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Optimisation of Activated Carbon Preparation by Chemical Activation of *Ayous* Sawdust, *Cucurbitaceae* Peelings and Hen Egg Shells Using Response Surface Methodology

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Authors' contributions

This work was carried out in collaboration between all authors. Authors NSC, NHM and ASG designed the study, wrote the first draft and managed the literature searches. Authors TTDR and NSC managed all the analysis of the study and corrected the first manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Aims: Explore the effects of the mixture of biomass materials: *ayous* sawdust, *cucurbitaceae* peelings and hen eggs shells in the preparation of activated carbon by chemical activation using the Methodology of Experimental Design.
Study Design: Determination of the physicochemical properties of the prepared activated carbons by the mixture of biomass materials: *ayous* sawdust, *cucurbitaceae* peelings and hen eggs shells using the Methodology of Experimental Design.

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Place and Duration of Study: Laboratory of Noxious Chemistry and Environmental Engineering, Department of Chemistry, University of Dschang, between February 2015 and November 2016.
Methodology: A mixture of *ayous* sawdust, *cucurbitaceae* peelings and hen egg shells were used to prepare activated carbon by chemical activation using H_3PO_4. The effect of the composition of the components on the iodine number of the activated carbon was investigated. The Methodology of Experimental Design (MED) was used to optimize the production conditions of the activated carbons, while the analysis of variance (ANOVA) was used to determine the significance of the various variables.
Results: Using a carbonization temperature of 450°C and a H_3PO_4 concentration of 0.79 M, the optimum conditions for preparing the activated carbon were 2 hours of resident time, 0.549 as mass fractions for *ayous* sawdust, 0.450 for *cucurbitaceae* and 2.296×10^{-10} for hen egg shells. The resultant activated carbon was found to have a BET surface area of 311.23 m^2/g, an iodine number of 656.70 mg/g and a carbon yield of 44.88%. It was also observed that the experimentally obtained correlation coefficient of 0.999 was in good agreement with the values predicted by the model. The iodine number, which decreased with the fraction of hen eggs shells used portrayed an antagonist effect, while its increase with the increase in *ayous* sawdust and *cucurbitaceae* peelings showed the synergetic effect.
Conclusion: The optimized activated carbon was the one produced from the near 50/50 combination of *ayous* sawdust and *cucurbitaceae*, and its BET surface area, and iodine number were superior to those from the individual materials.

Keywords: Activated carbon; phosphoric acid; Ayous sawdust; Cucurbitaceae peelings; hen eggs shells; optimization; response surface methodology.

1. INTRODUCTION

Activated carbon, a widely used adsorbent in industrial processes, is composed of a microporous, homogenous structure with high surface area. Worldwide, the process for producing high-efficient activated carbon is not completely investigated [1]. Nowadays, there is a great interest in finding inexpensive and effective alternatives to the existing commercial activated carbons [2,3]. Lignocellulosic materials are gaining more and more interest as raw materials in the manufacture of adsorbents [4,5]. Many researchers have prepared activated carbons using *ayous* sawdust, and *cucurbitaceae* peelings [6-9]. There was a great loss in mass when activated carbon was obtained from these materials, especially when the temperature used was above 500°C [10-12].

Although cellulosic materials have been used to produce activated carbons, a combination of biomaterials obtained from both animals and waste vegetables is not often found in the literature. Given that eggs shells is constituted mainly of oxides [13] three starting materials could be mixed and pyrolysed to form a porous material that can be suitable to be used as adsorbents.

In the literature, physical activation and chemical activation are the two methods used to produce activated carbons [5,14,15]. Based on previous works on these methods of preparation, chemical activation has been chosen as the method to prepare activated carbon in this work. Its advantages are simplicity, shorter production time, lower activation temperature, good development of the porous structure and higher yield [16-18].

In the chemical activation process, both carbonization and activation steps proceed simultaneously by carrying out thermal decomposition of the raw material impregnated with an appropriate chemical agent, such as phosphoric acid in an inert atmosphere [19,20]. These impregnating materials are used as dehydrating agents and oxidants that influence pyrolytic decomposition and inhibit the formation of tar, thus enhancing the yield of activated carbon [17,21]. The quality of activated carbon obtained depends mainly on the precursor material used and the preparation conditions [22,23].

To investigate the effect of some factors on the production of activated carbons, a number of the factors are kept fixed at a certain level, while varying one other so as to determine the best condition for this parameter. The disadvantage of this method is that, there is a lack of research on the interactive effects of these factors studied, and that, there is a large

number of experiments required, which consequently requires more time [24,25]. The evaluation of the interactions between these factors is essential in determining the characteristics of the prepared activated carbons.

A number of researchers have used the Methodology of Experimental Design to optimize the production of activated carbons. This method consists of diverse mathematical models and statistical techniques based on the adjustment of equations or models to experimental data to describe the behavior of independent variables [25,26]. In recent years, some authors have applied this optimization method to the production of activated carbons [1,24-36]. These works have rarely studied the use of a mixture of biomass materials.

The aim of this research was to explore the effects of the mixture of three biomass materials: *ayous* sawdust, *cucurbitaceae* peelings and hen eggs shells in the preparation of activated carbon by chemical activation by using the Methodology of Experimental Design.

2. MATERIALS AND METHODS

2.1 Sample Collection and Preparation of Activated Carbon

The raw materials for the preparation of activated carbon were *ayous* sawdust, *cucurbitaceae* peelings and hen egg shells. They were collected from Etoudi wood market, Nanga-Eboko market and 'Marché A' in Bafoussam respectively; all of them being locations in Cameroon. The materials were carefully washed with distilled water to reduce impurities, and then dried in an oven set at 110°C to remove excess water. The dried materials were then stored at room temperature for further use.

2.2 Experimental Methods

Exact amounts of each biomass material (3 mm of diameter) were weighed and added into beakers containing a solution of phosphoric acid (0.79 M). Each mixture, maintained at ambient temperature, was manually shaken for 2 hours, and then dried in an oven set at 105°C overnight so that, any water present in the sample was completed evaporated. The reactor was loaded with 10 g of impregnated sample and then placed in a furnace. The furnace was heated at the rate of 5°C/min to the pre-determined or desired activation temperature of 450°C. It was then held at this temperature for a period of 1 hour. After allowing to cool to ambient temperature, the samples were washed several times with distilled water. The washing process was done until all the phosphate ions initially present in the sample were completely removed as was indicated by the final pH ranging between 6 and 7 [37]. The washed samples were dried in the oven set at 105°C for 24 hours. The resulting products were crushed into powder and well-kept for further tests.

2.3 Methodology of Experimental Design

The Methodology of Experimental Design (MED) is a multivariate statistical technique used to optimize processes, i.e., to discover the conditions in which to apply a procedure in order to obtain the best possible response in the experimental region studied [38]. The MED is utilized to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interactions between the parameters [1]. The three parameters investigated in the present study were coded as fraction of *Ayous* sawdust (X_1), fraction of *Curcubitaceae* peelings (X_2) and fraction of hen egg shells (X_3). The experimental design matrix of 10 experiments and results are given in the Table 1. Each row represents an experimental run, and each column represents the variables tested. The response analyzed was iodine number (Y) and it was used to develop a model which correlates the response to the three variables using a polynomial equation given by the following expression:

$$Y = b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 \quad (1)$$

where Y is the predicted response, b_i a linear coefficient; b_{ii}, a quadratic coefficient; b_{ij}an interaction coefficient, X_1, X_2 and X_3, the coded values of the activated carbons preparation variables. The sum of the three fractions must be equal to 1. The experimental data were analyzed using a statistical software design expert named Statgraphics Plus 5, for regression analysis to fit the equations developed and also to evaluate the statistical significance of the equations obtained.

Table 1. Experimental design matrix and the corresponding experimental responses

N° of experiment	*Ayous* sawdust (X_1)	*Cucurbitaceae* peelings (X_2)	Hen egg shells (X_3)	Iodine number (mg/g)
1	1	0	0	546.3
2	0	1	0	487.3
3	0	0	1	57.1
4	1/2	1/2	0	656.7
5	1/2	0	½	178.9
6	0	1/2	½	209.4
7	1/2	1/2	½	367.4
8	2/3	1/6	1/6	300.7
9	1/6	2/3	1/6	376.9
10	1/6	1/6	2/3	194.1

2.4 Activated Carbons Yield

The activated carbon yield, Y was calculated using the following formula:

$$Y = \frac{m}{m_o} \times 100 \quad (2)$$

where m and m_0 are the dry weight of final activated carbon (g) and dry weight of precursor (g), respectively [19].

2.5 Adsorption of Iodine: Adsorption Capacity of the Activated Carbons

Iodine was considered as probe molecule for assessing the adsorption capacity of adsorbents for solutes of molecular sizes <10 Å. The iodine number is defined as the milligrams of iodine adsorbed by 1 g of carbon. The quantity of iodine adsorbed was determined by volumetric analysis method using sodium thiosulfate [39]. The iodine number was estimated by shaking a mixture of activated carbon with 0.02 N iodine solution, and then titrating the resulting solution against $Na_2S_2O_3$ solution [37].

2.6 Bulk Density

A measuring cylinder was weighed empty. It was then filled with the prepared sample of activated carbon and gently tapped until no change in the level of the sample was observed. The volume occupied by the packed sample was recorded as V_s. Using W_c as the weight of the empty cylinder and W as the weight of the cylinder and sample, the weight of the sample W_s was obtained by: $W_s = W - W_c$. The bulk density was calculated using the following formula:

$$B_d = \frac{W_s}{V_c} \quad (3)$$

Where W_s and V_c are the weight of activated carbon (g) and, the volume occupied by the packed sample (mL) respectively.

2.7 Determination of pH Point of Zero Charge (pHpzc)

The determination of pH point of zero charge (pHpzc) for an activated carbon was carried out by adding 0.1 g of activated carbon to 40 mL solution of 0.1 M NaCl whose initial pH had been measured and adjusted with 0.1 M NaOH or 0.1 M HCl to vary between 3-11. The containers were sealed and placed on an agitator for 72 hours after which the pH was measured. The pHpzc occurs if there is no change in the pH after contact with the adsorbent [40,41].

2.8 Functional Groups on the Surface of Activated Carbon

The Boehm titration method was used for this analysis. 0.4 g each of the activated carbon was added to separate 20 mL solutions of $NaHCO_3$ (0.1 M), Na_2CO_3 (0.1 M) and NaOH (0.1 M) for acidic groups, and 0.1 M HCl for basic groups respectively at room temperature under agitation for 48 hours. Subsequently, the aqueous solutions were back-titrated with HCl (0.1 M) in the presence of methyl orange indicator for acidic groups, and NaOH (0.1 M) in the presence of phenolphthalein indicator for the basic groups. The number and types of acidic sites were calculated by considering that NaOH neutralizes carboxylic, lactonic and phenolic groups; Na_2CO_3 neutralizes carboxylic and lactonic groups while $NaHCO_3$ neutralizes only carboxylic groups [42].

Carboxylic groups were therefore quantified by direct titration with $NaHCO_3$. The difference between the groups titrated with Na_2CO_3 and those titrated with $NaHCO_3$ was assumed to be lactones and the difference between the groups titrated with NaOH and those titrated with Na_2CO_3 was assumed to be phenol. Basic sites were determined by titration with HCl. In order to

neutralize the remaining basic groups, HCl in the solution was back-titrated with 0.1 M NaOH.

2.9 Determination of Specific Area of Activated Carbon

Brunauer Emmett Teller (BET) analysis was carried out to determine the surface area and porosity of the activated carbons. The BET analysis was carried out by nitrogen adsorption-desorption method using nitrogen at -196°C and the autosorb BET apparatus, Micrometrics ASAP 2020, which has both surface area and porosity analyzers. The analytical procedure was automated and operated with static volumetric techniques. The samples were first degassed at 200°C for 2 h before each measurement was recorded.

3. RESULTS AND DISCUSSION

The runs from the experimental design and the respective responses are presented in Table 1 below, while Tables 2 to 5 show respectively, the ANOVA analysis, the value of 2^{nd} polynomial fit, the control points and the combinations of factors which give highest response values. Equation 4 is the mathematical model equation of the parameters of composite activated carbon that result from the experimental runs. The graphical presentation (Figs. 1a and 1b) plots visualize the fitted models in triangular surface plots.

3.1 Responses Analysis and Interpretation

The experiments at the center point of the complete design matrix (experiments n°8 to 10) were used to verify the reproducibility of experimental data. The adsorption capacities of I_2 ranged from 57.1 to 656.7 mg/g. The lowest value of 57.1 was obtained in experiment 3, where only hen egg shells were used, while the highest value of 656.7 mg/g was obtained in experiment 4, where the mixture ratio: X_1= 0.5, X_2 = 0.5 and X_3 = 0.0. The polynomial model equation in terms of coded factors is given as:

$$\text{Iodine number} = 544.66X_1 + 485.652X_2 + 55.4611X_3 + 592.507X_1X_2 - 458.223X_1X_3 - 218.383X_2X_3 \quad (4)$$

A positive sign in front of the coefficients indicates synergistic effects, whereas the negative sign indicates antagonistic effects. So, it can be observed from this equation that of the three components, the effect of hen egg shells was antagonistic on the response, implying that an increase of this factor results in the reduction of the response studied. This can be explained by the fact that, hen egg shells contain more oxides than carbon, which tend to reduce the development of porous structures during calcination, and consequently leading to a reduction in the iodine number during iodine adsorption [13,43].

Table 2 shows an analysis of variance for the currently selected quadratic model. The P-values were used as a tool to check the significance of each of the coefficients, which in turn was necessary in understanding the pattern of the mutual interactions between the test variables [44]. Since the P-value for this model is less than 0.10, there is a statistically significant relationship between iodine number and the components at the 90% confidence level. R^2 indicates the fraction of the total variables of response variable that has been explained by the predicator variables [45]. The R-squared statistics obtained indicates that the model as fitted explains 99.875% of the variability in iodine number. The adjusted R-squared statistics, which is more suitable for comparing models with different numbers of independent variables, is 99.252% and therefore indicates a high significance for the model [46-48]. The standard error of the estimate shows the standard deviation of the residuals to be 18.8873. Since the P-value is less than 0.10, there is an indication of possible serial correlation.

The control points results are shown in Table 4.

The difference between theoretical and experimental values of iodine number is due to the absence of the error coefficient ε in the defined mathematical model. By using table 3 we can estimate ε to be around 200.

Table 5 shows the combination of factor levels which maximize iodine number over the indicated region.

Table 2. Analysis of variance of iodine adsorption

Source	Sum of squares	Degree of freedom	Mean square	F-ratio	P-value
Quadratic model	285822.0	5	57164.5	160.24	0.0521
Total error	356.732	1	356.732	/	/

Table 3. Constants of the mathematical model and their statistical validation

Component	Model coefficient	Standard error	P-value
A : *Ayous* sawdust	544.66	18.8157	/
B : *Cucurbitaceae* peelings	485.652	18.8157	/
C : Hen egg shells	55.4611	18.8157	/
Mixture AB	592.507	86.4902	0.0923
Mixture AC	458.223	86.4902	0.1188
Mixture BC	218.383	86.4902	0.2401

Table 4. Control points results

Experiment No.	8	9	10
Theoretical iodine number (mg/g)	549.05	556.03	346.80
Experimental iodine number (mg/g)	300.37	376.89	194.16

Table 5. Combination of factor levels which maximize iodine number

Factors	Low	High	Optimum
Ayous sawdust	0	1	0.549
Cucurbitaceae peelings	0	1	0.450
Hen egg shells	0	1	2.296×10^{-10}

The Fig. 1 presents the response surface constructed to show the most important variable on the response Y.

It is observed that, the iodine number increases when the fraction of hen egg shells decreases (Fig. 1-a, b and c). This is because egg shells contain low amounts of carbon. The correlation coefficient of 0.999 (Fig. 1-d) shows that the model can be used to optimize the iodine number of an activated carbon starting from *ayous* sawdust, *cucurbitaceae* peelings and hen egg shells.

Since the results of the optimization studies show that it is the mixture of *ayous* sawdust and *cucurbitaceae* peelings that give good iodine number, the characterizations that follow will be carried out on activated carbon from *ayous* sawdust (B1M), *cucurbitaceae* peelings (P1M) and the mixture of the two materials (BP1M) with H_3PO_4 as activating agent (1M).

3.2 Functional Groups on the Surface of Activated Carbon

Table 6 shows the result of functional groups determined by the Boehm method.

The analysis of table 6 shows that all the activated carbons produced have mainly acidic character on their surfaces. This is due principally to the presence of carboxylic and phenolic functional groups present in the activated carbons.

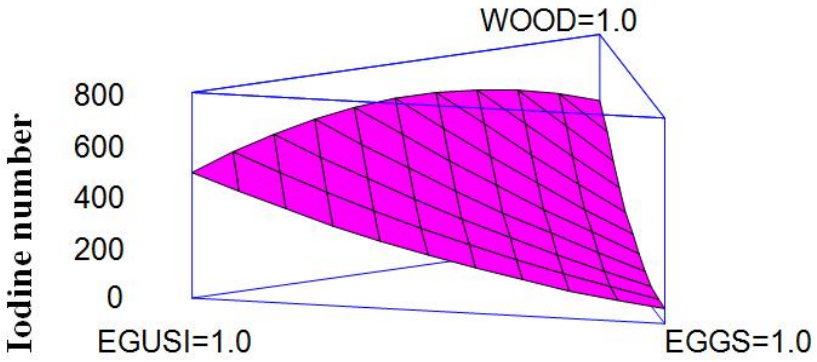

Fig. 1a. Estimation of the surface reponse in 3D (wood: *Ayous* sawdust; egusi: *cucurbitaceae* peelings and hen egg shells)

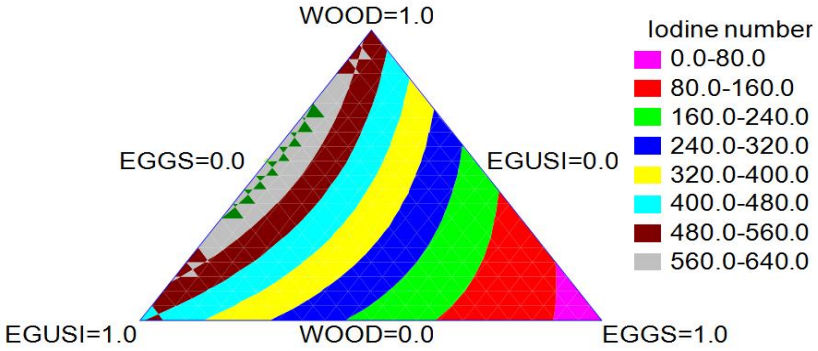

Fig. 1-b. Contours of estimation of the surface reponse in 2D (wood: *ayous* sawdust; egusi: *cucurbitaceae* peelings and eggs: hen egg shells); where each colour represents an interval of iodine number corresponding to a precise composition of the mixture

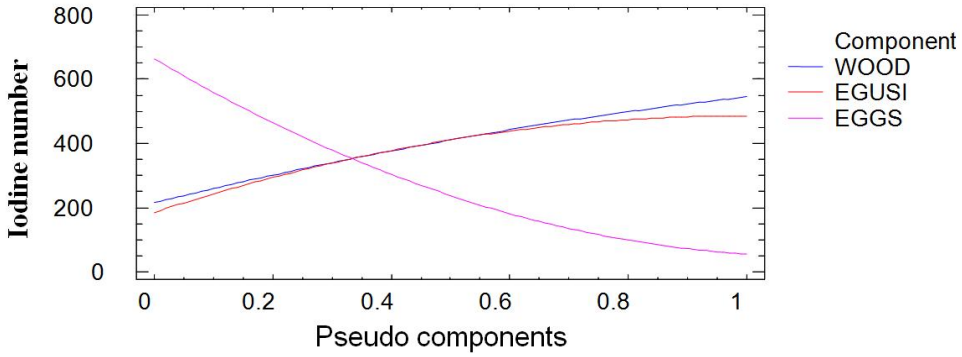

Fig. 1-c. Variation of iodine number as a function of components fractions (wood: *Ayous* sawdust; egusi: *cucurbitaceae* peelings and eggs: hen egg shells)

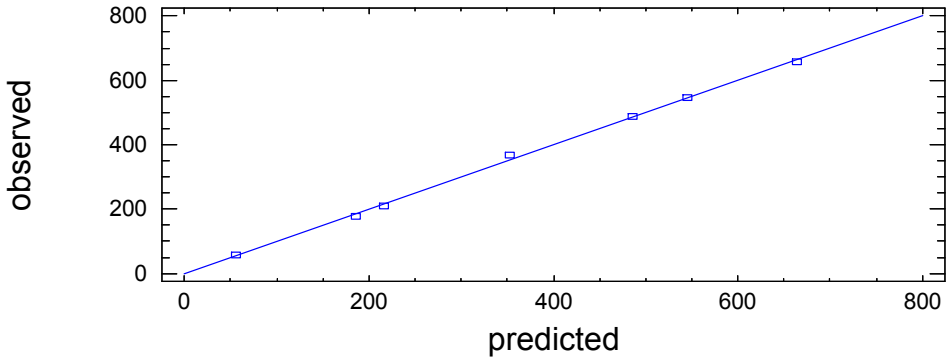

Fig. 1-d. Correlation between predicted and observed values of iodine number

Table 6. Number of gramme equivalents of functional groups on the activated carbon surface

Functional groups	Carboxylics (meq/g)	Lactones (meq/g)	Phenolics (meq/g)	Total basicity (meq/g)
B1M	0.18	0.32	0.14	0.084
P1M	0.22	0.00	0.28	0.042
BP1M	0.22	0.22	0.18	0.042

Where B1M is ayous sawdust activated with 1M H_3PO_4; P1M is cucurbitaceae peelings activated with 1M H_3PO_4, and PB1M is a mixture of the two biomass activated with 1M H_3PO_4.

3.3 Determination of pH Point of Zero Charge

The plot of final pH of an activated carbons as a function of its initial pH is given in Fig. 2.

Fig. 2 shows that, the pHpzc are 6.1, 6.5 and 6.3 respectively for B1M, P1M and BP1M. These values indicate that the activated carbons produced have acidic character. This result is consistent with that of the determination of the functional groups on the surface by the Boehm method, which shows the dominance of acidic groups. In fact, the pH at the point of zero charge is a very important parameter in the characterization of the activated carbons, which indicates the acid-base behavior of finely divided solids.

From these results it can be said that at pH less than 6, all the three activated carbons are positively charged, while beyond a pH of 6.6, they are negatively charged. Based on this result the adsorption of positively charged adsorbates must be done at pH greater than 6.6 while those that are negatively charged at pH less than 6.

3.4 Bulk Density, Activated Carbons Yield and pH of Activated Carbon

The result of bulk density and activated carbon yield is given in Table 7.

The activated carbon yield of close to 50 percent can be explained by the calcination temperature of 450°C and the activated agent H_3PO_4. At this temperature, the removal of carbon in the form of carbon dioxide is smaller than that for the temperature higher than 450°C, and probably that the phosphates in H_3PO_4 form linkages that retain carbon and avoid the loss of volatile material [49]. The value of the pH close to 7, which is the pH of drinking water, indicates that the resulting activated carbons can be used to treat water, since their agitation in water will not release compounds that will change the final pH of the water. Also, the amount of activated carbon used to treat water is inversely proportional to the bulk density. That is, if the bulk density is high, then the amount of activated carbon needed for a given adsorption capacity will be small. These values of bulk density can also be attributed to random arrangement of micro-crystallites and with strong cross-linking which produce porous structure.

3.5 Determination of Specific Surface Area of Activated Carbon

Table 8 summarizes the properties of activated carbon prepared under optimum conditions. From the table, it can be observed that activated carbon prepared from a mixture of *ayous* sawdust and *cucurbitaceae* peelings have a

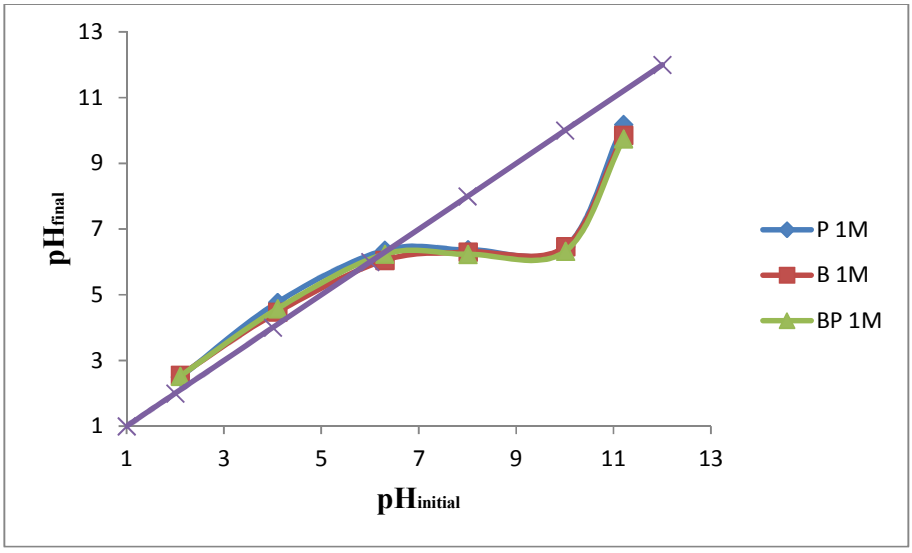

Fig. 2. pH point of zero charge (pHpzc) of activated carbons

Table 7. Bulk density, pH and yield of activated carbon

Activated carbon	Bulk density (kg/m^3)	Activated carbon yield	pH
B1M	517.33	49.50	7.398
P1M	772.80	44.28	7.472
BP1M	686.67	44.88	7.175

Table 8. Pore properties of the activated carbon prepared under optimum conditions

Activated carbon	Specific area using BET (m^2/g)	Total pore volume (cm^3/g)	Average pore diameter (nm)
B1M	151.52	8.102	2.138
P1M	5.10	2.365	18.539
BP1M	311.56	0.145	1.856

higher surface area and a lower pore diameter. On the other hand, activated carbon from *ayous* sawdust has pores less than those in activated carbon from *cucurbitaceae* peelings. This can be explained by the fact that materials with a greater content of lignin as grape seeds or cherry stones develop activated carbon with a predominance of macropores, while other raw materials characterized as having a higher content of cellulose produce activated carbons with a predominantly microporous structure [49]. When the two materials are mixed the increase in cellulose composition favors the formation of microspore and hence an increase in specific surface area [50].

4. CONCLUSION

The Methodology of Experimental Design was used to produce optimized activated carbons from mixtures of *ayous* sawdust, *cucurbitaceae* peelings and hen egg shells, in the mass ratios X_1, X_2 and X_3 respectively. Using iodine number as the response, the optimization process carried out using experimental data gave the polynomial model equation as:

$$\text{Iodine number} = 544.66X_1 + 485.652X_2 + 55.4611X_3 + 592.507X_1X_2 - 458.223X_1X_3 - 218.383X_2X_3$$

The lowest value of the iodine number of 57.1 mg/g was obtained when only hen egg shells, X_3 was used, while the highest value of 656.7 mg/g was obtained when the mixture ratio was X_1= 0.5, X_2 = 0.5 and X_3 = 0.0.

Through analysis of the response surfaces derived from the models, *ayous* sawdust and *cucurbitaceae* peelings were found to have significant effects on the responses (Iodine number). The R^2 values of the responses showed a good fit of the models with experimental data.

The activated carbon prepared under the optimum conditions were found to have well-developed pores on its surface according to their adsorption capacities of iodine. Among the three starting materials, *ayous* sawdust was the best constituent in terms of iodine number. Since optimization showed that the mixture of the two starting materials gave the highest iodine number, activated carbons made from *ayous* sawdust, *cucurbitaceae* peelings were also characterized. The mixture of the starting materials increase BET specific area from 151.520 m^2/g for the activated carbon obtained from *ayous* sawdust and 5.100 m^2/g for the activated carbon obtained from *cucurbitaceae* peelings to 311.560 m^2/g for the activated carbon obtained from a mixture of the two. Also, the pore diameter was significantly reduced from 2.138 nm and 18.539 nm respectively to 1.856 nm for the mixture. The surface of all the activated carbons produced more acidic functional groups than the basic ones. The activated carbons so prepared are potential adsorbents for the removal of pollutants from surface water.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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