PREDICTIVE MODELLING OF BIO-OIL AND BIO-CHAR PRODUCTION FROM MICROWAVE PYROLYSIS OF VARIOUS BIOMASS

PROJECT

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CANDIDATE'S DECLARATION

We hereby certify that the work which is being presented in the project entitled "**PREDICTIVE MODELLING OF BIO-OIL AND BIO-CHAR PRODUCTION FROM MICROWAVE PYROLYSIS OF VARIOUS BIOMASS**", in the partial fulfillment of the requirement for the award of the Degree of Master of Technology in Environmental Engineering and submitted in the School of Infrastructure, Indian Institute of Technology Bhubaneswar, is an authentic record of my own work carried out during a period from July 2023 to May 2024 under the supervision of **Dr. REMYA NEELANCHERRY**, IIT Bhubaneswar. The matter presented in the project has not been submitted by me for the award of any degree of this or any other Institute/ University

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This is to certify that the above statement made by the candidates is correct to the best of our knowledge.

Signature of Supervisor (S) **Signature of H.O.S.**

ABSTRACT

Accurately predicting the properties of bio-char and bio-oil holds paramount importance for their effective utilization. However, these predictions are significantly swayed by the diverse compositions of biomass and the specific conditions of pyrolysis. Biomass, encompassing crops, wood, agricultural residues, and forestry remnants, stands as a pivotal renewable resource for the generation of sustainable bioenergy and eco-friendly materials. Microwave heating presents itself as a superior alternative to conventional heating due to its myriad advantages. Moreover, the judicious inclusion of suitable catalysts exerts a profound impact on the product selectivity during microwave pyrolysis. This research endeavour offers a novel perspective on forecasting the attributes of bio-oil derived from various biomass compositions subjected to distinct pyrolysis conditions. It employs a range of machine learning and deep learning methodologies to facilitate these predictions. By doing so, it not only enhances our understanding of bio-char and bio-oil behaviour but also contributes to the optimization of biomass-based energy production and environmentally conscious materials manufacturing.

Keywords: bio-char; machine learning; linear regression; random forest regression

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CONTENTS

LIST OF FIGURES

LIST OF TABLES

NOTATIONS

VM	Volatile Matter
ASH	Ash content
HHV	High Heating Value
RT	Reaction Time
SSA	Specific Surface Area
HR	Heating Rate

Chapter 1
INTRODUCTION

1.1 General

The excessive depletion of non-renewable fossil fuels has led to profound concerns regarding energy security and environmental degradation. Consequently, the focus has shifted towards harnessing biomass energy, which is abundant and sustainable. Thermochemical conversion stands out as an efficient and cost-effective means of transforming biomass into biofuels, which can be further synthesized into desired chemicals or directly utilized. This conversion process encompasses combustion, gasification, and pyrolysis, with pyrolysis, a thermally induced decomposition method conducted in an oxygen-free environment, emerging as a promising technique due to its simplicity and rapidity of operation. Pyrolysis holds great potential for biomass conversion, offering a pathway to address energy and environmental challenges.

Assessing the suitability of various biomass feedstocks for energy conversion processes, including combustion, pyrolysis, gasification, liquefaction, and hydrothermal processing, necessitates comprehensive characterization and experimental investigations. Among the critical properties for these thermal conversions, the Higher Heating Value (HHV), also known as gross calorific value or gross energy, plays a pivotal role. HHV quantifies the heat released when a unit mass of fuel undergoes complete combustion, producing water in a condensed state.

Traditionally, researchers measure the HHV of biomass and other fuel sources using an adiabatic oxygen bomb calorimeter, a widely accepted, precise, but time-consuming and cost-intensive method. Unfortunately, this method isn't readily accessible to all researchers interested in HHV determination for biomass. Pyrolysis, a process involving thermal decomposition in the absence of oxygen, constitutes the initial step in generating primary products for combustion and gasification processes, followed by the total or partial oxidation of intermediate products. Biomass pyrolysis yields solid char, liquid bio-oil, and gaseous fuel, with their proportions influenced by operational variables. Lower process temperatures and extended vapor residence times favour char production, while higher temperatures and longer vapor residence times enhance gas production. Optimal conditions for liquid production involve moderate temperatures and shorter vapor residence times. Much research on pyrolysis employs conventional heating

sources like electric and gas heaters. However, utilizing microwave irradiation for biomass pyrolysis shows promise due to several advantages over conventional methods.

Evaluating biomass suitability for energy conversion processes necessitates characterizing properties like HHV, while pyrolysis serves as a critical step in producing biofuels. Microwave-assisted pyrolysis presents a promising avenue for its efficiency and potential benefits compared to traditional methods. In this work, a comprehensive analysis of the bio-oil and bio-char yield, HHV and specific surface area prediction was presented. Using machine learning tools the quality will be predicted according to biomass compositions and pyrolysis conditions, and the correlation analysis will be carried out. This study provides feasible thinking for the prediction of the characteristics of bio-oil and bio-char obtained by biomass with different compositions under different pyrolysis conditions.

Chapter 2
LITERATURE REVIEW

2.1 General

Predictive modelling of bio-oil and bio-char production will require the use of machine learning algorithms and the process involved for experimentation. In this chapter we have covered the method involved for production of biofuel. It has describe about the Microwave pyrolysis process, the different feedstocks used and what features are considered important to predict the yield, HHV and specific surface area. This chapter also provides the information about the basic machine learning algorithms like linear regression and some advanced algorithms like random forest, adaptive boosting regression and artificial neural network also provides a detail overview of how this algorithms work.

2.2 Microwave Pyrolysis

Microwave pyrolysis is an innovative and efficient technique employed in the production of bio-oil and bio-char from biomass. This method leverages microwave radiation to induce thermal decomposition of biomass in the absence of oxygen, leading to the creation of valuable products.

2.2.1 Principle of Microwave Pyrolysis

Microwave pyrolysis relies on the principle of selective heating. When microwave radiation is applied to biomass, water molecules within the material absorb the microwave energy and rapidly heat up, leading to the formation of steam. As the temperature increases, the biomass undergoes pyrolysis, breaking down into its constituent components: solid bio-char, liquid bio-oil, and gaseous products.

2.2.2 Advantages of Microwave Pyrolysis

1. Speed: Microwave pyrolysis is significantly faster compared to conventional heating methods. The selective heating of water molecules within the biomass allows for rapid and uniform heating throughout the material.
2. Energy Efficiency: It is more energy-efficient because it directly targets the water content within the biomass, reducing energy wastage.
3. Enhanced Product Quality: The controlled and uniform heating in microwave pyrolysis can lead to higher yields of bio-oil and bio-char with improved quality.

4. Reduced Environmental Impact: Microwave pyrolysis can be carried out in an oxygen-free environment, reducing the production of harmful by-products and minimizing emissions.

2.2.3 Process and Products

In a typical microwave pyrolysis process:

1. Biomass is loaded into a microwave-reactive vessel.
2. Microwaves are applied, causing rapid heating of the biomass.
3. As the temperature increases, the biomass undergoes pyrolysis, releasing volatile compounds that can be condensed into bio-oil.
4. The remaining solid residue is bio-char, which is rich in carbon and can be used as a valuable soil conditioner or for carbon sequestration.
5. Gaseous by-products can also be collected and used for energy generation or other applications.

2.2.4 Applications

- Bio-Oil: The liquid bio-oil obtained from microwave pyrolysis can be a valuable feedstock for the production of biofuels, chemicals, and other high-value products. Its composition can vary depending on the biomass source and pyrolysis conditions.
- Bio-Char: Bio-char, the solid residue, finds applications in agriculture as a soil amendment to improve soil fertility, water retention, and carbon sequestration. It can also be used as a precursor for activated carbon production.

Microwave pyrolysis is a promising method for the sustainable production of bio-oil and bio-char from biomass. Its advantages in terms of speed, energy efficiency, and product quality make it an attractive option for biomass conversion, contributing to the development of renewable energy and environmentally friendly materials.

2.3 Biomass feedstocks dataset

The experimental data of bio-oil and bio-char were collected from the literature. The dataset consists of a wide range of biomass feedstocks such as commercial fuels, industrial wastes, forest wastes (including branches and leaves), energy crops and cereals, which all can be used to build a model valid in a wide range of biomass feedstocks to predict the yield, specific surface area and HHV. Table 2.1 gives the sample dataset of process parameters from the original dataset which were used for prediction.

Table 2.1. Sample Dataset of Process Parameters and target parameters

biomass type	C (%)	H (%)	N (%)	O (%)	VM (%)	Ash (%)	FC (%)	T (°C)	RT (min)	HR (°C/min)	N-char (%)	Yield-char (%)	SSA-char (m^2/g)	HHV (MJ/KG)
Durian Wood	33.77	11.40	6.06	48.77	85.80	10.52	3.68	350	180	1.94	5.12		2.57	14.31
Durian Wood	33.77	11.40	6.06	48.77	85.80	10.52	3.68	450	180	2.5	2.01		45.68	14.31
Durian Wood	33.77	11.40	6.06	48.77	85.80	10.52	3.68	550	180	3.05	0.55		221.00	14.31
marine macroalgae	39.78	6.35	5.40	48.46		24.51		200	120	1.66	5.24	78.34	1.91	0.00
marine macroalgae	39.78	6.35	5.40	48.46		24.51		400	120	3.33	4.74	63.64	70.29	0.00
marine macroalgae	39.78	6.35	5.40	48.46		24.51		600	120	5	4.47	37.96	61.81	0.00
empty fruit bunch (EFB)	53.04	6.39	0.87	39.37	82.07	4.88	13.05	500	60	8.33			1.41	16.66
empty fruit bunch (EFB)	53.04	6.39	0.87	39.37	82.07	4.88	13.05	300	120	2.5			1.42	16.66
empty fruit bunch (EFB)	53.04	6.39	0.87	39.37	82.07	4.88	13.05	700	120	5.83			1.13	16.66
rice husk (RH)	52.85	6.19	0.44	40.22	70.78	12.00	17.22	500	60	8.33			7.84	16.23
rice husk (RH)	52.85	6.19	0.44	40.22	70.78	12.00	17.22	300	120	2.5			0.39	16.23
corn straw pellets	40.02	6.01	0.88	52.86	72.29	10.02	17.68	400	30	13.3	1.29	50.17	4.26	16.61
corn straw pellets	40.02	6.01	0.88	52.86	72.29	10.02	17.68	450	30	15	1.20	49.57	12.61	16.61
Saccharina japonica algae	40.18	5.55	2.00	51.39				300	60	5			1.36	0.00
Saccharina japonica algae	40.18	5.55	2.00	51.39				450	60	7.5			210.00	0.00
Saccharina japonica algae	40.18	5.55	2.00	51.39				600	60	10			266.00	0.00
rice husk (RH)	40.65	5.95	1.07	52.19		16.53		400	60	6.66	8.65	60.57	5.49	0.00

rice husk (RH)	40.65	5.95	1.07	52.19		16.53		700	60	11.6	6.59	52.58	7.12	0.00
sewage sludge (SS)	45.74	10.76	7.04	34.91	49.36	42.02	8.63	400	60	6.66	4.83	53.84	4.39	10.26
sewage sludge (SS)	45.74	10.76	7.04	34.91	49.36	42.02	8.63	700	60	11.6	3.35	40.35	10.71	10.26
bamboo sawdust (BS)	49.51	3.42	0.77	46.02		2.04		400	60	6.66	4.43	44.51	2.78	0.00
Raw maize straw	42.19	7.21	1.28	49.19		5.31		250	60	4.16			5.20	0.00
Raw maize straw	42.19	7.21	1.28	49.19		5.31		300	60	5	1.68	36.70	7.80	0.00
Raw maize straw	42.19	7.21	1.28	49.19		5.31		500	60	8.33	1.71	28.90	33.20	0.00
Raw sugarcane bagasse	48.88	7.11	1.02	42.88		4.98		500	60	8.33	0.68	23.70	97.80	0.00
Raw sugarcane bagasse	48.88	7.11	1.02	42.88		4.98		500	480	1.04		20.30	105.40	0.00
Raw sugarcane bagasse	48.88	7.11	1.02	42.88		4.98		600	480	1.25		18.80	108.70	0.00
Raw pine sawdust	50.61	6.18	0.05	43.10		3.76		500	60	8.33	0.15	25.80	68.40	0.00
Raw pine sawdust	50.61	6.18	0.05	43.10		3.76		500	480	1.04		24.30	79.30	0.00
sewage sludge	43.97	13.67	7.24	32.84	55.80	33.70	10.50	300	10	30	8.64		14.00	11.84
sewage sludge	43.97	13.67	7.24	32.84	55.80	33.70	10.50	500	10	50	6.76		55.72	11.84
sewage sludge	43.97	13.67	7.24	32.84	55.80	33.70	10.50	600	10	60	9.20		92.45	11.84
soybean oil cake	44.48	6.28	8.21	40.49	77.50	6.47	16.03	600	60	10	8.48	29.72	9.47	16.89
soybean oil cake	44.48	6.28	8.21	40.49	77.50	6.47	16.03	800	60	13.3	5.51	27.77	5.37	16.89
Switchgrass	44.70	7.24	1.19	46.87	94.16	2.47	3.37	600	60	10	0.60	23.30	47.50	15.49
Switchgrass	44.70	7.24	1.19	46.87	94.16	2.47	3.37	800	60	13.3	0.46	21.34	62.50	15.49
Water Oak	53.65	7.45	1.05	37.84	85.80	1.87	12.33	200	60	3.33	0.87	74.88	2.92	17.01
Water Oak	53.65	7.45	1.05	37.84	85.80	1.87	12.33	400	60	6.66	0.74	38.86	17.71	17.01
Water Oak	53.65	7.45	1.05	37.84	85.80	1.87	12.33	600	60	10	0.62	27.04	31.87	17.01
Biosolids	58.49	7.71	5.70	28.11	54.67	38.19	7.14	400	60	6.66	7.29	48.74	67.29	10.61

Biosolids	58.49	7.71	5.70	28.11	54.67	38.19	7.14	600	60	10	7.54	33.01	55.42	10.61
Biosolids	58.49	7.71	5.70	28.11	54.67	38.19	7.14	800	60	13.3	6.36	30.12	53.12	10.61
goat manure	45.02	6.57	2.19	46.22	76.12	18.95	4.93	400	30	13.3	2.74	44.50	3.27	13.22
goat manure	45.02	6.57	2.19	46.22	76.12	18.95	4.93	500	30	16.6	2.75	40.60	1.68	13.22
goat manure	45.02	6.57	2.19	46.22	76.12	18.95	4.93	600	30	20	1.78	37.90	13.92	13.22
Municipal biosolids	45.18	10.63	6.95	35.71		41.30		400	60	6.66	8.61	60.57	5.49	0.00
Municipal biosolids	45.18	10.63	6.95	35.71		41.30		450	60	7.5	9.13	59.55	7.21	0.00

2.4 Machine Learning Algorithms

2.4.1 Linear Regression

Linear regression may be defined as the statistical model that analyses the linear relationship between a dependent variable with given set of independent variables. Linear relationship between variables means that when the value of one or more independent variables will change (increase or decrease), the value of dependent variable also change accordingly (increase or decrease).

Mathematically, the relationship can be represented per equation 2:

$$Y = mX + b \quad (1)$$

Where, Y is the dependent variable (to be predicted)

X is the dependent variable used to make predictions.

m is the slope of the regression line which represents the effect X on Y

b is a constant, known as the Y-intercept. If $X = 0$, Y would be equal to b.

In case the best fit line passes through origin, the equation becomes

$$Y = bX \quad (2)$$

Generalized linear regression model for any response (y) and its predictors (x_1, x_2, ..., x_n), i.e., biodiesel production process parameters in this case can be stated as:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_n x_n \quad (3)$$

Furthermore, the linear relationship can be positive or negative in nature as explained below:

2.4.1.1 Positive Linear Relationship

A linear relationship will be called positive if both independent and dependent variable increases. It can be understood with the help of following graph:

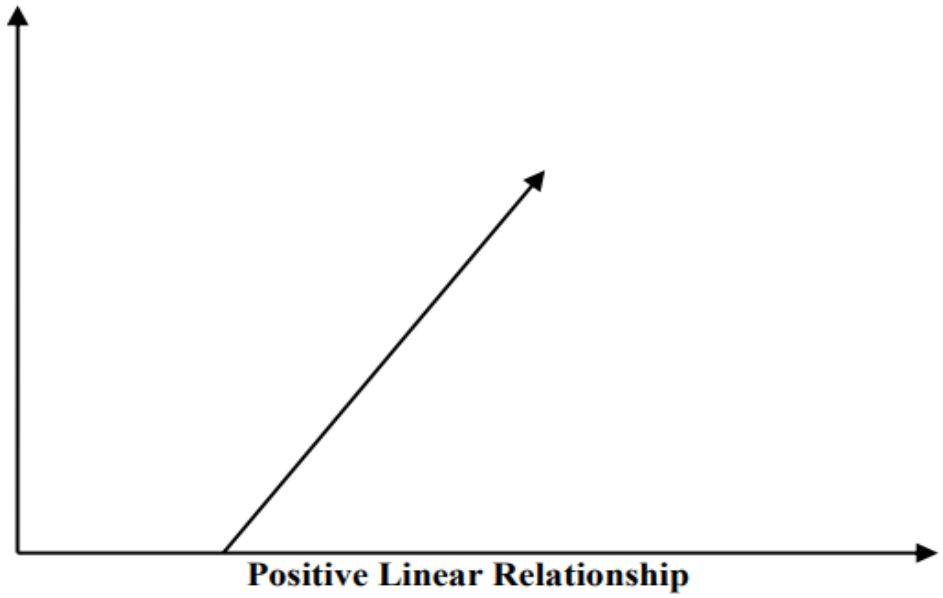

Figure 2.1 Positive Linear Relationship Graph

2.4.1.2 Negative Linear relationship

A linear relationship will be called negative if independent increases and dependent variable decreases. It can be understood with the help of following graph:

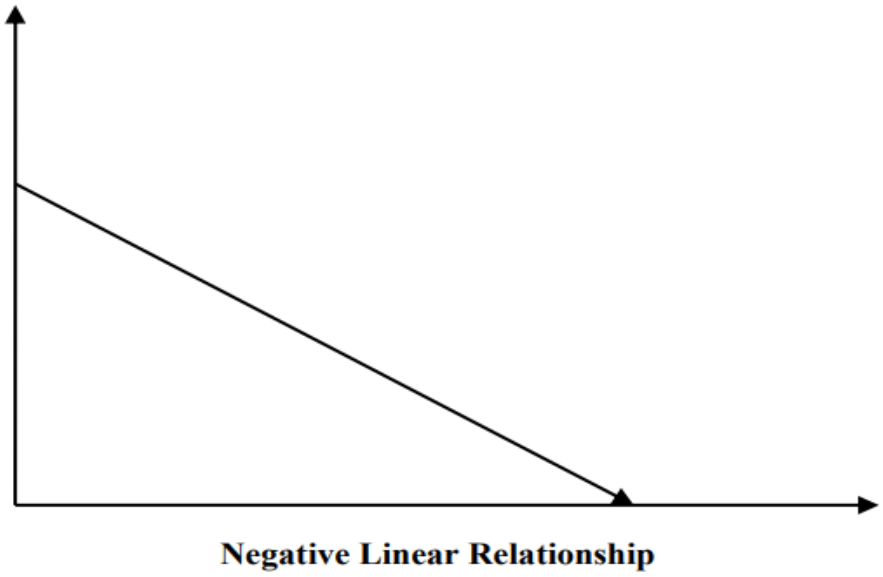

Figure 2.2 Negative Linear Relationship Curve

2.4.2 Random Forest Regression

The Random forest is based on applying bagging to decision trees, with one important extension: in addition to sampling the records, the algorithm also samples the variables. To determine how to create a sub partition of a partition A, the algorithm chooses the variable and split point by minimising a criterion such as Gini impurity with random forests, the choice of variable is limited to a random subset of variables at each stage of the algorithm. Compared to the basic tree algorithm, the random forest algorithm adds two more steps: the bagging and the bootstrap sampling of variables at each split:

1. Take a bootstrap (with replacement) subsample from the *records*.
2. For the first split, sample $p < P$ *variables* at random without replacement.
3. For each of the sampled variables X_{j1} , X_{j2} , ..., X_{jp} , apply the splitting algorithm:
    a. For each value s_{jk} of X_{jk} :
        i. Split the records in partition *A*, with $X_{j(k)} < s_{j(k)}$ as one partition and the remaining records where $X_{jk} \geq s_{jk}$ as another partition.
        ii. Measure the homogeneity of classes within each subpartition of *A*.
    b. Select the value of s_{jk} that produces maximum within-partition homogeneity of class.
4. Select the variable X_{jk} and the split value s_{jk} that produces maximum with in partition homogeneity of class.
5. Proceed to the next split and repeat the previous steps, step 2 onwards.
6. Continue with additional splits, following the same procedure until the tree is grown.
7. Go back to step 1, take another bootstrap subsample, and start the process over again.

2.4.3 Adaptive Boosting Regression

Adaptive boosting, also known as AdaBoost, is a sequential ensemble technique that develops several weak learners using different training subsets drawn at random from the original training dataset [13-14]. Weights are assigned during each training and are used when learning each hypothesis. The weights are used to compute the hypothesis error on the dataset and act as an indicator of the relative importance of each instance. After each iteration, the weights are recalculated so that instances incorrectly classified by the previous hypothesis receive higher weights. This allows the algorithm to concentrate on more difficult-to-learn instances. The algorithm's most important task is to assign revised weights to incorrectly classified instances. In regression, unlike in

classification, the instances are not correct or incorrect; rather, they constitute area-value error. The computed error can be labelled as an error or not error by comparing it to a predefined threshold prediction error, and thus the AdaBoost classifier can be used. Instances with larger errors on previous learners are more likely (i.e., have a higher probability) of being chosen to train the next base learner. Finally, the weighted average or median of the individual base learner predictions is used to provide an ensemble prediction.

2.4.4 Predictive Model Performance Evaluation Metrics

The residue, ε_i between the i^{th} original value, yi and predicted value, y_0 is calculated as,

$$\varepsilon_i = y_i - y_0 \quad (4)$$

The coefficient of determination, R^2 is computed using the y_i, y_0 and the mean of the dataset, y_m.

$$R^2 = 1 - \sum (y_i - y_{0i})^2 / \sum (y_i - y_m)^2 \quad (5)$$

The mean-absolute-error is computed based on the number of the samples, n as

$$\text{MAE} = \sum |y_i - y0_i| / n \quad (6)$$

The mean-squared-error, MSE, is computed as

$$\text{MSE} = \sum (y_i - y_{0i}) / n \quad (7)$$

The root-mean-squared-error, RMSE, is computed as

$$\text{RMSE} = (\sum (y_i - y_{0i})^2 / n \,)0.5 \quad (8)$$

The maximum error is computed as

$$\text{Max. Error } (y, y_0) = \text{max. } (|y_i - y_{0i}|) \quad (9)$$

The median error is computed as

$$\text{Med. Error } (y, y_0) = \text{median } (|y_i - y_{01}|, \ldots , |y_n - y_{0n}) \quad (10)$$

Chapter 3
METHODOLOGY

3.1 General

In this research endeavour, the primary focus revolves around the meticulous analysis of experimental data with the aim of discerning underlying patterns and trends. The data in question holds the key to understanding and potentially predicting the bio-oil and bio-char production processes. To harness the full potential of this data, a crucial preliminary step involves data pre-treatment to render it suitable for utilization within predictive models. The foundation of this research rests on the utilization of cutting-edge Machine Learning algorithms, which serve as the cornerstone of our predictive endeavours. These algorithms are strategically applied to predict the outcomes of the bio-oil and bio-char production processes. The predictive models are designed to take into account various process parameters, which play a pivotal role in determining the final outcomes.

Among the ensemble of Machine Learning algorithms at our disposal, we will employ linear regression and random forest regression. These techniques have been chosen based on careful observation and analysis of the experimental data. Their application is rooted in the belief that they possess the capability to effectively capture and extrapolate the intricate relationships between the process parameters and the production responses. To gauge the effectiveness and accuracy of these predictive models, several error metrics are employed. These metrics serve as benchmarks against which the performance of the Machine Learning regression models is evaluated. By quantifying the disparities between predicted values and actual outcomes, these error metrics provide invaluable insights into the predictive power and reliability of the models.

The significance of this research extends beyond its immediate objectives. It holds the potential to revolutionize the bio-oil and bio-char production processes. The ability to predict these processes with a high degree of accuracy can pave the way for enhanced efficiency, reduced costs, and environmentally friendly production methods. Moreover, the insights gleaned from this research can be extrapolated to other domains, demonstrating the broader applicability of Machine Learning in industrial and scientific endeavors. In conclusion, this research embarks on a journey into the realm of data-driven analysis and prediction in the context of bio-oil and bio-char production processes. Through the meticulous application of Machine Learning algorithms and comprehensive evaluation using error metrics, we aim to unlock the secrets hidden within

the experimental data. Ultimately, this research has the potential to reshape the landscape of biofuel production and contribute to a more sustainable and eco-conscious future.

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