Journal of Energy, Environmental & Chemical Engineering
2021; 6(4): 114-123
http://www.sciencepublishinggroup.com/j/jeece
doi: 10.11648/j.jeece.20210604.13
ISSN: 2637-4331 (Print); ISSN: 2637-434X (Online)

Optimization of Selected Pyrolysis Parameters in Pyroligneous Acid Production Rate and Quality from Acacia Twigs

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To cite this article:
Baqe Sharu Doti, Samwel Nyakach, Jane Nyaanga, Oscar Ingasia, Daudi Nyaanga. Optimization of Selected Pyrolysis Parameters in Pyroligneous Acid Production Rate and Quality from Acacia Twigs. *Journal of Energy, Environmental & Chemical Engineering*. Vol. 6, No. 4, 2021, pp. 114-123. doi: 10.11648/j.jeece.20210604.13

Received: September 25, 2021; **Accepted**: October 12, 2021; **Published**: October 21, 2021

Abstract: Biomass material is a renewable source of energy which are readily available and being produced in large quantities as most of it goes to waste. These materials can be recovered through pyrolysis process in order to produce usable products like biochar and pyroligneous acid. These products can be used as bio-fertilizer and bio-pesticides. The aim of this research was to optimize the selected pyrolysis parameters in pyroligneous acid production rate and quality from acacia twigs. The parameters varied were feedstock moisture content (10%, 15% and 20%), Pyrolysis residence time (90 minutes, 135 minutes and 180 minutes) and chimney inclination angle (30°, 45° and 60°). Smoke condensation system also known as heat exchanger was used for condensing the pyrolysis smoke in to pyroligneous acid. Response Surface Methodology technique by using *Box-Behnken Design* was used to develop a mathematical equation to predict the production rate and quality of the pyroligneous acid with respect to varied parameters which was later optimized to determine the optimal conditions for pyroligneous acid production rate and quality. The pyroligneous acid quality was based on its pH and density. The combined optimal conditions were 20% feedstock moisture content, 137.27 min pyrolysis residence time and 60° chimney inclination angle resulting to a density of 1.03 gcm^{-3}, pH of 3.01 and production rate of 0.19 kg/min (26.08%). The mathematical equation developed had a composite desirability of 0.9663 for pyroligneous acid production rate at p-value ≤0.05 which made it viable. These research findings are of importance since pyrolysis of the biomass material will maintain a balance in the environment and also serve as a source of livelihood when the products are sold as bio-fertilizer or bio-pesticides.

Keywords: Biomass, Pyrolysis, Pyroligneous Acid, Optimization, Heat Exchanger, Response Surface Methodology

1. Introduction

Biomass being a renewable source of energy and environmental friendly can be converted to liquid fuels, bio-ethanol and chemicals through biochemical and thermochemical processes [5]. Utilization of biomass energy has been put in to considerations with increase in environmental pollution and shortage of fossil fuels and it has been reported that the estimated potential of annual biomass production from agriculture and forestry is approximately 1.08 x 10^{11} tons [8]. Biomass produces a zero net emissions due to carbon offsetting which makes them have less effect on the ozone [17]. Therefore, there is need for diversified utilization of biomass and improvement in conversion technologies that is being used for converting biomass material in to high value added products.

Pyrolysis which can be defined as the direct thermal decomposition of biomass in absence of oxygen is an efficient way to facilitate the comprehensive utilization of biomass material and the process produces high value biochar, bio oil and non-condensable gases [13]. Pyrolysis process consists of primary and secondary stages which involves devolatisation of the main components and thermal cracking of heavy compounds in to gases such as carbon monoxide, carbon dioxide, methane and hydrogen [1]. The process results to formation of various products and has minimal emissions of greenhouse gases to the atmosphere [4]. Bio-oil which is the dark brown liquid obtained after pyrolysis process has two layers where the top layer is in aqueous phase with low viscosity and large amount of water soluble compounds known as pyroligneous acid and the bottom layer consisting of organic material with high viscosity referred to as tar [13].

Medeiros et al [9] reported that about 70% of the carbonization products are released as volatiles and this is a proof that there is need for a desirable technologies which can convert these substances in to usable products or heat through pyrolysis in a carbonization kiln hence reducing air pollution generated through traditional charcoal making process. Burning biomass directly results to minimal energy transformation efficiency since it has low heating value [17].

Pyroligneous acid is a natural distillate extracted from the pyrolysis process as a by-product of charcoal production and used as plant growth regulator and pesticides hence improving the quality and medicinal value of fruits and vegetables [14]. In producing pyroligneous acid, the pyrolysis smoke is condensed and it can be generated from any type of organic waste that is readily available. Browning et al [4] reported that pyroligneous acid is environmental friendly, economically viable, socially acceptable and used in many parts of the world especially in developing countries. The product has more than 200 natural compounds with its principal components being acetic acid and phenols which can be used as soil disinfectant to increase crop yield while utilizing less water [15]. The quality and the yield of the product depends on the type of pyrolysis process, biomass and operating parameters such as pyrolysis temperature, heating rate, sweep gas flow rate, feedstock type and pyrolysis residence time [6]. Lu et al [7] reported that a good quality pyroligneous acid should have a pH that ranges between 2 and 4. Pyroligneous acid should have a standard specific gravity around 1.010 to 1.050, pale yellowish color, reddish brown, marked smoky odor, the dissolved tar content of less than 3%, exhibit transparency without any suspended solid matter and ignition residue of less than 0.2% [16].

Optimization using Response Surface Methodology is applied when the response of interest is affected by different variables. The objective of this mathematical and statistical technique is to determine the optimal operating conditions of the system, either by maximizing or minimizing the response to reach a specific value and identify a suitable approximations for the relationship among the variable through polynomial regressions [3].

The ever growing population is a threat to food security and this has prompted farmers to use chemical fertilizers and synthetic pesticides to produce food from crops. This results to air, soil and water pollution further depleting ozone layer resulting to global warming. Over using of these chemicals has made pests develop resistance to it [2]. To avoid this and to save the environment, there is need to produce fertilizers and pesticides from the pyrolysis of biomass material.

In this research, acacia twigs were used as feedstock material for slow pyrolysis process. The twigs are being burnt directly as firewood hence releasing smoke to the atmosphere yet it can be recovered in to a clean usable product known as pyroligneous acid. A pyrolysis system consisting of a carbonization kiln and the smoke condensation system was developed in order to carry out the study. Therefore, the aim of this study was to optimize the selected pyrolysis parameters in pyroligneous acid production rate and quality from acacia twigs. The pyrolysis parameters varied were feedstock moisture content, pyrolysis residence time and chimney inclination angle. A mathematical equation to predict the production rate and quality of pyroligneous acid was developed for all the varied parameters and optimized using minitab18 software. The pyroligneous acid production rate was significantly influenced by pyrolysis residence time and chimney inclination angles while quality was influenced by the pyrolysis residence time.

2. Materials and Methods

2.1. Research Site

The research was carried out at Egerton University energy laboratory, Department of Agricultural Engineering. Egerton University is located in Njoro, Nakuru County (Latitude 0°22’11.0”S Longitude 35°55’58.0”E). Laboratory analysis was carried out at Egerton University’s Chemistry and Food Science and Technology Department.

2.2. Experimental Setup

Acacia twigs were sun dried to a moisture content of 20%, 15% and 10% and this was determined using a muffle furnace. The material for each moisture content was weighed to 100 kg of mass and then fed in to the carbonization kiln where it was carbonized at a pyrolysis temperature of approximately 400°C. The experiment was carried out at pyrolysis residence times of 90 minutes, 135 minutes and 180 minutes and chimney inclination angles of 30°, 45° and 60°. The carbonization kiln used had a height of 1 m and diameter of 0.5 m. The smoke outlet chimney from the carbonization kiln which had an outer diameter of 3 inch (0.0762 m) was connected to a smoke condensation system of height 1 m and diameter 0.32 m. Water was used as a cooling agent to condense the pyrolysis smoke into a pyroligneous acid. The pyrolysis system used is as shown in (Figure 1).

The production rate of pyroligneous acid obtained was determined using (Equation 1).

$$y(t) = \frac{M_{pa}}{x_2} \quad (1)$$

Where;

$y(t)$ = pyroligneous acid production rate (kg/min)
M_{pa} = mass of the pyroligneous acid obtained (kg)
x_2 = pyrolysis residence time (min)
The pyroligneous acid obtained from each selected parameter was weighed to determine its mass and the volume was determined using a graduated cylinder. The density was then calculated from the ratio of its mass to the volume. A pH meter was then used to determine the pH of the product.

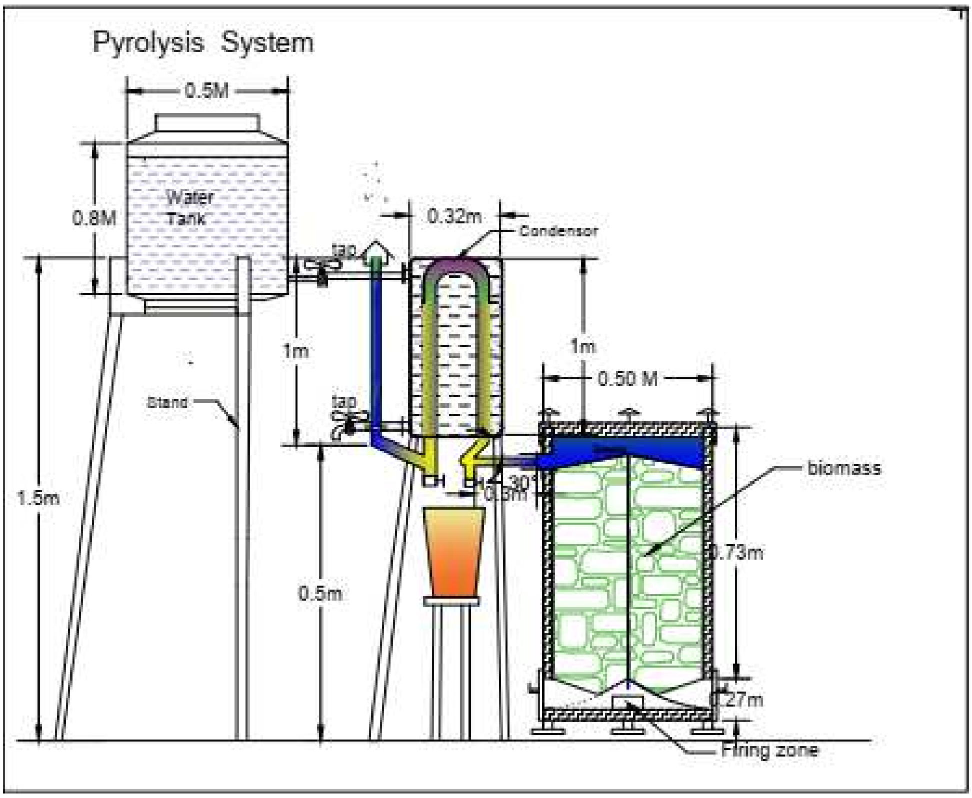

Figure 1. *The pyrolysis System.*

2.3. Experimental Design

Response Surface Methodology technique was used to optimize the selected pyrolysis parameters for optimum production rate and quality. It ensured that the correlation between the response (pyroligneous acid production rate and quality) and the experimental variables (pyrolysis residence time, feedstock moisture content and chimney inclination angle) were examined. *Box-Behnken Design* was chosen for the experiment design since it can observe the interaction effect of the independent variables on the response [11]. The three factors and the design point for Response Surface Methodology (low, medium and high) in coded and un-coded independent variables as shown in (Table 1) was used. The technique was then applied to analyze the effect of independent variables on the response parameter and this was done by matching the responses studied (y(t), ρ and pH) using the second order polynomial equation which is given in (Equation 2).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=2}^{k} \beta_{ii} x_i^2 + \sum_i \sum_j \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

Where; β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients for intercepts, linear, quadratic and interaction terms respectively, x_i and x_j are the independent variables and ε is error term.

Table 1. *The level of variable used for Box-Behnken Design.*

Independent variables	**Symbol**	**Coded variable level**		
		Low	**Medium**	**High**
		-1	**0**	**1**
Feedstock moisture content (%)	x_1	10	15	20
Pyrolysis residence time (min)	x_2	90	135	180
Chimney inclination angle (°)	x_3	30	45	60

The experiments were carried out according to the design point with the independent variables including the feedstock moisture content at 20%, 15% and 10%, pyrolysis residence times of 90 minutes, 135 minutes and 180 minutes and the chimney inclination angles of 30^0, 45^0 and 60^0 with a total of 15 experimental runs.

2.4. Statistical and Optimization Analysis

Analysis of Variance (ANOVA) was used to analyze the regression model developed and all the treatments were subjected to a p-level ≤ 0.05. The optimum conditions for the three variables namely; feedstock moisture content, pyrolysis residence time and chimney inclination angle was obtained using statistical analysis data. Minitab18 software was then used to fit the mathematical equation developed and prepare the response surfaces and surface plots.

3. Results and Discussion

3.1. Experimental Results

The results obtained showed that as the pyrolysis residence time and feedstock moisture content decreased, the production rate of pyroligneous acid increased while an increase in chimney inclination angle resulted in to decrease in production rate as shown in (Table 2).

Table 2. *Effect of selected pyrolysis parameters on the pyroligneous acid production rate.*

x_1	x_2	x_3	y(t)
		30	0.34
20	90	45	0.32
		60	0.30
		30	0.22
20	135	45	0.22
		60	0.21
		30	0.17
20	180	45	0.16
		60	0.15
		30	0.35
15	90	45	0.34
		60	0.33
		30	0.22
15	135	45	0.21
		60	0.20
		30	0.17
15	180	45	0.15
		60	0.14
		30	0.34
10	90	45	0.32
		60	0.31
		30	0.22
10	135	45	0.21
		60	0.20
		30	0.16
10	180	45	0.15
		60	0.14

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°), y(t) = production rate (%).

The production rate obtained at feedstock moisture content of 20% was 0.34 kg/min (30.60%), 0.32 kg/min (28.80%) and 0.30 kg/min (27.00%) for pyrolysis residence time of 90 minutes and chimney inclination angle of 30°, 45° and 60°, respectively. The highest production rate obtained was 0.35 kg/min (31.50%) at feedstock moisture content of 15%, pyrolysis residence time of 90 minutes and chimney inclination angle of 30° and the lowest was 0.14 kg/min (25.20%) at feedstock moisture content of 10%, pyrolysis residence time of 180 minutes and chimney inclination angle of 60°. The highest production rate obtained was slightly lower than the one obtained by [8] (34.09%) and this is attributed to difference in feedstock material (Chinese Fir waste), pyrolysis residence time (30 minutes) and pyrolysis temperature (250°C). An increase in pyrolysis residence time results to increase in the production of uncondensed gas and the formation of tar thus resulting to decrease in the pyroligneous acid production rate [6]. The chimney inclination angle also had an effect on the yield of pyroligneous acid due to variation in the speed flow of the pyrolysis smoke in the chimney. The speed flow of the pyrolysis smoke was determined using an anemometer and the results obtained were 0.45 m/s, 0.58 m/s and 0.72 m/s for chimney inclination angle of 30°, 45° and 60°, respectively. This shows that at lower speed, the smoke condensation system had enough time to condense the pyrolysis smoke hence resulting to higher yield of the product. The pyroligneous acid obtained is as shown in (Figure 2).

Figure 2. *The pyroligneous acid obtained.*

The density of pyroligneous acid was observed to remain constant for each feedstock moisture content, pyrolysis residence time and chimney inclination angle but its pH decreased with an increase in pyrolysis residence time as shown in (Table 3). Similar results were obtained by [7] after producing pyroligneous acid from *cunninghamia lanceolate* waste with its pH reducing from 3.47-2.49 and density remaining stable at 1.010 g/ml to 1.025 g/ml.

Table 3. *Effect of selected pyrolysis parameters on the quality of pyroligneous acid.*

x_1	x_2	x_3	ρ (g/cm³)	pH
		30	1.01	3.09
20	90	45	1.04	3.08
		60	1.02	3.07
		30	1.03	3.03
20	135	45	1.02	3.02
		60	1.04	3.03
		30	1.02	2.90
20	180	45	1.01	2.90
		60	1.02	2.80
		30	1.01	3.08
15	90	45	1.01	3.06
		60	1.01	3.08
		30	1.04	3.04
15	135	45	1.01	3.04
		60	1.01	3.03
		30	1.03	2.90
15	180	45	1.05	2.80
		60	1.03	2.80
		30	1.03	3.06
10	90	45	1.02	3.09
		60	1.04	3.08
		30	1.04	3.03
10	135	45	1.04	3.03
		60	1.04	3.03
		30	1.03	2.78
10	180	45	1.03	2.93
		60	1.02	2.93

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°).

The pyroligneous acid density obtained was 1.01 gcm^{-3}, 1.03 gcm^{-3} and 1.02 gcm^{-3} while the pH was 3.09, 3.03 and 2.90 for pyrolysis residence times of 90 minutes, 135 minutes and 180 minutes respectively at feedstock moisture content of 20% and chimney inclination angle of 30°. Similar trends were obtained when the feedstock material was carbonized at feedstock moisture content of 15% and 10%. The pH and density of the pyroligneous acid obtained agrees with the results obtained by [7]. This shows that the product obtained is of high quality and can be used as bio fertilizer or bio pesticide since it falls within the required range as reported by [16] and [7].

3.2. Statistical and Regression Analysis of Pyroligneous Acid Model

The statistical analysis was done in this study to determine the relationship that exists between the varied parameters namely; feedstock moisture content, pyrolysis residence time and chimney inclination angle and the response (pyroligneous acid production rate and quality). The result obtained is illustrated in (Tables 4, 5 and 6).

Table 4. *Reduced regression model co-efficient for pyroligneous acid production rate.*

Term	Co-efficient	SE-Coefficient	t-value	*p*-value
Constant	0.2100	0.00408	51.44	0.000
x_1	0.00250	0.00250	1.00	0.363
x_2	-0.08750	0.00250	-35.00	0.000
x_3	-0.01000	0.00250	-4.00	0.010
x_1*x_1	-0.00375	0.00368	-1.02	0.355
x_2*x_2	0.03125	0.00368	8.49	0.000
x_3*x_3	0.00625	0.00368	1.70	0.150
x_1*x_2	0.00250	0.00354	0.71	0.511
x_1*x_3	0.00250	0.00354	0.71	0.511
x_2*x_3	-0.00250	0.00354	-0.71	0.511

$\alpha = 0.05$, $R^2 = 99.62\%$, Adj $R^2 = 98.94\%$, Error term = 0.0071
x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°).

The statistical significance of the factors was examined using *p*-values at $\alpha = 0.05$ generating regression co-efficient and ANOVA for the quadratic model of the response surfaces. The factors which had *p*-values less than 0.05 were termed significant hence otherwise. The result obtained had a high R^2 of 0.99 and small error of 0.0071. This shows that the linear, quadratic and interaction of all the factors, regression and ANOVA models was well fit. From the model obtained, it can be noted that x_2 ($p = 0.000$) and x_3 ($p = 0.010$) have significant effect on the production rate of pyroligneous acid since $p<0.05$ while x_1 ($p = 0.363$) had no significant influence. The quadratic term x_2^2 ($p = 0.000$) had significant effect on the production rate. Conversely, the quadratic terms for x_1^2 ($p = 0.355$) and x_3^2 ($p = 0.150$) had no significant influence on the pyroligneous acid production rate since $p>0.05$. The interaction term x_1 and x_2 ($p = 0.511$), x_1 and x_3 ($p = 0.511$) and x_2 and x_3 ($p = 0.511$) had no significant effect on the production rate of pyroligneous acid.

Table 5. *Reduced regression model co-efficient for the pyroligneous acid density.*

Term	Co-efficient	SE-Coefficient	t-value	*p*-value
Constant	1.01000	0.00563	179.48	0.000
x_1	-0.00125	0.00345	-0.36	0.732
x_2	0.00250	0.00345	0.73	0.501
x_3	0.00125	0.00345	0.36	0.732
x_1*x_1	0.01625	0.00507	3.20	0.024
x_2*x_2	-0.00125	0.00507	-0.25	0.815
x_3*x_3	0.01125	0.00507	2.22	0.077
x_1*x_2	-0.01000	0.00487	-2.05	0.095
x_1*x_3	0.00250	0.00487	0.51	0.630
x_2*x_3	0.0000	0.00487	0.000	1.000

$\alpha = 0.05$, $R^2 = 79.87\%$, Adj $R^2 = 43.64\%$, Error term = 0.0098
x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°).

The results obtained from (Table 5) shows that only quadratic term x_1^2 (p = 0.024) had significant influence on the density of pyroligneous acid while all other terms were insignificant since they all had p-values greater than 0.05.

Table 6. *Reduced regression model co-efficient for the pyroligneous acid pH.*

Term	Co-efficient	SE-Coefficient	t-value	*p*-value
Constant	3.0400	0.0147	206.53	0.000
x_1	-0.0050	0.00901	-0.55	0.603
x_2	-0.1000	0.00901	-11.09	0.000
x_3	-0.01250	0.00901	-1.39	0.224
x_1*x_1	0.0125	0.0133	0.94	0.389
x_2*x_2	-0.0525	0.0133	-3.96	0.011
x_3*x_3	-0.0225	0.0133	-1.70	0.151
x_1*x_2	-0.0050	0.0127	-0.39	0.711
x_1*x_3	0.0000	0.0127	0.00	1.000
x_2*x_3	-0.0250	0.0127	-1.96	0.107

$\alpha = 0.05$, $R^2 = 96.75\%$, Adj $R^2 = 90.89\%$, Error term = 0.0255
x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°).

The analyzed data from Table 6 shows that x_2 ($p = 0.000$) and quadratic term x_2^2 ($p = 0.011$) had significant influence on the pyroligneous acid pH while all the other terms had no significant influence having *p*-values greater than 0.05.

A mathematical equation was developed from un-coded co-efficient terms in order to carry out further investigation on the production rate and quality of pyroligneous acid. The equation was developed using minitab18 software where *Box-Behnken Design* was used. Omulo et al [10] reported that positive term in the equation indicates increase in the response with increase of the variable while a negative term indicates a decrease in the response with increase in the variable. The equations developed considered all the three varied pyrolysis parameters (feedstock moisture content, pyrolysis residence time and chimney inclination angle) with response to the pyroligneous acid production rate, density and pH. The mathematical equations developed to predict the pyroligneous acid production rate, density and pH is as shown in (Equations 3, 4 and 5), respectively.

$$y(t) = 8.213 \times 10^{-1} + 2.000 \times 10^{-3}x_1 - 6.111 \times 10^{-3}x_2 - 3.170 \times 10^{-3}x_3 - 1.500 \times 10^{-4}{x_1}^2 + 1.500 \times 10^{-5}{x_2}^2 + 2.800 \times 10^{-5}{x_3}^2 + 1.100 \times 10^{-5}x_1x_2 + 3.300 \times 10^{-5}x_1x_3 - 4.000 \times 10^{-6}x_2x_3 \quad (3)$$

$$\rho = 1.171 \times 10^{0} - 1.525 \times 10^{-2}x_1 + 8.890 \times 10^{-4}x_2 - 4.920 \times 10^{-3}x_3 + 6.500 \times 10^{-4}{x_1}^2 - 1.000 \times 10^{-6}{x_2}^2 + 5.000 \times 10^{-5}{x_3}^2 - 4.400 \times 10^{-5}x_1x_2 + 3.300 \times 10^{-5}x_1x_3 \quad (4)$$

$$pH = 2.560 \times 10^{0} - 1.300 \times 10^{-2}x_1 + 6.780 \times 10^{-3}x_2 + 1.317 \times 10^{-2}x_3 + 5.000 \times 10^{-4}{x_1}^2 - 2.600 \times 10^{-5}{x_2}^2 - 1.000 \times 10^{-4}{x_3}^2 - 2.200 \times 10^{-5}x_1x_2 - 3.700 \times 10^{-5}x_2x_3 \quad (5)$$

Where;
y(t) = production rate (kg/min)
ρ = density (gcm^{-3})
pH = pyroligneous acid pH
x_1 = feedstock moisture content (%)
x_2 = pyrolysis residence time (min)
x_3 = chimney inclination angle (°)
The regression models developed was analyzed using ANOVA and the result obtained is as shown in (Tables 7, 8 and 9).

The analyzed data shows that linear ($p = 0.000$) and quadratic regression ($p = 0.002$) were significant since $p<0.05$ while the interaction ($p = 0.698$) regression was insignificant. The surface plot graphs for (Equation 3) was plotted as shown in (Figure 3).

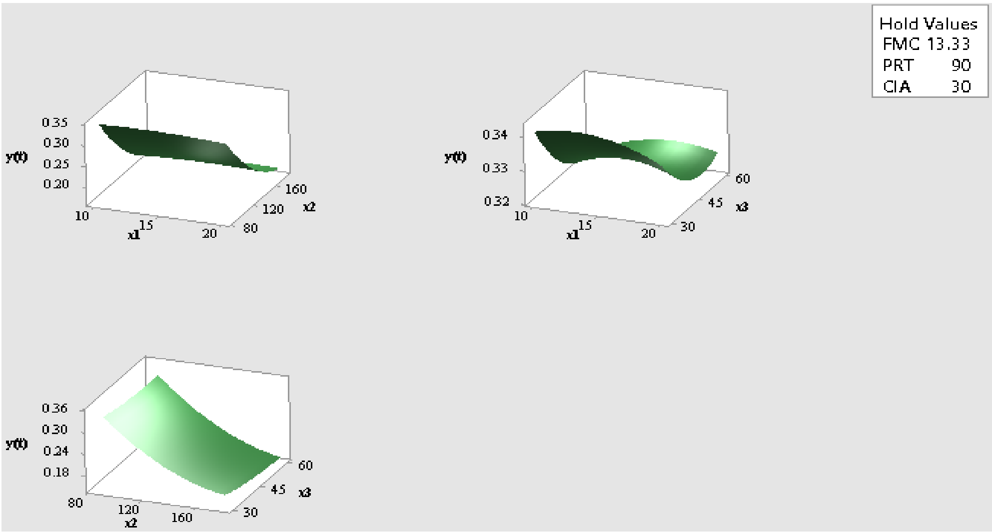

Figure 3. *Surface plots for the pyroligneous acid production rate.*

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°), y(t) = production rate (kg/min), FMC = optimum feedstock moisture content (%), PRT = optimum pyrolysis residence time (min), CIA = optimum chimney inclination angle (°).

Table 7. *ANOVA for the pyroligneous acid production rate regressions.*

Source	DF	SS	MS	F-Value	p-value
Linear	3	0.062100	0.020700	414.00	0.000
Quadratic	3	0.003815	0.001272	25.43	0.002
Interaction	3	0.000075	0.000025	0.50	0.698
Error	5	0.000250	0.000083		
Total	14	0.066240			

Table 8. *ANOVA for the pyroligneous acid density regressions.*

Source	DF	SS	MS	F-Value	p-value
Linear	3	0.000075	0.000025	0.26	0.849
Quadratic	3	0.001385	0.000462	4.86	0.061
Interaction	3	0.000425	0.000142	1.49	0.324
Error	5	0.000475	0.000095		
Total	14	0.002360			

The linear ($p = 0.849$), quadratic ($p = 0.061$) and interaction ($p = 0.324$) regressions were insignificant since they have p-values greater than 0.5. The surface plot graphs for (Equation 4) was plotted as shown in (Figure 4).

Table 9. *ANOVA for the pyroligneous acid pH regressions.*

Source	DF	SS	MS	F-Value	p-value
Linear	3	0.081450	0.027150	41.77	0.001
Quadratic	3	0.012633	0.004211	6.48	0.036
Interaction	3	0.002600	0.000867	1.33	0.362
Error	5	0.003250	0.000650		
Total	14	0.099933			

The analyzed data from (Table 9) shows that both linear and quadratic regressions were significant having *p*-value less than 0.05 while the interaction regression was insignificant. The surface plot graphs for (Equation 5) was plotted as shown in (Figure 5).

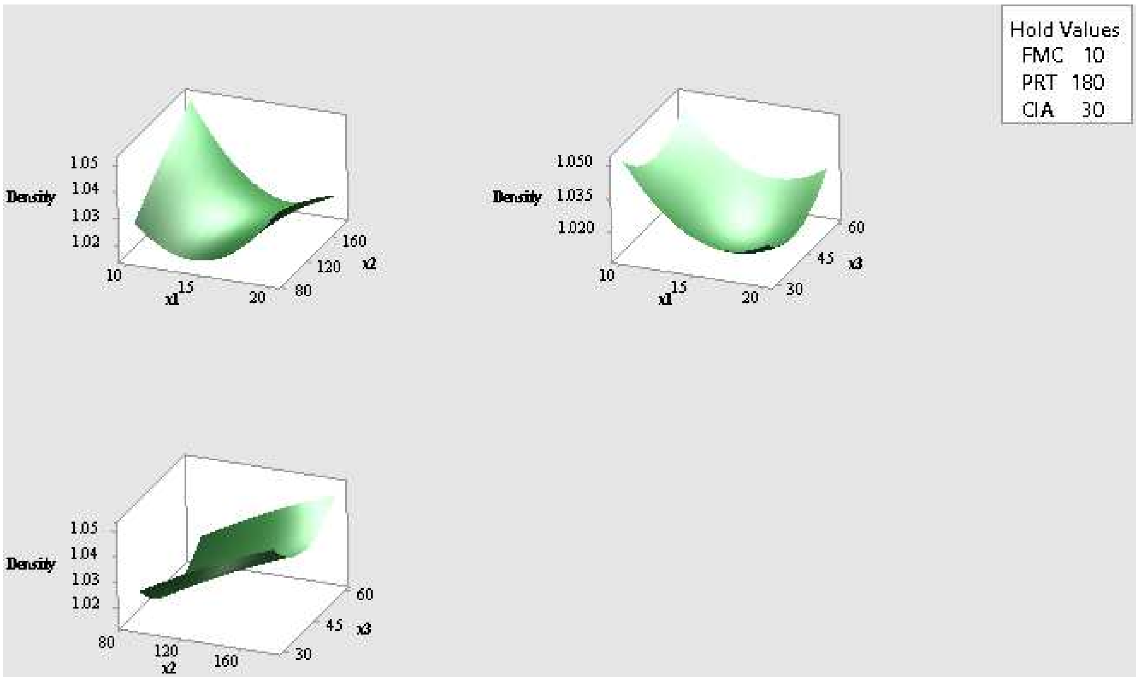

Figure 4. *Surface plots for the pyroligneous acid density.*

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°), Density = pyroligneous acid density (gcm^{-3}), FMC = optimum feedstock moisture content (%), PRT = optimum pyrolysis residence time (min), CIA = optimum chimney inclination angle (°).

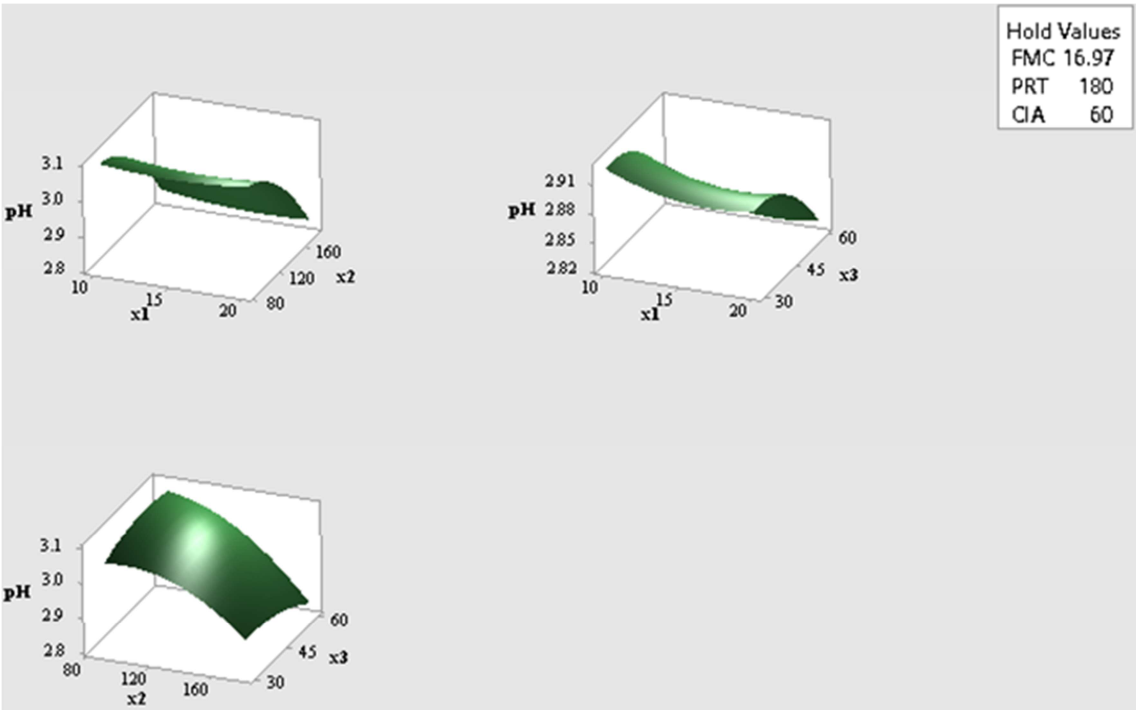

Figure 5. *Surface plots for the pyroligneous acid pH.*

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°), FMC = optimum feedstock moisture content (%), PRT = optimum pyrolysis residence time (min), CIA = optimum chimney inclination angle (°).

The predicted values for pyroligneous acid production rate, density and pH were determined using (Equations 3, 4 and 5) and then compared with the observed ones as shown in (Tables 10 and 11).

***Table 10.** The pyroligneous acid production rate for the observed and predicted value.*

Std. Order	Run	x_1	x_2	x_3	Pyroligneous acid Observed (kg/min)	Pyroligneous acid Predicted (kg/min)
13	1	0	0	0	0.21	0.20
2	2	1	-1	0	0.32	0.32
7	3	-1	0	1	0.20	0.19
14	4	0	0	0	0.21	0.20
3	5	-1	1	0	0.15	0.13
8	6	1	0	1	0.21	0.20
1	7	-1	-1	0	0.32	0.32
5	8	-1	0	-1	0.22	0.21
6	9	1	0	-1	0.22	0.21
11	10	0	-1	1	0.31	0.32
10	11	0	1	-1	0.17	0.16
9	12	0	-1	-1	0.35	0.34
4	13	1	1	0	0.16	0.14
12	14	0	1	1	0.14	0.13
15	15	0	0	0	0.21	0.20

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°).

The normal probability and residual versus fitted value plots developed at α = 0.05 was developed for pyroligneous acid production rate as shown in (Figure 6).

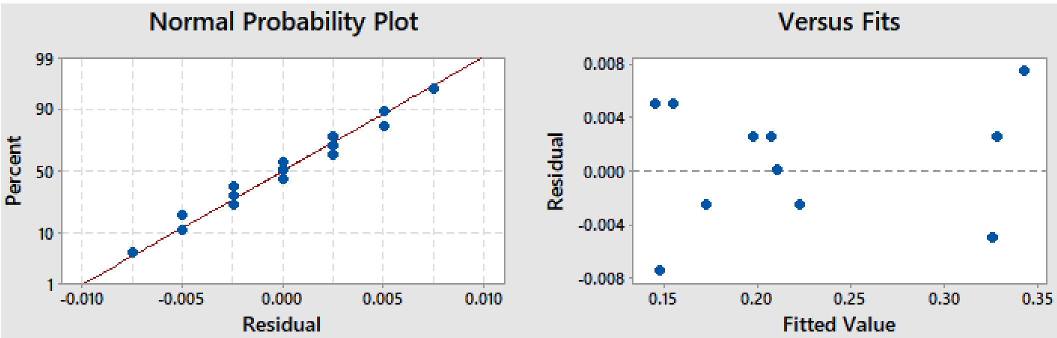

***Figure 6.** Residual plots for the pyroligneous acid production rate.*

***Table 11.** The pyroligneous acid quality for the observed and predicted value.*

Std. Order	Run	x_1	x_2	x_3	ρ (g/cm^3) Observed	ρ (g/cm^3) Predicted	pH Observed	pH Predicted
13	1	0	0	0	1.01	1.00	3.04	3.04
2	2	1	-1	0	1.04	1.03	3.08	3.10
7	3	-1	0	1	1.04	1.03	3.03	3.02
14	4	0	0	0	1.01	1.00	3.04	3.04
3	5	-1	1	0	1.03	1.03	2.93	2.91
8	6	1	0	1	1.04	1.03	3.03	3.01
1	7	-1	-1	0	1.02	1.01	3.09	3.10
5	8	-1	0	-1	1.04	1.03	3.03	3.05
6	9	1	0	-1	1.03	1.03	3.03	3.04
11	10	0	-1	1	1.01	1.02	3.08	3.08
10	11	0	1	-1	1.03	1.01	2.90	2.90
9	12	0	-1	-1	1.01	1.01	3.08	3.05
4	13	1	1	0	1.01	1.01	2.90	2.89
12	14	0	1	1	1.03	1.01	2.80	2.83
15	15	0	0	0	1.01	1.00	3.04	3.04

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°).

The normal probability and residual versus fitted value plot for the pyroligneous acid density and pH is as shown in (Figures 7 and 8).

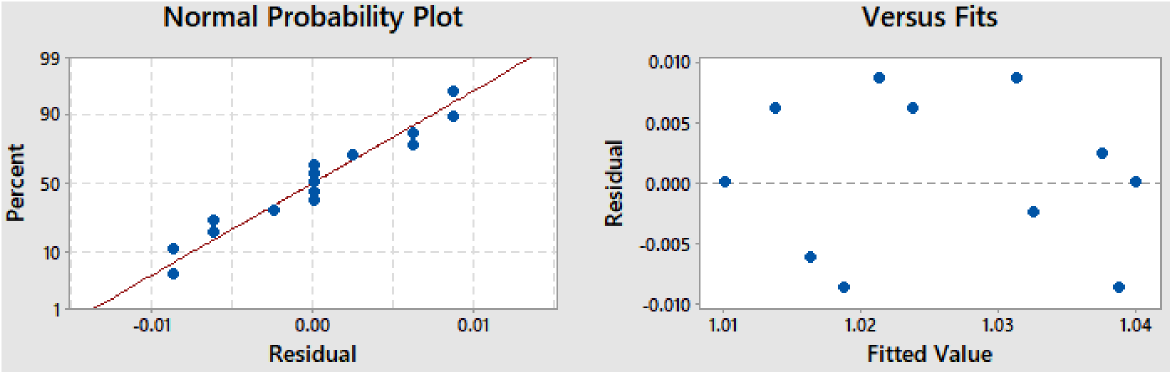

Figure 7. *Residual plots for the pyroligneous acid density.*

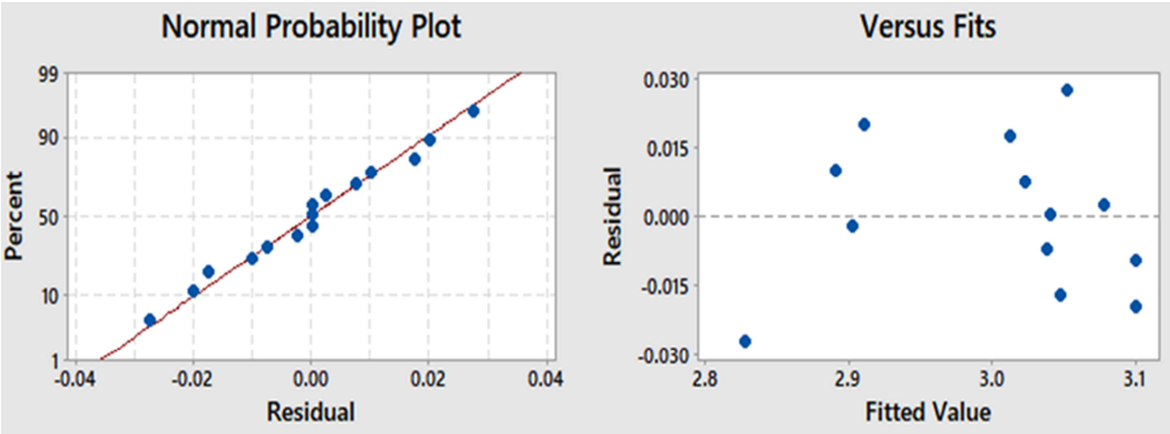

Figure 8. *Residual plots for the pyroligneous acid pH.*

From the plots in (Figures 6, 7 and 8), it can be observed that the points on the plot were a straight line and this indicates normality in the residual distribution. It can also be observed that there is a random pattern of residuals on either sides of 0.00 from the residual versus fitted value plot. This indicates that the error was randomized and the predictor mathematical equation was efficient in analyzing the effects of feedstock moisture content, pyrolysis residence time and chimney inclination angle on the production rate, density and pH of pyroligneous acid.

3.3. Optimization Analysis

(Equation 3) was maximized to determine the optimal condition for the pyroligneous acid production rate resulting to y(t) of 0.34 kg/min (30.60%) at x_1 = 13.33%, x_2 = 90 minutes and $x_3 = 30^0$. The result obtained agrees with the ones obtained by [11] and [12] after obtaining the maximum yield of pyroligneous acid (30.31%) at x_1 = 13.95% and x_2 = 93 minutes. The developed mathematical equations for the production rate and quality of the pyroligneous acid were combined and then optimized in such a way that y(t) and ρ were maximized while pH was minimized. The optimized equation resulted in y(t) of 0.19 kg/min (26.08%), ρ of 1.03 gcm^{-3} and a pH of 3.01 at x_1 of 20%, x_2 of 137.27 minutes and x_3 of 60° with a composite desirability of 0.4418. The optimized combinations for the pyroligneous acid production rate and quality are summarized in (Table 12).

Table 12. *The optimized combination for pyroligneous acid production rate and quality.*

x_1	x_2	x_3	y(t)	ρ	pH
20	137.27	60	0.19	1.03	3.01

x_1 = feedstock moisture content (%), x_2 = pyrolysis residence time (min), x_3 = chimney inclination angle (°), y(t) = production rate (kg/min), ρ = density (gcm^{-3}).

By using the pyrolysis system designed and developed, the pyrolysis conditions in (Table 12) will guide the production of high yield and quality of pyroligneous acid. From the results obtained for the predicted value of production rate and quality of pyroligneous acid, it shows that the predicted value is closer to the observed ones which indicates a relatively good fit to predict the response variables.

4. Conclusions

The developed pyrolysis system was suitable for the production of pyroligneous acid from acacia twigs and this is as a result of high production rate and quality of pyroligneous acid. The system developed can not only be used for the acacia twigs but also for other biomass materials especially the ones termed as wastes such as maize cobs, sawdust, rice husks, mowed grasses, flower trimmings among others. In examining the correlation between the varied pyrolysis parameters (feedstock moisture content, pyrolysis residence time and chimney inclination angle) with the response (pyroligneous acid production rate, density and pH), the *Box-Behnken Design* was very successful. The mathematical equation developed predicted efficiently the production rate and quality of the pyroligneous acid to be obtained. The optimized pyrolysis conditions obtained will guide any user of the developed pyrolysis system so that it can be used efficiently.

Acknowledgements

The Study was supported and funded by Center of Excellence in Sustainable Agriculture and Agribusiness Management (CESAAM), Egerton University-World Bank funded project and Kenya Climate Smart Agriculture Projects (KCSAP) Bioenergy Researchers, Egerton University.

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