law for non-steady state. The value of 0.685·10-12 m^2 /s fully matched reports on effective diffusion coefficient for other solids. It was also verified from the statistical analysis and a linear fit for experimental data that the model can be used to describe the oil extraction process of Moringa oleifera in the Soxhlet extractor, responding to the diffusive phenomenon (process controlled by internal resistance).

Shaah *et al.*, [65] determined the feasibility of biodiesel production from candlenut oil using supercritical methanol (scMeOH) as a non-catalytic transesterification process. The influence of the scMeOH transesterification process was determined with varying pressure (85–145 bar), temperature (260–300 ˚C), methanol to oil (M: O) ratio (15: 1–35: 1), and reaction time (15–25 min). The experimental conditions of the scMeOH transesterification process were designed using central composite design (CCD), and the process was optimized using response surface methodology (RSM). It was found that scMeOH temperature, pressure, M: O ratio, and reaction time substantially influenced the transesterification process. The maximum biodiesel yield of 96.35 % was obtained at an optimized scMeOH transesterification process at the pressure of 115 bar, the temperature of 285 ˚C, M: O ratio of 30: 1, and reaction time of 22 min. A second-order kinetics model and Eyring equations were utilized to determine the kinetics and thermodynamics of biodiesel production from candlenut oil. The activation energy value was determined to be 28.35 KJ mol^{-1}. Analyses of the thermodynamic properties of biodiesel revealed that the transesterification process was nonspontaneous and endothermic. The findings of their study reveal that the scMeOH is an effective non-catalytic transesterification process for biodiesel production from candlenut oil.

Oladipo and Betiku [66] extracted Moringa oleifera seed oil (MOSO) from its kernels using solvent extraction method. They assessed the effect of extraction time (1–3 h), solid/solvent ratio (0.050–0.200 g/ml) and type of solvent (ethyl acetate, ethanol and n-hexane) on MOSO yield. The coefficient of determination - R^2, adjusted R^2, and predicted R^2 of 0.9993, 0.9986 and 0.9965, respectively obtained demonstrate the model developed can effectively explain the extraction process. Optimal condition of extraction time of 3 h, solid/solvent ratio of 0.050 g/ml and n-hexane established for the process could be used to obtain MOSO yield of 41.72 ± 0.45 wt.%. Extraction efficacy test showed ethanol, ethyl acetate and n-hexane gave maximum MOSO yield of 8.24 ± 0.00, 14.28 ± 0.22 and 42.00 ± 0.45 wt.%, respectively. The physical and chemical properties of the MOSO differed on the basis of choice of solvent used for the extraction. The acid values of the oil were 8.25 ± 0.01, 3.40 ± 0.04 and 2.88 ± 0.07 mg KOH/g oil with ethanol, ethyl acetate and n-hexane. Fatty acid profile analysis of the MOSO samples confirms that a high-oleic oil could be produced from Moringa oleifera seeds, and thus, could potentially serve as feedstock for chemical and food processing industries.

The study of Ighose *et al.*, [67] focused on the application of adaptive neuro-fuzzy inference system (ANFIS) and response surface methodology (RSM) as predictive tools for production of fatty acid methyl esters (FAME) from yellow oleander (Thevetia peruviana) seed oil. Two-step transesterification method was adopted, in the first step, the high free fatty acid (FFA) content of the oil was reduced to < 1% by treating it with ferric sulfate in the presence of methanol. While in the second step, the pretreated oil was converted to FAME by reacting it with methanol using sodium methoxide as catalyst. To model the second step, central composite design was employed to study the effect of catalyst loading (1–2 wt.%), methanol/oil molar ratio (6:1–12:1) and time (20–60 min) on the T. peruviana methyl esters (TPME) yield. The reduction of FFA of the oil to 0.65 ± 0.05 wt.% was realized using ferric sulfate of 3 wt.%, methanol/FFA molar ratio of 9:1 and reaction time of 40 min. The model developed for the transesterification process by ANFIS (coefficient of determination, R^2 = 0.9999, standard error of prediction, SEP = 0.07 and mean absolute percentage deviation, MAPD = 0.05 %) was significantly better than that of RSM (R^2 = 0.9670, SEP = 1.55 and MAPD =

0.84 %) in terms of accuracy of the predicted TPME yield. For maximum TPME yield, the transesterification process input variables were optimized using genetic algorithm (GA) coupled with the ANFIS model and RSM optimization tool. TPME yield of 99.8 wt.% could be obtained with the combination of 0.79 w/v catalyst loading, 12.5:1 methanol/oil molar ratio and time of 58.2 min using ANFIS-GA in comparison to TPME yield of 98.8 wt.% using RSM. The results of this work established the superiority of predictive capability of ANFIS over RSM.

Chisa *et al.*, [68] transesterify oil extracted from Moringa seed into biodiesel and use factorial design of experiment of 2^4 to obtain different combination factors at different level of reaction temperature, catalyst amount, reaction time and alcohol to oil ratio, that gave rise to 48 experimental runs. Factorial design model was developed using Design Expert 7.0, the model generated R of 0.987 and Mean Square Error (MSE) of 5.0453 and was used to predict and optimize biodiesel yield. Artificial Neural Network (ANN) model from MATLAB R2016a was developed using 4 input variables and 30 runs, the remaining 18 runs were tested with the ANN model to predict and compare the biodiesel yield with the experimental biodiesel yield, the model generated R value of 0.99687 and MSE of 3.50804. It was found that optimum biodiesel yield of 91.45 % was obtained at 5:1 alcohol/ oil molar ratio, 18.89 wt.% catalyst amounts, 45 minutes reaction time and at 45 °C reaction temperature. The experimental validation yielded 88.33 % biodiesel. The ANN model adequately predicted the remaining 18 runs with R^2 value of 0.99649 and MSE of 4.914243. Both models proved adequate enough to predict biodiesel yield but ANN model proved more adequate.

Amenaghawon *et al.*, [69] used Genetic algorithm (GA), to optimize the RSM model for optimum biodiesel production. The optimization was carried out using the GA toolbox of MATLAB R2015a with the RSM model as the fitness function. The RSM model was found to be very adequate in modeling biodiesel production (R^2 = 0.9939). The catalyst was highly stable with high biodiesel yields (>80 %) recorded after six cycles.

Sharma *et al.*, [70] proposed a nature-inspired algorithm named Laplacian Harris Hawk Optimization (LHHO) to optimize biodiesel production instead of the well-known transesterification process. Three independent variables were optimized by this algorithm such that the biodiesel production is maximized. The optimal output of biodiesel obtained by this algorithm was 96.8199 % at parameter values of 65 °C temperature, 12.4245 methanol to oil ratio, and 1.0435 % (w/v) catalyst concentration which is higher than that of other algorithms. From these results, it can be concluded that LHHO can be a preferred choice of a biodiesel manufacturer for obtaining higher profits

5. Conclusion

n this review, several optimization studies have been reported in the production of biodiesel including Central composite face centered design of Response surface methodology, factorial design of experiment of 2^4, Analysis of Variance (ANOVA), Central Composite Rotatable Design matrix (CCRD). Kinetics: second order model, Factorial design model was developed using Design Expert 7.0, Artificial Neural Network (ANN) model, Tukey's test and Duncan's Multiple Range Analysis, Box-Behnken Model, adaptive neuro-fuzzy inference system (ANFIS). Investigation of kinetics, thermodynamics and transesterification essentially provides in-depth knowledge on oil extraction and biodiesel production. Advanced optimization and modelling software's such as Artificial Neural Network (ANN), Laplacian Harris Hawk Optimization (LHHO), and adaptive neuro-fuzzy inference system (ANFIS) were observed to be efficient in the production of high yield (91.45 %, 96.8199 %, 99.8 %) biodiesel.

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