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Predictive capability evaluation and optimization of sustainable biodiesel production from oleaginous biomass grown on pulp and paper industrial wastewater

Madhu Vasaki E [a], Rama Rao Karri [b, *], Gobinath Ravindran [c], Balasubramanian Paramasivan [d]

[a] Department of Environmental Engineering, Government College of Technology Coimbatore, India
[b] Petroleum and Chemical Engineering, Faculty of Engineering, Universiti Teknologi Brunei, Brunei Darussalam
[c] Department of Civil Engineering, S R Engineering College Warangal, Telangana, India
[d] Department of Biotechnology & Medical Engineering, National Institute of Technology Rourkela, India

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ABSTRACT

Biodiesel, as a green fuel, acts as a potential candidate to supplement conventional fossil fuels. This research study targets green environment (*using biodiesel*) and clean environment (*reduce wastewater*) by producing biodiesel through oleaginous biomasses (*Yarrowia lipolytica*, *Metschnikowia pulcherrima* and *Lipomyces starkeyi*) grown on pulp and paper industrial wastewater. Batch culture studies were explored for the potential feedstock of the oleaginous organism by the synthesis of single cell oil and fatty acid methyl ester (FAME) yield. Response surface methodology (RSM) was used to design the optimal experimental matrix and identify the optimal process conditions that enhance the FAME yield. To determine the inherent characteristics of the growth of oleaginous biomasses on the industrial wastewater, a data-driven adaptive neuro-fuzzy inference system (ANFIS) is implemented. *Y. lipolytica* strain cultured shown high biomass concentration of 32.36 g/l with biomass productivity of 5.39 g/l/d was considered for further scale-up for the transesterification process. Results indicated that the maximum yield of 0.48 (g-biodiesel/g-lipid) was obtained under the 2.5 g of lipid dosage with 0.02 g/ml of catalyst concentration by constant stirring at 70 °C. The optimum conditions to achieve maximum FAME yield of 1.154 g/g was obtained at 2.485 g, 70.87 °C and 0.021 g/ml for lipid dosage, temperature and catalyst concentrations, respectively.

1. Introduction

Most of the energy needs of human mankind are met through fossil fuels. With the rapidly growing population, and depleting oil reserves, there is a constant stress to extract more oil from the earth. This process is not only disturbing the ecological behaviour of earth but also contributing to carbon dioxide (CO_2) emission, climatic changes and global warming [1]. Since the transportation sector is essential for industrialization and urbanization, so the need for petroleum products never ends. With limited oil reserves and high demand, the cost of petroleum products is rapidly growing. In order to maintain a sustainable environment, an alternative source of energy is necessary. One such source is the usage of biofuels, especially biodiesel, which has similar characteristics as petroleum products [2]. The significant advantage of biofuel is that it is renewable and can be produced in massive quantity from biological sources within a short interval of time. It can be directly used in engines without significant modifications, and it can also be blended with bioethanol, bio-oil and nano-additives in order to increase engine performance and reduce the CO_2, oxides of Nitrogen (NO_X) and unburned hydrocarbon emission into the atmosphere [3].

Biodiesel is chemically known as fatty acid methyl ester (FAME) can be produced from edible and non-edible oil sources (such as soya bean, sunflower, cottonseed, rapeseed, palm, canola, and peanut) [4,5], animal fats [6] as well as from other plant sources (such as *Raphanus sativus, Brassica carinata, Jatropha curcas* [7],

* Corresponding author.
E-mail addresses: madhuvasaki@gmail.com (M. Vasaki E), kramarao.iitd@gmail.com, karri.rao@utb.edu.bn (R.R. Karri), gobinathdpi@gmail.com (G. Ravindran), biobala@nitrkl.ac.in (B. Paramasivan).

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Pongamia pinnata and *Madhuca indica* [8] by transesterification process). Some oleaginous microorganisms such as microalgae, fungi, yeast and bacteria have the capacity to produce single cell oil (SCO) also known as lipids (or triglycerides), whose composition is similar as that of the plant oils [9]. They accumulate more than 20–70% of lipid by the mass fraction of biomass. SCO reduces the raw material cost for the production of biodiesel, and it is also a renewable and sustainable source for biodiesel production [10]. The use of edible/fatty oils for biodiesel production increases the price of food. The cultivation of non-edible plants requires large area; these disadvantages can be overcome by the usage of oleaginous species, which grow and multiply themselves by consuming the nutrients and carbohydrates in the media composed of high carbon and low nitrogen content. As the nitrogen gets exhausted, the oleaginous organism stops multiplying and convert the carbon into intercellular lipid. The lipid accumulation is carried out up to a certain limit until the cell reaches the limit of obesity.

For the growth of oleaginous microorganisms, there should be a medium that can continuously supply the required nutrients for the growth. This requirement should be met through a source which is available abundantly. A low-cost substrate such as molasses from the sugar manufacturing industry, food waste, agricultural waste, lignocellulosic biomass obtained from agro-based industry [11], glycerol [12], and waste sludge [11,13,14], with high organic content, could be used as a feedstock for culturing of oleaginous organisms.

Several wastewater streams from various process industries are rich in cellulose fibre. Pulp and paper manufacturing industry generate wastewater that contains cellulose fibre, lignin, dioxins, volatile fatty acids and nutrients such as sulphate, phosphate and nitrates that can be a good source of nutrients for the growth of oleaginous organisms. The pulp and paper industry is the largest consumer of freshwater and generates lignocellulose rich wastewater. They produce a large amount of wastewater at different operations such as chipping, digestion, delignification, and paper manufacturing [15,16]. There, by utilizing this wastewater, the oleaginous organisms can be grown abundantly, and that can be used for further production of biodiesel [17]. This approach not only helps in producing biofuel but also reduces the amount of wastewater released into water bodies.

Research studies have been carried out by various researchers worldwide who utilized different combinations of the highly organic palm oil industrial effluent, serum latex, cured glycerol and molasses for stimulating the growth of oleaginous yeast (*Yarrowia lipolytica)* [18]. Distillers and domestic mixed wastewater also used as an effective medium for co-culturing of microalgae and yeast for lipid accumulation [19]. Along with the lipid production, nutrients such as chemical oxygen demand (COD), total nitrogen, and total phosphate can be successfully removed from the distillery and domestic mixed wastewater by cultivating *Rhodosporidium toruloides* [20]. A symbiotic relationship exists between algae *Chlorella vulgaris* and wastewater bacteria when cultivated in municipal wastewater, which results in efficient nutrient removal along with biodiesel production [17,21]. Hence there is a need to identify other carbon sources that are rich in nutrients and available as industrial waste. These oleaginous microorganisms growth is very high, if the nutrients has more of cellulosic components. So, this approach will create a potential to generate energy from the waste.

Therefore, in this research study, the production of FAME from the oleaginous biomass grown from pulp and paper industry wastewater was investigated. The factors that affect the yield of FAME are systematically analyzed by conducting various experimental runs. Response surface methodology (RSM) is used to investigate the interaction between the independent process variables on the FAME yield and estimate the optimal conditions that can lead to maximum yield. Also, to capture the critical inherent characteristics of the esterification process and further predict the enhanced FAME yield at different process conditions, a data-driven adaptive neuro-fuzzy inference system (ANFIS) is implemented. The study demonstrates the exploitation of modeling techniques for identifying the optimal conditions as well as capturing the inherent characteristics for better yield predictions, which could minimize the time-consuming and energy-intensive scale-up experiments before preceding for real-time applications. The significant outcomes of this research study can lead to achieving sustainable biodiesel production.

2. Materials and methods

2.1. Wastewater collection

For this research study, the wastewater is collected from the Tamilnadu Newsprint and Papers Limited (TNPL) industry in the Karur District of Tamilnadu, India. This industry produces stationery paper and newsprint paper using sugarcane bagasse and hardwood. Generally, in most pulp and paper mills, the raw material sequentially undergoing a number of unit operations such as raw material preparation, pulp digestion, washing, bleaching and finally papermaking. Pulping and bleaching processes consume lots of freshwaters and release a considerable amount of contaminated wastewater. Generally, the characteristics of wastewater depend upon the process, and hence the wastewater collected from different sections will be highly varying.

Therefore, in this research study, the three varieties of high polluted samples were collected from this paper industry. These samples were termed as Bagasse seepage water (BSW), Bagasse wash water (BWW) and Hardwood wash water (HWW). BSW was collected from the bagasse storage yard; BWW was obtained from bagasse digestion, pulp washing and bleaching process and HWW were collected from hardwood digestion, pulp washing and bleaching process as shown in Fig. 1. All these samples from the plant were brought to the storage chamber within 7 h and were stored at −4 °C to prevent any microbial growth.

2.2. Pre-treatment and preparation of wastewater

The wastewater obtained from the industry (pulp and paper) contains suspended solids and floating matter, which cannot be directly used for culturing. In order to enhance the growth and lipid accumulation in oleaginous yeasts, all these extraneous materials should be filtered out. Using Whatman filter paper, all the suspended solids and floating matter in the wastewater was removed.

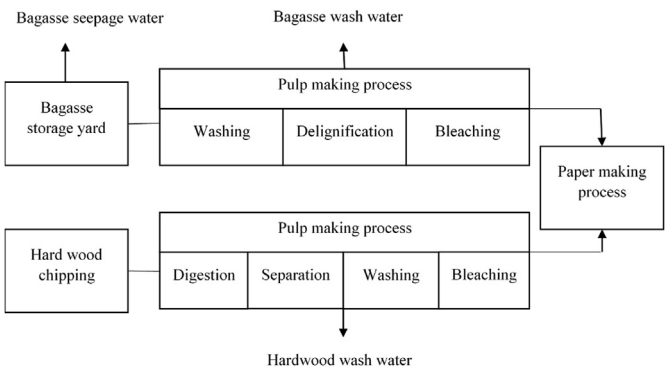

Fig. 1. Schematic diagram of the papermaking process and various sources of wastewater.

Before inoculation of a yeast strain in wastewater, sterilization of wastewater was done using autoclave at 121 °C with the pressure of 15 psi and allowed to react for 20 min to avoid the growth of other microorganisms present in the wastewater. The features of pulp and paper industry wastewater like colour, pH, total solids (TS), total suspended solids (TSS), total dissolved solids (TDS), dissolved oxygen (DO), chemical oxygen demand (COD) and glucose were analyzed according to the standard procedures [37].

2.3. Oleaginous yeast cultivation

Oleaginous organisms such as *Yarrowia lipolytica* (MTCC 789), *Metschnikowia pulcherrima* (MTCC 1678) and *Lipomyces starkeyi* (MTCC 1388) was obtained from Microbial type culture collection and gene bank (MTCC), Chandigarh, India in the form of freeze-dried culture. It was revived in 1.50 mL nutrient broth and kept in an orbital shaker at 28 °C for 24h. This seed culture was transferred to 100 mL nutrient media, making a 6% inoculum concentration. The subcultures were then used for inoculation into the wastewater for further studies. Three types of pre-treated wastewater samples are adjusted to different pH ranges as per the requirement of three different strains of oleaginous yeasts. *Y. lipolytica, M. pulcherrima* and *L. starkeyi* were grown in the pH of 4, 6–7 and 7.5–8 respectively. The yeast strains were inoculated into the 100 ml of filtered wastewater sample taken in Erlenmeyer flasks (250 ml) under laminar airflow chamber to prevent the entry of other microorganisms, and it was kept under the aerobic condition at 25 °C in a rotary shaker at 150 rpm. The purpose of the batch culture experiment was to observe cell dry weight production at a given temperature, pH, rpm and concentration of medium in 9 combinations of wastewater and determining the best values for further pilot-scale study.

2.4. Lipid extraction

After the growth of 6 days, inoculum added samples are centrifuged for 3h 30 min at 9000 rpm for 28 °C in a large volume centrifuge. The biomass was settled at the bottom of the centrifuge tube; which is extracted and washed twice with the distilled water. Then, cell wall disruption due to biomass is conducted via two approaches [22]. In the first method, biomass is mixed with 0.1N HCl in a mortar and pestle arrangement and homogenized for about 5 min to disrupt the cell. And in another method, biomass mixed with 0.1 N HCl by sending ultrasonic pulse wave in an ultrasonicator for about 30 min. Later both mixtures were dried in an oven at 40 °C until the HCl gets evaporated and the final weight of dried biomass is noted down.

This dry biomass was subjected to lipid extraction by means of modified Hara and Radin method [23] using hexane and isopropanol at the ratio of 5:3 and mixed using a magnetic stirrer at 400 rpm for over 2.5 h. Then the mixture was transferred to falcon tubes and centrifuged at 8000 rpm for 10 min in a large volume centrifuge. After centrifuging, the mixture was distributed as two layers. The top layer (lipid-rich) was pipetted and evaporated under mild heating in a thermomixer to obtain purified lipids.

2.5. Transesterification

A catalyst solution of 3% w/v of K_2CO_3 in methanol was initially prepared, which is later used for transesterification of lipids. It is then added to the desired amount of lipid in a screw cape conical flask. Then it is kept in a hot plate provided with a magnetic stirrer at 700 rpm for perfect mixing, for about 90 min at the desired temperature. Then the mixture was allowed to cool down to room temperature, and hexane is added at the ratio of 5:4 and magnetically stirred for about 30 min. An equal volume of distilled water was added to the mixture and centrifuged at 1000 rpm for 10 min. Slowly a layer separation was attained, and the top layer which is FAME is carefully extracted.

2.6. Design of experiments to verify the FAME yield through transesterification process

The production of FAME via transesterification process depends on the process variables (parameters) that dictate the performance of the process. Hence, to investigate the efficacy of FAME production and ascertain the interaction between the independent process variables on the production, bench experiments have to be conducted in a precise way. In this study, it was observed that the FAME production was found to be dependent on lipid dosage, catalyst concentration and reaction temperature. Depending on the magnitude of each variable, they may have a different influence on the production. Therefore, the experiments need to be carried for wide ranges for each process variable, to understand inherent characteristics of the transesterification process, as well as to ascertain the significance of each process variable on the overall process. Since, for wide ranges of each variable, there will be different permutations and combinations to conduct experiments. As a result, there is a need to plan a methodology and approach to design an experiment matrix that can minimize the number of tests and reduce the wastage of chemicals as well as minimize the human effort. With practical limitations and past experience, the experimental design range and coded levels of the process variables were chosen, as shown in Table 1. Response surface methodology (RSM) is a technique found to be a useful statistical technique for optimizing the complicated process. It produces an experimental design matrix, thus reducing the number of experiments which require evaluating multiple parameters and their interactions within [24–26]. This approach is less tedious and less time-consuming than other approaches [27–30].

For the given range of process variables, a systematic approach is followed, and an experimental matrix is designed using the Box-Behnken design (BBD) as shown in Table 2. This experimental design is obtained from the trial version of Design Expert 10. As seen in this table, 18 sets of experimental runs are conducted which represent different spatial distribution in a cubical representation. For validating the repeatability of the yield produced at various scenarios, each experiment was done in triplicates, and the average value was used for further analysis. Using RSM, the interaction between the independent process variables on the FAME yield and optimal conditions that can lead to maximum yield is investigated.

To predict the yield for different ranges of process variables, a multivariable data-driven model has to be identified. The response variable (FAME Yield, η_{FAME}) can be expressed as a function of the independent process variables lipid dosage (x_1), catalyst concentration (x_2), and reaction temperature (x_3),

$$\eta_{FAME} = f(x_1, x_2, x_3) \quad (1)$$

The function shown in **Eq. (1)** need to be estimated, which can be a quadratic or cubic or polynomial multiple regression expression.

Table 1
Experimental design range and coded levels of the process variables.

Variables (parameters)	Units	−1	0	1
Lipid dosage	g	2	2.5	3
Reaction Temperature	°C	60	70	80
Catalyst concentration	g/ml	0.01	0.02	0.03

Table 2
Box–Behnken experiment design matrix and corresponding FAME yield obtained from experiment.

Sl. No	A: Lipid dosage (g)	B: Temperature (°C)	C: Catalyst (g/ml)	FAME, (g/g)
1	2.5	70	0.02	1.20
2	2.5	80	0.03	0.90
3	2.5	70	0.02	1.15
4	2.5	80	0.01	0.79
5	2	80	0.02	0.70
6	3	70	0.03	0.67
7	2	60	0.02	0.65
8	2	70	0.01	0.54
9	2.5	70	0.02	1.15
10	3	60	0.02	0.57
11	2.5	70	0.02	1.10
12	2	70	0.03	0.68
13	2.5	70	0.02	1.10
14	3	80	0.02	0.61
15	2.5	60	0.03	0.78
16	3	70	0.01	0.55
17	2.5	60	0.01	0.83
18	2.5	70	0.02	1.20

2.7. An adaptive neuro-fuzzy inference system

Artificial neural networks (ANN) is a powerful tool mimicking the biological neural network to identify a reliable data-driven model for predicting the behaviour of any non-linear high dimensional process [31,32]. For nonlinear systems, to enhance the learning capabilities and capture the inherent characteristics, a fuzzy-based interference system will be a great tool. In this regard, to incorporate the features of both ANN and fuzzy logic principles and capture the benefits of both in a single framework, the well-known, adaptive neuro-fuzzy inference system (ANFIS) can be an efficient approach [33–35]. In this study, the ANFIS framework, which is based on the Takagi-Sugeno fuzzy interface system, is implemented. The ANFIS architecture is composed by five layers. The first layer takes the input values and determines the membership functions belonging to them. It is commonly called fuzzification layer. The membership degrees of each function are computed by using the premise parameter set. The second layer is responsible of generating the firing strengths for the rules. Due to its task, the second layer is denoted as "rule layer". The role of the third layer is to normalize the computed firing strengths, by dividing each value for the total firing strength. The fourth layer takes as input the normalized values and the consequence parameter set. The values returned by this layer are the defuzzificated ones and those values are passed to the last layer to return the final output [35,36].

3. Results and discussion

3.1. Biomass production

The growth of the oleaginous yeast depends upon the carbon source and nutrients present in the cultivation media and culture conditions. The prime characteristics of the wastewater collected from pulp and paper industry is shown in Table 3. In this study, yeast growth was determined in different wastewaters on a dry weight basis, under identical culture conditions and collected after six days of the cultivation period. A relevant criterion for the selection of yeast species is the high specific growth rate and high productivity. *Yarrowia lipolytica* strain cultured in the BWW shows the high biomass concentration of 32.361 g/l and biomass productivity of 5.395 g/l/d among the nine combinations of batch culture. Hence, *Yarrowia lipolytica* grown on BWW was taken for further pilot-scale study.

Table 3
Characteristics of different wastewater samples collected.

Parameters	Units	BSW	BWW	HWW
Colour	–	Dark brown colour	Brown colour	Yellow colour
pH	–	3.56–3.6	4–4.16	2.17–2.82
TS	mg/l	2220	1910	2850
TSS	mg/l	1258	984	281
TDS	mg/l	962	926	2569
DO	mg/l	3.3	3.4	3.4
COD	mg/l	5120	1920	1530
Glucose	mg/l	910	930	1050

BSW - Bagasse seepage water; BWW - Bagasse wash water; HWW - Hardwood wash water; TS – Total solids; TSS – Total suspended solids; TDS – Total dissolved solids; DO – Dissolved oxygen; COD – Chemical oxygen demand.

Biomass concentration and biomass productivity of nine batch culture were shown in Table 4. The pilot-scale study was meant for large volume cultivation of oleaginous organism. In this study, 5 L Bio-age autoclavable fermenter, which has the capability of maintaining constant pH, DO, and speed (RPM) was used. Aerobic condition is facilitated by means of an artificial air pump makes the oleaginous yeast to grow well as compared to batch study.

3.2. Lipid production

The lipid obtained from *Yarrowia lipolytica* using BWW by adopting mortar and pestle for cell wall disruption was found to be 0.34 g/g of the dry weight of biomass. Whereas, the lipid yield obtained by adopting ultrasonication for cell wall disruption was found to be 0.432 g/g of the dry weight of biomass. FTIR spectrum of lipid extracted from ultrasonication and transesterified biodiesel was shown in Fig. 2(a) and (b). FTIR for lipid confirms the presence of saturated fatty acids like butyl stearate and methyl palmitate, with a spectral match of 73.53% and 68.8%. The peak at 1746 cm^{-1} corresponds to the presence of the carbonyl group (-C═O) in lipids. The peaks at 2923 cm^{-1} and 2853 cm^{-1} indicates the presence of alkyl chains with carbon-carbon single and double bonds, implying the unsaturation of molecules. The peak at 3351 cm^{-1} is indicative of hydroxyl molecules (–OH). Therefore, the presence of long-chain fatty acids in the sample is confirmed, hence produced lipid can be converted into biodiesel.

In case of FTIR spectrum for biodiesel, the peak at 721 cm^{-1} corresponds to the presence of the long alkyl chains. The result shows there are no peaks in the range of 4000-3450 cm^{-1}, which corresponds to the presence of –OH bond. This designates that the hydroxyl molecules were replaced by ester during the transesterification reaction. The peaks at 2921.89 cm^{-1} (-CH_2), 2853.15 cm^{-1} (-CH_2), a strong peak at 1741.07 cm^{-1} (-C═O), 1462.59 cm^{-1} (-CH_2), 1361.21 cm^{-1} (-CH_3), 1169.52 cm^{-1} (C–O–C), 1015.72 cm^{-1} (C–O–C) and 722.14 cm^{-1} (-CH_2) are clearly present in the spectrum of FAME [38,39]. The library searches from FLUKA

Table 4
Biomass concentration and productivity.

Organism	Biomass concentration (g/l)			Biomass productivity (g/l/d)		
	BSW	BWW	HWW	BSW	BWW	HWW
Yarrowia lipolytica	9.642	32.361	27.862	1.607	5.394	4.643
Metschnikowia pulcherrima	1.918	3.819	5.769	0.319	0.636	0.961
Lipomyces starkeyi	6.162	8.956	16.441	1.027	1.493	2.740

BSW - Bagasse seepage water; BWW - Bagasse wash water; HWW - Hardwood wash water.

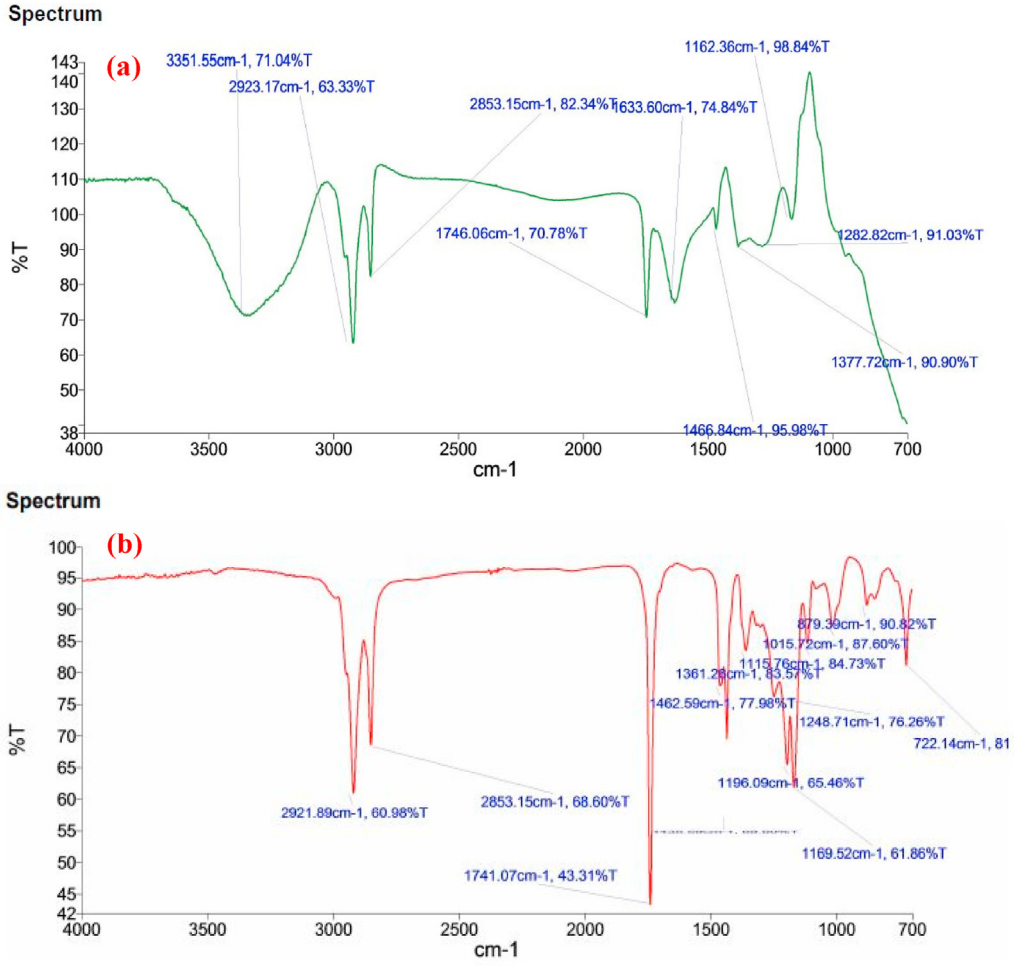

Fig. 2. FTIR spectrum for (a) lipid and (b) biodiesel.

yielded spectral match of 0.85 and 0.68 with long-chain unsaturated fatty acid esters like methyl linoleate and saturated methyl palmitate respectively. The depth of the peaks is a measure of the purity of the extracted samples.

3.3. Model development for mimicking the transesterification process for producing FAME

To investigate the performance of FAME production and ascertain the interaction between the three independent process variables (lipid dosage, catalyst concentration and reaction temperature) on the yield, a total of 18 experiments were conducted as indicated in Table 2. This experimental matrix is designed using the BBD approach in RSM. To predict the yield for different ranges of process variables, a multivariable data-driven model is identified using analysis of variance (ANOVA) approach. Results from the transesterification process yielding FAME are presented in Table 5. The statistical significance was tested by determining F and p-values. The model shows high significance with the F-value of 47.14 and p-value of <0.0001. P-values less than 0.05 indicate that those model terms are significant. In this case, X_3, X_1^2, X_2^2 and X_3^2 are significant model terms. The lack of fit (LOF) value of 1.44 implies that the LOF is not significant relative to the pure error, indicating that the model fit is good.

For the experimental data, it was found that the variations in the transesterification process can be best explained using a quadratic model, which is expressed as in Eq. (2).

$$\text{FAME} = -15.81250 + 7.3375 * X_1 + 0.20712 * X_2 + 48.0 * X_3 - 0.0005 * X_1 * X_2 - 1.0 * X_1 * X_3 + 0.4 * X_2 * X_3 - 1.465 * X_1^2 - 0.00151 * X_2^2 - 1737.5 * X_3^2 \quad (2)$$

The goodness of fit of the obtained quadratic expression (model) can be validated by calculating the coefficient of determination (R^2) by comparing the quadratic model predictions with experimental values. Interestingly, the R^2 value is found to be 0.9809, indicating the high degree of correlation between the predicted response and experimental values. As well, the difference between predicted and adjusted R^2 is less than 0.2, indicating that they are reasonably good agreement with each other.

3.4. Data-driven adaptive neuro-fuzzy inference system (ANFIS) outcomes

From the experimental analysis, it was observed that FAME yield highly depends on the process variables like lipid dosage, catalyst concentration and reaction temperature. As shown in Table 2, eighteen experiments were conducted as per the design of experiments (DOE) – BBD framework. To capture the inherent characteristics of the esterification process and better predict the yield at different process conditions, a data-driven ANFIS is implemented. This data-driven model is developed using the input/output

Table 5
Analysis of variance for the fitted quadratic polynomial model for optimization of FAME content.

Source	Sum of Squares	degrees of freedom	Mean Square	F-value	p-value	
Model	0.9877	9	0.1097	47.14	<0.0001	significant
Lipid (X_1)	0.0036	1	0.0036	1.55	0.2481	
Temperature (X_2)	0.0036	1	0.0036	1.55	0.2481	
Catalyst (X_3)	0.0128	1	0.0128	5.50	0.0471	
$X_1 * X_2$	0.0000	1	0.0000	0.0107	0.9200	
$X_1 * X_3$	0.0001	1	0.0001	0.0430	0.8410	
$X_2 * X_3$	0.0064	1	0.0064	2.75	0.1359	
X_1^2	0.5853	1	0.5853	251.42	<0.0001	significant
X_2^2	0.0998	1	0.0998	42.88	0.0002	significant
X_3^2	0.1317	1	0.1317	56.58	<0.0001	significant
Residual	0.0186	8	0.0023			
Lack of Fit	0.0086	3	0.0029	1.44	0.3365	not significant
Pure Error	0.0100	5	0.0020			

Table 6
Table showing different ANFIS architecture and their performance evaluated in terms statistical metrics (RMSE).

No.	No. of Membership Function	Function Type	Output Function	RMSE	
1	2	triMF	Constant	0.7881	0.7870
2			Linear	0.7078	0.7078
3		trapMF	Constant	0.6239	0.6200
4			Linear	0.5698	0.5657
5		gbellMF	Constant	0.6554	0.6534
6			Linear	0.5138	0.5130
7		gaussMF	Constant	0.7106	0.7085
8			Linear	0.4358	0.4323
9		gauss2MF	Constant	0.5754	0.5682
10			Linear	0.4763	0.4707
11		piMF	Constant	0.5324	0.5287
12			Linear	0.4537	0.4498
13		dsigMF	Constant	0.5261	0.5223
14			Linear	0.4727	0.4692
15		psigMF	Constant	0.5261	0.5223
16			Linear	0.4358	0.4323
17	3	triMF	Constant	0.1280	0.1277
18			Linear	0.1139	0.1139
19		trapMF	Constant	0.1441	0.1432
20			Linear	0.0928	0.0924
21		gbellMF	Constant	0.1328	0.1324
22			Linear	0.1163	0.1150
23		gaussMF	Constant	0.1335	0.1332
24			Linear	0.0922	0.0920
25		gauss2MF	Constant	0.1298	0.1294
26			Linear	0.0667	0.0667
27		piMF	Constant	0.1501	0.1495
28			Linear	0.0908	0.0903
29		dsigMF	Constant	0.1398	0.1388
30			Linear	0.0848	0.0840
31		psigMF	Constant	0.1398	0.1388
32			Linear	0.0848	0.0840
33	4	triMF	Constant	0.0352	0.0349
34			Linear	0.0346	0.0346
35		trapMF	Constant	0.0365	0.0364
36			Linear	0.0117	0.0114
37		gbellMF	Constant	0.0370	0.0369
38			Linear	0.0210	0.0210
39		**gaussMF**	Constant	0.0376	0.0376
40			**Linear**	**0.0107**	**0.0106**
41		gauss2MF	Constant	0.0337	0.0336
42			Linear	0.0113	0.0112
43		piMF	Constant	0.0373	0.0372
44			Linear	0.0117	0.0114
45		dsigMF	Constant	0.0365	0.0364
46			Linear	0.0221	0.0220
47		psigMF	Constant	0.0365	0.0364
48			Linear	0.0148	0.0239

membership functions via the Sugeno type fuzzy rule. In this framework, the membership functions are updated using the hybrid method (backpropagation + least squares estimation). The structure (rules) of the tuned ANFIS architecture with the membership rules and AND/OR logical connectors are shown in Fig. S1. The structure connects through different input membership functions, rules and output membership functions, to capture the inherent characteristics and thus providing a better predictable outcome.

The performance of ANFIS varies with chosen membership functions and the type of membership function. In ANFIS architecture, there are different types of fuzzy-based membership functions like Triangular, Trapezoidal, Bell, Gaussian, pi-shaped, Sigmoidal and Asymmetric functions [36,40]. To identify the best fuzzy-based membership function along with a number of membership functions, different numerical runs were conducted, as shown in Table 6. The performance of each run was verified in terms of root mean square error (RMSE) which is useful statistical metrics. From these numerical runs, it was observed that four Gaussian membership functions resulted in lower RMSE (indicated in bold values in Table 6) for a linear output function. Hence this topology is considered as the best or optimal network. Therefore, for further analysis in this research study, the optimal ANFIS topology (4 linear Gaussian membership function) is used.

3.5. Investigation of process parameters on FAME yield

It was identified that the yield of FAME depends on the three independent process variables (lipid dosage, catalyst concentration and reaction temperature), but the interaction within these variables should be investigated. One variable may have a positive impact on the response in the presence of another variable, whereas it may have a reverse effect in the presence of the third variable. In order to understand the process effectively, it is always good to investigate the individual impact on the response as well as the interaction between the process variables and their overall influence on the final response.

The effect of perturbation of each independent process variables on the FAME yield is shown in Fig. 3 (a). It can be observed that each independent variable has a different effect on the yield. For instance, perturbation of lipid dosage has a significant impact on the yield. It can be observed that, when the lipid concentration is increased from 2.0 g to 2.5 g, this has resulted in an increase in FAME yield from 0.805 g/g to 1.15 g/g. However, any further increase has a reverse effect. When the lipid concentration is further increased to 3.0 g, it has resulted in a drastic decrease in FAME yield to 0.765 g/g. Remarkably, temperature and catalyst concentration has exhibited a similar trend, but not so strong like lipid dosage. When the temperature is increased from 60 °C to 70 °C, the yield has risen from 0.98 g/g to 1.15 g/g, however, further increase in temperature to 80 °C, the yield has decreased to 1.02 g/g. Similarly, when the catalyst concentration is increased from 0.01 g/ml to 0.02 g/ml, the yield has increased from 0.94 g/g to 1.15 g/g, however,

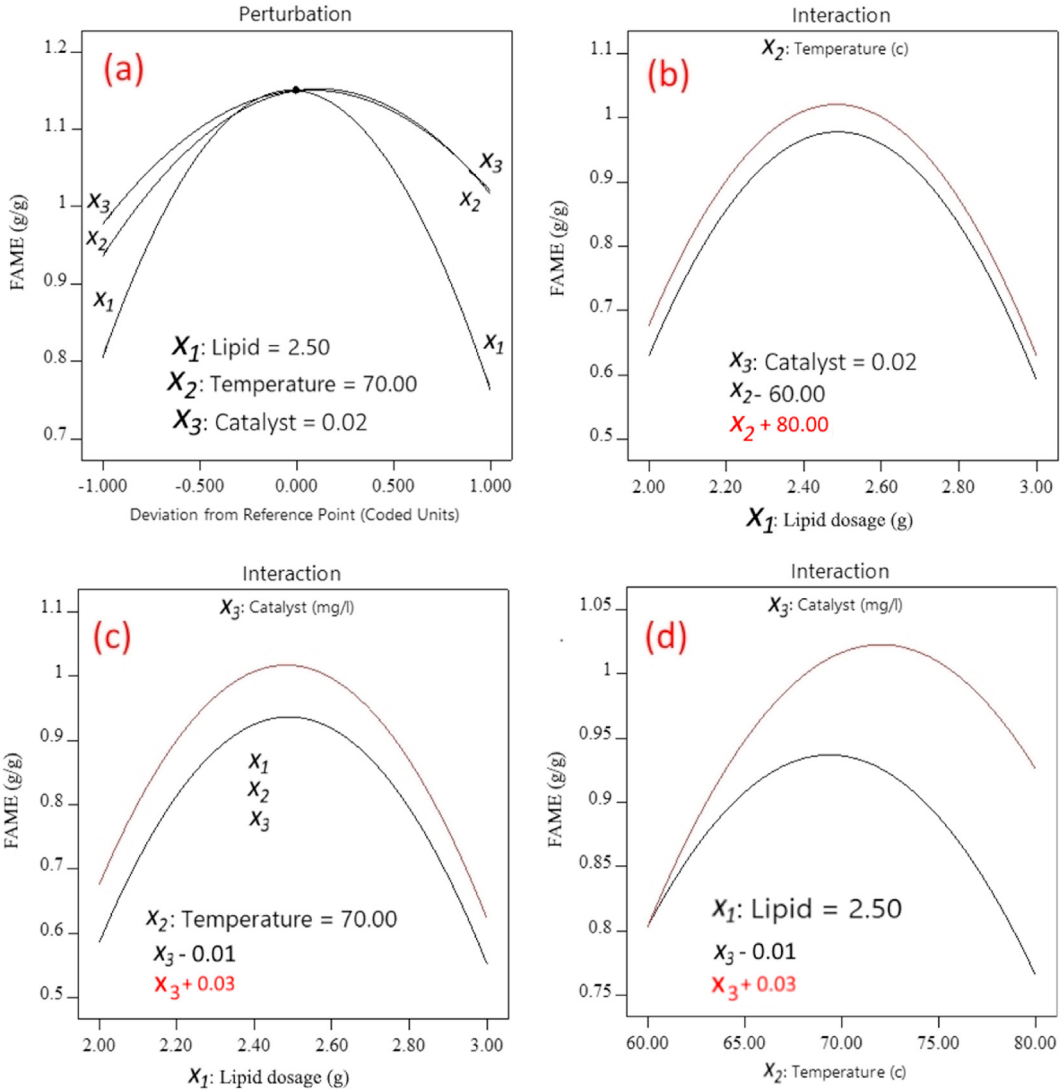

Fig. 3. (a) Effect of perturbation of each process variable on FAME yield; (b)–(d) Interaction effect of all the process variables on FAME yield.

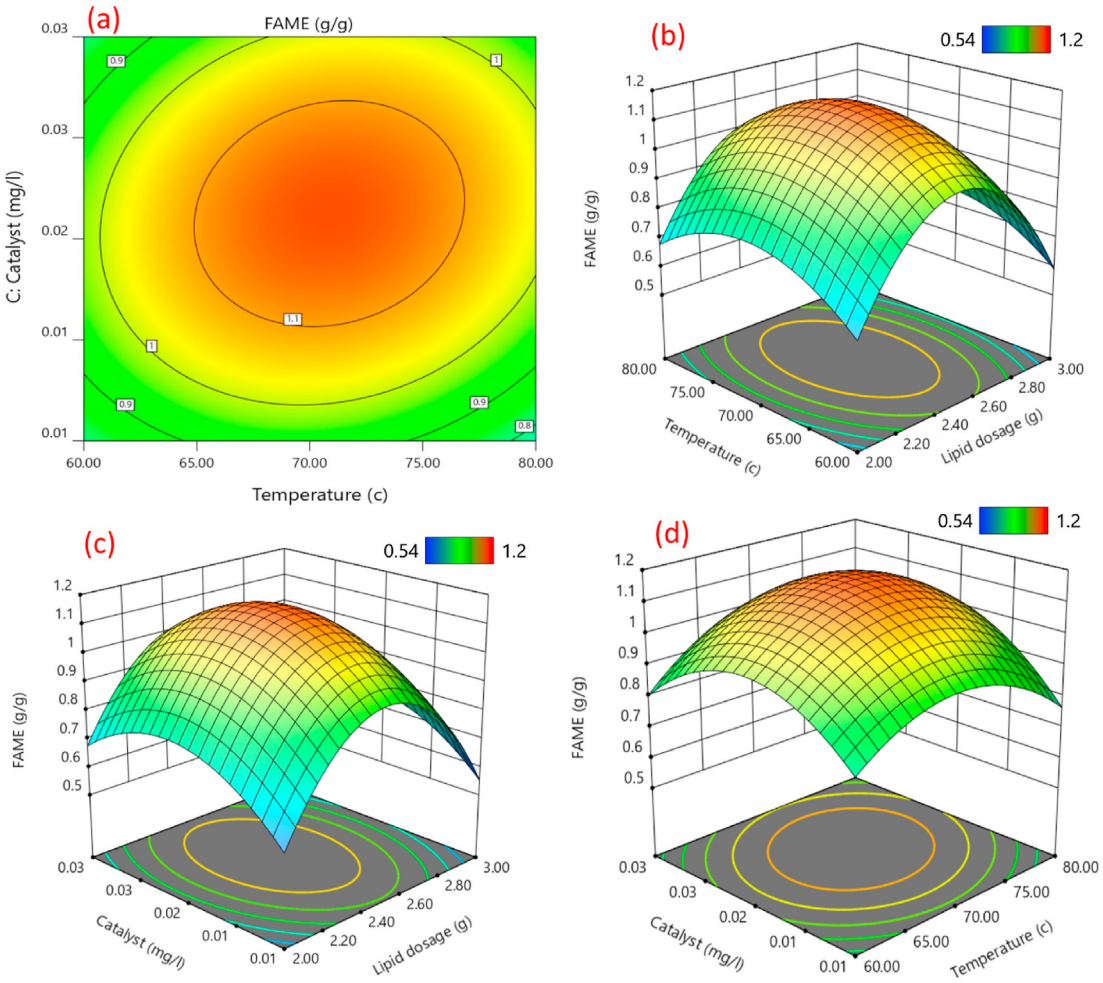

Fig. 4. Response surface curve for FAME production by employing alkaline transesterification at 90 min showing combined effects of variables. (a) contour plot; (b) lipid dosage and temperature (c) lipid dosage and catalyst (d) temperature and catalyst.

further increase in catalyst concentration to 0.03 g/ml, the yield has decreased to 1.016 g/g. These results further confirm that the perturbation of each process variable has a different effect on the overall yield.

The interaction effect of lipid dosage-temperature on FAME yield at constant catalyst concentration is shown in Fig. 3 (b). It can be seen that by increasing the temperature enhances the yield for any lipid dosage. This indicates that a higher temperature is preferable in this esterification process. Similarly, the interaction effect of lipid dosage-catalyst concentration on FAME yield at constant temperature is shown in Fig. 3 (c). This profile indicates that the higher catalyst concentration is preferable for higher yields. Whereas, the interaction effect of temperature-catalyst concentration on yield at lipid dosage relative to the methanol volume (see Fig. 3 (d)) shows a different trend. For a given lipid dosage, an increase in catalyst concentration has a drastic influence on the yield. These profiles indicate that there is a strong interaction between the independent process variables, thus have a significant effect on FAME yield.

To further investigate, the optimal values of independent process variables that can result in a higher yield, the contours and 3D response surface plots curves are reproduced, as shown in Fig. 4. The contours showing the yield for different catalyst concentration and temperatures (constant lipid dosage – 2.5 g) is shown in Fig. 4 (a). These contours provide a clear picture of yield variation on a global range. It can be noted that higher yield will be obtained for the values of catalyst and temperature in the range of 2.0–2.2 g/ml and 70–72 °C respectively.

Fig. 4 **(b)**–**(d)** illustrates, the 3-dimensional response surface plots for different independent process parameters with respect to FAME yield. Each plot was generated by varying two individual variables in their corresponding experimental ranges, while the third parameter was kept constant. It can be noticed that (see Fig. 4 (b)) the yield was lesser at lower temperature and lipid dosage. The maximum yield can be achieved at a lipid dosage of 2.4–2.6 g. Fig. 4 (c) & **(d),** elucidates that the maximum yield can be attained when the catalyst concentration is in the range of 1.8–2.2 g/ml. The yield increased with increasing lipid dosage up to a specific value and eventually decreased.

3.6. The predictive capability of RSM and ANFIS approaches

In order to validate and test the predictive capabilities of both the RSM and ANFIS approaches, the model predictions should be compared with the experimental values. The scatter plots are shown in Fig. 5 (*a*) & (*b*) illustrates the performance of the model predictions (RSM & ANFIS) when compared with the experimental values of FAME yield. It can be observed from Fig. 5 (*a*), that the RSM predictions are statistically good and hence show a noteworthy correlation between the actual and predicted values. This

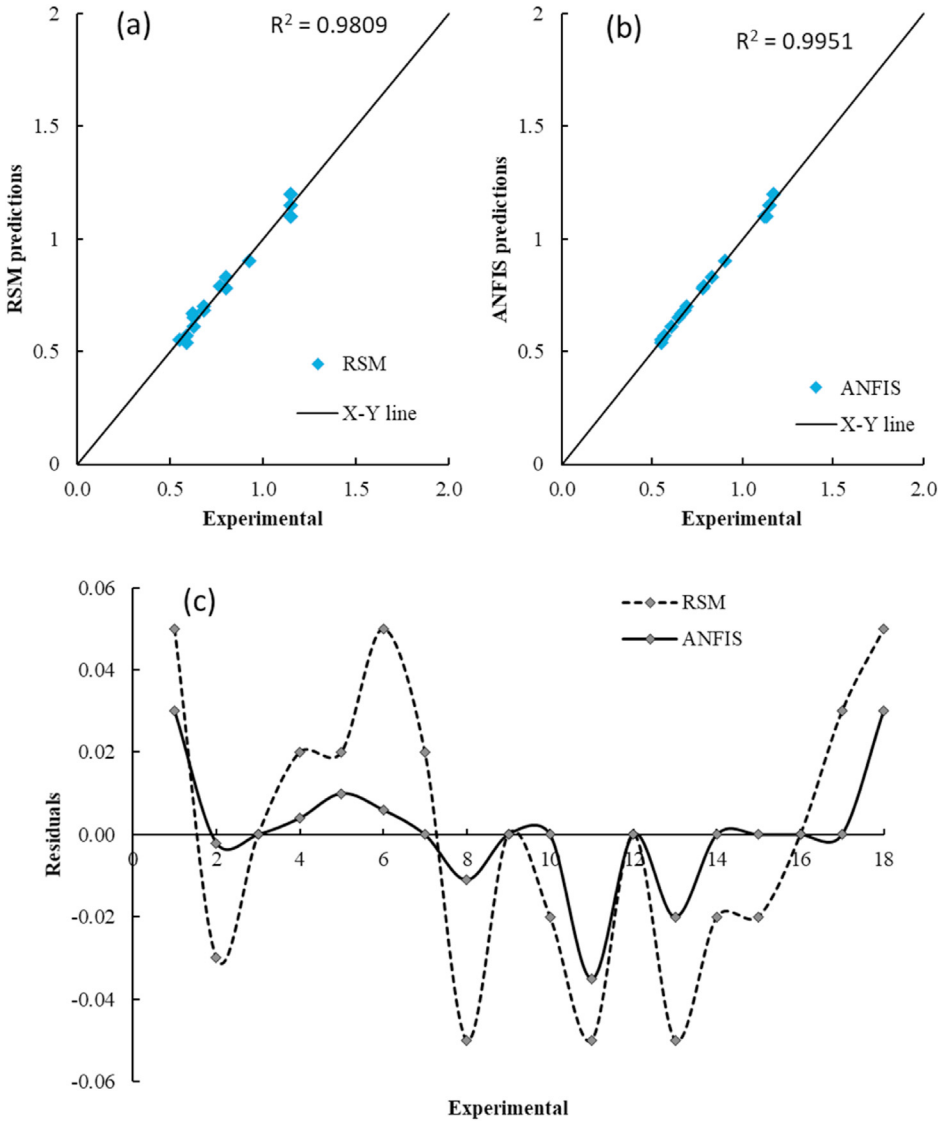

Fig. 5. (a) Response surface methodology predictions vs experimental values; (b) adaptive Neuro-fuzzy inference system predictions vs experimental values; (c) residuals of Response surface methodology & adaptive Neuro-fuzzy inference system model predictions.

has resulted in a coefficient of determination, R^2 of 0.9809. In comparison, the model predictions from ANFIS is shown in Fig. 5 (*b*). This scatter plot indicates that the model predictions are very close to the experimental values, thus resulted in higher R^2 of 0.9951. These data-driven model predictions are significantly good over RSM predictions, thus showcasing the superior performance of ANFIS. For a better understanding of deviations in model predictions against the experimental values and detailed comparison of both the RSM and ANFIS approaches, the residual plots are presented in Fig. 5 (*c*). It can be observed that the residuals of RSM predictions are ranging between [-0.05 0.05]; whereas the residuals of ANFIS predictions are ranging between [-0.035 0.030]. The optimal ANFIS architecture couldn't explain only less than 1% of total variations. Therefore, overall results indicate that the ANFIS approach better captures the esterification process dynamics and hence resulted in better model predictions.

3.7. Process optimization and model validation

Since each independent process variable significantly impacts the FAME yield obtained, it is essential to identify the optimal values of each process variables that can result in a higher yield. Also, few process variables like lipid dosage or catalyst may come with a cost, so optimal use of the variables not only a feasible solution for large scale industrial use but also provide the required yield. In order to estimate the optimum values of corresponding process parameters influencing the FAME yield, the desirability index can be an excellent statistical metric. The list of possible optimal solutions for obtaining higher FAME yields that result in high desirability index is shown in Table S1. These optimal values have more significance when the esterification process is used on a commercial scale, where the total expenses of the process operation depend on the cost of the catalyst and lipid dosage.

Based on the optimal solutions resulted with high desirability, maximum FAME yield (1.154 g/g) was obtained under optimized conditions of lipid dosage, temperature and catalyst concentrations at 2.50 g, 70.0 °C and 0.02 g/ml respectively. For the same optimal conditions, the ANFIS model is also tested and resulted in FAME yield of 1.149 g/g. To validate the optimal values obtained, the experiment was conducted for the same conditions and the FAME yield obtained were 1.148 g/g. It was observed that the experimental values of FAME yield were in good agreement with the

Table 7
Lipid yield obtained from different feed stock at different pH and reactor conditions.

Feedstock	Feedstock concentration (g/L)	pH	Reactor conditions	Lipid yield (g/L)	Ref
Acetic acid	12	5.6	Batch cultivation in a 500 mL bioreactor	1.84	[41]
	70	10	Batch cultivation in a 250 mL	10.11	[42]
	90	11	Erlenmeyer flask	7.9	[42]
	110	12		3.85	[42]
	30	8		5.24	[42]
	50	9		8.01	[42]
Butyric acid	12	5.6	Batch cultivation in a 500 mL bioreactor	0.85	[41]
Butyric acid	50	8	Batch cultivation in a 250 mL Erlenmeyer flask	7.22	[42]
Glucose-based media with olive mill wastewater	0–1.57	–	Batch cultivation in a 250 mL Erlenmeyer flask	0.2–1.7	[43]
Food waste fermentate	35.35	8	1.5 L Anaerobic digestion reactor	3.2	[42]
Fruit and vegetable waste fermentate	22.18			3.08	[42]
Glucose	80	5.6	Fed Batch cultivation in a 500 mL bioreactor	15.93	[41]
Glucose + acetic acid	Initially grown on glucose at 40 g/L followed by sequential additions of VFA at 1.5 g/L/h	5.6	Two stage fed batch cultivation in a 7 L bioreactor	12.36	[41]
Glucose + butyric acid				7.62	[41]
Glucose + propionic acid				14.84	[41]
Glucose + VFAs				16.5	[41]
Glycerol + acetic acid				15.73	[41]
Glycerol + butyric acid				11.19	[41]
Glycerol + propionic acid				14.29	[41]
Glycerol + VFAs				14.19	[41]
Glycerol	80	5.6	Batch cultivation in a 500 mL bioreactor	16.11	[41]
Glycerol with C/N ratio of 75	50	6	Batch cultivation in a 300 mL Erlenmeyer flask	2.31	[44]
Glycerol with C/N ratio of 100	50			2.06	[44]
Mixed VFAs (acetic: propionic: butyric acid = 5:2:3)	50	8	1.5 L Anaerobic digestion reactor	8.27	[42]
Nitrogen-limited glucose-based media with olive mill waste-waters	0–1.64	–	Batch cultivation in a 250 mL Erlenmeyer flask	0.2–1.9	[43]
Palm oil mill effluent	–	4.3	Batch cultivation in a 250 mL	1.25	[45]
	–	5	Erlenmeyer flask	1.46	[45]
	–	5.5		1.64	[45]
	–	6		0.82	[45]
Propionic acid	8	5.6	Batch cultivation in a 500 mL bioreactor	0.87	[41]
	50	8	1.5 L Anaerobic digestion reactor	4.48	[42]
Rapeseed oil	20	–	–	1.91	[46]
	30	–	–	3.15	[46]
	40	–	–	2.97	[46]
Waste cooking oil medium	100	–	–	5.97	[47]

ANFIS predicted values with a standard error of less than 2%, thus inferring the commandability of a data-driven model.

4. Comparison of relevant studies

4.1. Lipid production from various feedstocks

Cultivation of non-edible plants requires large area; these disadvantages can be overcome by the usage of oleaginous species, which grow and multiply themselves by consuming the nutrients and carbohydrates in the media composed of high carbon and low nitrogen content. Oleaginous organisms accumulate more than 20–70% of lipid by the mass fraction of biomass.

As the nitrogen gets exhausted, the oleaginous organism stops multiplying and convert the carbon into intercellular lipid. The lipid accumulation is carried out up to a specific limit until the cell reaches the limit of obesity. Production of FAME via transesterification process mainly depends on lipid dosage. Table 7 shows the Lipid yield obtained from different feedstock at different pH and reactor conditions. These studies indicate that pH influences the lipid yield and alkali medium preferred for higher lipid yield. It can be seen that lipid yield is varying from 0.2 to 16.5 g/L, depending on the feedstock and process variables (Feedstock concentration, pH) and reactor conditions. Glycerol as feedstock with 80 g/L of Feedstock concentration and pH of 5.6 in a bioreactor has produced highest yield of 16.11 g/L.

4.2. FAME obtained from different oleaginous microorganism grown on various carbon sources

For the growth of oleaginous microorganisms, there should be a medium that can continuously supply the required nutrients for the growth. This requirement should be met through a source which is available abundantly. A low-cost substrate derived from wastewater, food waste or agricultural waste having high organic content, will be a potential feedstock for culturing of oleaginous organisms. Various researchers have studied the production of FAME using different oleaginous microorganisms. Table 8 shows the FAME yield obtained from different oleaginous microorganism grown on various carbon sources. It can be seen that researchers have explored different carbon sources to grow the oleaginous microorganisms. From activated sludge to industrial crude glycerol,

Table 8
FAME yield obtained from different oleaginous microorganism grown on various carbon sources.

Species	Carbon source	Trans esterification method	Maximum FAME to lipid ratio	Ref
R. Toruloides	Distillery and domestic waste water	Acid catalyst	70%	[20]
N. liquefaciens	Predigested municipal waste activated sludge	Garbage lipase catalyst	97.13%	[48]
R. kratochvilovae	Cassias fistula L. fruit pulp	BF_3 methanol reagent	94.5%	[49]
Meyerozyma caribbica	Industrial crude glycerol	Two step trans esterification	90.20%	[50]
Rhodotorula mucilagina	Marine microalga chlorella salina oil	Biocatalyst	85.29%	[51]
Trichosporon oleaginous	Glycerol medium	Ultrasonication aided In-situ trans-esterification using alkali catalyst	92.1%	[52]
Yarrowia lipolitica	Palm oil mill effluent + serum latex + crude glycerol + molasses	Alkali catalyst	73%	[18]
	Palm oil mill effluent	Acid catalyst	94.99%	[53]
	Crude glycerol	Petroleum diesel co-solvent	97.2%	[54]
	Starch industrial waste	In-situ trans- esterification using acid catalyst	96.1%	[55]
	Bagasse wash water from paper industry	Alkali catalyst	96.43%	Current work

agricultural biomass to industrial waste, the maximum FAME to lipid ratio is varying from 70% to 97%. It can be seen that Yarrowia lipolytica was most commonly used species. Also, it can be noted that among the various oleaginous microorganisms, Yarrowia lipolytica is more efficient and produce a higher yield. Hence most researchers prefer this microorganism for FAME production. This table also presents the transesterification method that was used to produce FAME. It can be noted that the highest FAME yield produced using Bagasse wash water as the carbon source is very much comparable with the other reported studies.

5. Conclusion

The present study demonstrated the growth of oleaginous biomass (*Yarrowia lipolytica*) in bagasse wash water of pulp and paper industry as an excellent sustainable feedstock for the biodiesel production under optimized reaction conditions. FTIR analysis confirmed the prime biodiesel components such as methyl linoleate and methyl palmitate. The enhanced FAME yield (1.154) was accomplished by RSM optimization of the transesterification reaction conditions to 2.50 g, 70.0 °C and 0.02 g/ml of lipid dosage, temperature and catalyst concentrations respectively. Further, the developed multivariate regression model by ANFIS demonstrated the capturing of intrinsic characteristics of the transesterification process for the better prediction of FAME yield at varying process conditions. The significant outcomes of this research study could lead to achieve a sustainable biodiesel production through resource recovery while ensuring greener technologies for waste to wealth in pulp and paper industries.

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CRediT authorship contribution statement

Madhu Vasaki E: Data curation, Formal analysis, Investigation, Writing - original draft. **Rama Rao Karri:** Methodology, Writing - review & editing, Software, Visualization. **Gobinath Ravindran:** Supervision, Conceptualization, Project administration. **Balasubramanian Paramasivan:** Resources, Writing - review & editing.

Declaration of competing interest

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

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.renene.2020.12.038.

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