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Optimization of Pyrolysis Process to Produce Biochar from Poultry Waste

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Abstract:- Recent attractions toward biochar production centered on its wide applications in this 21st century. Advocate for green environment and strategy for mitigating global warming require appreciable reduction in the concentration of carbon dioxides present in the atmosphere. Biochar, which is the solid product obtained from the carbonization of biomass can sequesters carbon in a stable carbon pool. In addition, high heating value and low emissions make biochar as the most suitable substitute for solid fossil fuels. In the context of biochar production, slow pyrolysis was identified most advantageously. However, the distribution, property and the quality of the resultant pyrolysis products are dependent on the type of feed stock and pyrolysis conditions under consideration which includes temperature, particle size, residence time and flow rate. In order to increase the yield of desired product and to maintain the products quality consistently, the pyrolysis process was optimized.This is considered one of the best quantitative tools in decision making during pyrolysis experiment. The goal was to maximize the biochar production while keeping all others within their constraints (bio-oil and biogas minimized). The computational software (Design Expert version 12.0) adopted for the optimization purpose divided the coded factor into low, mid and upper points corresponding to -1, 0 and +1 languages as understood by computer. Temperature had its points as 300, 450 and 600 °C, flow rates were 0.5, 1.75 and 3L/mins, particle size 0.5, 1.75 and 3mm, residence time 10, 35 and 60mins and kaolin 5, 17.5 and 30%. After 46 runs, the program delivered model equations for the production of biochar and other co-products. Ultimately, after the optimization process, the optimum pyrolysis conditions for the biochar production were identified and experimented which yielded favorable results.

I. INTRODUCTION

The fact that crude oil is non-renewable source of energy, it also has many disadvantages: burning of gasoline releases carbondioxide which contributes to the global warming, refining of petroleum creates air pollution, its drilling causes water pollution that prevent photosynthesis in plants, transforming crude oil into petrochemicals releases toxins into atmosphere etc. Due to these consequences, Nigeria government strongly advises diversification of economy with emphasis on agriculture. This, in addition to other factors like less investment capital, short growing period, readily available market and less attached risks necessitated interest in poultry farming. The Nigeria poultry industry is estimated at #80 billion and is comprised of 165 million birds (Sahel, 2015). The growing population resulted in high demand for the poultry products. As such, the poultry farming as emerged as one of the fastest growing businesses in Nigeria. It has been known to contribute about 10% of the total national meat production and identified among other livestock as fastest means of reducing protein deficiency in Nigeria (Ahmed et al., 2017). Consequently, the wastes such as litters, feed remnants, faces, death species and other byproducts causes enormous challenges which in some cases discourage people from venturing into the poultry production. The traditional methods of disposing includes: dumping to running water, utilization by local farmers, land filling etc. Most of these practices are harmful and resulted in environmental pollution. It is in light of above that conversion of poultry waste into biochar was proposed.

The utilization of poultry waste as an alternative carbon source for the production of a novel carbon-riched material could be considered as cost effective means of preventing environmental problems associated with disposing of poultry waste.The energy recovery from biomass is based on the conversion techniques employed. Thermochemical conversion is one of the basic technologies used for the conversion lignocellulosic biomass, including fast pyrolysis, slow pyrolysis, and hydrothermal carbonization (Liu et al., 2013). Many studies identified slow pyrolysis as the main conversion technology in the context of biochar production (Lehmann 2009: Demirbas et.al 2014). Biochar is the residual product left in the reaction zone after the biomass has undergo carbonization under limited condition of oxygen. By constituents, it contains large amount of carbon, followed by hydrogen, oxygen, ash content and also trace amounts of nitrogen and sulphur.The elemental compositions of biochar generally changes with the nature of feedstock and pyrolytic conditions such as carrier gas flow rate, catalyst, heating rate, pressure, reactor bed height, particle size, residence time and temperature (Tripathi et al. 2015). Its wide surface area, porosity and surface functionality identified biochar as a good candidate for the production of activated carbon. Conventionally, pyrolysis process produces three array of products namely: biochar, bio-oil and biogas. Studies on the effects of pyrolysis process conditions will be necessary to maximize the yields of the most economically valuable product of interest. Several researches had been conducted on the properties and the yield of biochar generated under different pyrolysis conditions. The compositions of the three main stream products of pyrolysis is a function of the process condition employed which include temperature, heating rate, sweeping gas flowrate, residence time and pressure. However, most previous studies focuses on either two or three of the parameters without considering the combined effect. The present study therefore optimized the process conditions for the production of biochar as prerequisite for activated carbon production.

II. MATERIALS AND METHODS

A. Research Biomass

The choice of poultry waste was as a result of it abundant availability in the environment mostly as wastes and it properties as revealed by proximate and ultimate analysis coupled with information from literature. It pyrolysis with kaolin was aimed at enhancing biochar yield, carbon retention and stability in the biochar produced. Poultry waste was obtained at Amasco Poultry Farm in Ilorin, whileKaolin was sourced from abundant kaolin area at Kutigi, in Lavun Local Government Area of Niger State, Nigeria.

a) Sample Preparation

The sample was air-dried as received prior to division and utilization in the experiment. The feedstock had undergone the sun drying and later conducted in conventional oven at 105 ^{o}C. Part of the samples were grinded and sieved to the particle sizes ranged from 0.5 – 3.0mm. Proximate analysis and Ultimate analysis were done to obtain its elemental compositions. The prepared samples were used in the pyrolysis experiment.

b) Preparation of Y-Alumina from Kaolin

a. Clay Beneficiation

With the aid of wooden pestle and mortar, the kaolin clay was ground. The ground clay sample was wet beneficiated using 100g/L clay to water ratio (Salahudeen et al., 2014) and allowed to settle overnight. The fine kaolin slurry was dewatered until a solid clay cake was obtained, then sieved with continuous manual shacking. The oversize was further ground followed by sieving on the same sieve. The procedures were repeated till the entire clay sample passed through the sieve of 3mm.

b. Metakaolinization Experiment

Ground clay sample was subjected to calcination. At 700 ^{o}C for 1 h, the clay was activated before acid treatment, which is reported in some previous work to be the recommended conditions for activation (Pandel et al 2010). The endothermic dehydration led to the formation of metakaolin and water.

c. Acid Leaching Experiments

Convectional activation approach was adopted in this research. This involved contacting the calcined clay with strong acid and then calcining at 500 ^{o}C using liquid to solid ratio of 10ml/g (Panda et al., 2010). To this effect, 1MH_2SO_4 solution was prepared, from which 500ml was carefully added to 50g of kaolin. The resulting mixture was stirred with the aid of magnetic stirrer at 100 ^{o}C for 5hrs. During the leaching of metakaolin in H_2SO_4 the alumina in metakaolin is extracted and dissolved in H_2SO_4 which leads to formation of aluminiumsulphate.

d. Precipitation Experiment

To precipitate aluminium hydroxide, caustic soda was added to the solution of aluminium. The mixture was cooled and excess acid first removed by filtration and finally washed severally with distilled water. This was followed by calcination at 700 ^{o}C for 1hr which produces γ -alumina. Cooled and sieved for use.

B. Experimental Design

To evaluate the effect of process parameters such as temperature, flow rate, particle size, residence time and kaolin ratio on the yields of pyrolysis products, an un-replicated 2 level factorial design was adopted. In order to generate the experimental design results and perform the appropriate statistical analysis, the Design Expert 12.0 (Stat Ease, Incorporated Minneapolis, MN, USA) was used for the computational program. Each range of factor was coded to a computer languages, says, -1, 0, +1 interval to represent low, middle and high levels of each parameters selected in this research.

III. RESULT AND DISCUSSION

A. *Proximate and Ultimate Analysis*

Table 1 shows the Proximate and Ultimate results of the poultry waste to determine their compositions.

Properties	Poultry Waste (%)	
	Present Study	Vamvuka et al 2013
Moisture Content	1.4	-
Ash Content	15.0	17.7
Volatile Matter	32.0	26.7
Fixed Carbon	51.6	55.6
Carbon	64.72	55.9
Hydrogen	13.82	8.2
Nitrogen	4.82	10.6
Oxygen	16.43	6.2
Sulphur	0.21	1.1

Table 1: Proximate and Ultimate Analysis of Biomass

The proximate and ultimate analysis show that Poultry waste have volatile content (32.0 %wt), carbon (64.72 %wt), and hydrogen (13.82 % wt). Chlorine and sulphur are the major contributing factor to ash formation as they facilitate the mobility of inorganic compounds from the fuel to surfaces where they form corrosive compounds (Wilson, 2014).

B. *XRF analysis of kaolin*

The chemical analysis of the Kutigi kaolin as shown in Table 2 indicates that it contains alumina, silica, iron and calcium in major quantities and other elements in minor quantities. Result as presented shows that percentage of SiO_2 is 27.67% while that of Al_2O_3 is 40.0%. The metal oxide compositions of Kutigi kaolin as presented is close to the reported literature value (Ahmed et al. 2014). The little variation could be attributed to different geographical and geological formation of kaolin. They reported that kaolin has approximately 45% SiO_2 and 37% Al_2O_3. Also in their report are 0.29% Fe_2O_3, 0.17% CaO, 0.96% Na_2O, 0.50% K_2O, and 0.95% MgO. The presence of these essential compounds in kutigi kaolin are believed to enhanced pyrolysis product distribution with much regard to biochar production. Just like in some biomass, when Al_2O_3 and CaO were added, the weight changed positively which indicated the presence of this mineral matter.

Compounds	**Values (wt%)**
SiO_2	27.90
Al_2O_3	40.9
Fe_2O_3	1.925
CaO	6.075
Mn_2O_3	0.004
TiO_2	3.141
LSF	4.783
$CaCO_3$	10.843
L.O.I	4.57

Table 2 XRF of Kutigi Kaolin

C. *Preliminary Results*

Table 3 shows the preliminary experiments conducted for the pyrolysis of poultry waste respectively at the chosen values of independent variables. The process parameters selected for the experiment include: temperature, flow rate, particle size, residence time and kaolin ratio. The range of each factor selected were shown in each Table 3 alongside the yields of pyrolysis products.

Process Parameters					**Product yields**		
TEMP (^{o} C)	**FLOWRATE (L/min)**	**PARTICLE SIZE (mm)**	**RESIDENCE TIME (mins)**	**KAOLIN RATIO (g)**	**BIOCHAR (g)**	**BIO-OIL (g)**	**BIOGAS (g)**
300	0.5	0.5	10	5	43.1	31.2	25.7
400	1	1	20	10	45.8	28.4	25.8
450	1.5	1.5	30	15	43.7	30.1	26.2
500	2	2	40	20	41.1	28.5	30.4
550	2.5	2.5	50	25	38.1	24.3	37.6
600	3	3	60	30	36	22.9	41.1

Table 3: Preliminary Experiment on PW Pyrolysis

The temperature in a pyrolysis process is the most significant operating parameter (Salehi, 2011). At 300 ^{o}C, the yield of biochar and bio-oil were 43.1 and 32.2% respectively. As the reaction temperature increases the biochar and liquid product yields increases but further increase in temperature (above 500 °C) shows decrease in their yields. The reason for the lower yield of biochar and bio-oil at lower temperature may be due to the fact that the reaction temperature becomes too low to complete pyrolysis process. On the other hand, upon increasing the temperature, secondary reactions of the high molecular weight compounds in the pyrolysis vapors or between the vapors and primary biochar dominated, resulting in a decrease of the bio-oil yield and an increase of the biogas yield (Park et al., 2009). The interaction of the hot pyrolysis vapors with surrounding solid environment lead to the formation of biochar. As thought of, the yield of biochar decreases as flow rate increases. Thus low flow rate favor biochar formation. Low flow rate of 0.5 L/min create more time for hot pyrolytic vapor to reside inside the reactor and thus maximizes the secondary reactions like thermal cracking, re-polymerization and re-condensation which favors biochar product yield (Uzunn et al., 2011). Larger particle sizes are expected to favor biochar production because of temperature gradient established between the particle core and its outer surface. However, the preliminary study shows decreases in biochar and bio-oil yields as particles sizes increase. This could be because of other conditions under which the process take place. Also from the preliminary studies, residence time was varied from 10 to 60mins and the yields of biochar and bio-oil correspond to 31.1 to 38.6% respectively. Fassinou et al. (2016) showed an interactive effect of temperature and residence time, wherein increased temperature and residence times resulted in increase in the biochar yield, whereas lower temperature and increase in contact time reduced the yield of biochar. It is therefore difficult to make conclusion regarding the relationship between the production of biochar and the residence times. The distribution of pyrolytic products can be affected by the presence of catalyst. Kaolin addition shows changes in the yield of pyrolysis product. Yields of biochar were optimum at low kaolin addition. Several catalysts such as alumina, Al-MCM-41, oxides of magnesium, oxides of nickel and ZSM-5 showed positive effects on the yield of biochar (Stefanidis et al 2011).

D. *Optimization of PW Pyrolysis*

This section depicted the optimization results of Poultry Waste as a function of some independent variables otherwise called process parameters (Temperature, Flow rate, Particle size, Residence Time and Kaolin ratio) to obtain various responses known as pyrolysis products (Biochar, Bio-oil and Biogas). As shown on the Table 4, the computational software (Design Expert version 12.0) adopted for the optimization purpose divided the coded factor into low, mid and upper points corresponding to -1, 0 and +1 languages understood by computer. In similar manner, the yields of responses 1 and 2 (Biochar and Bio-oil) attained high values around the midpoint of temperature, low value of residence and kaolin along with high residence time appeared to favor biochar production.

STD	**Runs**	**Temp (^{o}C)**	**Flow (L/min)**	**Rate Particle (mm)**	**Size Residence (min)**	**Time Kaolin (g)**	**Ratio Biochar (g)**	**Bio-Oil (g)**	**Biogas (g)**
15	1	300	1.75	3	35	17.5	30.4	35.2	34.4
45	2	450	1.75	1.75	35	17.5	49.6	32.5	18.1
6	3	450	1.75	3	10	17.5	49.4	32.3	18.3
18	4	450	1.75	1.75	60	5	49.8	32.2	18
8	5	450	1.75	3	60	17.5	49.7	32	18.3
17	6	450	1.75	1.75	10	5	49.2	32.7	18.1
44	7	450	1.75	1.75	35	17.5	49.6	32.3	21.1
7	8	450	1.75	0.5	60	17.5	48.6	33.3	18.1
46	9	450	1.75	1.75	35	17.5	49.6	32.3	18.3
37	10	450	0.5	1.75	10	17.5	48.4	33.3	18.7
3	11	300	3	1.75	35	17.5	30.4	35.1	34.5
41	12	450	1.75	1.75	35	17.5	49.6	32.3	18.1
29	13	450	1.75	0.5	35	5	48.3	33.3	23.4
22	14	450	3	0.5	35	17.5	49.6	32.3	18.1
2	15	600	0.5	1.75	35	17.5	28.9	32.4	42.6
19	16	450	1.75	1.75	10	30	49.6	32.3	18.1
24	17	450	3	3	35	17.5	49.6	32.2	18.2

26	18	600	1.75	1.75	10	17.5	29.1	33.2	42.6
28	19	600	1.75	1.75	60	17.5	28.7	33.8	37.5
40	20	450	3	1.75	60	17.5	49.6	32.3	18.1
35	21	300	1.75	1.75	35	30	30.1	34.7	35.2
31	22	450	1.75	0.5	35	30	49.6	32.3	18.1
16	23	600	1.75	3	35	17.5	30.1	31.5	42.6
38	24	450	3	1.75	10	17.5	48.6	33.3	18.1
20	25	450	1.75	1.75	60	30	49.6	32.3	18.1
36	26	600	1.75	1.75	35	30	29.7	31.1	42.6
9	27	450	0.5	1.75	35	5	49.6	32.3	18.1
33	28	300	1.75	1.75	35	5	31.4	32.8	35.8
1	29	300	0.5	1.75	35	17.5	30.8	32.4	36.8
5	30	450	1.75	0.5	10	17.5	49.6	32.3	18.1
10	31	450	3	1.75	35	5	49.6	32.3	18.1
25	32	300	1.75	1.75	10	17.5	30.7	35.5	34.8
21	33	450	0.5	0.5	35	17.5	49.6	32.3	18.1
4	34	600	3	1.75	35	17.5	28.3	33.3	42.6
34	35	600	1.75	1.75	35	5	29	32.5	42.6
43	36	450	1.75	1.75	35	17.5	49.6	32.3	18.1
32	37	450	1.75	3	35	30	49.6	32.3	18.1
14	38	600	1.75	0.5	35	17.5	28.8	34.1	42.6
27	39	300	1.75	1.75	60	17.5	30.5	34.8	34.7
12	40	450	3	1.75	35	30	49.6	32.3	18.1
42	41	450	1.75	1.75	35	17.5	49.6	32.3	18.1
13	42	300	1.75	0.5	35	17.5	30.1	35.7	34.2
23	43	450	0.5	3	35	17.5	49.6	32.3	18.1
11	44	450	0.5	1.75	35	30	49.6	32.3	18.1
39	45	450	0.5	1.75	60	17.5	49.6	32.3	18.1
30	46	450	1.75	3	35	5	49.6	32.3	18.1

Table 4: Effect of Low, Mid and Upper points of Optimization on Poultry Waste Pyrolysis

a) ANOVA for Quadratic Model of Response 1: Biochar

The analysis of variance and fit statistics of biochar resulted from optimization of PW pyrolysis were shown on Table 5 and 6 respectively. The **Model F-value** of 1214.03 implies the model is significant. There is only a 0.01% chance that an F-value could occur due to noise.**P-values**of less than 0.0500 indicate that model terms are significant. In this case A, C, AE, A² are significant model terms. Basically, values greater than 0.1000 indicate the model terms are not significant. Many insignificant model terms are removed for the reduction of model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	4032.60	20	201.63	1214.03	< 0.0001	Significant
A-Temperature	8.70	1	8.70	52.40	< 0.0001	
C-Particle Size	0.9025	1	0.9025	5.43	0.0281	
AE	1.0000	1	1.0000	6.02	0.0214	
A^2	3382.67	1	3382.67	20367.31	< 0.0001	
Residual	4.15	25	0.1661			
Lack of Fit	4.15	20	0.2076			
Pure Error	0.0000	5	0.0000			
Cor Total	4036.75	45				

Table 5: ANOVA for Biochar

Fit Statistics			
Std. Dev.	0.4075	**R^2**	0.9990
Mean	42.61	**Adjusted R^2**	0.9981
C.V. %	0.9564	**Predicted R^2**	0.9959
PRESS	16.61	**Adeq Precision**	77.9825

Table 6: Fit Statistics

The **Predicted R²** of 0.9959 is in reasonable agreement with the **Adjusted R²** of 0.9981; i.e. the difference is less than 0.2. **Adequate Precision**usually measures the signal to noise ratio. A ratio greater than 4 is desirable. Thus, the ratio of 77.983 indicates an adequate signal.

- **Final Equation in Terms of Actual Factors**

Biochar=-124.29350+0.776517 * Temperature-0.214000 * Particle Size+0.000267 * Temperature * Kaolin Ratio-0.000875 * Temperature² (6.4)

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels are specified in the original units for each factor. Thus, the equation revealed those factors with single and interactive effects for the biochar production from poultry waste.

b) Model Validation for PW Biochar

- **Normal Residual Plot**

Stat Ease Design Expert version 12.0 automatically calculates and plots the residual plot (Fig 1) to validate the model. Since the points in the residual plot are randomly distributed on the vertical axis, a nonlinear (quadratic) is appropriate for the data and model prediction can be considered accurate.

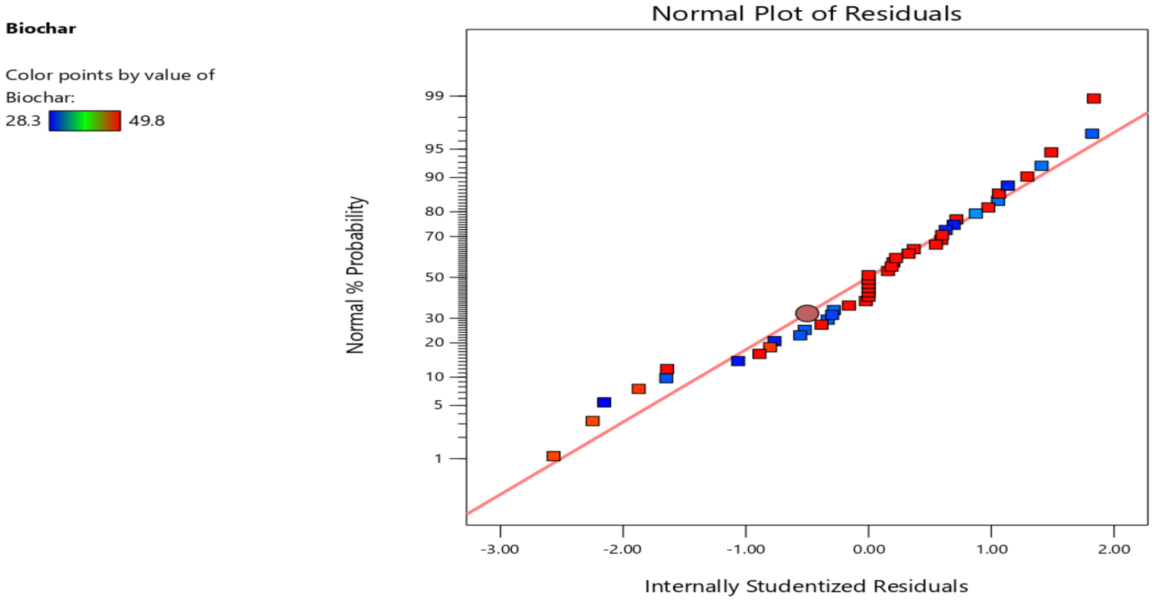

Fig. 1: Normal Residual Plot for PW Biochar

- **Single Factor Effect on PW Biochar**

The single effects of temperature and particle size on the optimization of PW for biochar production were shown in Figure 2 and 3 respectively. It could be observed that the yield of biochar initially increased up to approximately 450 °C and later decreases to the final temperature of 600 °C. The yields of biochar also increase with increasing particle size. This could be as a result of temperature gradient established between the biomass core and its outer surface.

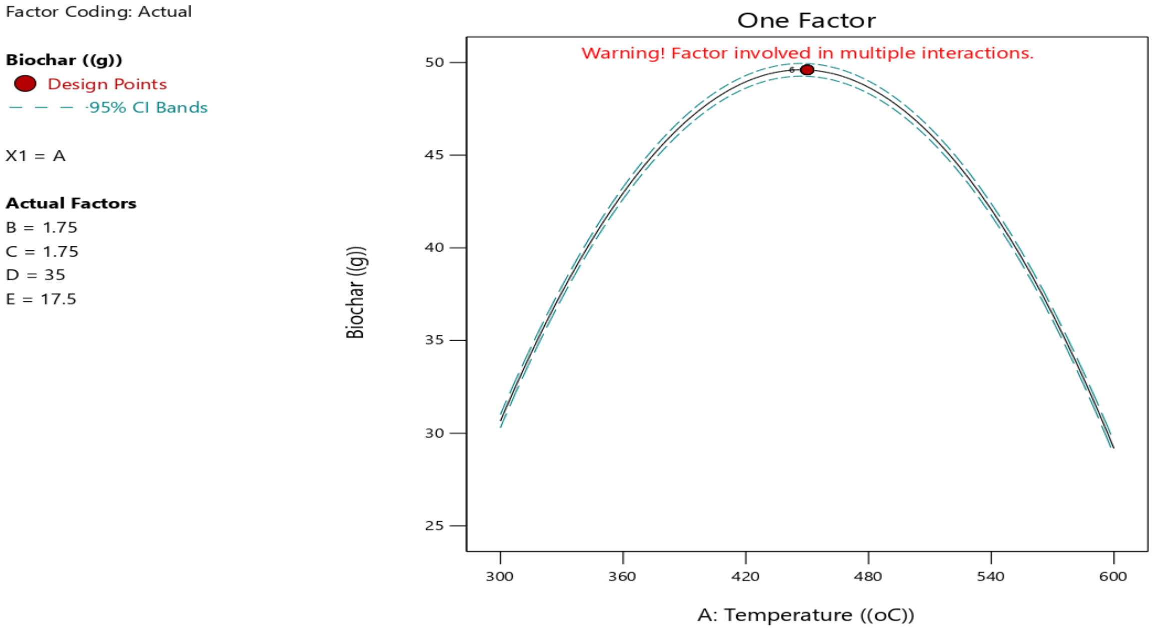

Fig. 2: Single Effect of Temperature on PW Biochar

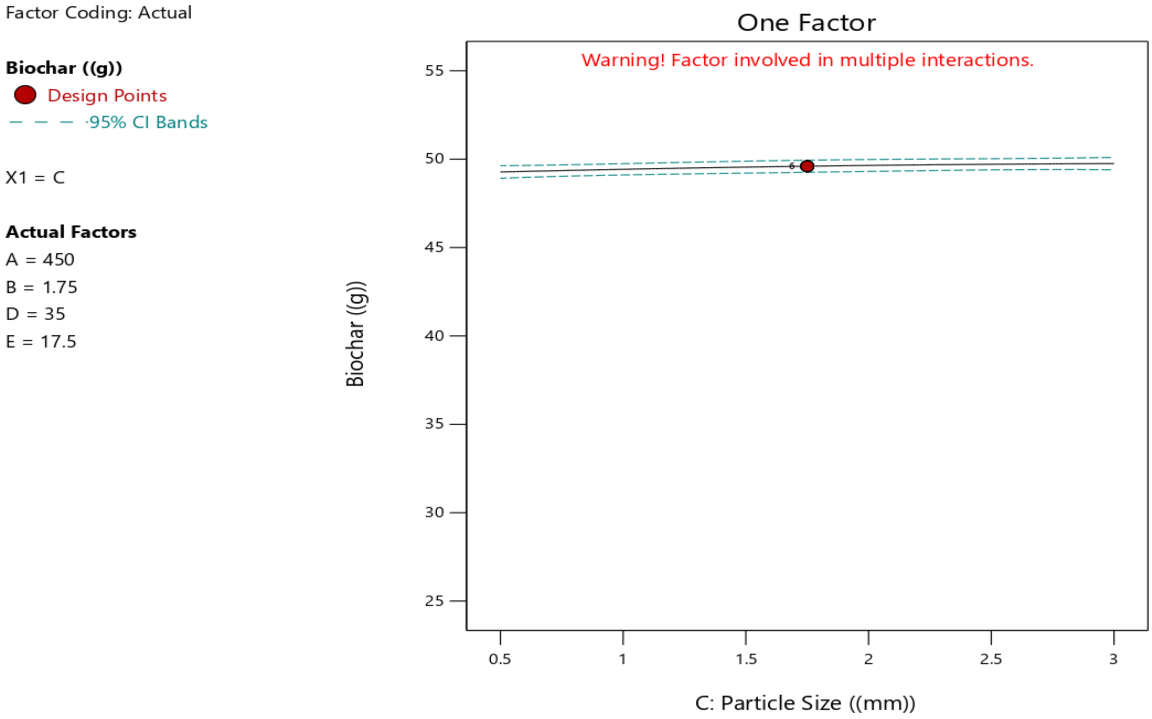

Fig. 3: Single Effect of Particle Size on PW Biochar

- **Contour and Interaction Plots for PW Biochar**

  The two plots (Contour and Interaction) represented on Figure 4 and 5 (for temperature and Kaolin ratio) were presented to visualize the response (biochar) yields as a function of two separate independent variables that are significant and had interactive effect on biochar production. For contour plot, the yields were represented on the contour line while temperature and Kaolin ratio were represented on horizontal and vertical axis. For Interaction plot, yields of biochar, temperature and kaolin ratio were represented on y, x and z planes.

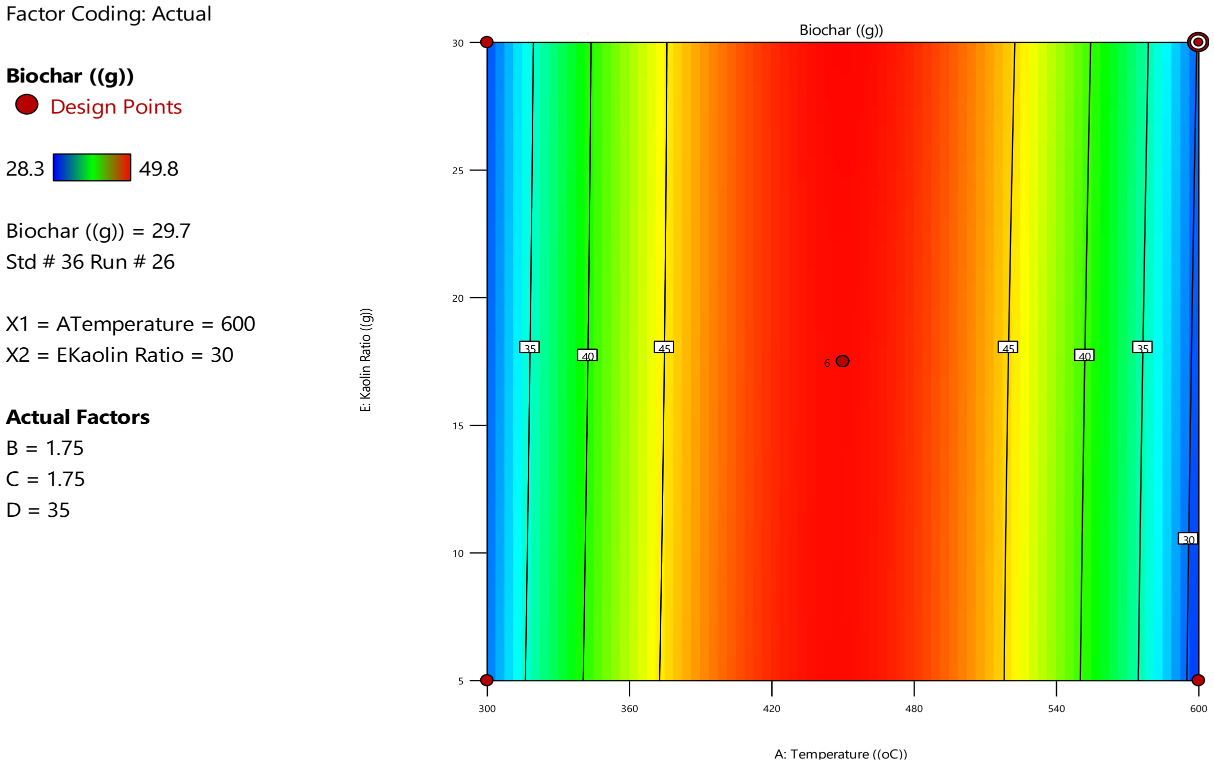

Fig. 4: Contour Plot for Temperature and Kaolin ratio on PW Biochar yields

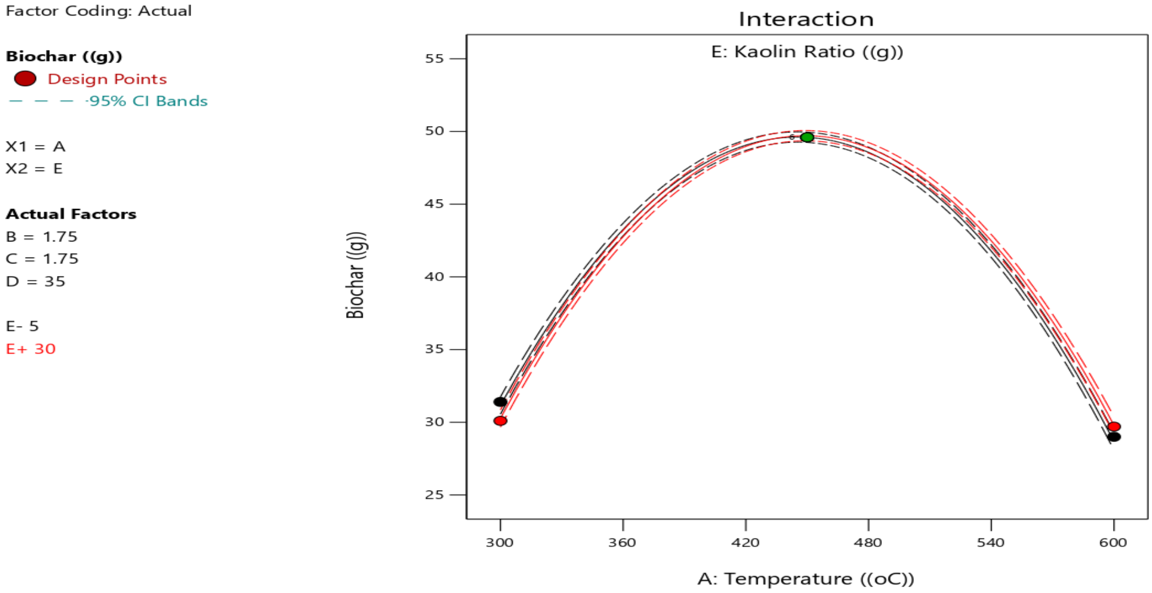

Fig. 5: Interaction Plot for Temperature and Kaolin ratio on PW Biochar yields

IV. CONCLUSION

Economy diversification with emphasis on agriculture had been the demand of many developing countries. The numerous advantages of poultry farming had necessitated many efforts in it operation. The resultant wastes were pyrolysed to obtain array of products with interest on biochar. The computational software (Design Expert version 12.0) adopted for the optimization purpose divided the coded factor into low, mid and upper points corresponding to -1, 0 and +1 languages understood by computer. In similar manner, the yields of responses 1 and 2, that is, Biochar and Bio-oil attained high values around the midpoint of temperature, low value of flow rate and kaolin along with high residence time. 450ºC, 1.75L/min, 60 min and 5g of kaolin appeared to favor biochar production.

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