Chapter 3

Torrefaction of pigeon pea stalk and eucalyptus along with their statistical analysis and process optimization using RSM

3.1 Overview

In this chapter, torrefaction for both pigeon pea stalk and eucalyptus have been carried out in a tubular quartz reactor under nitrogen atmosphere. Central composite design (CCD) in response surface methodology (RSM) has been employed to study the individual and the interactive influence of operating parameters (temperature, residence time, and heating rate) on HHV and energy yield of torrefied biomass. This chapter also includes the process optimization and its validation for the torrefaction of pigeon pea stalk and eucalyptus which has been based on maximum HHV and energy yield of the torrefied biomass. Efficient and accurate mathematical models for predicting HHV and energy yield of torrefied pigeon pea stalk and eucalyptus within the operating limits of experimental domain have been also included in this chapter.

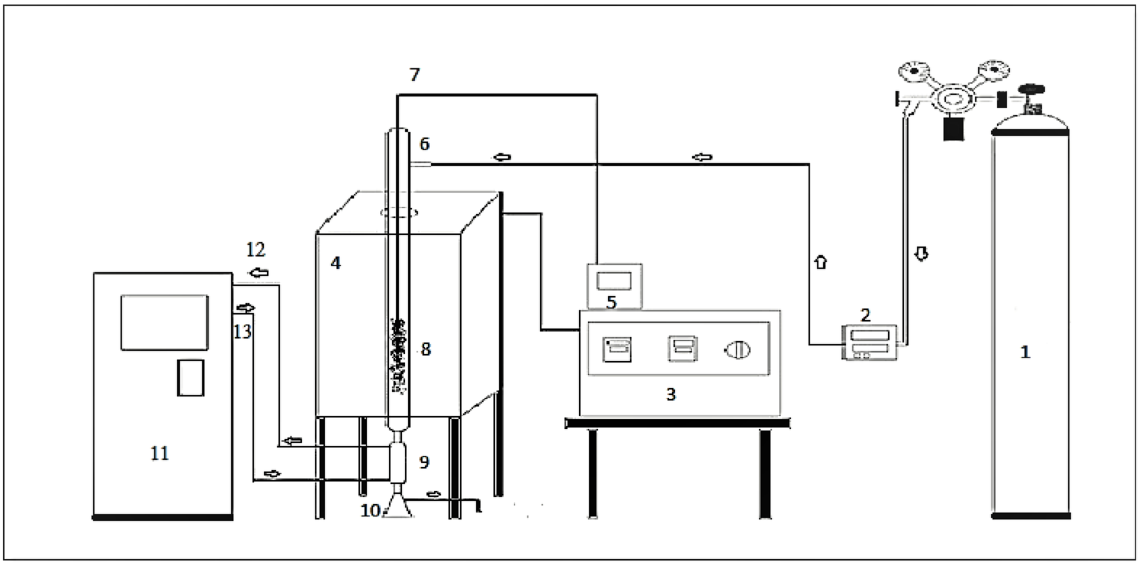
3.2 Materials and Methods

3.2.1 Material selection

Pigeon pea stalk and wooden blocks of eucalyptus, from the nearby areas of the Banaras Hindu University, India, have been cut into smaller blocks and dried in the presence of sunlight. They have been fed separately to a cutting mill (SM300, Retsch, Germany) and the disintegrated biomass materials have been sieved in sieve shaker, and the fraction between 0.7 to 1.2 mm have been collected and further dried in a hot air oven at 105 ^{o}C for 2-3 hours to remove any unbound moisture, before carrying out any further experiment.

3.2.2 Experimental setup and procedure

Fig. 3.1 Pictorial view of the experimental setup



1-Nitrogen Cylinder, 2-Mass Flow Controller, 3-Temperature Controller Unit, 4- Split Tube Furnace (NSW-104), 5-K-Type Thermocouple, 6-Long Tube Fixed Bed Reactor, 7-Thermocouple Probe, 8-Biomass With Ceramic Wool Bed, 9-Condenser, 10-Oil Collector, 11-Chiller (Eyela CA-1112CE), 12-Fluid Inlet, 13-Fluid outlet

Fig. 3.2 Schematic of the experimental setup.

Figs. 3.1 and 3.2 present the pictorial view and the schematic of the experimental setup, respectively. The experimental setup mainly consists of an electrically heated split-tube furnace housing 80 cm long quartz tube reactor. The PID controller has been used to control the temperature, residence time, and heating rate with an accuracy of ±1 °C, ±1 min, and ±0.1 °C/min, respectively. In each experiment, the fixed bed reactor has been fed with 6-8 g sample of biomass supported with ceramic wool. The bed height has been kept to 10 cm with the tip of the probe of a K-type thermocouple just touching top surface of the biomass-bed to observe the reaction temperature. Reactor with biomass has been purged for 30 min using nitrogen gas of 99.99 % purity (Sigma Gases, New Delhi) at a flow rate of 40 mL/min to get rid of any trapped oxygen before heating commences. For the torrefaction process, the temperature varies from 200-300 °C; residence time varies from 0-60 min, and heating rate varies from 5-20 °C/min. During each experiment, the condensable part has been condensed using a counter current condenser running on a recirculating bath (CA-1112 CE, Eyela, Japan). While non-condensable gases have been collected in Tedlar bags and the reactor after the heating stops has been allowed to cool down, and the solid residue or product has been collected. The mass of non-condensable gases (m_{ncg}) and solid yield (Y_{solid}) have been calculated using Eqs. (3.1) and (3.2):

$$\mathbf{m_{ncg} = m_{raw} - (m_{liquid} + m_{solid})} \quad (3.1)$$

$$\mathbf{Y_{solid} = \frac{m_{solid}}{m_{raw}}} \quad (3.2)$$

A bomb calorimeter (Rajdhani Scientific Instruments Co., New Delhi, R-S-B-3/2204-7-3) has been used to measure the HHV by following the procedure as mentioned in the standard UNE-EN 14918:2011.

Using Eq. (3.3) energy yield (Y_E) has been calculated.

$$\mathbf{Y_E} = \mathbf{Y_{solid}} \times \frac{\mathbf{HHV_{Torrefied}}}{\mathbf{HHV_{raw}}} \tag{3.3}$$

3.2.3 Experimental design

State-Ease Design Expert has been used for the design and statistical analysis of the torrefaction process. CCD, which is one of the response surface methodologies has been used for determining the optimum conditions, quadratic effects, main effects and interaction effects of operating parameters (temperature, residence time and heating rate) on the HHV and energy yield. The other operating parameters such as biomass particle size (0.7 to 1.2 mm) and nitrogen sweeping rate (40 ml/min) have been kept constant during the torrefaction process. In CCD option central face-centered (α=1) has been chosen. As per the conditions chosen in the software, there have been 18 sets of experiments for each pigeon pea stalk and eucalyptus, which includes eight factorial points, 4 center points, and six axial points. The coded levels of 200, 250, and 300 °C temperature, and 0, 30, and 60 minutes residence time, and 5, 12.5, and 20 °C/min have been marked with -1, 0, and +1, respectively. To fit the second order polynomial to the experimental data, a non-linear regression method has been employed. The predictive polynomial quadratic equation in general form is given by Eq. (3.4):

$$\boldsymbol{A} = \boldsymbol{\beta_0} + \sum_{j=1}^{k} \boldsymbol{\beta_j X_j} + \sum_{j=1}^{k} \boldsymbol{\beta_{jj} X_j^2} + \sum_{i<j} \boldsymbol{\beta_{ij} X_i X_j} \tag{3.4}$$

where A has been the response (HHV and energy yield), β_0 has been the intercept coefficient, β_i, βi_j and β_{jj} have been the interaction coefficient of the linear, the second order terms and quadratic terms, k has been the number of independent parameters (k=3 in this study), X_j has been the independent variables (temperature, residence time and heating rate). ANOVA has been performed to understand the fitness and statistical significance of the regression models, with a 95 % confidence level. The model having a coefficient of determination (R^2) more than 0.95 is acceptable, suggesting that the model can explain ~95 % of the data variability (Bajar et al., 2016; Buratti et al., 2018). The expected difference between the values of predicted coefficient of determination (R^2_{pred}) and adjusted coefficient of determination (R^2_{Adj}) should be less than 0.2 (Lou et al., 2013). Signal to noise ratio is represented by adequate precision (Adeq precision), and a ratio greater than 4 is desirable (Singh and Bishnoi, 2013). LOF represents the inadequacy of the predicted model to estimate the values within the experimental domain. For the experimental data used for the statistical analysis and optimization to have good agreement with the model, the p-value of the lack of fitness test has to be insignificant (p-value >0.05).

3.3 CCD and statistical analysis

(a) **(b)**

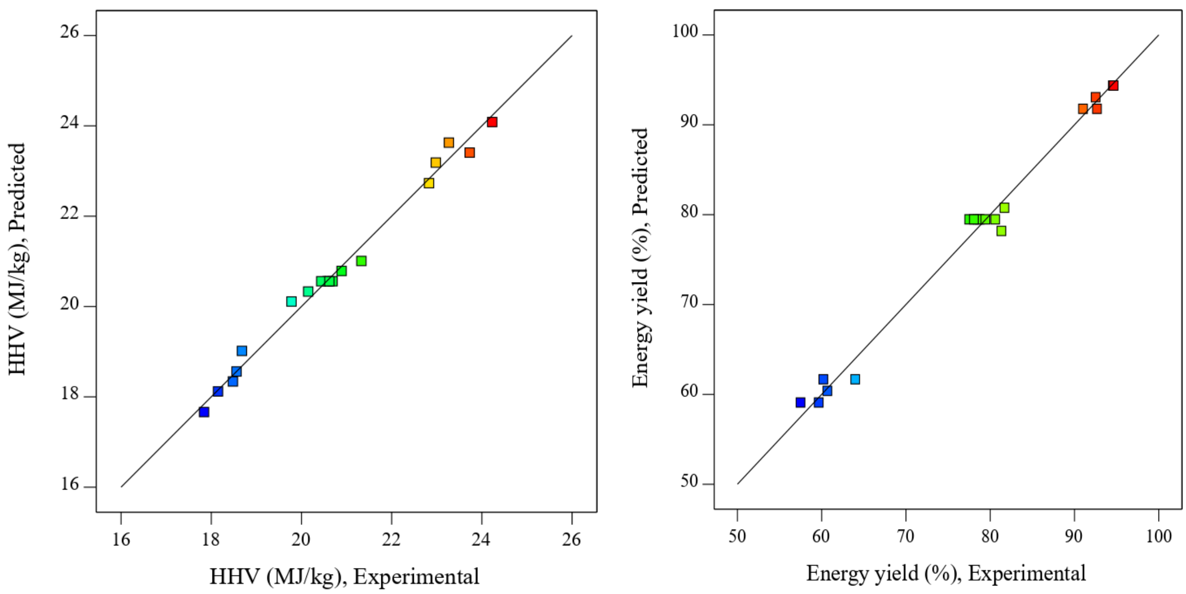


Fig. 3.3 Experimental versus predicted values for the responses of a) HHV, and b) energy yield of torrefied pigeon pea stalk.

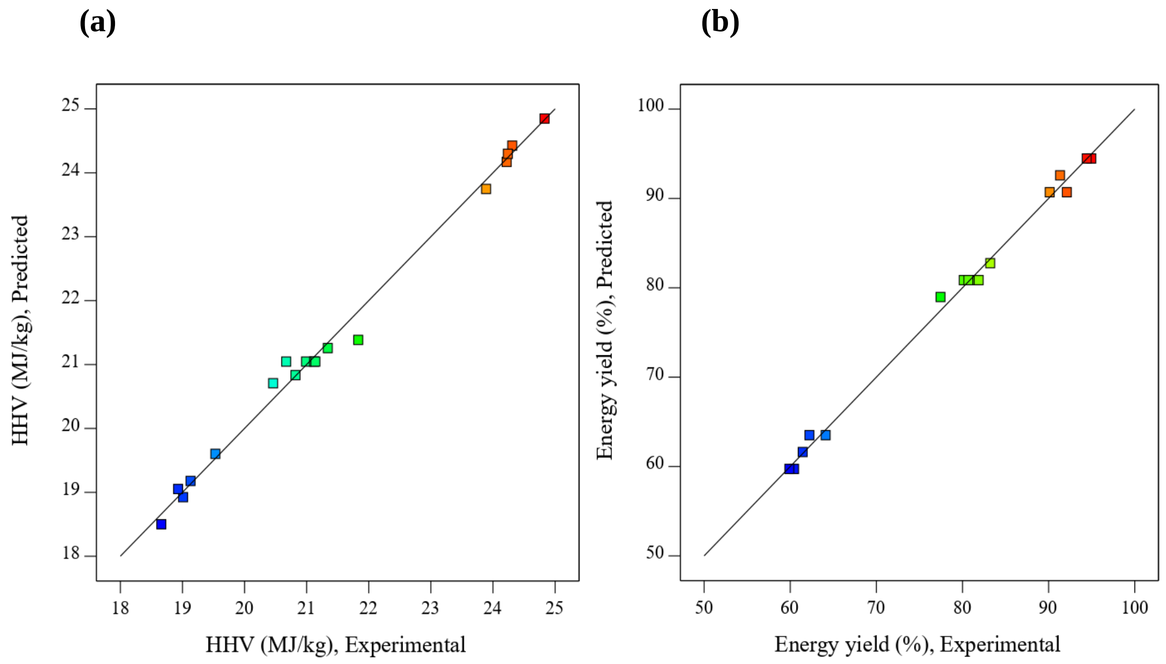


Fig. 3.4 Experimental versus predicted values for the responses of a) HHV, and b) energy yield of torrefied eucalyptus.

During the torrefaction process, each experiment has been repeated twice, and the average value has been quoted in the present study. However, in the case of center point of CCD (TPS-250-30-12.5 and TEC-250-30-12.5), the experiment has been performed four times to determine the experimental error and reproducibility of the proposed models. Tables 3.1 and 3.2 represents the experimental conditions along with their corresponding experimental and predicted values as obtained from CCD. The regression analysis has been carried out in order to a establish relationship between the dependent variable (HHV and energy yield) and independent variables (temperature, residence time, and heating rate). Fig. 3.3 ((a) and (b)) and Fig. 3.4 ((a) and (b)) represents the predicted versus the experimental data of the responses for the torrefaction of pigeon pea stalk and eucalyptus, respectively, and these plots helps in checking the adequacy of the mathematical models used in the present study. It can be illustrated from Figs. 3.3 and 3.4, that most of the data points lie very close to the straight line, suggesting that there has been an excellent agreement between the model and the experimental data. In all four plots, the actual and the predicted values have been close to each other, hence, the deviation in the results have been negligible. Also, the values of R^2 in all the observed cases have been close to 1, suggesting that the reported models can be used to predict the value of HHV and energy yield.

Table 3.1 Experimental responses for the torrefaction process of pigeon pea stalk

Run	X_1	X_2	X_2	HHV (MJ/kg)			Energy yield (%)		
	Temperature (ºC)	Residence time (min)	Heating rate (ºC/min)	Experimental value	Predicted value	Residual	Experimental value	Predicted value	Residual
1	250	30	12.5	20.62	20.56	0.06	78.06	79.48	-1.42
2	250	30	5	20.15	20.33	-0.18	80.59	79.48	1.11
3	300	60	20	24.23	24.09	0.14	57.49	59.11	-1.62
4	250	60	12.5	21.33	21.01	0.32	81.33	78.19	3.14
5	300	30	12.5	23.73	23.41	0.32	60.67	60.40	0.27
6	300	0	5	22.83	22.73	0.10	63.98	61.69	2.29
7	250	30	20	20.89	20.79	0.10	78.75	79.48	-0.73
8	250	30	12.5	20.69	20.56	0.13	78.52	79.48	-0.96
9	200	0	5	17.84	17.67	0.17	94.56	94.37	0.19
10	200	60	5	18.56	18.56	0.00	92.67	91.79	0.88
11	250	30	12.5	20.58	20.56	0.02	77.51	79.48	-1.97
12	300	0	20	22.98	23.19	-0.21	60.20	61.69	-1.49
13	300	60	5	23.27	23.63	-0.36	59.65	59.11	0.54
14	250	0	12.5	19.78	20.11	-0.33	81.69	80.78	0.91
15	200	60	20	18.68	19.02	-0.34	91.01	91.79	-0.78
16	200	0	20	18.15	18.12	0.029	94.65	94.37	0.28
17	200	30	12.5	18.48	18.34	0.14	92.51	93.08	-0.57
18	250	30	12.5	20.44	20.56	-0.12	79.42	79.48	-0.06

Table 3.2 Experimental responses for the torrefaction process of eucalyptus

Run	X_1	X_2	X_2	HHV (MJ/kg)			Energy yield (%)		
	Temperature (ºC)	Residence time (min)	Heating rate (ºC/min)	Experimental value	Predicted value	Residual	Experimental value	Predicted value	Residual
1	250	30	5	20.82	20.83	-0.01	81.45	80.87	0.58
2	200	60	20	19.53	19.60	-0.07	90.13	90.70	-0.57
3	300	0	20	24.22	24.17	0.05	62.23	63.52	-1.29
4	200	60	5	19.13	19.18	-0.05	92.12	90.70	1.42
5	200	0	5	18.66	18.50	0.16	94.96	94.48	0.48
6	300	30	12.5	24.24	24.30	-0.06	61.45	61.63	-0.18
7	300	60	20	24.83	24.85	-0.02	59.89	59.74	0.15
8	200	30	12.5	18.93	19.05	-0.12	91.33	92.59	-1.26
9	250	30	12.5	21.14	21.05	0.09	80.68	80.87	-0.19
10	250	30	12.5	20.99	21.05	-0.06	80.99	80.87	0.12
11	250	30	12.5	20.67	21.05	-0.38	81.88	80.87	1.01
12	300	0	5	23.89	23.75	0.14	64.11	63.52	0.59
13	250	30	12.5	21.12	21.05	0.07	81.12	80.87	0.25
14	250	30	20	21.34	21.26	0.08	80.14	80.87	-0.73
15	250	60	12.5	21.83	21.39	0.44	77.45	78.98	-1.53
16	250	0	12.5	20.46	20.71	-0.25	83.22	82.76	0.46
17	300	60	5	24.31	24.43	-0.12	60.45	59.74	0.71
18	200	0	20	19.01	18.93	0.09	94.42	94.48	-0.06

3.4 ANOVA analysis

In the present study, the insignificant terms have been excluded from the quadratic models to get the reduced quadratic models, which have better accuracy, reliable and reproducibility in predicting the values within the operating limits of experimental domain. Tables 3.3 and 3.4 represent the results of ANOVA obtained for HHV and energy yield of torrefied pigeon pea stalk and eucalyptus. To understand the influence of operating parameters during torrefaction on HHV and energy yield, F-value has been evaluated, and for each coefficient in the reduced quadratic model, F-value has been presented in Tables 3.3 and 3.4.

Table 3.3 ANOVA for the responses of the reduced quadratic models for the torrefaction of pigeon pea stalk

Source	Sum of squares	DF	Mean square	F-value	p-value
HHV (MJ/kg)					
Model	67.14	4	16.78	285.93	<0.0001
X_1	64.16	1	64.16	1093.02	<0.0001
X_2	2.02	1	2.02	34.34	< 0.0001
X_3	0.51	1	0.51	8.86	0.0107
X_1^2	0.44	1	0.44	7.51	0.0168
Residual	0.76	13	0.06	-	-
Lack of fit	0.7298	10	0.1	6.58	0.0740
Pure error	0.3	3	0.01	-	-
Standard deviation=0.24, mean=20.73, co-efficient of variation(%)=1.16, R^2=0.99, R^2_{Adj}=0.98, R^2_{Pred}= 0.98, Adeq precision= 50.28					
Energy yield (%)					
Model	2720.48	3	906.83	402.16	<0.0001
X_1	2670.28	1	2670.28	1184.21	<0.0001
X_2	16.72	1	16.72	7.41	0.0165
X_1^2	31.48	1	33.48	14.85	0.0018
Residual	31.57	14	2.25	-	-
Lack of fit	29.61	11	2.69	4.12	0.1353
Pure error	1.96	3	0.65	-	-
Standard deviation=0.93, mean=77.96, co-efficient of variation(%)=1.19, R^2=0.99, R^2_{Adj}=0.99, R^2_{Pred}= 0.98, Adeq precision=49.82					

In order to approve any proposed model, it is essential to analyze it's the p-value and the F-value; when the F-value is high, the reliability of the model is greater, while with a lower p-value, the significance of the model becomes higher (Arvindekar and Laddha, 2016; Gupta and Mondal, 2019; Nizamuddin et al., 2016). The F- values for the reduced quadratic models of HHV and energy yield have been 285.93 and 402.16, respectively for torrefied pigeon pea stalk while for eucalyptus it has been 451.24 and 1031.69, respectively. The F-value being so high clearly suggest that all four models have adequate reliability and there could be only 0.01 % chance that this could occur due to any noise. Also the p-values of all four proposed models have been less than 0.0001, suggesting that the regression models are high significant.

Table 3.4 ANOVA for the responses of the reduced quadratic models for the torrefaction of eucalyptus

Source	**Sum of squares**	**DF**	**Mean square**	**F-value**	**p-value**
HHV (MJ/kg)					
Model	72.16	4	18.04	451.24	<0.0001
X_1	68.80	1	68.80	1721.03	<0.0001
X_2	1.15	1	1.15	28.75	0.0001
X_3	0.44	1	0.44	11.24	0.0052
X_1^2	1.76	1	1.76	43.95	<0.0001
Residual	0.52	13	0.04	-	-
Lack of fit	0.38	10	0.03	4.80	0.655
Pure error	0.14	3	0.05	-	-
Standard deviation=0.12, mean=21.40, co-efficient of variation(%)=0.93, R^2=0.99, R^2_{Adj}=0.99, R^2_{Pred}= 0.98, Adeq precision= 60.23					
Energy yield (%)					
Model	2495.70	3	831.90	1031.69	<0.0001
X_1	2397.23	1	2397	2972.73	<0.0001
X_2	35.72	1	35.72	44.30	<0.0001
X_1^2	62.74	1	62.74	77.81	<0.0001
Residual	11.29	14	0.81	-	-
Lack of fit	10.51	11	0.96	6.68	0.156
Pure error	0.78	3	026	-	-
Standard deviation=0.89, mean=78.78, co-efficient of variation(%)=1.14, R^2=0.99, R^2_{Adj}=0.99, R^2_{Pred}= 0.99, Adeq precision=82.08					

The value of R^2 for both the models of HHV and that of energy yield have been 0.99, which clearly explains the precision of all the reduced quadratic models. It is worth mentioning that R^2 is more sensitive to the degree of freedom, which increases with the number of model terms due to which R^2_{Adj} value becomes more useful in checking the model adequacy (Glyk et al., 2015). The value of R^2_{Adj} for the reduced quadratic models of HHV and energy yield have been 0.98 and 0.99, respectively for torrefied pigeon pea stalk and it has been 0.99 each for both the models of torrefied eucalyptus. These values being more than 0.95 have been in accordance with the results quoted by other researchers (Buratti et al., 2018; Glyk et al., 2015). The values of R_{pred} and R_{Adj} have been in reasonable agreement with each other as the difference has been less than 0.2, indicating that the proposed regression models satisfactorily represents the true relationship between the chosen variables. Values of Adeq precision for HHV (50.28 and 60.23) and energy yield (49.82 and 82.08) have been in the desired limit (Adeq precision >4). Tables 3.1 and 3.2 also include the values of residuals for the present study, and their values have been investigated in sequence to confirm the normal distribution of the data. In Figs. 3.3 and 3.4, the predicted data and the experimental data lie very close to the straight line, confirming the normal distribution of data with the variation being random in nature (Swamy et al., 2014).

To check the acceptance of the proposed models, LOF test has been performed. LOF compares the residual error (error related to the fitted model) to the pure error obtained from the replicated design points (Luo et al., 2010). In the present study, for HHV and energy yield the p-values for LOF have been 0.0740 and 0.1353, respectively during torrefied pigeon pea stalk and for torrefied eucalyptus it has been 0.655 and 0.156, respectively. These values clearly suggest that LOF in all four proposed models have not been significant. F-value of LOF provides additional information regarding the suitability of the recognized quadratic regression model, and high values render insignificant behavior (Šumić et al., 2016). F-value of LOF for HHV and energy yield during torrefied pigeon pea stalk have been found to be 6.58 and 4.12, respectively, and for torrefied eucalyptus it has been 4.80 and 6.68, respectively. These values confirm that LOF for the reduced quadratic models have been insignificant. Eqs. (3.5), (3.6), (3.7) and (3.8) represents the developed quadratic models in reduced form which can be used for predicting the HHV (MJ/kg) and the energy yield (%) of the torrefied pigeon pea stalk and eucalyptus within the experimental domain.

$$HHV_{Pigeon\ pea\ stalk} = 20.56 + 2.53X_1 + 0.45X_2 + 0.22X_3 + 0.32X_1^2 \quad (3.5)$$

$$Energy\ yield_{Pigeon\ pea\ stalk} = 79.48 - 16.34X_1 - 1.29X_2 - 2.74X_1^2 \quad (3.6)$$

$$HHV_{Eucalyptus} = 21.05 + 2.62X_1 + 0.34X_2 + 0.21X_3 + 0.63X_1^2 \quad (3.7)$$

$$Energy\ yield_{Eucalyptus} = 80.87 - 15.48X_1 - 1.89X_2 - 3.76X_1^2 \quad (3.8)$$

In Eqs. (3.5), (3.6), (3.7) and (3.8), the terms X_1, X_2, and X_3 represent the coded values of temperature, residence time, and heating rate, respectively. The terms present in the equations obtained after statistical analysis are said to be significant when the p-value is <0.05 (Gupta and Mondal, 2019). Hence, based on this the terms which have been significant for HHV have been X_1, X_2, X_3, and X_1^2 for both the biomass suggesting that irrespective of biomass the significant terms would be the same. It can also be observed from Eq. (3.5) and (3.7) that all the significant terms have a synergic effect on HHV and based on F-value for the significant terms it can be said that the sequence of impact of synergistic effect has been $X_1>X_2>X_1^2>X_3$. There were studies where higher impact of temperature as compared to residence time on HHV was observed by the researchers in their respective studies (Buratti et al., 2018; Chiou et al., 2015; Mundike et al., 2016; Nam and Capareda, 2015). Similarly based on ANOVA in the case of energy yield for the reduced quadratic models, the terms which have been significant for both the biomass have been X_1, X_2, and X_1^2. This observation means that the heating rate has a nominal or insignificant effect on the energy yield as compared to temperature and residence time. On the basis of positive and negative signs prior to the terms of the reduced quadratic models, it can be attributed that for energy yield, the significant terms have an antagonistic effect. Also on the basis of magnitude of F-value, the sequence of influence of antagonistic effect on energy yield have been $X_1 > X_1^2 > X_2$ for both the biomass.

In Table 3.3 and 3.4 for ANOVA also depicts the model summary for HHV and energy yield. Standard deviation anything <1 is considered to be satisfactory and manifests the excellent model fitting for optimization. In the present study, standard deviation for HHV and energy yield of torrefied pigeon pea stalk have been 0.24 and 0.93, respectively, and for torrefied eucalyptus it has been 0.12 and 0.89, respectively. The co-efficient of variation is calculated as the ratio of the standard deviation to mean, and it depicts the error in mean percentage. The regression model to have high reliability and better reproducibility, the value of co-efficient of variation less than 10 is desirable (Mohammed et al., 2017). The co-efficient of variation have been 1.16 and 1.19 % for HHV and energy yield, respectively for torrefied pigeon pea stalk and for torrefied eucalyptus it has been 0.93 and 1.14 %, respectively.

3.5 Optimization

To maximize the HHV and energy yield, the operating parameters considered in the present study have been optimized. Torrefaction temperature, residence time, and heating rate have been maintained within the range of experimental conditions. Other constraints have been represented in Table 3.5, where equal weightage have been given to both HHV and energy yield. In the present study, Derringer's desirability function method has been employed to optimize the torrefaction process of pigeon pea stalk and eucalyptus. Based on the highest desirability, the optimum conditions for both the biomass have been obtained.

Table 3.5 Optimization condition (constraints).

Constraints name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Temperature	is in range	200	300	1	1	3
Residence time	is in range	0	60	1	1	3
Heating rate	is in range	5	20	1	1	3
Pigeon pea stalk						
HHV	maximize	17.84	24.23	1	1	5
Energy yield	maximize	57.49	94.65	1	1	5
Eucalyptus						
HHV	maximize	18.66	24.83	1	1	5
Energy yield	maximize	59.89	94.96	1	1	5

Table 3.6 HHV and energy yield at optimized condition and corresponding experimental values.

Run	Temperature (ºC)	Residence time (min)	Heating rate (ºC/min)	HHV (MJ/kg)	Energy yield (%)	Desirability (%)
Pigeon pea stalk						
Predicted	248.20	60	20	21.15	78.80	61.4
Experimental	248	60	20	21.49±0.22	79.91.±0.43	-
Deviation (%)				1.61	1.41	-
Eucalyptus						
Predicted	252.87	60	20	21.75	78.10	63.2
Experimental	252	60	20	22.38±0.34	80.26.±0.43	-
Deviation (%)				2.90	2.77	-

The optimum condition for the torrefaction of pigeon pea stalk and eucalyptus have been obtained at 248.20 °C, 60 min, 20 °C/min with desirability of 61.1 % and at 252.87 °C, 60 min, 20 °C/min with desirability of 63.2 %, respectively. Based on these results it can be observed that the optimum conditions for both the biomass have been quite similar clearly suggesting that moderate severity torrefaction is suitable for most of the biomass obtained from agricultural residue or wood. Similar results were reported by Buratti et al. (Buratti et al., 2018) for the torrefaction process optimization, where the desirability was limited to 52.1% and 56.2% for coffee chaff and spent coffee grounds, respectively.

3.6 3-D plots for individual and interactive influence of operating parameters on responses

In order to study the individual and interactive effect of process variables during torrefaction on the response values (HHV and energy yield) 3-D plots have been used. It has not been possible to represent the effect of all three parameters in a single 3-D plot hence, in the present study, a single RSM 3-D plot for either HHV or energy yield represents the variation of response versus two varying operating parameters and the remaining one operating parameter remains constant, as shown in Fig. 3.5, 3.6, 3.7 and 3.8.

Fig. 3.5 (a), 3.6 (a), 3.7 (a) and 3.8 (a) represents the 3-D plots between temperature and residence time versus HHV or energy yield obtained at a constant heating rate (20 °C/min for both pigeon pea stalk and eucalyptus). On analyzing these plots for the individual effect on responses it can be observed that for the increase in either temperature or residence time there has been increase in HHV and decrease in energy yield where temperature has more prominent impact on both the responses as compared to residence time. This increase in HHV may be viewed as a loss of energy lean components and residence or formation of energy rich components (Anukam et al., 2015). Similar results were observed by other researchers where higher heating value increased after the torrefaction process (Martín-Lara et al., 2017; Ohliger et al., 2013; Prins et al., 2006).

Energy yield indicates the total energy preserved during the torrefaction process, and it depends on both HHV and solid yield of torrefied biomass. The solid yield for torrefied biomass decreased sharply with the increase in severity of torrefaction, which results in a decrease in energy yield even though the HHV increased (Singh et al., 2019). Similar results were also observed by other researchers in their work for torrefaction of biomass, where energy yield decreased significantly with an increase in severity of torrefaction (Arias et al., 2008; Martín-Lara et al., 2017; Prins et al., 2006). Based on observation that both HHV and energy yield have opposite response towards the operating parameters; hence, the optimum condition or recommended region of operation would be moderate temperature range with high residence time.

The 3-D plots of HHV and energy yield for varying temperature and heating rate at constant residence time (60 min for both biomass) have been shown in Fig. 3.5 (b), 3.6 (b), 3.7 (b) and 3.8 (b). It can be observed that individual variation of temperature has an appreciable effect on HHV and energy yield; however when only heating rate has been varied a less impact or insignificant effect has been observed. Also, on observing their combined effect no appreciable influence on both responses has been observed. 3D plots for HHV and energy yield variation with residence time (0-60 min) and heating rate (5-20 °C/min) at a constant temperature (248.20 and 252.87 °C) have been depicted in Fig. 3.5 (c), 3.6 (c), 3.7 (c), and 3.8 (c). On analyzing the combined effect of residence time and the heating rate, it can be observed that that their combined variation has little or nominal effect on HHV and energy yield of torrefied biomass. However when these two parameters have been compared to each other at constant torrefaction temperature, the residence time has more effect on HHV and energy yield as compared to the heating rate.

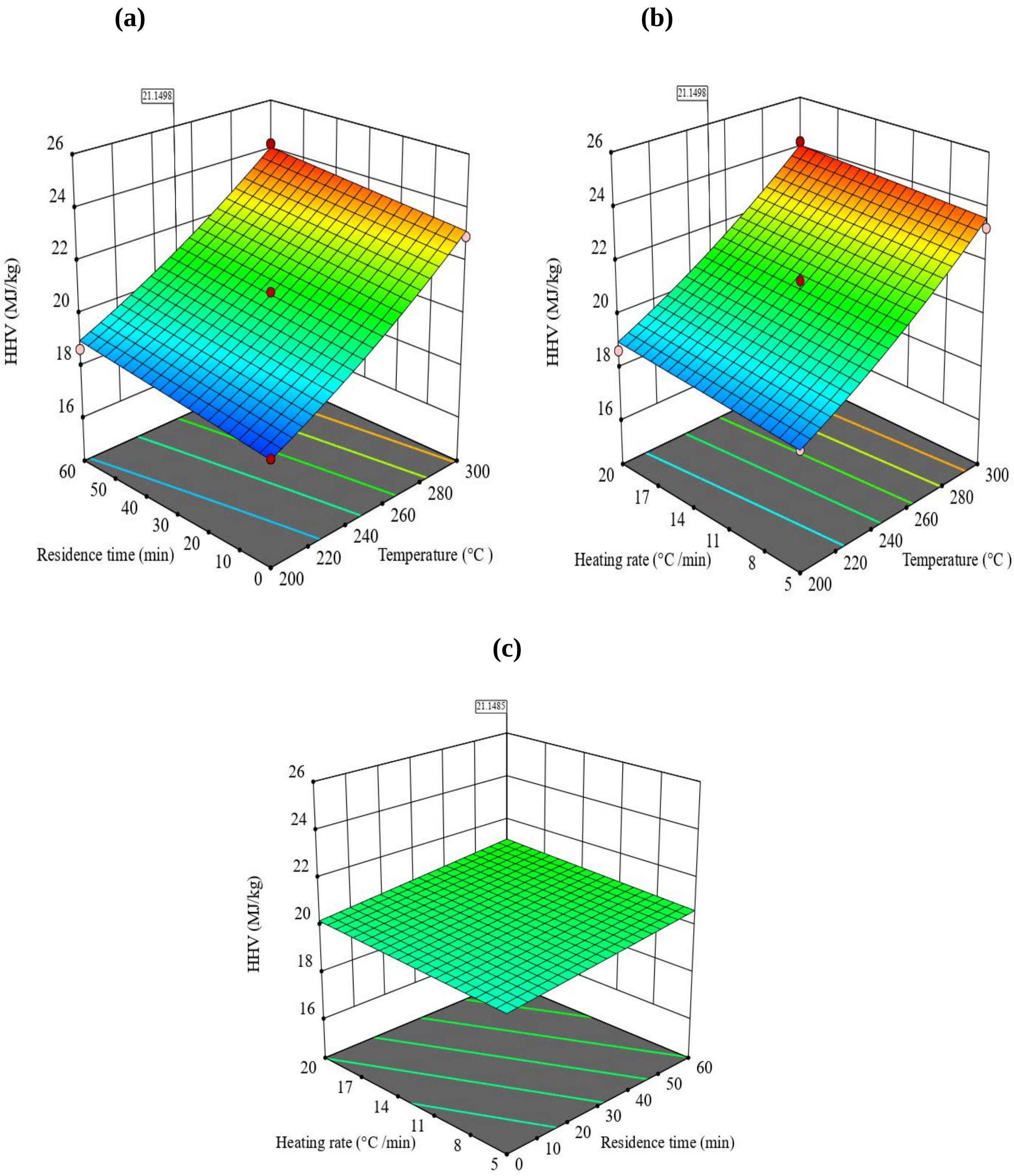


Fig. 3.5 Response surface 3D plots for the HHV of torrefied pigeon pea stalk showing the effect of (a) temperature and residence time, (b) temperature and heating rate, (c) residence time and heating rate.

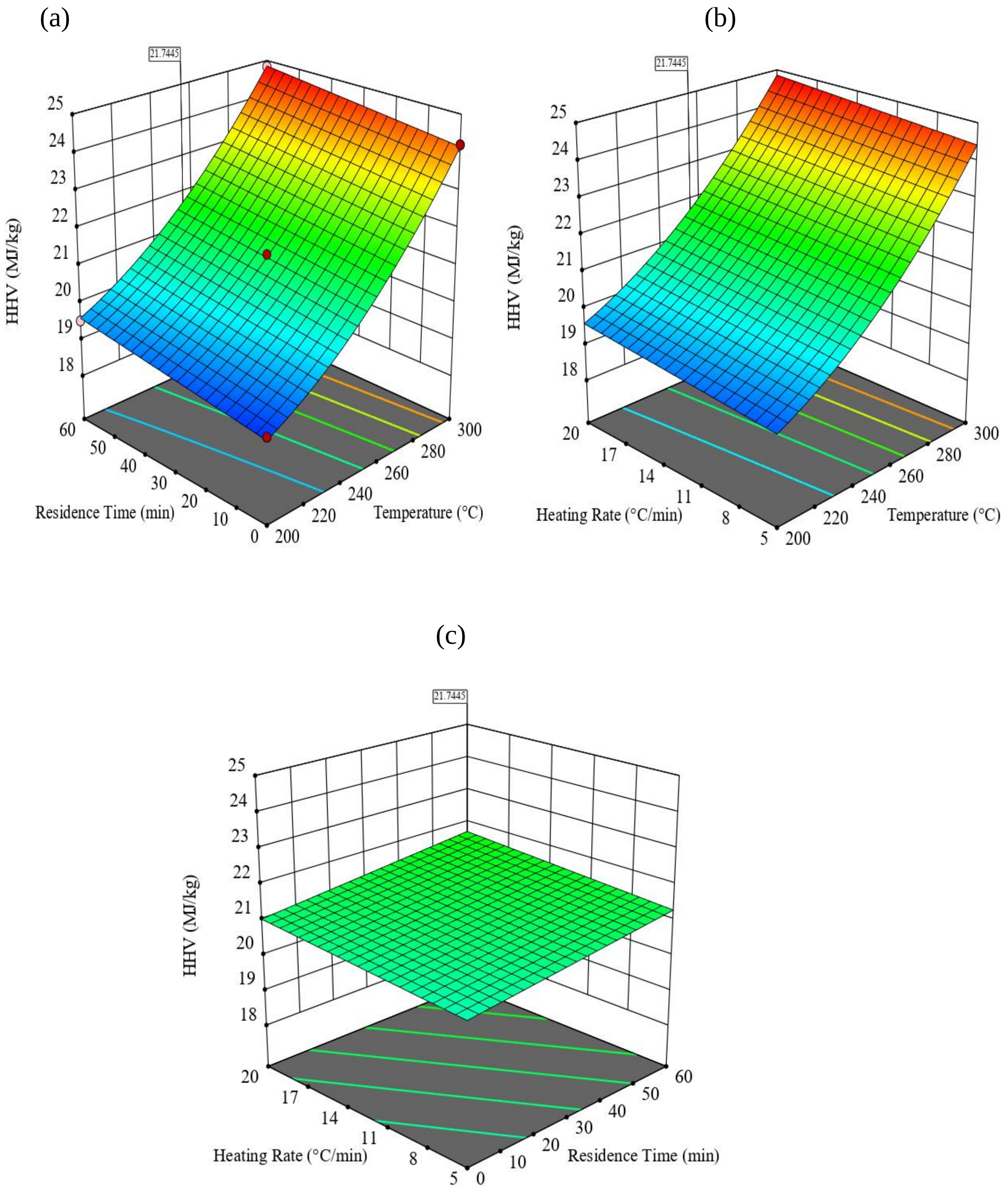


Fig. 3.6 Response surface 3D plots for the HHV of torrefied eucalyptus showing the effect of (a) temperature and residence time, (b) temperature and heating rate, (c) residence time and heating rate.

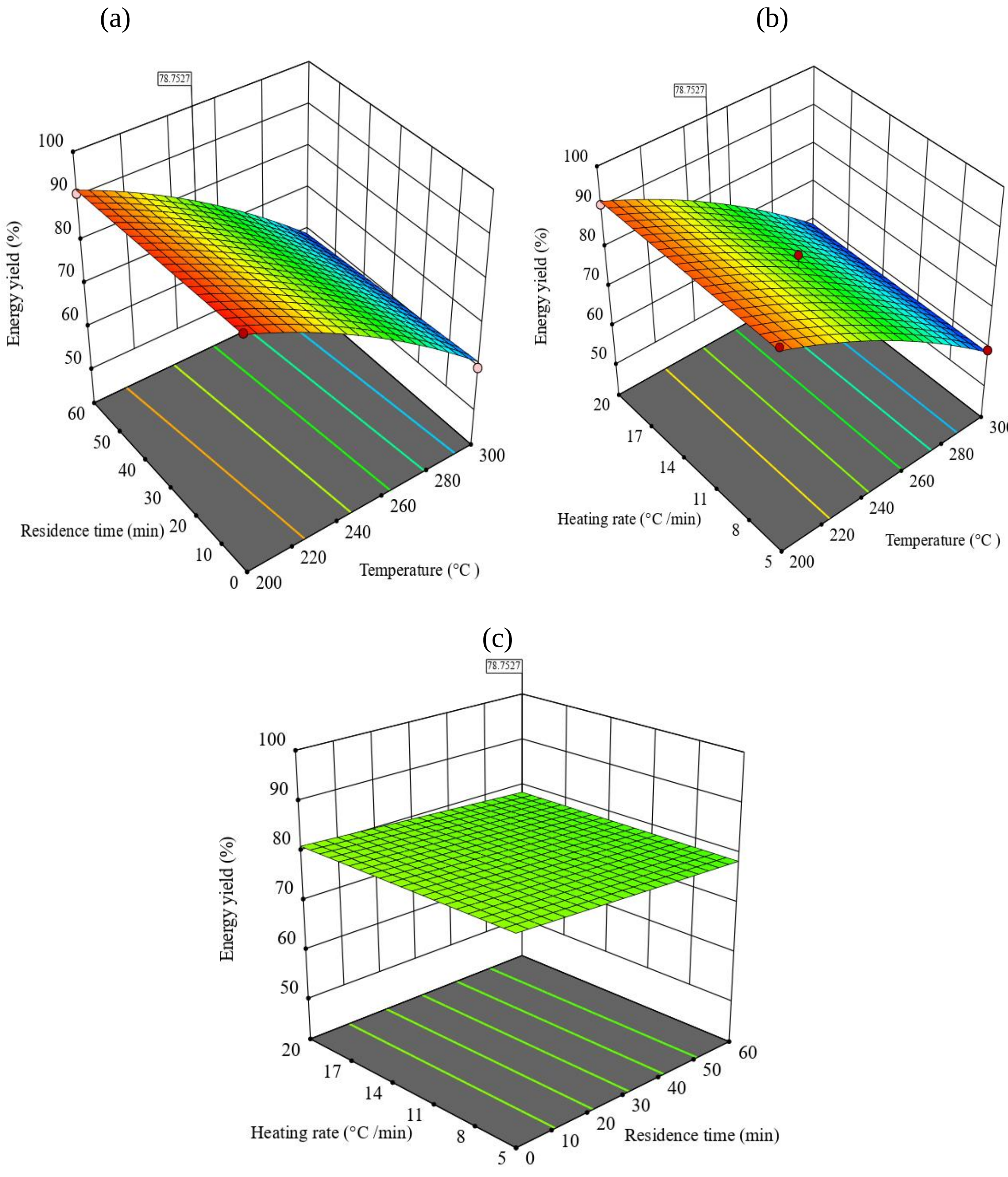


Fig. 3.7 Response surface 3D plots for the energy yield of torrefied pigeon pea stalk showing the effect of (a) temperature and residence time, (b) temperature and heating rate, (c) residence time and heating rate.

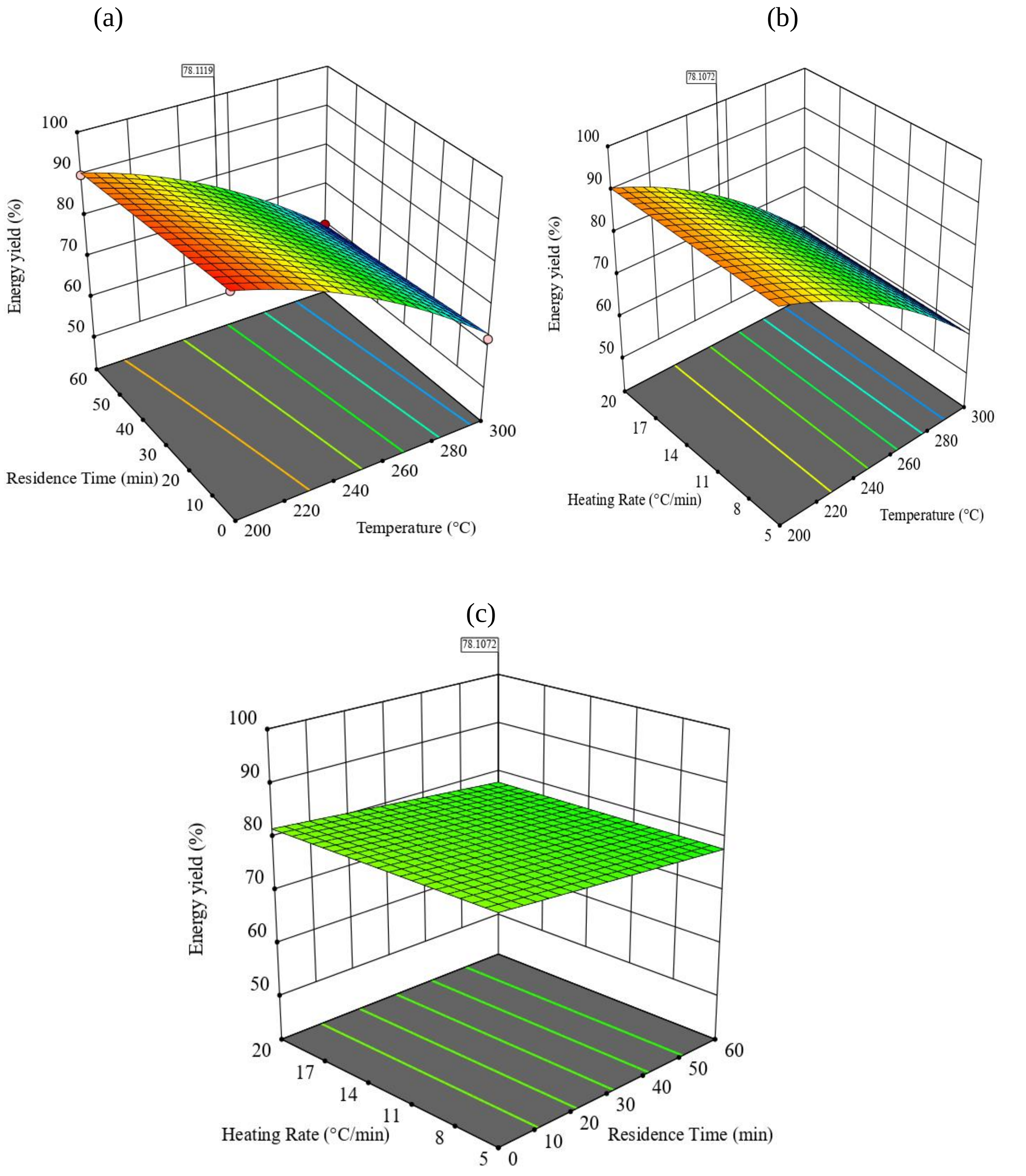


Fig. 3.8 Response surface 3D plots for the energy yield of torrefied eucalyptus showing the effect of (a) temperature and residence time, (b) temperature and heating rate, (c) residence time and heating rate.

3.7 Experimental validation of optimum condition

In order to validate the results obtained at the optimized condition, torrefaction for both pigeon pea stalk and eucalyptus have been performed thrice and their average values have been compared with their predicted values which have been obtained from the proposed mathematical model. Table 3.6 represents the predicted values from the model and average experimental values obtained while performing at optimum operating conditions. It can be observed that the experimental values for HHV and energy yield of both biomass materials have been in excellent agreement with their respective predicted values. The average experimental values for HHV have been 21.49±0.22 and 22.38±0.34 MJ/kg for torrefied pigeon pea stalk and eucalyptus, respectively, which have been sufficiently close to their respective predicted values with a deviation of only 1.61 and 2.90 %, respectively. Similarly, for energy yield, the experimental values have been 79.91±0.43 and 80.26±0.43 % for torrefied pigeon pea stalk and eucalyptus which have been very close to their respective predicted values with small deviation of 1.41 and 2.77 %, respectively. Hence, the proposed model finds its suitability in predicting the HHV and energy yield for torrefied pigeon pea stalk.

3.8 Summary

As a closure, this chapter helped in determining the optimum torrefaction conditions which could lead to a paradigm shift in the pretreatment process in terms of heating value, and energy yield. Based on the statistical analysis, the results suggest that the temperature had the most significant effect on HHV and energy yield, followed by residence time and heating rate. Based on ANOVA and validation of optimum condition, it can be attributed that the reduced quadratic models for predicting HHV and energy yield for the torrefied pigeon pea stalk and eucalyptus were efficient to operate in the design space. Based on statistical analysis and the optimum conditions obtained for both the biomass which were quite similar, it can be recommended that the moderate severity torrefaction is suitable for most of the biomass obtained either from agricultural residue or wood.