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Optimisation of Sapindus Mukorossi Drying Efficiency using Response Surface Methodology

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ABSTRACT

The Sapindus Mukorossi has to be pre-dried before it can be pyrolysed into bio-oil, biochar, and biogas. Traditionally, biomass is pre-dried for a full day at 105 degrees Celsius before pyrolysis. The optimum drying temperature, timing, and heating rate are essential for effective drying operation. The drying process parameters considered in the optimisation studies include temperature, duration and heating rate, and the optimisation was conducted using the response surface approach. Central Composite Design (CCD) was used to construct 20 experiment sets based on three factors (n = 3). The objective of each experiment was to observe weight reduction by applying a minimum load of 100g of Sapindus Mukorossi. The Raspberry Pi platform controlled a tailor-made 500-watt heater to adjust the temperature, rate of heating, and time. Pulse width modulation (PWM) was used to control the heating rate. The three factors and the results were generated in three-dimensional models and graphs, which finally helped to identify the best drying oven response. The optimisation method effectively resulted in a weight loss of 11.3685 g after 8 hours at a temperature of 200 °C and a heating rate of 14.15 °C/min. The CCD chooses the most efficient working parameters for the dryer to minimise operational costs. The design expert suggested a best-fitted correlation using the quadratic model with an R^2 coefficient of determination of 0.9369 and a standard deviation of 0.7762.

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1. Introduction

The world is becoming increasingly dependent on fossil-fuel-driven engines due to different technological breakthroughs. The demand for fossil fuels is significant, leading to a gradual supply reduction. Since the global oil crises of the 1970s, which caused a shortage of fossil fuels, there has been a lot of focus on producing renewable fuels [1].

Bio-oil, which is produced from biomass, has the potential to replace fossil fuels as the main fuel source [2]. This fuel has a significantly lower environmental impact compared to fossil fuels. Lignocellulosic biofuels, in particular, have a significantly reduced carbon footprint during both their production and combustion phases [3]. Biomass is organic matter sourced from living or recently deceased beings, mainly plants and animals.

In biofuel generation, "biomass" commonly denotes lignocellulosic biomass, encompassing plants and trees' inedible, fibrous, and structural components. Biomass contains various chemical ingredients and moisture when taken from the environment or as a residue of daily living. Three primary thermal treatment procedures can transform lignocellulosic biomass into bio-oil, biogas, and biochar. The most prevalent technologies for converting biomass into these three bioproducts are pyrolysis, gasification, and microwave treatment [4-6]. Water in biomass has a significant influence on the output yields [7-9]. As a result, thermal pretreatment is necessary to prepare the feedstock. The three most significant factors for pretreatment are temperature, residence time, and heating rate [10,11]. Temperature substantially impacts the disruption of internal hydrogen bonds and the enhancement of biomass porosity, facilitating the breakdown of its complex structure. The time spent in residence is a key feature. Extended periods of residency support enhanced fragmentation and dissolution of the biomass constituents. Accelerated heating rates can cause the breakdown of the biomass structure and improve the porosity, resulting in enhanced accessibility for subsequent hydrolysis or conversion processes. The moisture level of the biomass or water affects the pyrolysis rate because higher heat must be applied to vaporise the water, and additional cooling loads must be applied to return the gas to a liquid state.

Additionally, moisture significantly affects the bio-oil's density, pH, and viscosity. It is important to keep the biomass's moisture content below 10 wt% during pyrolysis to produce a higher-quality bio-oil. Drying before primary pyrolysis is crucial to maintaining its moisture. Biomass can continue to absorb moisture from the environment even after it has been dried. To preserve the quality of the bio-oil, one of the most important procedures before the pyrolysis process is drying. Some oven-drying processes require a temperature of less than 60°C and a minimum of four days before pyrolysis [12]. Despite this, moisture extraction will eventually reach its threshold over time, and it may be regarded as a decrease in weight. If the system operates for an extended period, it consumes excessive electricity, which is far from economical. The conventional drying process could take up to 24 hours at 103±5 °C until a constant weight is obtained [13,14]. Although the 24-hour drying method is widely employed, there is a scarcity of research that concentrates on optimising the process to enhance efficiency. In particular, there is a lack of thorough research examining the effects of operational temperature, heating rate, and drying time. Response Surface Methodology (RSM) has been widely used by current investigators to determine optimal parameters across several study fields, particularly in materials science and engineering [15-17]. RSM is a statistical and mathematical methodology that facilitates the development, improvement, and optimisation of processes by analysing the output of experiments based on a limited number of variables. This research aims to fill the gap by studying the optimisation of the drying process from the wet basic feedstock.

This research aims to accelerate the drying process by determining the optimal drying process for wet basic feedstock based on the heating rate, duration, and temperature. A comparison was made between the conventional drying process, which takes 24 hours, and the current optimisation method. Soap nut, a type of biomass, was chosen as one of the alternatives for the experiment. The soap-nuts (Sapindus Mukorossi) are non-edible fruits that grow in forests. The soap nut is a berry that grows up to 4000 feet in the Himalayan foothills and mid-hills. Sapindus mukorossi is a significant member of the Sapindaceae family. It is primarily found in the hilly region of Garhwal Himalaya in India [18].

2. Methodology

Figure 1 illustrates a flow chart of the overall process. In the first stage, the sorted soap nuts were processed further by cracking the seed's outer layer to uncover the inner kernel. The remaining unused soap nut was submitted to a crushing procedure. Approximately 100 grams of crushed soap seed feedstock was used for each experimental run. An experimental matrix was built using Stat-Ease Inc., Minneapolis, MN, USA, Software Design Expert version 10.0 to generate parameters. By applying the principles of design of experiments (DOE), central composite design (CCD) in Response Surface Methodology was used to generate a set of 20 distinct experimental runs from the three (3) parameters [19-21]. The DOE methodology systematically adjusts the process parameters to align with the design parameters. This approach minimises the resources required for experimental studies, such as tests, materials, time, and manpower, while enabling precise prediction of key outcomes and their interactions and examining numerous parameters [22]. The experimental data was examined to establish the best values for each process parameter. The purpose was to determine the parameter values that would give the best-desired output.

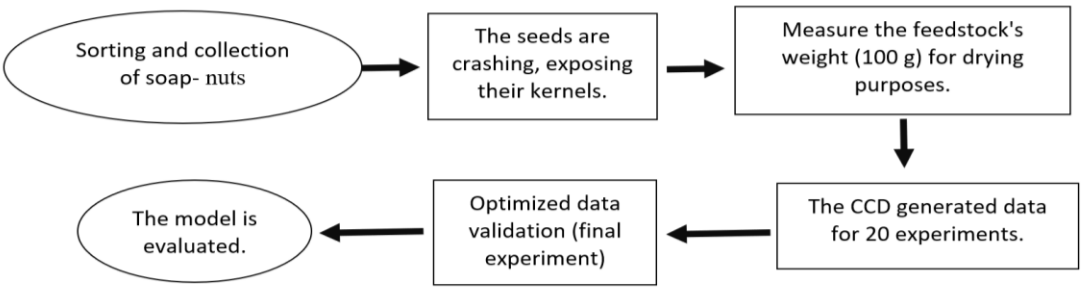


Fig. 1. Flow chart of the entire procedure

2.1 Materials and Methods

Figure 2 shows a soap-nuts for experiments. Only the seeds and kernel of the soap-nuts were utilised as biomass, as both the seeds and kernel were regarded as waste material. The nut needs to be cracked. The soap-nuts seed has a hard exterior. A moist, yellow kernel is embedded in the solid surface. The soap-nuts need to be fractured for maximum moisture removal. The seed kernel has the highest water content of any part of the seed. Once exposed, place 100 grams of wet basic crashed seed in the oven to remove moisture.

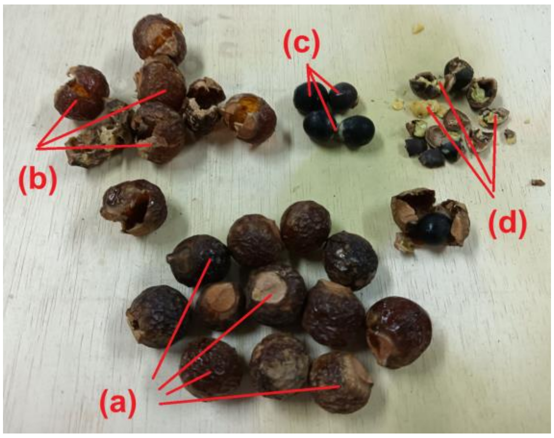


Fig. 2. (a) Soap-nuts (Sapindus Mukorossi), (b) skin for soap extraction, (c) seeds, (d) kernel

2.2 Determination of Alpha

The alpha (α) value enables simultaneous rotation and orthogonality [23]. To ensure the capacity to rotate, the alpha value is determined by the number of experimental runs in the factorial component of the central composite design. The alpha value can be determined using Eq. (1). The computed alpha value is 1.68:

$$\alpha = [2^k]^{1/4} \tag{1}$$

where α is the value to be keyed into design expert, and k is the number of parameters or factors.

2.3 Evaluation of Orthogonally Blocked CCD

Exactly 20 trials will be carried out to meet the criteria of 20 sets to obtain the coefficients of a second-order polynomial regression model for three variables. The calculation was performed using the equation provided below:

$$N = 2^n + 2n + n_c = 2^3 + 2(3) + 6 = 20 \tag{2}$$

where n is the number of parameters, and n_c represents the number of experiments that must be repeated. The study examined five levels for each variable, namely +α, -1, 0, 1, and –α.

2.4 Experimental Procedures

Table 1 displays CCD-based experimental details for 20 experiments used to examine the practical response and determine how the independent variables affect the moisture content. To identify the most optimal conditions, the experiments examined three process variables that could be influenced by each other: the duration of heating time (measured in hours), the temperature (measured in degrees Celsius), and the heating rate (measured in degrees Celsius per minute) [21]. A Raspberry Pi microcontroller was used to observe the experiment. The Raspberry Pi monitored the duration, temperature, and heating rate. The microcontroller's PWM (pulse width modulation) function controlled the heating rate. A total of 20 experiments were carried out to provide weight loss data readings. The experiments were accomplished once all of the predefined conditions had been met.

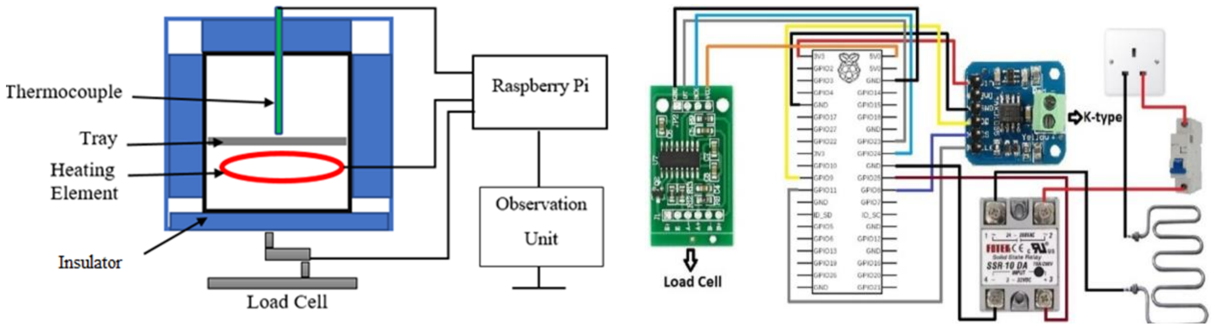
2.5 Schematic Diagram of the Hardware

Figure 3 provides a comprehensive overview of the heater. The schematic diagram below depicts only the project's essential features. The main components were the Raspberry Pi, monitor (observation unit), heating element, thermocouple, insulation, and load cell. The Raspberry Pi tracked the timing in relation to the input preset time. The microcontroller's PWM controlled the heating rate (temperature increment over time). The tested PWM responses ranged between 30 and 100 and were timed. PWM 30% can achieve a heating rate of 1.7034 °C/min.

In contrast, PWM 100% generates 14.712 °C/min. The microcontroller's data logger collected the weight reading every five (5) seconds, and the data was recorded as a CSV (Comma-Separated Values) file. The weight was collected by the load cell.

Table 1
The experimental parameters generated via CCD for 20 runs

Run	Factor 1 A: Time(hrs)	Factor 2 B: Temperature (°C)	Factor 3 C: Heating rate(°C/min)
1	6.5	234	11
2	10	200	7
3	6.5	150	11
4	6.5	150	11
5	6.5	150	11
6	6.5	66	11
7	0.5	150	11
8	10	200	15
9	3	100	15
10	10	100	7
11	6.5	150	18
12	3	200	7
13	6.5	150	11
14	6.5	150	11
15	3	200	15
16	10	100	15
17	6.5	150	4
18	3	100	7
19	6.5	150	11
20	12	150	11



(a) (b)

Fig. 3. (a) Schematic diagram of pretreatment dryer unit, (b) Electrical diagram of pretreatment dryer

3. Results and Analysis

Three independent variables—time (A), temperature (B), and heating rate (C)—were employed in this experiment to calculate the ultimate weight loss of the biomass (soap nuts). Design Expert software produced 20 sets of experimental parameters based on the three factors and the behaviour of the hardware component. Actual experiments were carried out in accordance with this 20-experiment data mapping. The CCD program responds to the weight loss of the biomass (soap nuts), which is the main goal of the experiment. Maximum weight loss implies the highest moisture removal impact on the biomass (soap nuts).

3.1 Central Composite Design (CCD) and Model Analysis

The centre values of the single-factor experiments were used to apply CCD in the optimisation studies. The experimental design consisted of three variables and five stages, as indicated in Table 2.

Table 2
Coded and actual levels of an independent variable

Factors /Parameters	Symbol	-1	0	1	$-\alpha$	$+\alpha$
Drying time (hours)	A	3	6.5	10	0.5	12
Temperature (°C)	B	100	150	200	66	234
Heating rate ((°C/min)	C	7	11	15	4	18

The ANOVA details for the model are provided in Table 3. The model's F-value is 16.50, indicating that the model is statistically significant. The probability of an F-value of this magnitude occurring only due to noise is only 0.01%. P-value, which falls below 0.0500, implies that the model terms are statistically significant. Therefore, the relevant model terms are A, B, A^2, and B^2. Values exceeding 0.1000 imply that the model terms are not statistically significant. If there exist many inconsequential model terms (except those necessary to enable hierarchy), model reduction may enhance the model. With a lack of fit F-value of 4.29, there is a 6.80% probability that this magnitude could occur due to random variation.

Table 3
ANOVA result for quadratic regression model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	89.48	9	9.94	16.50	< 0.0001	significant
A-Time	19.21	1	19.21	32.22	0.0002	significant
B-Temperature	55.6	1	55.6	102.71	< 0.0001	significant
C-Heating Rate	5.471E-06	1	5.471E-06	9.082E-06	0.9977	
AB	1.12	1	1.12	1.85	0.2031	
AC	0.0903	1	0.0903	0.1499	0.7067	
BC	0.0630	1	0.0630	0.1046	0.7531	
A^2	8.67	1	8.67	14.39	0.0035	significant
B^2	3.99	1	3.99	6.63	0.0277	significant
C^2	0.7757	1	0.7757	1.29	0.2830	
Residual	6.02	10	0.6025			
Lack of Fit	4.89	5	0.9770	4.29	0.0680	not significant
Pure Error	1.14	5	0.2279			
Cor Total	95.50	19				

The coded and actual equation which best fits the experimental data can be expressed as Eq. (3) and Eq. (4), respectively, upon the removal of the insignificant terms:

$$Y = 9.63 + 1.14\mathrm{A} + 2.02\mathrm{B} - 0.8374\mathrm{A}^2 - 0.5516\mathrm{B}^2 \quad (3)$$

$$Y = -6.40061 + 1.21548\mathrm{Time} + 0.106563\mathrm{Temp} - 0.068359\mathrm{Time}^2 - 0.000221\mathrm{Temp}^2 \quad (4)$$

3.2 CCD Result and Actual Result Comparison

Table 4 clearly shows that the error ratio is small. The actual weight loss from the lab test and the expected weight loss from the CCD software are nearly identical. The average value of the actual weight loss is 8.5665 g, whereas the anticipated weight loss is 8.5675 g. The experimental data has been examined and computed using the liner model by the Design Expert program.

Table 4
Actual and CCD generated result

Run	Factor 1 A: Time (hrs)	Factor 2 B: Temperature (°C)	Factor 3 C: Heating rate (°C/min)	Response 1 Weight Loss (gram)	
				Actual	Prediction
1	6.5	234	11	11.32	11.96
2	10	200	7	10.5	11.85
3	6.5	150	11	8.56	8.57
4	6.5	150	11	9.5	8.57
5	6.5	150	11	9.23	8.57
6	6.5	66	11	4.36	5.17
7	0.5	150	11	3.97	6.22
8	10	200	15	10.92	12.11
9	3	100	15	5.18	5.29
10	10	100	7	7.36	7.81
11	6.5	150	18	9.83	8.79
12	3	200	7	9.97	9.06
13	6.5	150	11	9.6	8.57
14	6.5	150	11	9.77	8.57
15	3	200	15	10.17	9.32
16	10	100	15	7.63	8.08
17	6.5	150	4	10.14	8.34
18	3	100	7	5.54	5.02
19	6.5	150	11	9.87	8.57
20	12	150	11	10.3	10.91

3.3 Model Summary

It is evident from the results that the maximum-to-minimum ratio is 2.85139. Manual transformation must be chosen from the "Transformation" option if the ratio exceeds 10. None of the transformations are required for the analysis because the ratio is less than 10. The design expert software suggests a quadratic model in accordance with the analysis. From Table 5, the quadratic model is the best-fitted model, with the R^2 and adjusted R^2 values as 0.9433 and 0.8923, respectively. The equation accurately represents the experimental data if the R^2 value is closer to 1. R^2 is called the "regression value" of the system.

Table 5
Model summary statistics and fit summary generated by Design Expert

	Model summary statistics				Fit Summary			
Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	Sequential p-value	Lack of Fit p-value	Adjusted R^2	
Linear	1.13	0.7864	0.7463	0.6367	< 0.0001	0.0178	0.7463	
2FI	1.21	0.7997	0.7072	0.4831	0.8334	0.011	0.7072	
Quadratic	0.7357	0.9433	0.8923	0.6518	0.0043	0.0866	0.8923	Suggested
Cubic	0.4774	0.9881	0.9547		0.0866			Aliased

3.4 Model Validation

The optimal parameters must lie within the maximum condition set by the CCD method. The optimal result was determined based on the 20 experimental values and the machine's behaviour. The RSM analysis began with a fixed range for the three factors. The range of the factors was fixed based on the machine's physical behaviour. Therefore, the CCD preset factors were set in range. The CCD software analysed 20 experimental values and generated the most precise optimum value. In optimum conditions, the time is 8.217 hours, the temperature 199.553 °C (~200°C), and the heating rate is 14.1565 °C/min (~15 °C/min) accordingly (see Table 6). The CCD evaluation predicts that the average weight loss in optimal circumstances will be 11.875 g. Table 7 uses expected standard error (SE) to assess the accuracy of the estimated mean response. The 95% confidence interval (95% CI) represents the predicted range of the experimental mean value. Based on this value, the actual optimum value will be generated. The 95% confidence interval is between 10.3774 and 12.359. The 95% PI value ranges from 9.37503 to 13.3614. These intervals show the acceptable ranges for weight loss calculated using the optimum parameters. Table 7 demonstrates that the actual weight loss is 11.09 g. The percentage error of the drying process from the fit value can be calculated via Eq. (5).

$$percentage\ error\ = \frac{Actual\ value - Fit\ value}{Fit\ value} x\ 100\% \quad (5)$$

Table 6
Model summary statistics optimum value

Factors	Optimum value	Desirability
A (Time)	8 hours	
B (Temperature)	200°C	100 %
C (heating rate)	15°C/min	

Table 7
Model validation

Response	Fit	SE Fit	95% CI low	95% CI high	95% PI low	95% PI high	Actual
Weight loss(g)	11.3682	0.894543	10.3774	12.359	9.37503	13.3614	11.09

The soap nut's weight loss was acceptable if it fell between the 95% PI and 95% CI limits. The result indicates that the developed model successfully predicted the response. The effects of time, temperature, and heating rate on the response variable are shown in Table 8. The optimal parameters for the procedure are as follows: Time: 8.22, Temperature: 199.55°C, and Heating Rate: 14.16°C/min. These settings are anticipated to yield the most favourable outcome within the range of the response variable.

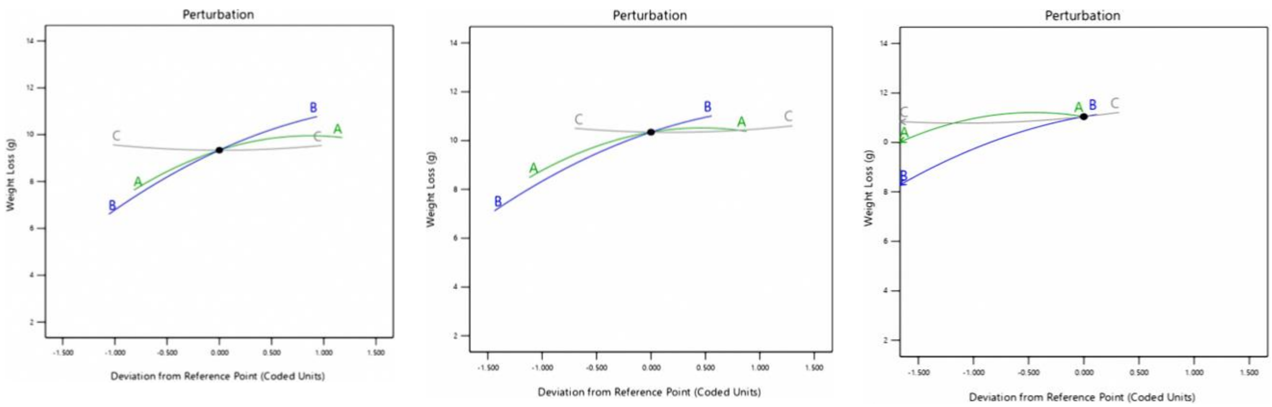
3.5 Analysis of CCD Response

Figure 4(a) shows the middle point of actual weight loss in the optimum scenario. In this situation, the drying process takes 6.5 hours when the reaction temperature and heating rate are 150°C and 11°C/min, respectively. The cost of the drying process increases with longer reaction times. Therefore, a drying time of 6.5 hours, which generates a maximum weight loss of 9.8 g, is the most economical option. On the other hand, when the heating rate decreases, the drying time is slightly longer. The weight loss of the biomass increases under these circumstances with the cost of the process's timing. Even though the heating rate decreased, the cost increased due to a longer reaction time. Here, the observed weight loss of 10.2 g is trivial compared to the expenditure. Figure 4(c) shows the highest rise in time, temperature, and heating rate. In this circumstance, there has been approximately a 10.5 g weight reduction.

For this experiment, the reaction time, temperature, and heating rate were all adjusted to the upper limits: the highest temperature of 200°C, the reaction time of 10 hours, and the maximum heating rate of 15 °C/min. The difference in weight loss under the maximum operating conditions compared with the optimal situation is 0.7 g. Timing in the optimum condition is only 6.5 hours instead of the maximum timing of 10 hours. Moreover, the maximum and optimal temperature is 200 °C and 150 °C, respectively. It is clear that there is a 4°C difference in the heating rate as well. This comparison provides a good picture of the optimum weight loss and weight loss under various circumstances. As of now, the cost of drying is anticipated to increase almost fourfold, while the weight loss is negligible.

Table 8
Final optimum result summary generated by design expert

Factor	Name	Level (Optimum)	Low Level	High Level	Std. Dev.	Coding
A	Time	8.22	3.00	10.00	0.0000	Actual
B	Temperature	199.55	100.00	200.00	0.0000	Actual
C	Heating Rate	14.16	7.00	15.00	0.0000	Actual



(a) (b) (c)

Fig. 4. (a) Actual weight loss by CCD, (b) Tuning three factors in different conditions (CCD), (c) Maximised the three factors (CCD)

The inspection of the 3D graphs (Figure 5(a) and Figure 5(b)) shows that the heating rate does not play a significant role in offsetting the weight loss ratio. Weight loss was around 10.5 g when the heating rate was 7 °C/min. When the heating rate was increased to 15 °C/min, the weight loss was roughly 10.7 g. In this situation, weight reduction is incredibly negligible. The heating rate is called the total heat increment over time. In this study, pretreatment, a biomass drying process conducted in the presence of oxygen, has a negligible impact on the biomass. Removing the moisture from the biomass (soap nuts) entirely requires a certain amount of time. The 3D analysis's effect on the heating rate can be shown by maintaining a constant heating rate while adjusting the other two factors (time and temperature). According to Figure 5(a), if the biomass (soap nuts) is dried at 140 °C for 10 hours at 7° C/min, the weight loss is approximately 8 g. Figure 5(b) shows that even after drying the biomass (soap nuts) at 140 °C for 10 hours at 15° C/min, the weight reduction is somewhat less than 8 g. Therefore, the weight does not change significantly even as the heating rate is raised.

It is clear from Figure 5(c) and Figure 5(d) that temperature change causes considerable amounts of weight loss. Increasing the temperature results in significant weight loss when the heating rate and time are both constant. At a temperature of 100 °C, approximately 8.9 g of weight is lost. However, at a temperature of 200 °C, a weight loss of at least 11.5 g can occur. Although the temperature increase suggests a notable weight reduction, this scenario undermines cost-effectiveness. Figure 5(c) and Figure 5(d) demonstrate a simple shifting of the 3D surface from down to up, indicating greater weight loss due to the temperature change.

This graph shows the biomass's weight loss over time. In this case, it was assumed that temperature and heating rate were constants. At the 3-hour reaction time, the weight reduction was approximately 8.9 g. Conversely, at a reaction time of 10 hours, it was observed that there was a weight loss of 11.8 g. The red surface of the 3D graph indicates the maximum and optimum consequences of the experiment.

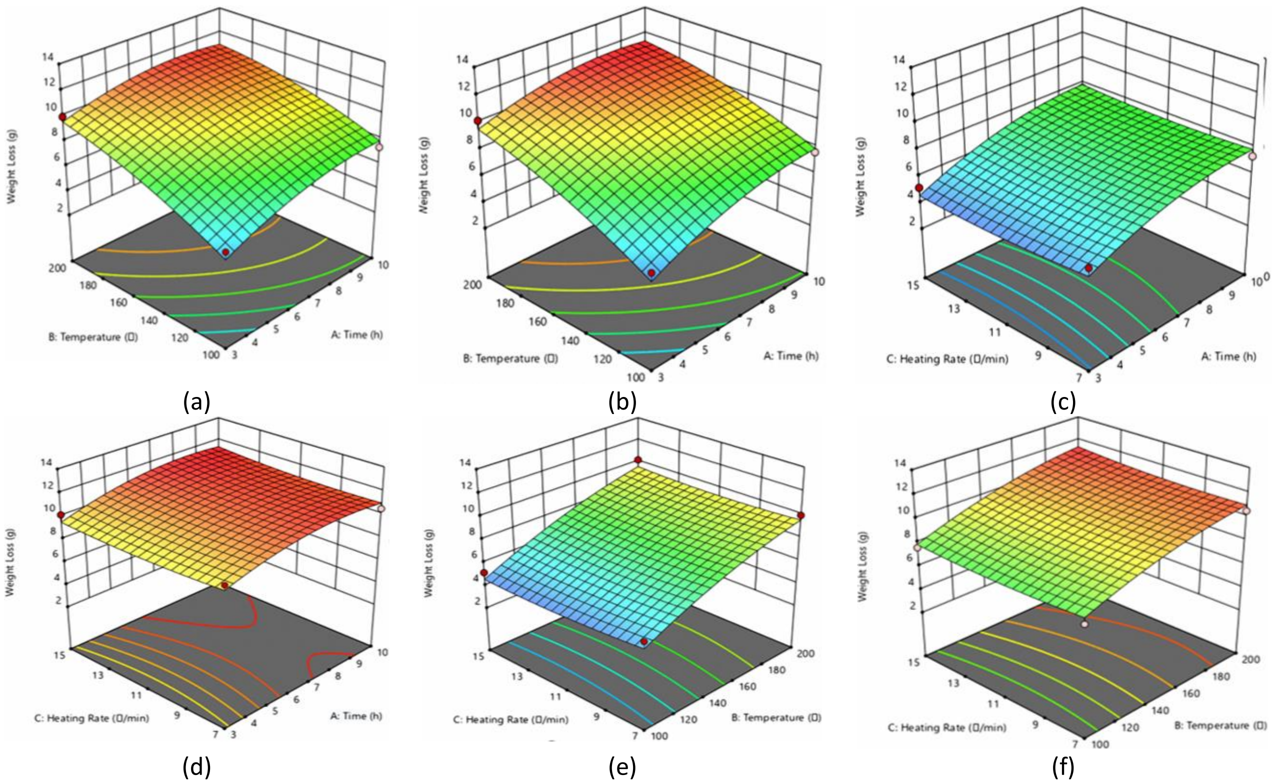


Fig. 5. 3D graphs. (a) Heating rate 7° C/min, (b) Heating rate 15° C/min (CCD), (c) Temperature 100°C, (d) Temperature 200°C, (e) Time on 3 hours, (f) Time on 10 hours

4. Conclusion

The current study's objective was to investigate potential sources of soap nut oil as a bio-oil feedstock. The moisture content of the feedstock should be below 10% to ensure a high-quality yield. Otherwise, it will just produce water and sludge. Long drying times can raise costs and increase the risk of biomass burning. The experiment examined the impacts of time, temperature, and heating rate on the dependent variable. The analysis produced the following conclusions:

i. Main Effects of Time (A): The main effect of time is statistically significant, with a p-value less than 0.05. The reaction directly correlates with time, progressively increasing from 3.00 to 10.00 hours. The peak response is observed at 8.22, indicating that the process is affected by the passage of time, and longer durations lead to more advantageous outcomes.
ii. Main Effects of Temperature (B): The effect of temperature is highly significant, with a p-value below 0.01. The reaction directly correlates with temperature, increasing consistently from 100.00 to 200.00. The maximum response is observed at a temperature of 199.55, indicating the ideal condition. Therefore, temperature significantly affects the process, and a temperature of 199.55°C yields the best outcomes.
iii. Main Effects of Heating Rate (C): The statistical analysis shows that the heating rate has a significant impact, with a p-value of less than 0.05. The reaction is directly related to the increase in heating rate, ranging from 7.00 to 15.00. The maximum response is observed at an ideal Heating Rate of 14.16. This result suggests that the process is sensitive to the rate at which it is heated, and heating rates of around 14.16°C per minute lead to more favourable outcomes.
iv. Interaction Effects of Time*Temperature (A*B): The statistical analysis indicates a significant interaction effect between time and temperature, with a p-value of less than 0.05. This data implies that the effect of time on the reaction depends on the temperature and vice versa. The reaction shows a more noticeable and continuous increase over time as the temperature rises.
v. Interaction Effects of Time*Heating Rate (A*C): The interaction effect between time and heating rate is not statistically significant (p-value > 0.05). Hence, it can be concluded that the impact of time on the reaction is independent of the Heating Rate.
vi. Interaction Effects of Temperature*Heating Rate (B*C): The interaction effect of temperature and heating rate is statistically significant (p-value < 0.05). These results demonstrate that temperature's impact on the reaction depends on the heating rate, and vice versa. With rising temperatures, the reaction experiences a more notable increase at greater heating rates.

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