Uptake of Ethylacetate from Ethylacetate – Water Mixture Using Activated Carbon

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***Abstract*– The adsorption capacity of activated carbon produced from hardwood sawdust on ethyl acetate-water has been investigated. The activated carbon was prepared using thermal activation by heating the hardwood sawdust in the absence of air using a muffle furnace at 200^{o}C, 350^{o}C, 500^{o}C, 600^{o}C for 30, 60, 90,120 minutes. The effect of three factors; temperature of activation, process duration and particle size were carried out using the Analysis of Variance. The optimum conditions were activation of the hardwood sawdust at 500^{o}C for 60mins. The ash content, iodine number, pH, bulk density and moisture content produced at these condition were 3%, 76.143, 4.75, 0.42mg/ml and 10%for the wood Saw dust. The adsorptive capacity of the adsorbent was tested using Langmuir and Freundlich isotherm. It was noted that the data fitted better in the Linearized Langmuir adsorption isotherm.**

***Keywords*– Adsorption, Activated carbon, Sawdust, Ethylacetate and Adsorption Isotherms**

I. INTRODUCTION

The value-adding step in the process industry is often found in the separation and purification of products. Adsorption technology has an important share in this step. Also, adsorption is important in the removal of undesired components from, for example wastewater and air streams. Ethylacetate are harmful to both human, animals. In human, it causes irritation of the eye and upper respiratory tract at concentration above 400ppm.it also causes eczema and depression of the heart function. At high concentration, it causes narcosis and depression of the heart function in animals [1].

There are many materials, both natural and from synthetic that have sorption capacity. The most important commercial adsorbents are activated carbon, molecular-sieve zeolites, silica gel and activated alumina. The applications of these adsorbents depend on their particular sorptive properties (Crittenden and Thomas, 1998). Applications and the use of activated carbon are, among other things: recovery of nitrogen from air, ethene from methane and hydrogen, removal of odours from gases in gas masks and water purification, including removal of phenol, halogenated compounds, pesticides, caprolactam and chlorine. The adsorption of organic pollutants can be carried out employing different adsorbents.

Among them, activated carbons are one of the best options due to their hydrophobic properties and their high surface area [2], [3].The main objective of this research work is to study the adsorption of ethyl acetate from ethylacetate-water mixture using activated carbon. Also, to determine the optimum conditions of the factors (time, temperature, and particle size) needed to achieve the best adsorption performance using ANOVA.Ethyl acetate-water mixture is a heteroazeotropic mixture, but in this study, it was dealt as a homoazeotropic mixture.

II. MATERIALS AND METHODS

A) Materials

The widely available adsorbent used in this study waswood particle sawdust of hard mahogany wood type collected from timber market, Kenyetta in Enugu State and screened through a set of sieves to geometrical size (75-600µm). The adsorbate used in this study was ethyl acetate gotten from Pymotech Laboratories in Enugu State.

B) Activated carbon preparation

Activated carbon was prepared using thermal activation by heating the sawdust in the absence of air using muffle furnace at 200^{o}C, 350^{o}C, 500^{o}C, 600^{o}C for 30,60, 90,120minutes.

C) Characterization of activated carbon

Determination of Iodine Number: Standard solution of 0.1N iodine solution was prepared. 3 drops of freshly prepared starch indicator was added to 20mls of 0.1N iodine solution and titrated with 0.05N sodium thiosulphate.

For the sample – 0.2g of the activated carbon was weighed and poured in the filter paper. 20ml of the standard (Std iodine solution was measured and poured in a filter paper. 2mls of standard (Std iodine solution was measured and poured in the sample in the filter paper). 10mls of the filtrate was pipette and 3 drops of freshly prepared starch indicator added and titrated with 0.005N Sodium thiosulphate.

Observation – Colourless

Moisture Content: The petridish was washed and dry in an oven and was weighed. 1g of the sample was weighed and added in the petridish, put in the oven and dry at 100^{o}C for 2hrs and cooled down in the desiccator and weighed. It was dried in an oven again at 100^{o}C for 30mins, cooled down and weighed again. Drying and weighing continued until a constant weight was obtained.

Bulk Densities: Bulk densities were determined using 50g (VP) of the starch powder. This was gently poured through a short stemmed glass funnel into a 100mls graduated cylinder.

Ash Content: Empty platinum crucible was weighed after which the empty crucible + 1g of the sample were weighed. The platinum Crucible and the content were heated over a flame from a Bunsen burner until the sample turned white colour. The crucible was cooled in desiccators and weighed again.

pH: The pH meter was deep into the samples and the value of the pH was taken.

D) Adsorption Experiment

The 0.4 and 0.05 normal solutions were prepared with the same volume 1000cm^3. Each of these solutions was divided into two equal parts of 500cm^3 each. One part was used to determine the exact concentration C^1 of the ethyl acetate before adsorption (initial concentration) by titrating an aliquot with NaOH. The other part was used for the adsorption by placing 1g of the activated carbon and shaking periodically for about 30mins until equilibrium has been established. The mixture was filtered and aliquot was titrated again with NaoH to determine the final concentration C^{11}.

E) Experimental Design

The experimental design was done using the Latin square experimental design method to determine the effect of the factors, factor X_1 (rows), Factor X_2 (column), Factor X_3 (Latin alphabets) for the Time, Temperature and Particle Size respectively on the adsorption of ethyl acetate.

F) FTIR Analysis

The FTIR Analysis was done using the FTIR Spectrometer to determine the type of functional group present in the samples taken and also about the concentration of the bonds.

III. RESULT AND DISCUSSION

Table 1 shows that the activation condition of Sample B gave the highest iodine number. High moisture contents contribute to low gas heating value. This is because, dry biomass burns at higher temperature and thermal efficiency than wet biomass. Sample B also gave the highest moisture content of 32%. High moisture content contributes to low thermal efficiency since the heat is used for drying purposes. Sample A and C gave the lowest moisture content at 500^{o}C and 600^{o}C within 60 and 30mins respectively. Flame temperature is directly related to the amount of heat necessary to evaporate in the biomass fuel. Mckendry (2002a, b) reported that biomass fuel with moisture content above 30% is difficult to ignite and reduces the heating value due to the need to evaporate the additional moisture before combustion.

A) Characterization Results

Table 1: Characterization of Activated Carbon

SAMPLE	TEMP (^{O}C)	TIME (mins)	IODINE No.	BULK DENSITY (g/mls)	ASH CONTENT (%)	pH	MOISTURE CONTENT (%)
A	500	60	76.143	0.42	3	4.75	10
B	-	-	96.448	0.29	1	4.70	32
C	600	30	83.658	0.45	4	4.96	8

Sample A ——— Activated Carbon at 600µm, 60mins, 500^{o}C
Sample B ——— Raw saw dust
Sample C ——— activated carbon at 180µm, 600^{o}C, and 30mins.

Table 2: ANOVA results for saw dust (0.05N)

SOURCE OF VARIATION	NO OF DEGREES OF FREEDOM	ESTMATION OF VARIATION	F RATIOS
ROWS Q(X1) 1.855×10^{-6}	$V_1 = 3$	$S_1^2 = Q(X_1)/V_1 = 6.183 \times 10^{-7}$	2.904×10^{-1}
COLUMN Q(X2) 5.765×10^{-6}	$V_2 = 3$	1.922×10^{-5}	9.023
TREATMENTS A,B,C,D, Q(X3) 1.025×10^{-6}	$V_3 = 3$	3.417×10^{-7}	1.604×10^{-1}
RESIDUALS Q_{RES} 1.2775×10^{-5}	$V_{RES} = 6$	2.1298×10^{-6}	

Table 3: ANOVA results for saw dust (0.4N)

SOURCE OF VARIATION	NO OF DEGREES OF FREEDOM	ESTMATION OF VARIATION	F RATIOS
ROWS Q(X1) 6.652×10^{-6}	$V_1 = 3$	$S_1^2 = Q(X_1)/V_1 = 2.217 \times 10^{-6}$	7.163×10^{-1}
COLUMN Q(X2) 4.31688×10^{-6}	$V_2 = 3$	1.439×10^{-6}	4.645×10^{-1}
TREATMENTS A,B,C,D, Q(X3) 8.167×10^{-6}	$V_3 = 3$	2.722×10^{-6}	8.787×10^{-1}
RESIDUALS Q_{RES} 1.859×10^{-5}	$V_{RES} = 6$	3.098×10^{-6}	

For level of significance $\alpha = 0.05$ and degree of freedom V1 = V2 = V3 = 3, $V_{res} = 6$. $F_{Table} = 4.76$.
Comparing between FI (I = 1, 2, 3) and F_{Table}.
X_1 (Process Duration) – 30, 60, 90, and 120mins.
X_2 (Temperature of Activation) – 200, 350, 500 and 600°C.
X_3 (Particle Sizes) – A = 75µm, B = 212µm, C = 180µm and D = 600µm.
The exact concentration C^1 of ethyl acetate is for:
0.4 – 0.39
0.05 – 0.045

B) ANOVA Results

From Table 2 and Table 3, it was observed that factor X_2 is significant, showing that the adsorption of ethyl acetate unto activated carbon using saw dust with initial concentration of 0.05N is significantly affected by the temperature, while factor X_1 and X_3 are insignificant showing that the adsorption of ethyl acetate using saw dust with initial concentration of 0.05N is not significantly affected by process duration and particle sizes.

C) Adsorption Isotherms

Freundlich Adsorption Isotherm: The Linear plots of log A against logC_e shows that the adsorption follows Freundlich Isotherm model. K and n were calculated from the intercept and slope of the plots. K is the adsorption or distribution coefficient and represents the quantity of ethylacetate adsorbed onto activated carbon adsorbent for a unit equilibrium concentration. The higher the K, the higher the adsorption efficiency. K value of the plot of Linearized Freundlich Isotherm of ethylacetate onto activated carbon at 500°C, with initial concentration of 0.05N gave the highest value (0.83mg/g) as shown in (Fig. 1). This shows that the activated carbon at 500°C with initial concentration of 0.05N gave the highest adsorption efficiency.

The slope 1/n ranging between 0 and 1, is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as it gets closer to zero as observed by Haghscresht, F [4]. According to Treyball, it has been shown using mathematical calculation that values of n between I and 10 represents beneficial adsorption.

Langmuir Isotherm for Activated Carbon: The linear plots of C_e/q_e Versus C_e shows that the adsorption obeys Langmuir Isotherm model.Q_0 and b were determined from the slope and intercept of the plot Langmuir constant related to the adsorption capacity and energy and presented into adsorption.

R_L values between 0 and I indicate favourable adsorption of ethylacetate untoactivated clay. The plot of the Langmuir isotherm for activated carbon in(Fig. 2) shows the highest isotherm line at 350°C with initial concentration of 0.05N indicating the best adsorptive capacity as was also observed by Nester *et al*, 2004 [5].

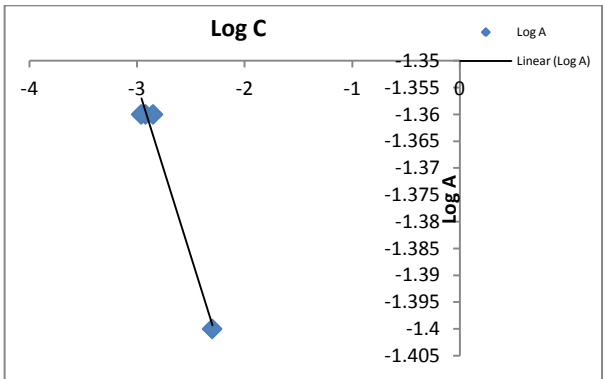

Fig. 1: Plot of the Linearized Freundlich Adsorption Isotherm of ethylacetate unto activated carbon (sawdust) at 500°C and initial concentration of 0.05N

Table 4: Parameters for Freundlich Isotherm for saw dust (0.05N) AT 500°C

Log A	Log C	k	1/n	N
-1.36	-2.92	0.83	0.15	6.7
-1.4	-2.3			
-1.36	-2.85			
-1.36	-2.96			

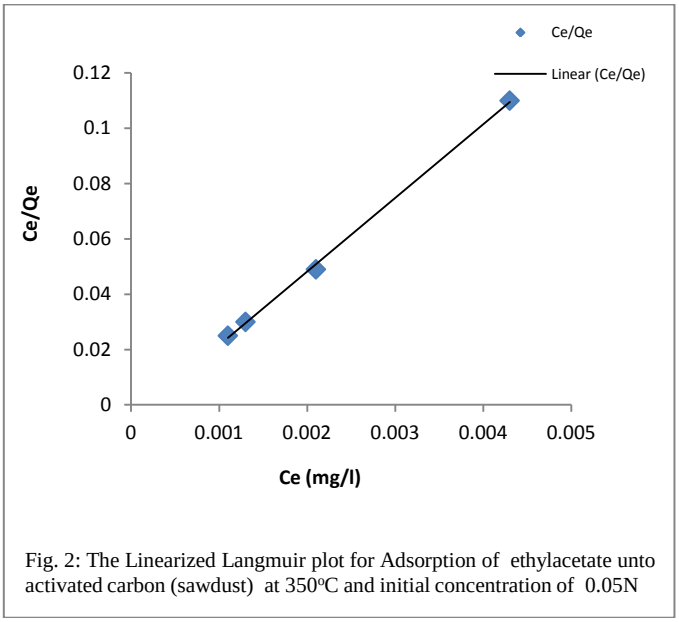

Fig. 2: The Linearized Langmuir plot for Adsorption of ethylacetate unto activated carbon (sawdust) at 350°C and initial concentration of 0.05N

At 350°C

C_e/Q_e	C_e	Q_o	B	R_L	R^2
0.11	0.0012	0.0375	-0.00001876	1	0.999
0.049	0.0021				
0.03	0.0013				
0.025	0.0011				

D) Infrared Position of Various Bond Variations

Table 5: Tables Showing Infrared Position of Various Bond Variations for different samples

Sample A. (Activated Carbon) at 600µm, 60mins, 500°C.

Peaks Cm^{-1}	Bond	Mode	Concentration
3417.19	O-H	Stretch	Strong
1640.93	C=O	Stretch	Strong
1033.93	C-O	Stretch	Strong
914.24	C-O	Stretch	Strong
783.16	C-H	Bend	Strong
689.11	C-H	Bend	Strong

Sample B. (Raw Saw dust)

Peaks Cm^{-1}	Bond	Mode	Concentration
3428.37	O-H	Stretch	Strong
2891.42	C-H	Stretch	Strong
1740.67	C=O	Stretch	Strong
1612	C-H	Bend	Weak
1626.68	C-H	Bend	Weak
1464.24	C-H	Bend	Variable
1156.47	C-O	Bend	Strong
1016.83	C-O	Bend	Strong
897.15	C-h	Bend	Strong
680.56	C-O	Bend	Broad

Sample C. (Activated Carbon) at 180µm, 600°c, and 30mins

Peaks Cm-1	Bond	Mode	Concentration
3434.28	O-H	Stretch	Strong
2925.71	C-H	Stretch	Strong
1615.28	N-H	Bend	Medium
1022.53	C-N	Stretch	Medium
891.45	C-N	Bend	Strong
794.55	C-N	Bend	Strong

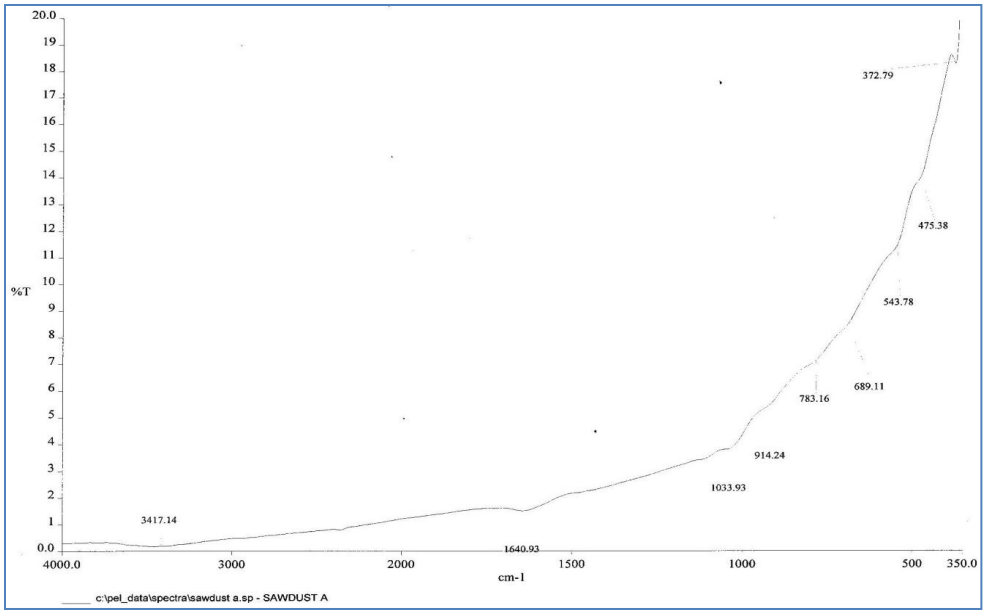

Fig. 3: FTIR Graphical Analysis of Sample A

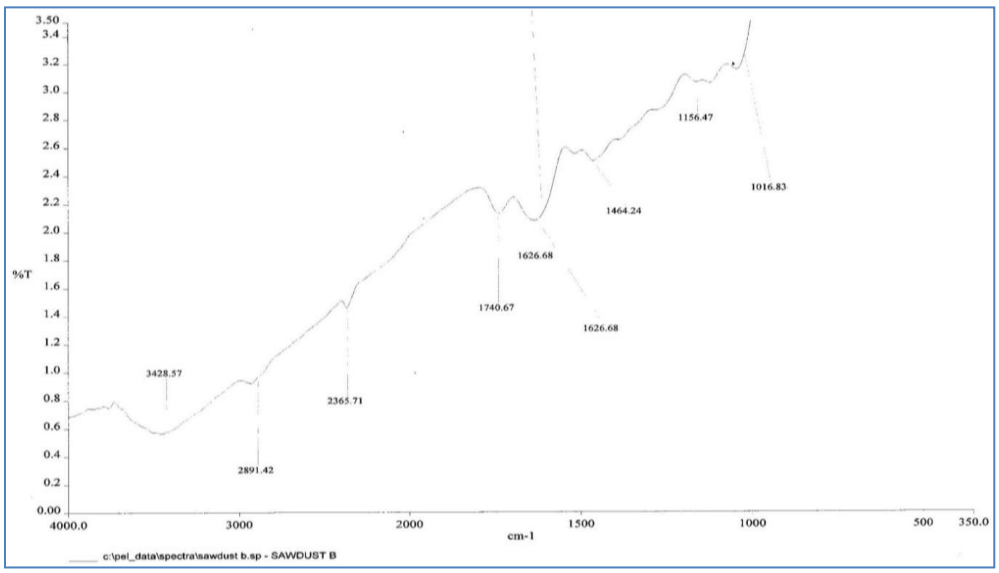

Fig. 4: FTIR Graphical Analysis of Sample B

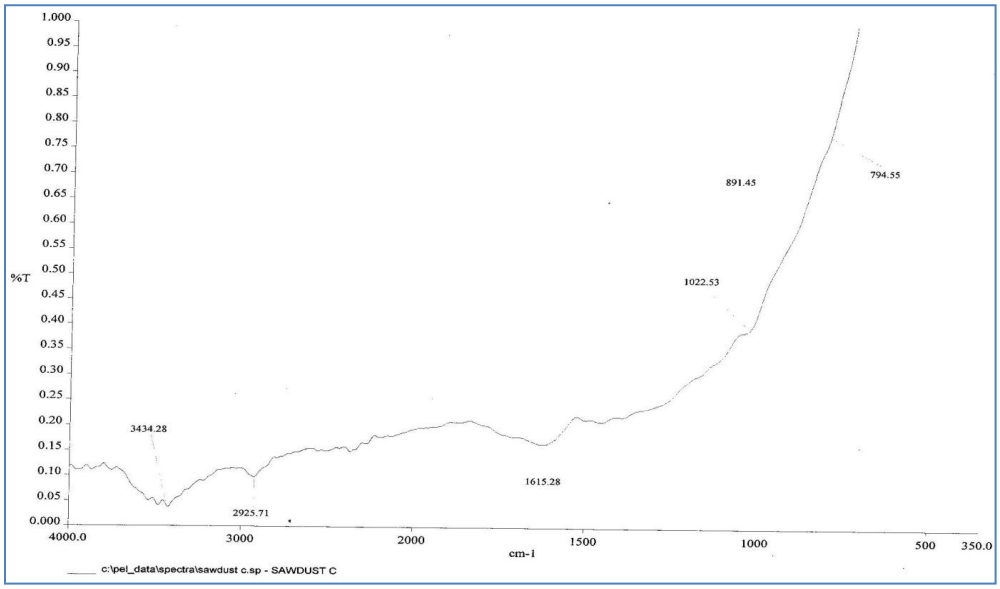

Fig. 5: FTIR Graphical Analysis of Sample C

FTIR of sample A (Fig. 3) shows all the functional groups concentration to be strong. It was found in sample A that the functional groups that are abundantly present is the C-H group.

Sample B (Fig. 4) shows that the C-H group are Present in abundance though other groups like the C-O, C=O and O-H are present too.

Analysis of the FTIR spectra of sample C (Fig. 5) shows that the compound mostly present is the amide group (C-N) showing strong and medium concentrations.

IV. CONCLUSION

Activated Carbon was shown in this study to be an effective adsorbent in the removal of ethyl acetate from ethyl acetate - water mixtures. The maximum adsorption takes place in the PH of 4.75. The optimum conditions were activation of the hardwood sawdust at 500^{o}C for 60mins.The Langmuir and Freundlich isotherms were used for fitting the experimental data in the adsorption studies to understand the extent and degree of favourability of adsorption. The range of n 1 to 10 indicated favourable

adsorption for the Freundlich plots while R_L values between 0 to 1 indicated favourable adsorption for Langmuir plots. The FTIR analysis on the samples indicated higher wavelengths (cm^{-1}) for the activated samples.Ethyl acetate-water mixture is a heteroazeotropic mixture, but in this study, it was dealt as a homoazeotropic mixture.

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