

COB-2023-0154

IMPLEMENTING THE TAGUCHI METHOD IN RICE HUSK BIOMASS TORREFACTION

Maristela Ribeiro de Oliveira

Ivonete Ávila

Miriam Ricciulli de Oliveira

UNESP - Universidade Estadual Paulista, Faculdade de Engenharia de Guaratinguetá, Departamento de Energia, Laboratório de Combustão e Captura de Carbono (LC3), Av. Dr. Ariberto Pereira da Cunha, 333, 12.516-410, Guaratinguetá-SP, Brazil. Dr. Ariberto Pereira da Cunha, 333, 12.516-410, Guaratinguetá-SP, Brazil.

maristela.olivera@unesp.br, ivonete.avila@unesp.br, miriam.ricciulli@unesp.br

Summary. *Due to the negative impacts caused by greenhouse gases (GHGs) on the environment, global policies are needed to implement renewable energy sources that benefit the well-being of the population and the global economy. Lignocellulosic biomass is a promising source of renewable energy used in several countries and is a sustainable source that contributes to reducing GHG emissions in the environment. This work aims to torrefaction rice husk lignocellulosic biomass in order to analyze the influence of pretreatment parameters. To this end, the Taguchi experimental design with an L9 orthogonal matrix was proposed, using factors such as temperature, residence time, mass, and granulometry at three different levels, high, medium, and low. By carrying out the experiments, it was possible to identify the condition with the best experimental fit (300°C; 50 min; 3g; 231 mm) for the variables, percentage of moisture and volatile released, fixed carbon, and higher heating value. The ANOVA analysis showed that temperature was the most significant factor in the torrefaction process, since the calculated F value was greater than the critical F value.*

Keywords: *Torrefaction, biomass, rice husk, Taguchi.*

1. INTRODUCTION

Currently, there is a great need to replace fossil fuels, as they are exhaustible sources and their extraction generates toxic gases such as carbon monoxide, sulfur dioxide, and nitrogen oxides, which are very harmful to the environment and the health of the population (TAYRA *et al.*, 2020). Studies have been carried out in search of new alternative energy sources to contribute to sustainable development and reduce global warming.

One of the advantages of renewable energy is that it is easy to obtain and significantly reduces pollutant emissions, as it is constantly regenerating and is easily found in nature. Renewable energies are classified as wind, solar, hydroelectric, geothermal, and biomass (INTELBRAS, 2022).

Lignocellulosic biomass is the most abundant renewable raw material available for the production of bio-oil, biochar, biogas, and synthesis gas. Some of the benefits of implementing biomass as a renewable raw material are the low cost of acquiring the raw material, ease of storage, ease of transportation, and conversion into energy (VUPPALADADIYAM *et al.*, 2022).

Brazil is one of the largest rice producers in the world, with a global production of 10 million tons; between 2022 and 2023, rice production is expected to reach 11.2 million tons (CONAB, 2022). Rice husk is a product of plant origin, composed of cellulose (34.0–53.2%), hemicellulose (4.6–28.0%), and lignin (16.0–24.0%). cellulose and hemicellulose correspond to 50% of the dry weight of the husk; lignin corresponds to 26% of the dry mass; and oils and proteins correspond to 4% of the dry mass. (MARCONCINI JOSÉ MANOEL, 2007). Rice husks are the main raw material for biomass plants in Rio Grande do Sul, and their energy potential is 175 MW per year, according to a report by the Institute for Applied Economic Research (Ipea).

The challenge in generating energy using biomass, with an emphasis on rice husks, is to improve its physical and chemical properties. As it has high humidity, many minerals, and a low calorific value, pre-treatments such as torrefaction help to improve the energy efficiency of biomass in terms of increased calorific value, lower atomic H/C and O/C ratios, better mobility and greater hydrophobicity. (MANATURA, 2020). Torrefaction is a process in which the material is burned in the absence of oxygen at a low temperature, usually between 200-300°C (CHEN *et al.*, 2015).

In the experiment in question, rice husk torrefaction was carried out by applying the design of experiments developed by Genichi Taguchi to carry out controlled experiments and processes, reducing the number of runs, and analyzing the significance of the parameters simultaneously to find the best compositions (GOOGERDCHIAN *et al.*,

2018). To do this, it is necessary to define the parameters that affect the experimental procedure, where for each parameter different levels and an orthogonal matrix are assigned; the matrix will guide how the experiments will be carried out; and finally, the results of the experiments are analyzed according to the chosen condition, and the result can be interpreted as "higher is better", lower is better," or "nominal is better" (CHEN *et al.*, 2022).

The experimental design was implemented with the aim of investigating the performance of the parameters that affect roasting and their interactive behavior. The study used variations in parameters such as temperature, residence time, mass, and granulometry; the output variable was the percentage of moisture and volatile released, the value of fixed carbon, and the highest calorific value, where in the end the significance or otherwise of the variables on the roasting parameters was identified.

2. MATERIALS AND METHODS

This study was carried out in the carbon capture and combustion laboratory (LC3) belonging to the energy department of FEG-UNESP. The rice husks used in the study were supplied by rice producers in Guaratinguetá, in the state of São Paulo.

2.1 Roasting

The vertical tubular reactor FORTLAB, model FT-1200/I2, available from LC3, was used for the roasting tests. The "Flexlab" software was used, which allows the temperature, heating rate, and isotherm time to be programmed. A heating rate of 10 °C/min was used under an inert nitrogen atmosphere at a flow rate of 100 mL/min, and the samples were placed in an alumina crucible with dimensions of 50 mm x 10 mm.

Figure 1. Vertical tubular reactor.



Source: author

2.2 Immediate analysis

A TA Instruments - SDT TGA-DSC Q 600 simultaneous system, available in LC3 and alumina crucibles, was used to study the thermal behavior of the torrefied biomass. To check the volatile material (VM), fixed carbon (FC), ash, and moisture (M) contents present in all the samples, the procedure developed by (KARATEPE; KÜÇÜKBAYRAK, 1993). To determine the moisture content, a nitrogen atmosphere was used at a flow rate of 40 mL/min with a heating rate of 20 °C/min from 30 °C to 110 °C, where the temperature of 110 °C was maintained for 30 min, the heating rate was increased to 40 °C/min, and the temperature was raised to 950 °C maintaining an isotherm for 7 min to quantify the volatile material, after lowering the temperature to 750 °C with a cooling rate of 20 °C/min, it was then changed to a synthetic air atmosphere, maintaining an isotherm for 30 minutes, so that oxidation of the fixed carbon could take place and the ash content could be determined.

2.3 Determination of calorific value by immediate analysis

To determine the heating value, an average of the results from Equations (1) and (2) was used (NHUCHHEN; ABDUL SALAM, 2012) and the values of fixed carbon (FC), ash (ASH), and volatile material (VM) were determined by immediate analysis.

$$HHV \left(\frac{MJ}{kg} \right) = 20,7999 - 0,3214 \left(\frac{VM}{FC} \right) + 0,0051 \left(\frac{VM}{FC} \right)^2 - 11,2277 \left(\frac{ASH}{VM} \right) + 4,4953 - 0,7223 \left(\frac{ASH}{VM} \right)^3$$

$$+0,0383 \left(\frac{ASH}{VM} \right)^4 + 0,0076 \left(\frac{FC}{ASH} \right) \quad (1)$$

$$HHV \left(\frac{MJ}{kg} \right) = 20,7623 - 0,4400 \left(\frac{VM}{FC} \right) + 0,071 \left(\frac{VM}{FC} \right)^2 - 5,2902 \left(\frac{ASH}{VM} \right) + 4,4921 + 0,0143 \left(\frac{FC}{ASH} \right) \quad (2)$$

2.4 Experimental design

To analyze the torrefaction of biomass, the L9 orthogonal arrangement proposed by Taguchi was used, totaling nine experiments. Four factors were considered (A, B, C, and D) that influence the torrefaction process, represented by temperature (°C), isotherm time (min), sample quantity (g), and particle size (). Based on the values for each defined level shown in Table 1, combinations between the factors were considered according to the Taguchi method (Table 2), in which the experiments were carried out considering the response variables (Yi) such as the percentage of moisture and volatiles released (UV), fixed carbon (FC), and higher calorific value (HHV) of the samples after roasting.

Table 1. Level values

Factors	Levels		
	Low	Medium	High
A - temperature	200	250	300
B - isotherm time	30	40	50
C - value	1	3	5
D - particle size	231	275	363

Table 2. Taguchi orthogonal matrix values

Experiments	factor A	factor B	C factor	D factor	Response variable UV; CF; HHV
1	200	30	1	231	Y _{i1}
2	200	40	3	275	Y _{i2}
3	200	50	5	363	Y _{i3}
4	250	30	3	363	Y _{i4}
5	250	40	5	231	Y _{i5}
6	250	50	1	275	Y _{i6}
7	300	30	5	275	Y _{i7}
8	300	40	1	363	Y _{i8}
9	300	50	3	231	Y _{i9}

To calculate the effect of the factors on the response variable, Equation (3) was used, with M_{j,k} being the average of the response variables, Y_i the response variable, J the factor (A, B, C, D), k the level of the factor (1, 2, 3) and n the number of repetitions of level k in factor j.

$$M_{j,k} = \frac{\sum Y_i}{n} \quad (3)$$

In the Taguchi method, the controlled factors are called parameters, and the uncontrolled factors are called noise. For this study, the condition "bigger is better" is considered when analyzing the influence of the factors on the signal-to-noise (S/N), where y is the output response and n is the number of replicates.

$$(S/N)_{j,k} = -10 \log \frac{\sum y^2}{n} \quad (4)$$

Analysis of variance (ANOVA) and the F-test were used to analyze the factors with a significant influence on the roasting process.

3. RESULTS AND DISCUSSION

3.1 Analysis of response variables

Table 5 shows the response variables, which refer to the percentage of moisture and volatiles released, the fixed carbon value, and the highest calorific value, obtained by the roasting tests in each experimental design test.

Table 5: Average values of the duplicate tests of the responses obtained in the L9 orthogonal arrangement.

Experiments	Response variables		
	% UV	% FC	HHV
1	6,39	15,21	17,62
2	6,79	15,66	18,08
3	4,71	16,11	18,58
4	10,18	17,01	18,09
5	12,42	18,28	18,35
6	10,18	16,49	17,85
7	25,33	22,49	17,92
8	28,08	23,98	17,98
9	34,31	26,95	17,23

Analyzing Table 5, in experiment 9, there was a greater loss of moisture and volatiles and also a higher percentage of fixed carbon, results that were expected given that a higher temperature of 300°C was used. At temperatures above 250 °C, a greater amount of hemicellulose, cellulose, and volatile organic materials begin to volatilize from the biomass in the torrefaction process. Lignin, therefore, becomes more concentrated, favoring the biomass's energy gain since its chair has ether and carbon-carbon bonds with a high energy density. (LEE *et al.*, 2012). Experiment 3, which used a rice husk sample with a mass of 5g and a particle size of 363 mm, resulted in a higher calorific value. These factors are important because the biomass's burning capacity increases or decreases in relation to its surface area.

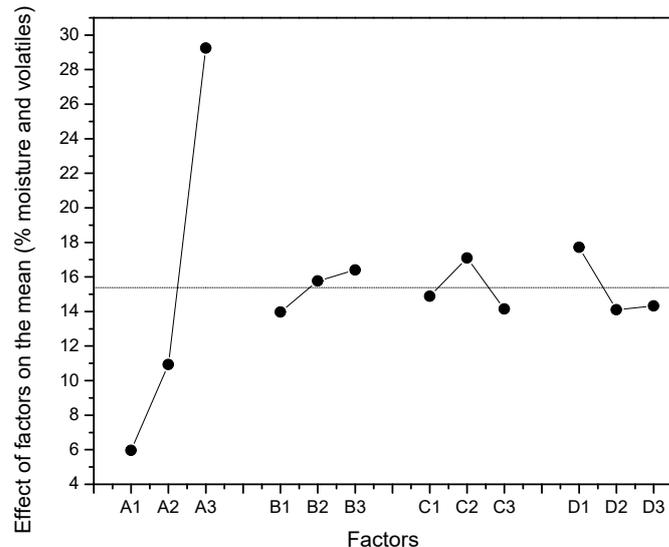
3.2 Analysis of the effects of the factors on the response variables.

Based on the results obtained for all the roasting tests, the effects of the factors on the average, Equation (3), and on the signal/noise (S/N), Equation (4), of the response variables were analyzed. The results are shown in Table 6.

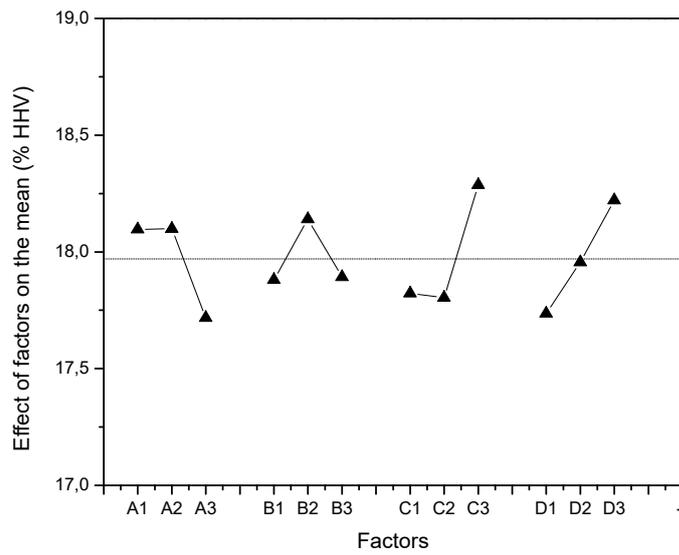
Table 6. Effects of the factors on the mean and signal/noise of the response variables.

Factors	% UV		% FC		%HHV	
	Mj,k	S/Nj,k	Mj,k	S/Nj,k	Mj,k	S/Nj,k
A1	5,96	15,40	15,66	23,89	18,10	25,15
A2	10,93	20,73	17,26	24,73	18,10	25,15
A3	29,24	29,25	24,47	27,74	17,72	24,93
B1	13,97	21,45	18,24	25,09	17,88	25,04
B2	15,76	22,50	19,31	25,57	18,14	25,16
B3	16,40	21,44	19,85	25,69	17,89	25,02
C1	14,88	21,74	18,56	25,19	17,82	25,01
C2	17,09	22,50	19,87	25,70	17,80	24,98
C3	14,15	21,14	18,96	25,47	18,29	25,24
D1	17,71	22,90	20,15	25,83	17,74	24,95
D2	14,10	21,62	18,21	25,09	17,96	25,07
D3	14,32	20,86	19,03	25,45	18,22	25,20

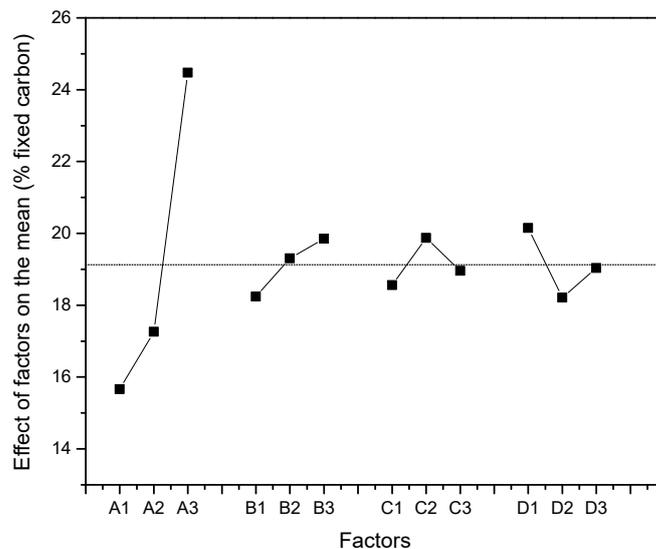
To better visualize the results, we used a graphical representation of the effects of the factors on the average, shown in Figures 1a, 1b and 1c. The overall average is represented by the dashed line.



(a)



(b)



(c)
 Figure 1: Effects of the factors on the mean of the response variables.

Figure 1 shows that the results with the best significance are those with the highest values and above the global average, since we chose to analyze the results as "bigger is better". Table 7 shows the above-average values from Figure 1, which are represented by the experiment's best-fit condition.

The best-fit condition for the higher heating value variable, Figure 1b, showed values above the average (A1A2; B2; C3; D3), which were not tested in the L9 matrix. This may be because Taguchi's method is fractional and does not consider all possible combinations of factors.

For the average responses, percentage of moisture and volatiles and fixed carbon (Figures 1a and 1c), it was possible to identify the best-fit condition, as all the values above the average were tested in experiment 9 (A1, B1, C2, and D1), making it the best experimental condition.

Table 7. Best-fit condition for the response variables.

Answer (Y) Factors on average	Factors			
	A (°C)	B (min)	C (g)	D (ϕ)
% UV	300 °C	50 min	3 g	231 mm
% FC	300 °C	50 min	3 g	231 mm
% HHV	200 / 250 °C	40 min	5 g	363 mm

3.3 Analysis of variance (ANOVA)

Based on the results obtained in the ANOVA, the factor in the experimental matrix with the greatest significance is evaluated. For the analysis, the quadratic sum of the factors (SQ), the degree of freedom (gl), the mean quadratic sum (SMQ) and the F of the F Test are used, which indicate the level of significance of the experimental parameter. Considering 95% (0.05) confidence with degree of freedom and degree of freedom of the error (n-1) equal to 2, the critical tabulated F value $F_{0.05;2;2}$ is 19, for the condition "bigger is better" the F value must be greater than 19 for the factors to be significant (BARROS NETO; SCARMINIO; ROY EDWARD BRUNS, 2010). Tables 8, 9 and 10 show the ANOVA results for the mean and signal/noise of the response variables.

Table 8. ANOVA analysis for % UV.

Source of variation	Average				S/N			
	Add	gl	SMQ	F	Add	gl	SMQ	F
SQA	901,82	2	450,91	94,43	292,70	2	146,35	132,44
SQB	9,55	2	4,78	1,00	2,21	2	1,11	1,00
SQC	14,06	2	7,03	1,47	2,79	2	1,40	1,26
SQD	24,51	2	12,25	2,57	6,37	2	3,19	2,88

SQ (A+B+C+D)	949,94	304,08
SQ error	0,00	0,00

Table 9: ANOVA analysis for % CF.

Source of variation	Average				S/N			
	Add	gl	SMQ	F	Add	gl	SMQ	F
SQA	132,27	2	66,13	48,63	24,67	2	12,34	61,68
SQB	4,04	2	2,02	1,48	0,61	2	0,30	1,51
SQC	2,72	2	1,36	1,00	0,39	2	0,20	0,98
SQD	5,65	2	2,82	2,08	0,82	2	0,41	2,04
SQ (A+B+C+D)	144,68				26,49			
SQ error	0,00				0,00			

Table 10: ANOVA analysis for HHV.

Source of variation	Average				S/N			
	Add	gl	SMQ	F	Add	gl	SMQ	F
SQA	0,29	2	0,14	2,41	0,10	2	0,05	2,65
SQB	0,13	2	0,06	1,08	0,04	2	0,02	1,03
SQC	0,45	2	0,22	3,74	0,11	2	0,06	3,24
SQD	0,35	2	0,18	2,94	0,10	2	0,05	2,74
SQ (A+B+C+D)	1,22				0,35			
SQ error	0,00				0,00			

In Table 8, considering the percentage of volatiles and moisture released, the most significant factor was the SQA for temperature, since the calculated $F > \text{critical } F$ ($450.91 > 19$), an expected result since the higher the temperature, the more moisture and volatiles are released in the roasting process. In the ANOVA result for S/N, considering the analysis of external factors, temperature is considered a significant factor, as the calculated F was greater than the critical F .

Table 9 shows the ANOVA for the fixed carbon content, and the most significant factor was also SQA. Since fixed carbon is the amount of carbon resulting after burning the biomass, temperature is considered an important factor since the higher the temperature, the more the sample is carbonized and the greater the amount of carbon remaining in the chemical structure. In the ANOVA result for S/N, considering the analysis of external factors, temperature is considered a significant factor, as the calculated F was greater than the critical F .

In the experimental conditions carried out using the ANOVA results in Table 10, for the higher heating value, no factor had a calculated F greater than the critical F , but temperature, mass, and granulometry (SQA, SQC and SQD) had a significant influence on roasting according to Phadke's criterion ($F > \text{or equal to } 2$).

4. CONCLUSIONS

In order to carry out the Taguchi experimental design, various experimental combinations were tested, using the L9 orthogonal arrangement with 4 factors at 3 levels. In the factorial effect analysis, the conditions with the best fit for the percentage of moisture and volatiles and fixed carbon were A3, B3, C2, and D1 (300°C, 50 min, 3g, 231 mm). It can also be seen that the result of the experiment carried out in this condition was the one with the highest percentage of volatilized material and fixed carbon, confirming that the parameters used in experiment 9 were the best for the roasting process for these responses.

For the highest heating value, the best fit was A1 or A2, B2, C3, D3 (200/250°C, 40 min, 5g, 363 mm) not foreseen in the L9 experimental setup used; in this case, the condition would have to be validated to confirm whether the parameters would really give a higher result than experiment 3 (18.58%), which had a higher HHV value. Finally, the analysis of variance showed that temperature was the most significant factor for good efficiency in the rice husk torrefaction process.

5. ACKNOWLEDGEMENTS

The authors would like to thank the Coordination for the Improvement of Higher Education Personnel (CAPES) for its financial support.

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