

# Production And Characterisation Of Activated Carbon From Melon Husks By Chemical Activation For The Removal Of 2, 3-Divinyl Dimethyl Phosphate (DDVP) From Aqueous Solution

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**Abstract:** *In this study, the removal of DDVP from aqueous solution was investigated with the aim of detoxifying contaminated bodies of pesticides. Three low-cost activated carbon prepared from Melon husks using 3 activating agents (phosphoric acid, potassium carbonate and nitric acid), were used to remove DDVP from aqueous solution. The one step activation process was adopted, using three activating agents: phosphoric acid, potassium carbonate and nitric acid. Adsorption Isotherm (Freundlich, Langmuir and Temkin) and Kinetic studies were carried out. The Isotherm studies revealed the experimental data fitting Freundlich model while the kinetics showed that the data fits best into the pseudo-second order kinetic model. The research concludes that melon husk is a good precursor for use for the production of low cost activated carbon, thus a cheaper alternative to the highly expensive commercial activated carbon.*

**Keywords:** *Melon husks, DDVP, Activating agents, Activated carbon*

## I. INTRODUCTION

Adsorption is the adhesion of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface. This process creates a film of the adsorbate on the surface of adsorbent. This process differs from absorption in which a fluid (adsorbate) permeates or is dissolved by a liquid or solid (adsorbent). Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent, or metallic) of the constituent atoms of the material are filled by other atoms of the material. However, atoms of the surface of the adsorbent are not wholly surrounded by other adsorbent atoms and therefore can attract adsorbates. The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption

(characteristic of weak Van der Waals forces) or chemisorption (characteristic of covalent bonding) (Ferrari *et al.*, 2010). Adsorption is usually described through isotherms; that is, the amount of adsorbate on the adsorbent is a function of its pressure (if gas) or concentration (if liquid), at constant temperature.

The continued use of commercial activated carbon for adsorption has become unbearably expensive. There has been an urgent need to get alternatives. The sources from agricultural wastes are gaining ground in this regard. These sources are abundantly available and are being synthesized and used as adsorbent for a variety of applications today. The dumping of agricultural wastes in the environment has become a source of concern, as they constitute solid waste. These wastes distort the aesthetic quality of the environment, leading to serious deleterious effects such as obnoxious odour and

associated diseases. The utilization of these wastes could be an excellent method of solid waste management technique that converts waste to wealth. The incidences of deaths, reported as a result of food poisoning in Nigeria is on the increase, particularly deaths recorded as a result of consuming porridge beans that are preserved using pesticides. Reports by the World Health Organisation (WHO) estimates that at least three million cases of acute poisoning and 29,000 deaths occur annually in the world due to exposure to pesticides. The report indicates a rapid annual increase in sales, use and dependence on pesticides in developing countries including Nigeria. It is estimated that up to 40% of harvested food can be lost if adequate storage and preservation measures are not employed. The need to store and preserve food which has been harvested is therefore as important as its production. However, the storage and preservation of grains and pulses is only valuable in meeting food storages, if after storage, the harvested produce is still fit for human consumption and poses no health hazard (Awofadeji, 2008).

The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption (characteristic of weak Vander Waals forces) or chemisorption (characteristic of covalent bonding). It may also occur due to electrostatic attraction (Ferari *et al.*, 2010).

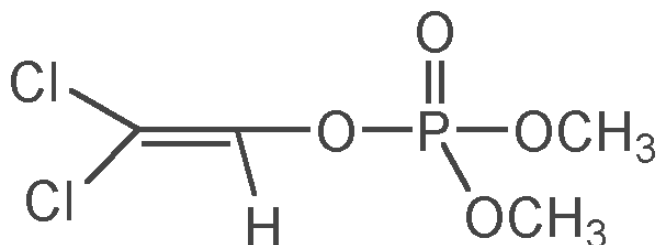


Figure 1: Structure of DDVP

Dichlorvos (2, 3-dichlorovinyl dimethyl phosphate) (Fig.1) is one of the classes of insecticides referred to as organophosphates used to control household and stored products insects. It is effective against mushroom flies, aphids, spider mites, caterpillars, and white flies in greenhouse, outdoor fruits, and vegetable crops (Lotti, 2001). It acts against insects as both a contact and a stomach poison (Lotti, 2001). Dichlorvos pesticide self-poisoning is an important clinical problem in the developing world, and kills an estimated 200 000 people every year (Michael *et al.*, 2008). Concentrates of dichlorvos is mildly irritating to skin and may cause localized sweating, involuntary muscle contractions and burning sensations or actual burns (Aaron, 2001). When inhaled, the first effects are usually respiratory and may include bloody or runny nose, coughing, chest discomfort, difficult or short breath, and wheezing due to constriction or excess fluid in the bronchial tubes. Eye contact will cause pain, bleeding, tears, pupil constriction, and blurred vision.

## II. MATERIALS AND METHODS

Melon husks samples were collected from local farmers in Keffi, Nasarawa State during harvest and thoroughly washed with water to remove all surface impurities, after which it was sun-dried. The sample was then ground with mortar and pestle

and sieved with a 2mm mesh size sieve and then dried further in an oven overnight.

The selected pesticide, 2, 3-dichlorovinyl dimethyl phosphate (DDVP), was purchased from designated shops for use in the research as adsorbate. This was selected because it is the pesticide majorly used for beans storage as well as the storage of many other legumes. It is also the pesticide commonly used in the home as insecticide. The analysis was carried out at the Laboratory Complex of Federal Ministry of Agriculture, Abuja road, Kaduna.

## REAGENTS, CHEMICALS AND INSTRUMENTS

All reagents and chemicals that were used are of analytical grade. Distilled water was used all through for the preparation of standard solutions. All equipment and apparatus used were standardized to specification.

## PREPARATION OF ACTIVATED CARBON FROM THE SAMPLES

The one-step activation process of adsorbent production was adopted. In this method, melon husk was activated separately by mixing each with standard solutions of  $H_3PO_4$ ,  $K_2CO_3$ , and  $HNO_3$ , respectively in the ratio of 1:1 (ie, 1g of sample to 1ml of activating agent) and allowed to stand for 24 hours (Gimba *et al.*, 2004; Malik, *et al.* 2006).

The separate samples in crucibles were loaded into a muffle furnace to attain a temperature of  $600^\circ C$  and maintained for 2h. It was allowed to cool to room temperature and then washed separately with 0.1M HCl in order to remove surface ash and further rinsed with distilled water to remove excess acid to a pH of between 6 to 8. Each of the samples was dried in an oven at  $105^\circ C$  to remove the moisture content. The different adsorbents produced from the melon husks using the 3 different activating agents were stored in a desiccator for further analysis.

## CHARACTERIZATION OF THE ACTIVATED CARBON

Different physicochemical analysis, including moisture content, ash content, bulk density and pH were all determined using standard methods (Nwufo & Ezeoma, 1995). Preliminary analysis was also carried out on the produced activated carbon by determining methylene blue number, Iodine content and Scanning electron microscopy (SEM).

## EFFECT OF INITIAL CONCENTRATION AND CONTACT TIME

The determination the equilibrium time for maximum DDVP uptake was carried out using different concentrations of DDVP (5, 10, 15, 20 and 25 mg/l) and contact time for adsorption. 1g of each of the activated carbons produced (MHAC<sub>x</sub>, MHAC<sub>y</sub> and MHAC<sub>z</sub>), was added into 100ml of the different concentrations of the adsorbate and shaken in a flask shaker at 150 rpm for different time ranges (10, 20, 30, 40, 50, 60, 70, 80 and 90 min). After each time range, the mixture was quickly filtered and the absorbance of the filtrate was determined using UV/Visible spectrophotometer at 232nm.

The absorbance readings of the filtrate were compared with the calibration curve of the DDVP and the concentration of each filtrate at each time interval was determined.

The concentration of DDVP adsorbed by the adsorbent,  $C_{ad}$  (initial concentration- concentration of filtrate), and adsorption capacity,  $q_e$ , were calculated.

$$\text{Adsorption capacity}(q) = (C_0 - C_f) * v/w$$

$C_0$  – initial concentration in mg/l

$C_f$  – final concentration from sample in mg/l

$q$ - adsorption capacity in mg/g

$v$ - volume in litre

$w$ - weight of the activated carbon in g

#### ADSORPTION ISOTHERMS STUDIES

The purpose of the adsorption studies is to relate the adsorbate concentration in the bulk and the adsorbed amount at the interface. The Langmuir model is based on the assumption that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface with no lateral interactions between the sorbed molecules (Nwufu & Onche, 2010). It assumes homogeneity of active sites.

The Freundlich Isotherm is an empirical equation employed to describe heterogeneous systems and Temkin considered the effects of some indirect sorbate/adsorbent interactions. The heat of adsorption of all the molecules in the layer decreases linearly with coverage.

It is worthy of note that at equilibrium,  $q_t = q_e$  and  $C_t = C_e$

#### FREUNDLICH ADSORPTION ISOTHERM

$$Q_e = K_f + C_e^{1/n}$$

Linearizing the above equation,

We have:

$$\ln Q_e = \ln K_f + 1/n \ln C_e$$

A plot of  $\ln Q_e$  against  $\ln C_e$  gives the Freundlich Isotherm where,

$K_f$  = Freundlich adsorption constant (mg/g), also called Freundlich adsorption capacity

$n$  = adsorption intensity or binding force

$C_e$  = the equilibrium concentration of adsorbate (mg/l) in solution

$Q_e$  = the amount of DDVP adsorbed per gram of the adsorbent at equilibrium

If  $n$  lies between 1 and 10, this indicates favourable adsorption i.e adsorption intensity is strong.

#### LANGMUIR ADSORPTION ISOTHERM

$$q_e = \frac{Q_0 K_1 C_e}{1 + K_1 C_e}$$

Linearizing the equation, and replacing  $K_1$  by  $b$ , it will be:

$$C_e/q_e = 1/Q_{0b} + C_e/Q_0$$

A plot of  $C_e/q_e$  against  $C_e$  gives the Langmuir adsorption Isotherm

$C_e$  = the equilibrium concentration of adsorbate (mg/l) in solution

$q_e$  = the amount of DDVP adsorbed per gram of the adsorbent at equilibrium

$Q_0$  = maximum monolayer coverage in mg/g

$b$  = Langmuir isotherm constant (L/mg)

$$r = \frac{1}{1 + bC_0}$$

$r$  = the separation ratio or adsorption dimensionless constant, also called equilibrium parameter

$C_0$  = initial concentration of the adsorbate in mg/l

If  $0 < r < 1$ , then the Langmuir adsorption Isotherm is favourable

The following equations were used to complete the data:

The amount of DDVP adsorbed by each activated carbon is given by:

$$q_e = [(C_0 - C_e)/w] * v \text{ in mg/g}$$

where,  $C_0$  is the initial concentration of the DDVP in mg/l before contact with the activated carbon.

$C_e$  is the concentration of the DDVP in mg/l after adsorption with the activated carbon.

$w$  is the weight of the activated carbon used, in grammes.

$v$  is the volume of the DDVP (L) used in contact with the different activated carbon.

$$\% \text{Removal} = [(C_0 - C_e)/C_0] * 100.$$

#### TEMKIN ADSORPTION ISOTHERM

The adsorption potential of adsorbent to adsorbate can also be tested by applying Temkin isotherm model. Temkin considered the effects of some indirect sorbate/adsorbate interactions on adsorption isotherms and suggested that because of these interactions, the heat of adsorption of all molecules in the layer would decrease linearly with coverage. Temkin isotherm has been generally applied in the form:

$$q_e = \left(\frac{RT}{b}\right) \ln AC_e, \text{ and when linearized it becomes:}$$

$$q_e = B \ln A + B \ln C_e,$$

where  $B = \left(\frac{RT}{b}\right)$ ,  $b$  is the Temkin constant related to heat of sorption (J/mol);  $A$  is the Temkin isotherm constant (L/g),  $R$  the gas constant (8.314J/mol K) and  $T$  is the absolute temperature (K). Therefore, by plotting a graph of  $q_e$  against  $\ln C_e$  enables one to determine the constants  $A$  and  $b$ .

#### ADSORPTION KINETIC STUDIES

Kinetic studies of the raw samples as well as the activated carbon were carried out at different concentration of the adsorbate (DDVP). The extent of adsorption was investigated as a function of time. The amount adsorbed at time,  $t$ ,  $q_t$  (mg/g) was calculated by

$$q_t = \left(\frac{C_0 - C_t}{w}\right)V$$

Two different models were used, the pseudo-first-order and pseudo-second-order.

The pseudo first order model was described by Lagergren in the form of a linear equation:

$$\log (q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t$$

A graph of  $\log (q_e - q_t)$  against time,  $T$ , is plotted. Values of  $q_e$  and  $q_t$  are the amounts of DDVP adsorbed (mg/g) at equilibrium and at time,  $t$  (min), respectively, while  $k_1$  is the rate constant of adsorption ( $\text{min}^{-1}$ ). The value of  $q_e$  is

calculated from the intercept and that of  $k_1$  is obtained from the slope of the graph.

If the plot is found to be linear with good correlation coefficient and  $q_e$  calculated agrees with  $q_e$ , experiment, then it indicates the Lagergren equation is appropriate to DDVP adsorption on the activated carbon, so the adsorption process is a pseudo first order process.

The pseudo second order kinetics is expressed in a linear form as:

$$t/q_t = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) t$$

Where  $q_e$  is the equilibrium adsorption capacity, and  $k_2$ , (g/mg min) is rate constant of the second order adsorption rate. A graph of  $t/q_t$  against  $t$  is plotted and the values of  $k_2$  and  $q_e$  are calculated from the intercept and slope of the graph respectively. The correlation coefficients,  $R^2$  is also obtained from the graph. A linear plot with very high values of  $R^2$  and good agreement of the values of the calculated and experimental  $q_e$  when compared for the pseudo second order, then the adsorption of DDVP onto the activated carbon is said to be a second order kinetics (Njoku, *et al.* 2015).

Kinetic models enable one to study the adsorption rate, model the process and predict information about adsorbent/adsorbate interaction (physisorption or chemisorption). Very high values of  $R^2$  in addition to the good agreement between experimental and calculated values of  $q_e$  indicate whether it is first or second order kinetic.

### III. RESULTS AND DISCUSSIONS

The different activated carbons produced from melon husks using three activating agents are labeled MHAC<sub>x</sub> (from phosphoric acid), MHAC<sub>y</sub> (from potassium carbonate) and MHAC<sub>z</sub> (from nitric acid). The untreated melon husk sample is labeled MH. The moisture content and bulk densities of the raw and activated carbon produced from melon husk all meet the requirements of being good precursors of adsorbents (Aziza, *et al.* 2009), (Table 3.1). The methylene blue number of MHAC<sub>x</sub> was found to be 120 and the other ones have MBN of less than 100 (Table 3.2). This indicates also that the activated carbon using phosphoric acid is the only one that is likely to be a good source of adsorbent with mesoporous structure. All activated carbon with MBN of 100 and above are said to be good adsorbents (Malik *et al.* 2006). The iodine value (Table 3.3) showed that all the activated carbon meet the specification for good adsorbents, having Iodine number of between 300-1200 (Etonihu, *et al.* 2011). The MHAC<sub>z</sub> has the highest iodine number, suggesting that its morphology is more microporous than the others. From the SEM morphology (plate I-V), it is clear that all the activated carbon produced show a mixture of micropores and mesopores, agreeing with the preliminary analysis of iodine and methylene blue number results. MHAC<sub>z</sub> showed the best pore distribution morphology.

S/NO	Sample	Moisture Content %	Ash Content %	pH	Bulk density g/cm <sup>3</sup>
1	MH	3.4	6.5	5.39	0.560
2	MHAC <sub>x</sub>	4.4	-	6.7	0.639
3	MHAC <sub>y</sub>	4.3	-	6.5	0.630

4	MHAC <sub>z</sub>	4.5	-	6.9	0.634
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Table 3.1: Physico-Chemical Parameters of the Samples

S/N	Sample	Methyleneblue number (MBN)
1	MHAC <sub>x</sub>	120.
2	MHAC <sub>y</sub>	97
3	MHAC <sub>z</sub>	67

Table 3.2: Methylene Blue Number (MBN) of the Activated Carbon

S/N	Sample	Iodine value
1	MH	804
2	MHAC <sub>x</sub>	423
3	MHAC <sub>y</sub>	559
4	MHAC <sub>z</sub>	868

Table 3.3: Iodine Value Determination for Raw and Activated Samples

### SEM FOR MELON HUSKS & MELON HUSKS ACTIVATED CARBON AT 1000 MAGNIFICATION

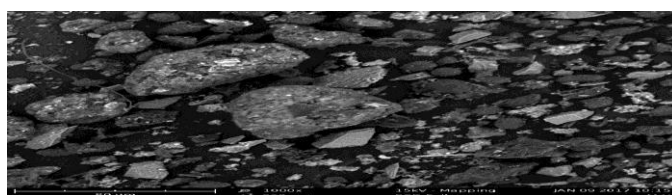


Plate II: SEM for Melon husk 1000 magnification

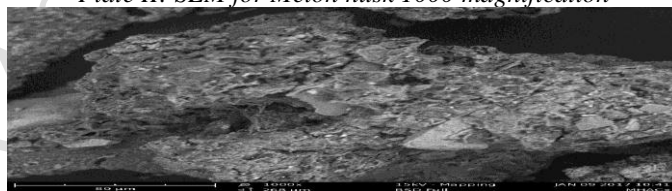


Plate IIa: SEM for Melon husk activated with phosphoric acid at 1000 magnification

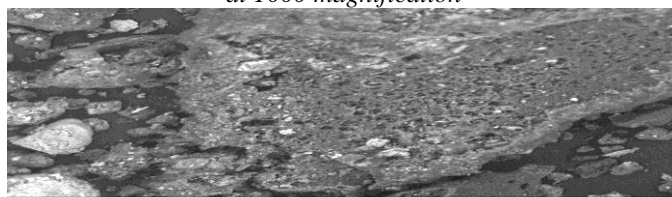


Plate IIb: SEM for Melon husk activated with potassium carbonate at 1000 magnification

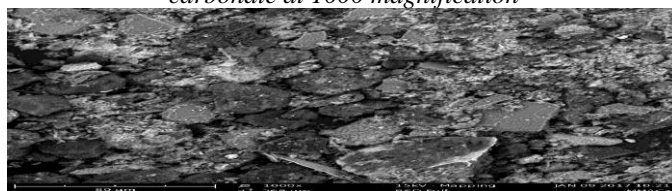


Plate IIc: SEM for Melon husk activated with nitric acid at 1000 magnification

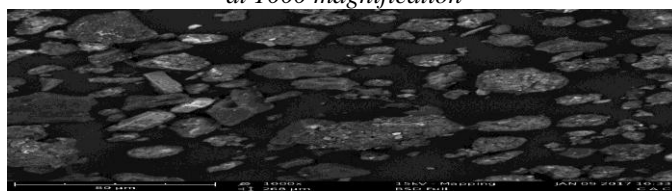


Plate 4: SEM for Commercial activated carbon at 1000 magnification

Sample	R <sup>2</sup>	Intercept	Slope	Binding force (n)	Adsorption Capacity(K)
MH	0.973	2.158	1.117	0.90	8.65
MHAC <sub>x</sub>	0.979	0.499	0.783	1.28	1.65
MHAC <sub>y</sub>	0.998	0.719	0.718	1.39	2.05
MHAC <sub>z</sub>	0.994	0.747	0.724	1.38	2.11
CAC	0.907	0.387	0.497	2.01	1.47

Table 3.4: Freundlich adsorption data for adsorption of DDVP on different Samples

Sample	R <sup>2</sup>	Intercept	Slope	Q <sub>0</sub>	Binding force(b)	Sep. Ratio (R <sub>L</sub> )
MH	0.164	7.882	0.117	8.54	1.09	0.035
MHAC <sub>x</sub>	0.978	1.402	0.319	3.13	2.23	0.018
MHAC <sub>y</sub>	0.907	1.891	0.259	3.86	2.04	0.019
MHAC <sub>z</sub>	0.838	2.039	0.232	4.31	2.11	0.018
CAC	0.992	0.882	0.486	2.06	2.34	0.017

Table 3.5: Langmuir adsorption data for adsorption of DDVP on different Samples

Sample	R <sup>2</sup>	Slope	Intercept	b (kJ/mol)	A (Temkin constant)
MH	0.960	0.784	0.437	2.90	1.16
MHAC <sub>x</sub>	0.980	0.768	0.644	2.96	1.24
MHAC <sub>y</sub>	0.938	0.677	0.456	3.35	1.15
MHAC <sub>z</sub>	0.916	0.706	0.421	3.21	1.14
CAC	0.974	0.474	0.743	4.79	1.17

Table 3.6: Temkin Adsorption data for adsorption of DDVP on various Samples

Tables 3.4, 3.5 and 3.6 show the Freundlich, Langmuir and Temkin adsorption data for adsorption of DDVP on melon husks and melon husks activated carbons. From the data, the correlation coefficient, R<sup>2</sup> for the Freundlich Isotherm model were found to be generally higher than that of Langmuir and Temkin models. The binding forces between DDVP and the activated carbon for Freundlich model are 0.9, 1.28, 1.39, 1.38 and 2.01 for MH, MHAC<sub>x</sub>, MHAC<sub>y</sub>, MHAC<sub>z</sub>, and CAC respectively. Binding force of activated carbon between of 1-10 indicates that the activated carbon can be utilized for use as adsorbent. The result however indicates that the raw sample of melon husk may not be adequate for use as adsorbent, but only the activated sample. Langmuir adsorption data shows their separation ratio (R<sub>L</sub>) (Table 3.5). From the results, it is evident that the values also meet the requirements of a good adsorbent, having values greater than 0, but less than 1 (Nwifo & Onche, 2010). The adsorption Isotherm models showed that melon husk activated carbon fits well into the freundlich adsorption Isotherm. This indicates that the adsorption that takes place is more likely to be multi-layer rather than monolayer.

Sample	R <sup>2</sup>	Slope	Intercept	Rate constant(k <sub>1</sub> )	q <sub>e</sub> , cal	q <sub>e</sub> , exp
MH	0.880	0.010	1.780	0.023	60.26	1.06
MHAC <sub>x</sub>	0.940	0.007	1.616	0.016	41.30	1.88
MHAC <sub>y</sub>	0.954	0.009	1.880	0.021	75.86	1.87
MHAC <sub>z</sub>	0.971	0.011	1.988	0.025	97.27	1.87
CAC	0.982	0.023	3.067	0.053	116.87	1.66

Table 3.7: Results of Pseudo-first-order kinetic model for raw sample and activated carbon

Sample	R <sup>2</sup>	Slope	Intercept	Rate constant(k <sub>2</sub> )	q <sub>e</sub> , cal	q <sub>e</sub> , exp
MH	0.995	0.823	8.926	0.076	1.215	1.06
MHAC <sub>x</sub>	0.999	0.519	1.094	0.246	1.927	1.88
MHAC <sub>y</sub>	0.999	0.519	1.277	0.211	1.927	1.87

MHAC <sub>z</sub>	0.999	0.519	1.260	0.214	1.927	1.87
CAC	0.999	0.561	3.355	0.094	1.783	1.66

Table 3.8: Results of Pseudo-second-order kinetic model for raw sample and activated carbon

Table 3.7 and 3.8 show the results of pseudo-first and pseudo-second order kinetic models. From the results, the coefficient correlation values, R<sup>2</sup> for the pseudo-first order kinetic models are generally high, but not as high as the R<sup>2</sup> values for the pseudo-second order kinetic models. The experimental values of the amount of DDVP adsorbed at equilibrium, q<sub>e</sub> for the adsorption data do not agree with the q<sub>e</sub> calculated using the pseudo- first order kinetic models. The q<sub>e</sub> values calculated from the pseudo-second order kinetic model, however are in close agreement with that of the experimental data. Very high values of R<sup>2</sup>, in addition to closeness in agreement between q<sub>e</sub> from experimental data and q<sub>e</sub> calculated from kinetic model indicates the fitness of the data to the type of kinetic models (Taha, *et al.* 2014). The kinetic studies therefore showed that the adsorption of DDVP on melon husk activated carbon is a pseudo-second order rate of reaction. A better fit to the pseudo- second order model suggests that the adsorption rate is dependent more on the availability of adsorption sites than the concentration of the DDVP in solution. The applicability of pseudo-second order to the data of DDVP onto melon husk activated carbon indicates that the concentration of both activated carbon and DDVP are involved in the rate determining step and the adsorption process may be chemisorptions.

#### IV. CONCLUSION

The study indicated that melon husk needs to be activated by an appropriate activating agent to make it feasible for use as an adsorbent. It also concludes that the most likely adsorption of DDVP onto melon husk activated carbon fits the Freundlich adsorption Isotherm, hence its tendency of multilayer adsorption and that the adsorption is dependent more on the availability of active sites of the adsorbent. Melon husk, can therefore be harnessed and put into use in the adsorbent technology, hence achieving the concept of converting waste to wealth.

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