



Removal of Phenol and Parachlorophenol from Synthetic Wastewater Using Prepared Activated Carbon from Agricultural Wastes

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Abstract: The aim of the present study is to remove Phenol and Parachlorophenol from synthetic wastewater using prepared activated carbon from agricultural wastes (rice husk, (RH) date stones, (DS) and palm fronds, (PF)) utilizing chemical and physical activation methods. Three principal operating parameters have been considered which are activation temperature, activation time, and impregnation ratio. The results showed that the activated carbon prepared from rice husk by chemical method at a temperature of 700 °C, activation time of 3 hours, and impregnation ratio of 3: 1 gives highest removal of phenol and Parachlorophenol. The results also showed that the activated carbon prepared by physical method from rice husk gives higher removal efficiency for phenol and Parachlorophenol than that of activated carbon prepared from date stones and palm fronds. The results also showed that the rate of removal of Phenol and Parachlorophenol increases with the increase of activation temperature, activation time, and impregnation ratio. The highest removal of phenol and Parachlorophenol are (84.38% and 94.65%) respectively. Application of the most prominent adsorption models shows acceptable agreement with Langmuir and Temkin models. Maximum adsorption capacity for the prepared activated carbon is found to be 39 mg/g and 38.82 mg/g for Phenol, 44.64 mg/g and 44.94 mg/g for Parachlorophenol respectively.

Keywords: Activated Carbon, Adsorption, Phenol, Parachlorophenol, Rice Husk

1. Introduction

Activated carbon is commonly defined as a carbonaceous material showing a well-developed surface area and porous texture. As a consequence, activated carbon has been widely used as adsorbent and in catalysis or separation processes (Kalderis et al., 2008; Tamai et al., 2009). The characteristics of activated carbon depend on the physical and chemical properties of the precursor as well as on the activation method (Demiral et al., 2008). Activated carbon can be produced from any carbonaceous solid precursor which may be either natural or synthetic. The choice of precursor is largely dependent on its availability, cost, and purity. Due to environmental considerations, agricultural wastes are considered to be a very important precursor because they are cheap, renewable, safe, and available at large quantities; in addition they have high carbon and low ash content (Kalderis et al., 2008).

Rice husk, date stones, and palms fronds are agricultural wastes, have been reported as a good adsorbents for many heavy metals and hydrocarbon compounds, lignocellulosic composition promotes the preparation of activated carbon from these precursors (Bouchelta et al., 2008).

There are two processes for the preparation of activated carbon: physical activation and chemical activation. Physical activation involves carbonization of a carbonaceous materials followed by activation of the resulting char in the presence of activating agents such as CO₂ or steam. In chemical activation, a raw material is impregnated with an activating reagent such as ZnCl₂, H₃PO₄, KOH, etc., and the impregnated material is heated in an inert atmosphere. There will be a reaction between the precursor and the activating agent leads to a development of porosity. Chemical activation is preferred over physical activation owing to the higher yield,

simplicity, lower temperature, and shorter time needed for activation, and good development of the porous structure (Guo and Rockstraw, 2007).

Phenolic compounds are classified to be extremely toxic for human beings and for all aquatic life. One of the most hazardous polluting phenolic compounds to the environment is phenol, which can exert negative effects on different biological processes and their presence even at low concentrations can cause unpleasant taste and odor of drinking water and can be an obstacle to the use of wastewater (Dabrowski et. al., 2005). Phenol was designated as priority pollutants by the USEPA, which takes the 11th place in the list of 129 chemicals; Environmental Protection Agency (EPA) has set a limit of 0.1 mg/L of phenol in wastewater (Salame and Bandosz, 2003). Other important polluting phenolic compound is Parachlorophenol. It can enter the human body through all routes and reacts easily in the blood to convert hemoglobin to methemoglobin, thereby preventing oxygen uptake even at low concentration.

Removing these pollutants or decreasing their concentration levels in the wastewater to the allowable or permitted levels is a target that needs to be achieved and reached by several environmental agencies and governments. Several ways have been developed to remove phenolic compounds from wastewaters, including electrochemical oxidation, chemical coagulation, solvent extraction, membrane separation, photo catalytic degradation, and adsorption. Among these methods adsorption is still the most popular and widely used technique for phenols removal, because of its simple design, easy operation, it can remove both organic as well inorganic constituents even at very low concentration, no sludge formation, and the adsorbent can be regenerated and reused again (Nevskaia et. al., 2004). Moreover the process is economic because it requires low capital cost and there are abundant low cost materials available which can be used as adsorbents (Halouli and Drawish., 1995). It is a mass transfer process that involves contact of a solid (adsorbent) with a fluid contacting the target solute (adsorbate). The efficiency of the adsorption process is mainly determined by the characteristic of the adsorbent such as high surface area, high adsorption capacity, microporous structure, and special surface reactivity. To the present day, activated carbon is the most widely used adsorbent because it has good capacity for adsorption of hydrocarbon compounds and heavy metals from wastewater.

Mohammed, N. A., Aseel, A. H., and Firas S. A. (2013) used Iraqi rice husk (IRH) to remove phenol from wastewater under various operating conditions. Results show that the higher removal efficiency was 89.73% and this efficiency is decreased with the increase of initial concentration, flow rate, and pH while it is increased with the increase of inlet concentration, bed height, and feed temperature.

Khu Le Van and Thu Thuy Luong Thi (2014) had prepared four activated carbon (AC) samples from rice husk under different activation temperatures. The specific surface area of AC sample reached $2681 \text{ m}^2 \text{ g}^{-1}$ under activation temperature of 800°C . The AC samples were then tested as an electrode

material.

Huaxing Xu et.al.(2014) prepared nanoporous activated carbon from the waste rice husks (RHs) by precarbonizing RHs and activating with KOH. The nanoporous carbon has the average pore size of 2.2 nm and high specific surface area of $2523.4 \text{ m}^2 \text{ g}^{-1}$.

Mervette, El Batouti, Abdel-Moneim M. Ahmed(2014) conducted batch adsorption of Ni(II) onto activated carbon prepared from rice husk and stated that the adsorption process depends on the initial concentrations, adsorbent dose, contact time, and pH.

Mohammad, Y.S.(2014)examined the applicability of rice husk activated carbon in an adsorption column for the treatment of phenolic refinery wastewater under various bed depth and flow rate. They found that the adsorption capacity is 28mg/g. The performance of the adsorption column is affected by bed depth and flow rate.

Chitaranjan Dalaia, Ramakar Jhab, and Desai a V.R. (2015) used rice husk based Activated Carbon (RHAC) to remove Iron and Manganese from groundwater. They stated that Iron and Manganese are 100% removed.

In spite of this; it suffers from a number of disadvantages. Activated carbon is expensive, and the higher the quality is the greater the cost. Consequently, there has been a growing interest in developing and implementing various potential adsorbents for the removal of hydrocarbon compounds and heavy metals from wastewater.

2. Experimental Work

Figure (1) represents a schematic diagram of the activated carbon preparation unit. It consists of the following parts:

1. Tubular reactor is made of stainless steel 310. Its height is 76cm and its internal diameter is 6.35cm, sample of raw material is placed in.
2. Electrical coil, to heat the reactor which contains the sample.
3. Nitrogen and carbon dioxide cylinders, to supply gas for the reactor during the preparation of activated carbon.
4. Flow meter, to measure the gas flow rate.
5. The reactor contains in the upper end an exhaust to release the produced gases during the process.
6. Temperature recorder (Digital Recorder), used to measure and control the temperature of reactor.

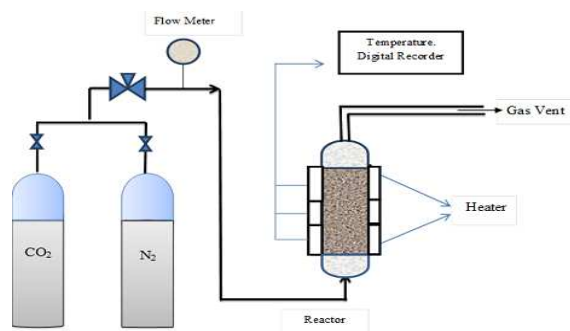


Fig. (1). Schematic diagram of the activated carbon preparation unit.

3. Material

Precursor: Rice husk, date stones, and palms fronds are used as the precursors in the preparation of activated carbon. These are first washed with water to get rid of impurities, dried at 110°C for 24 hour, and sieved. Only the fraction of particle sizes between 1 and 3 mm is selected for the preparation.

Activators: Zinc chloride (purchased from Didactic company) of 99.9% purity is used as a chemical reagents.

Adsorbate: Phenol (Ph) and Paraclorophenol (PCP) of purities higher than 99% are used as an adsorbate in this study.

Chemicals: Other chemical used are hydrochloric acid, sodium chloride, and acetone.

3.1. Preparation of Activated Carbon

3.1.1. Physical Activation Method

This method involves the following steps:

1. Raw material is washed with deionized water and subsequently dried at 105°C for 24 hour to remove moisture content.
2. The dried raw material is cut and sieved to a particle size of 1- 3mm.
3. Carbonization; raw material of step 2 is heated at a rate of 10°C/minute till the temperature reached 700°C under nitrogen of 99.9% purity flows at a rate of 150 ml/minute.
4. Activation; carbonized material is exposed to an oxidizing atmospheres, carbon dioxide at a temperature of 700°C for 3hours.
5. The activated product was then cooled to room temperature under nitrogen flow.
6. The samples are stored in a closed flask for adsorption applications.
7. Rice husk is washed with deionized water and subsequently dried at 105°C for 24 hour to remove moisture content.
8. The dried rice husk is cut and sieved to a particle size of 1- 3mm.

3.1.2. Chemical Activation Method

This method involves the following steps:

1. Chemical activation of the dried raw material was then done using activating agents $ZnCl_2$ using weight ratio of (1:1, 2:1, 3:1) and then deionized water is then added to dissolve $ZnCl_2$ pellets.
2. After chemical activation, sample is dried at 110°C for 15 hour.
3. Carbonization; rice husk of step 4 is heated at a rate of 10°C/minute till the temperature reached 500°C, 600°C, and 700°C under nitrogen of 99.9% purity flows at a rate of 150 ml/minute.
4. The activation step is done using the same reactor as in carbonization step. Once the final activation temperature reached, the gas flow is switched from nitrogen to CO_2 at a flow rate of 150 ml/minute for 1, 2,

and 3hours.

5. The activated product is then cooled to room temperature under nitrogen flow.
6. The activated product is washed with deionized water and hydrochloric acid (0.1M) until the pH of the washed solution reached 7 and subsequently dried at 105°C.
7. The sample is stored in closed flask for adsorption applications.

3.2. Batch Experiments

A series of batch experiments are carried out to determine the adsorption isotherms of Ph and PCP solutions onto activated carbon (AC), 0.2gram, at pH of 7. Activated carbon is placed into 250 ml flask containing phenol or parachlorophenol of 100ppm concentration. The mixture is stirred; the pH values are controlled by adding 0.1 N NaOH or 0.1 N HCl. The adsorption experiments are conducted at room temperature for 280 minute to achieve equilibrium. Then, the solutions are filtrated, and their concentration are determined by UV-Visible Spectrophotometer (Shimadzu UV-530) at $\lambda_{max} = 269nm$ and $\lambda_{max} = 280nm$ for Ph and PCP respectively. The adsorption capacity q_e (mg/g) of each solute onto AC was calculated using Equation (1)

$$q_e = \frac{V(C_o - C_e)}{W} \quad (1)$$

Where;

C_o and C_e are the initial and equilibrium concentration of adsorbate respectively (mg/L),

V is the volume of solution (L), and

W is the weight of AC (gram).

4. Results and Discussion

In the present work, samples of activated carbon are produced from different Iraqi waste materials (rice husk,(RH) date stones,(DS) and palm fronds(PF)) using two activation methods which are physical activation and chemical activation method. The prepared activated carbon samples are used to conduct many batch adsorption experiments for phenol and parachlorophenol of 100 mg/L concentration.

4.1. Results of Physical Activation Experiments

In physical activation method, 700°C is used as an activation temperature while the activation time is 3 hours. The results of batch adsorption experiments for phenol and parachlorophenol of 100mg/L concentration on raw materials (RH, DS, and PF) and on the prepared activated carbon samples by physical activation method (RH1, DS1, and PF1) are shown on Figures (2-5).

Figs.(2)&(3) indicated that phenol and parachlorophenol adsorption on raw materials (RH, DS, and PF) quickly reaches equilibrium (within 1 hour) with low removal for Ph and PCP. This is due to low surface area of the raw materials giving low adsorption sites. Adsorption process depends on

many parameters including the type and nature of adsorbent and adsorbate (Al-Sultani and Al-Seroury, 2012). However, parachlorophenol removal is higher than phenol due to its higher affinity (Ahmaruzzaman and Sharma, 2005). Moreover, rice husk, gives higher phenol and parachlorophenol removal (8.64% & 17.95% respectively) as compared with that of PF (4.38% & 10.65) and DS (5.69% & 8.36%). This is due to the main components of rice husk are carbon and silica; it has the potential to be used as an adsorbent. Thus, the ionic structure of silica provides a capability of adsorbing phenolic compounds which are polar molecules (Kumar, 1987).

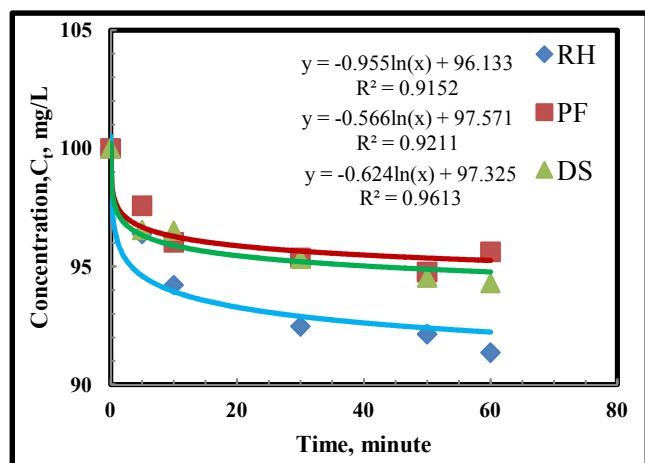


Fig. (2). Phenol adsorption on raw materials (RH, DS, and PF).

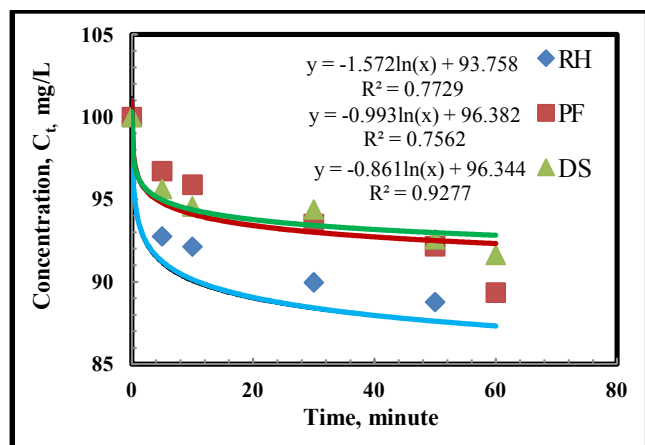


Fig. (3). Parachlorophenol adsorption on raw materials (RH, DS, and PF).

Figs.(4) & (5) represent the batch adsorption for phenol and parachlorophenol on the produced activated carbon samples (RH1, DS1, & PF1). These Figures indicated that (i) equilibrium time (about 4 hours) and phenol and parachlorophenol removal are higher than the corresponding of raw materials, (ii) the removal of parachlorophenol (64.42%, 51.78%, & 46.72% for RH1, PF1, & DS1 respectively) are higher than the corresponding values (58.4%, 40.21%, & 36.78) of phenol removal, and (iii) RH1 gives higher removal for both pollutants as compared with PF1 which is in turn gives higher removal as compared with DS1. These results are due to: physical activation enables

moisture loss as well as lignin decomposition of raw materials (Lapuerta et al., 2004). The treatment of raw material by physical activation also reduces the content of hemicellulose, lignin, and cellulose crystals which leads to an increase of the surface area compared to raw material (Daffalla et. al., 2010).

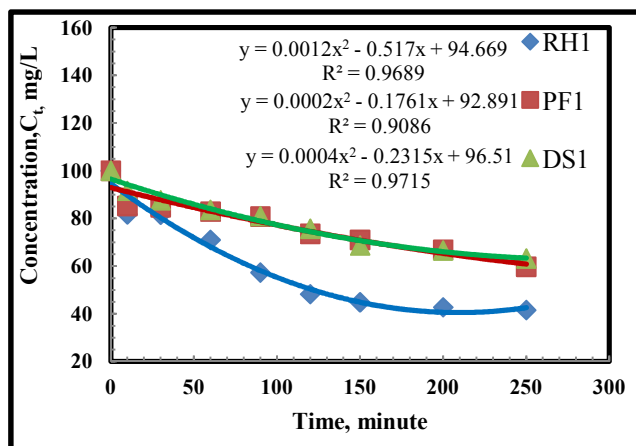


Fig. (4). Phenol adsorption on RH1, PF1, DS1.

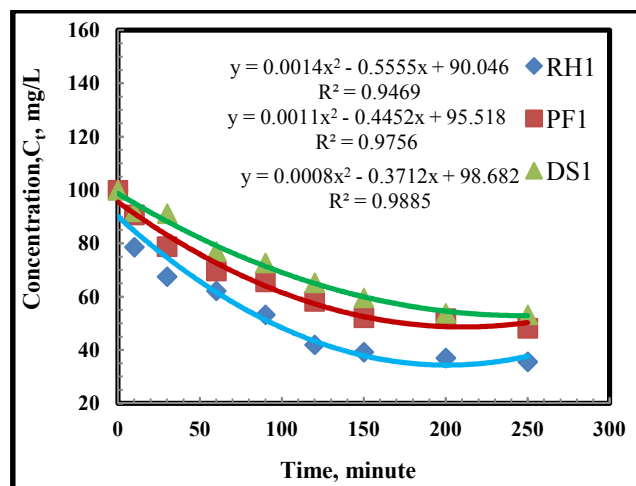


Fig. (5). Parachlorophenol adsorption on RH1, PF1, and DS1.

4.2. Results of Chemical Activation Experiments

Since rice husk gives the best adsorption performance for phenol and parachlorophenol when it is activated by physical activation method, it is used to perform chemical activation method under various operating conditions which are: activation temperature of 500°C, 600°C, and, 700°C activation time of 1, 2 and, 3 hour: impregnation ratio ZnCl₂/Rice husk of 1:1, 2:1, and 3:1. Table (1) represents the operating conditions and the name given for each sample of the prepared activated carbon.

Phenol and parachlorophenol batch adsorption experiment is conducted for each sample of the prepared activated carbon in order to test the effect of each operating parameter on the adsorption performance. The adsorption isotherms are shown graphically on Figs.(6-23).

Table (1). Operation conditions for chemical activation.

Run No.	Activation, Temperature, T, °C	Impregnation, Ratio, IR	Activation time, AT, hour	Produced, Activated, Carbon name
1	500	3:1	1	RHC1
2	500	3:1	2	RHC2
3	500	3:1	3	RHC3
4	500	2:1	3	RHC4
5	500	1:1	3	RHC5
6	600	3:1	1	RHC6
7	600	3:1	2	RHC7
8	600	3:1	3	RHC8
9	600	2:1	3	RHC9
10	600	1:1	3	RHC10
11	700	3:1	1	RHC11
12	700	3:1	2	RHC12
13	700	3:1	3	RHC13
14	700	2:1	3	RHC14
15	700	1:1	3	RHC15

4.3. Effect of Activation Temperature

Figures (6-11) represent adsorption data for phenol and parachlorophenol on various activated carbon samples under different operating conditions.

It is clear that the removal of phenol and parachlorophenol is increased with the increase of activation temperature with the highest removal for phenol (84.38%) and for parachlorophenol (94.06%) obtained for RH13 which is activated under 700 °C, 3:1 IR, and 3 hours activation time.

This is due to the formation of well-developed mesopores structures on the adsorbent during activation. The activation temperature is very essential parameter for pore structure of activated carbon, which determines the adsorption capacity (Hu et al., 1995). The volatiles from the samples continue to evolve with increasing carbonization temperature. The devolatilization process further develops the pore structure and creates new porosities and thus, increasing surface area (Kalderis et al., 2008). Increases in phenol and parachlorophenol removal with activation temperature indicate the increase in suitability of pore size to accommodate the phenol and parachlorophenol. Similar result was reported by (Theydan and Ahmed, 2012). According to (Guo and Lua, 2001), increasing temperature within the range of 500-900°C will increase the released volatiles. The decrease of yield is maximum between 200°C and 800°C due to rapid carbonization in this region. It is also unsuitable to prepare activated carbon when the carbonization temperature is higher than 800°C since the successive decrease in volatile matter is minimum above this range. This is usually accompanied with an increase of fixed carbon and ash content which may be attributed to the removal of volatile matter during carbonization process. It is also noticed that the adsorbed parachlorophenol is higher than phenol due to its lower solubility in aqueous solution and because of it is more non-polar, lesser affinity (Cooney, 1999). In addition, the difference in adsorption behavior of phenol and parachlorophenol might be due to the different affinities of the two phenolic species for the reactive

functional groups in the ARH. Similar result was reported by (Streat et al., 1994), (Jung et al., 2001), (Termoul et al., 2006) and (Al-Roubaiaay, 2011), where the adsorption capacity for parachlorophenol is greater than that for phenol.

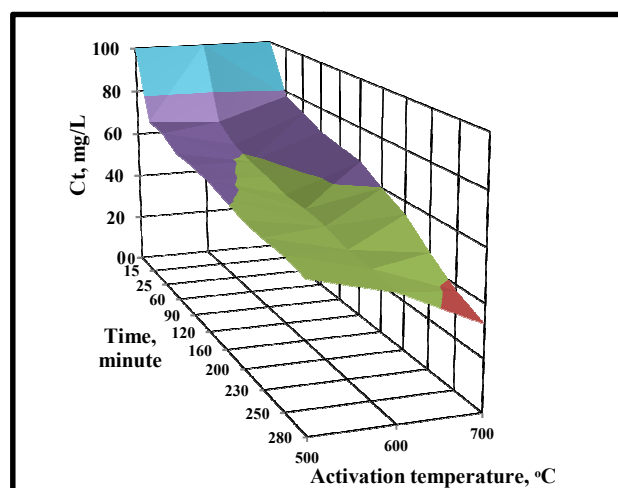


Fig. (6). Phenol concentration versus time for various activation temperature (T), IR= 3:1, and AT=1 hour.

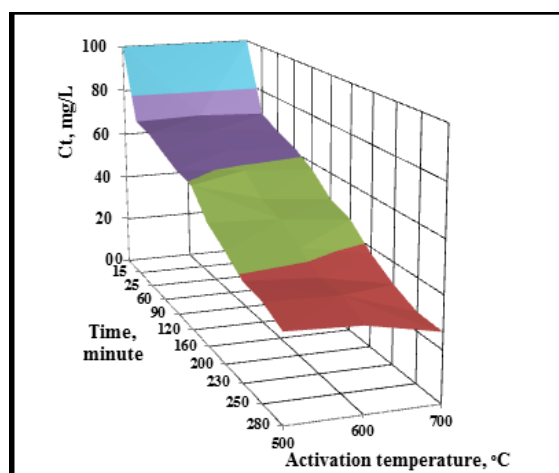


Fig. (7). Parachlorophenol concentration versus time for various activation temperature (T), IR= 3:1, and AT=1 hour.

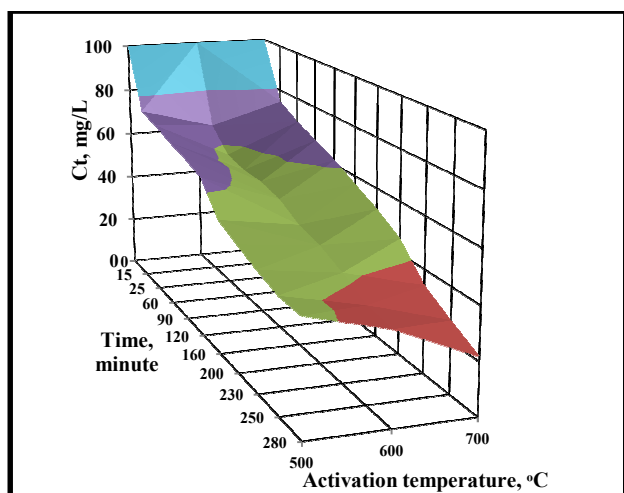


Fig. (8). Phenol concentration versus time for various activation temperature(T), IR= 3:1, and AT=2 hour.

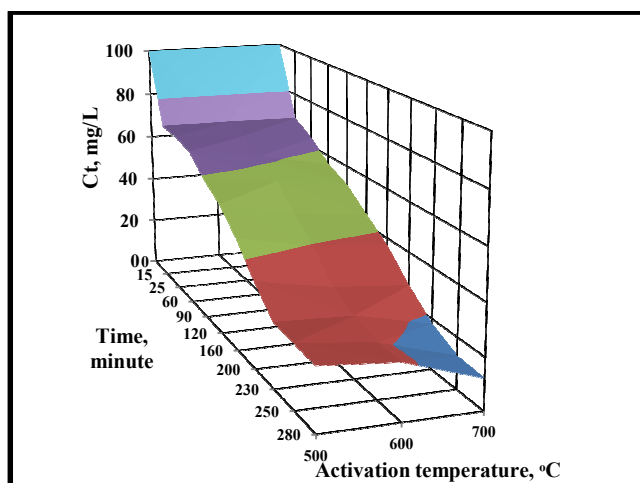


Fig. (9). Parachlorophenol concentration versus time for various activation temperature(T), IR= 3:1, and AT=2 hour.

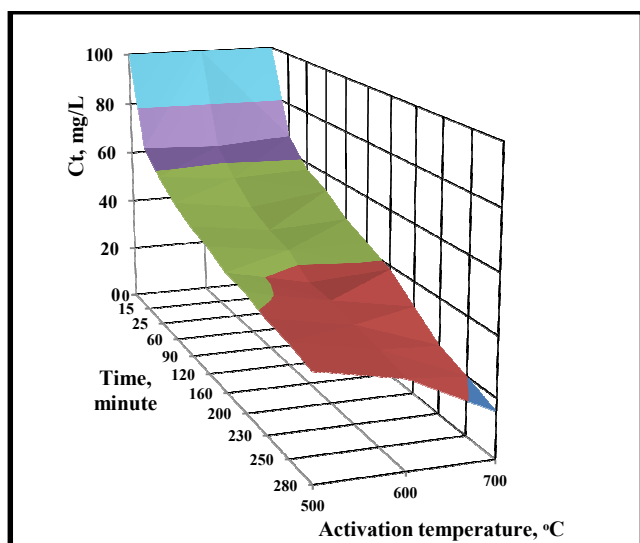


Fig. (10). Phenol concentration versus time for various activation temperature(T), IR= 3:1, and AT=3 hour.

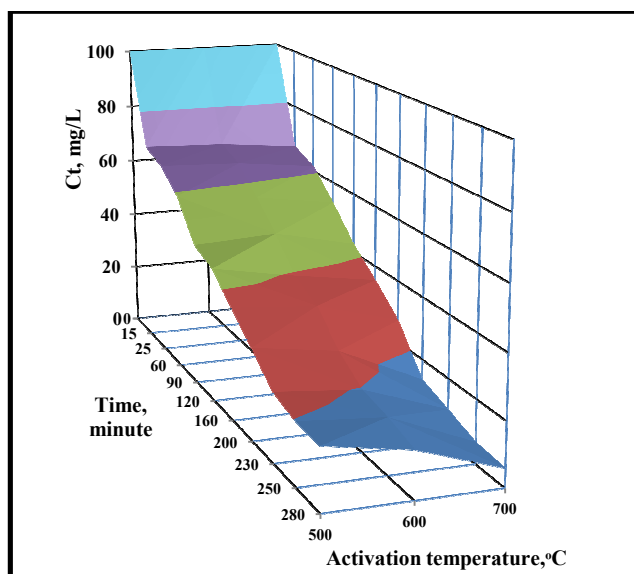


Fig. (11). Parachlorophenol concentration versus time for various activation temperature(T), IR= 3:1, and AT=3 hour.

4.4. Effect of Activation Time

The activation time has a significant effect on the porous network development in the activated carbon. Therefore, in order to obtain activated carbon having desired properties, precursor must be treated at a suitable carbonization time. Figs (12-17) represent adsorption data for phenol and parachlorophenol on various activated carbon samples under different activation time, 1, 2, & 3 hour. It is clear that the removal of phenol and parachlorophenol is increased with the increase of activation time. The highest removal for phenol (84.38%) and parachlorophenol (94.06%) obtained for RH13 which is activated under 700 $^{\circ}\text{C}$, 3:1 IR, and 3 hours activation time. This is because increasing activation time will give more chance for hot flowing gases to do its work in increasing the pores and surface area (Hameed et al., 2009). i.e. higher time will increase the amount of volatile matter during carbonization process and higher removal giving more pores and higher surface area. Also at high activation times, the change (quality and quantity) in functional groups may subsequently affect the adsorption of phenol and parachlorophenol. Generally at high activation times the increase in the aromatic content of AC functional groups has been well recorded (Baçaoui, Yaacoubi et al. 2001; Wang, S. et al. 2005). These results agree with the result published by (Chatterjee and Kumar, 2012; Yang and Lua, 2003).

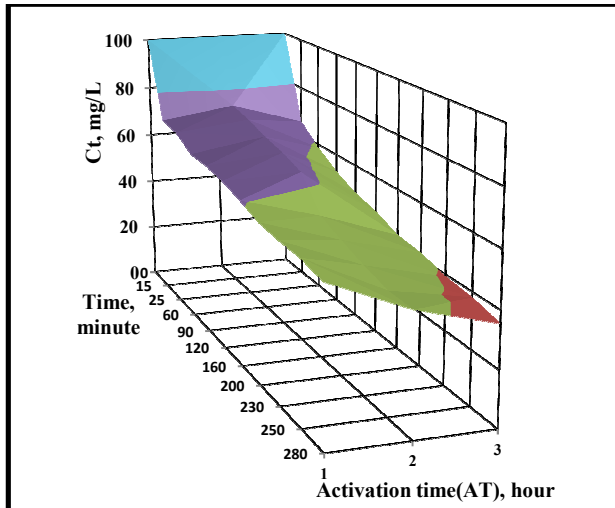


Fig. (12). Phenol concentration versus time for various activation time(AT), IR= 3:1, and $T=500$ °C.

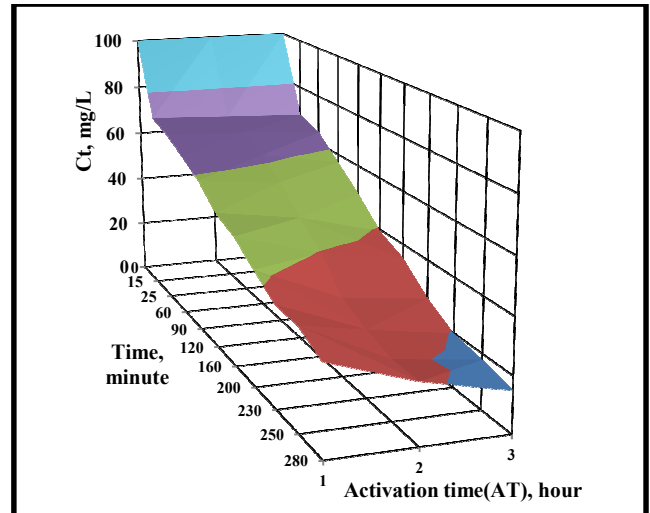


Fig. (15). Parachlorophenol concentration versus time for various activation time(AT), IR= 3:1, and $T=600$ °C.

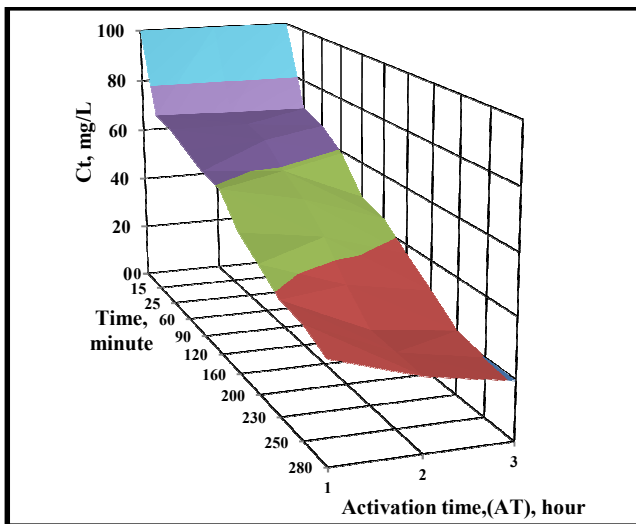


Fig. (13). Parachlorophenol concentration versus time for various activation time(AT), IR= 3:1, and $T=500$ °C.

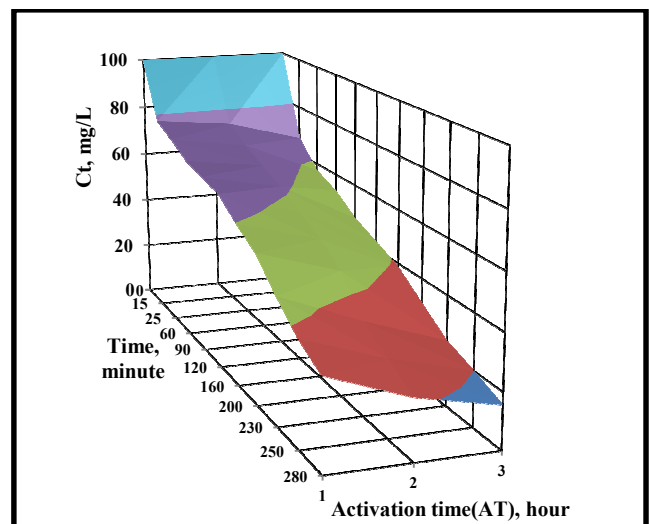


Fig. (16). Phenol concentration versus time for various activation time(AT), IR= 3:1, and $T=700$ °C.

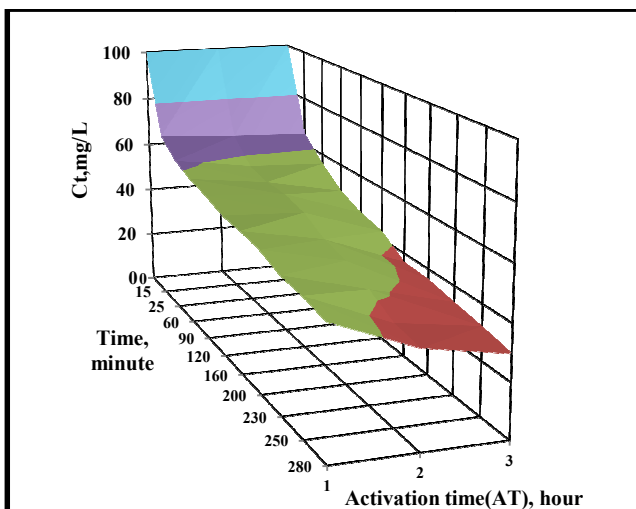


Fig. (14). Phenol concentration versus time for various activation time(AT), IR= 3:1, and $T=600$ °C.

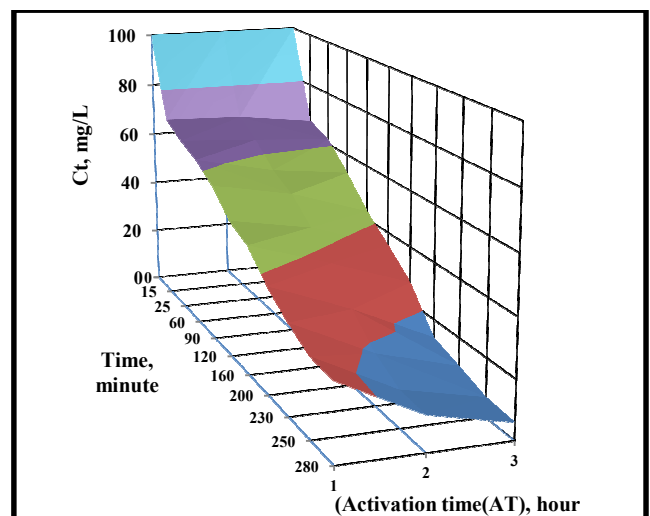


Fig. (17). Parachlorophenol concentration versus time for various activation time(AT), IR= 3:1, and $T=700$ °C.

4.5. Effect of Impregnation Ratio, IR, of $\text{ZnCl}_2/\text{Rice Husk}$

Figures (18-23) represent adsorption data for phenol and parachlorophenol on various activated carbon samples under different Impregnation ratio (IR) 1:1, 2:1, and 3:1. The highest removal for phenol (84.38%) and parachlorophenol (94.06%) obtained for RH13 which is activated under 700°C , 3:1 IR, and 3 hours activation time. It is clear that the removal of phenol and parachlorophenol is increased with the increase of IR. This is because rice husk which is treated with ZnCl_2 led to a removal of the cellulosic component of the adsorbent. It is well known that activation with zinc chloride prevents the accumulation of tar on the carbon surface and provides further decompositions and thus, develops the microporosity when using cellulosic and lignocellulosic precursors in the manufacture of activated carbon. More activator perhaps leads to excessive dehydration and destruction of mesopores turning them into larger pores which reduces the adsorption efficiency (Kim et al., 2001). Similar results are reported by (Al- Roubaiaay2011 and Kalderis.2008).

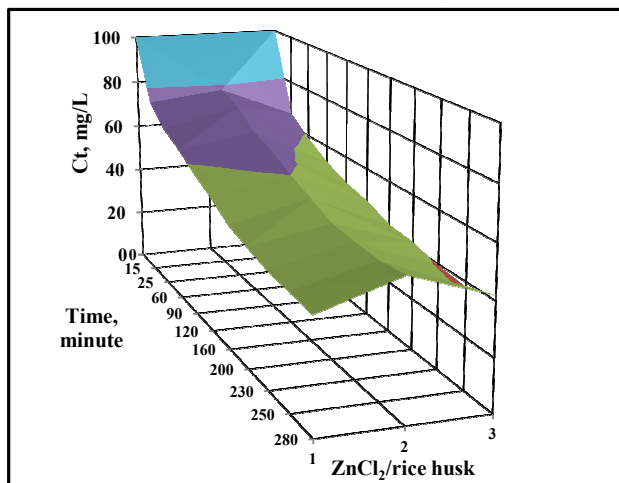


Fig. (18). Phenol concentration versus time for various Impregnation ratio, IR, (AT)=3hour, $T=500^\circ\text{C}$.

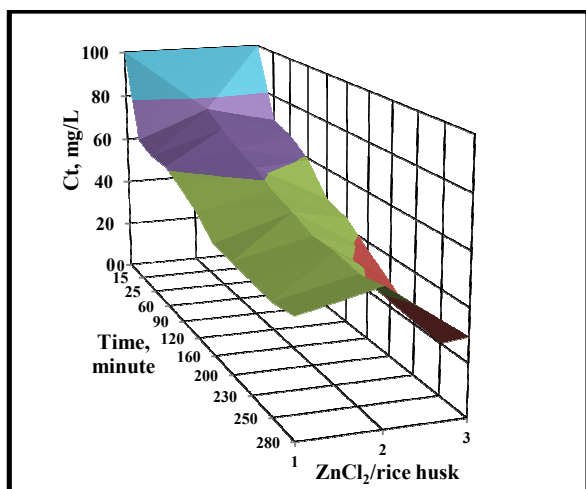


Fig. (19). Parachlorophenol concentration versus time for various Impregnation ratio, IR, (AT)=3hour, $T=500^\circ\text{C}$.

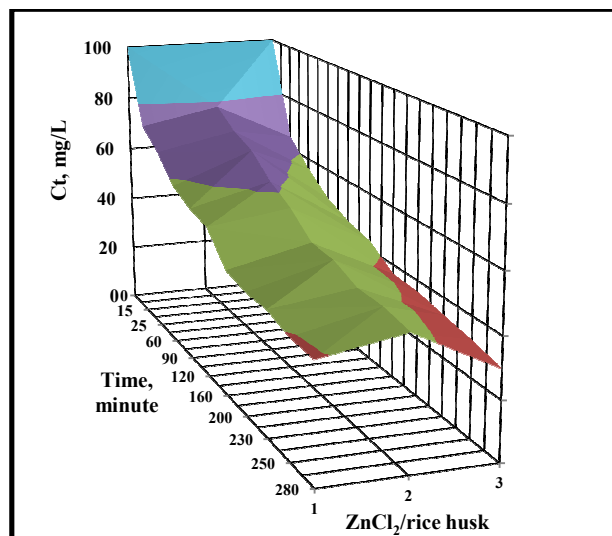


Fig. (20). Phenol concentration versus time for various Impregnation ratio, IR, (AT)=3hour, $T=600^\circ\text{C}$.

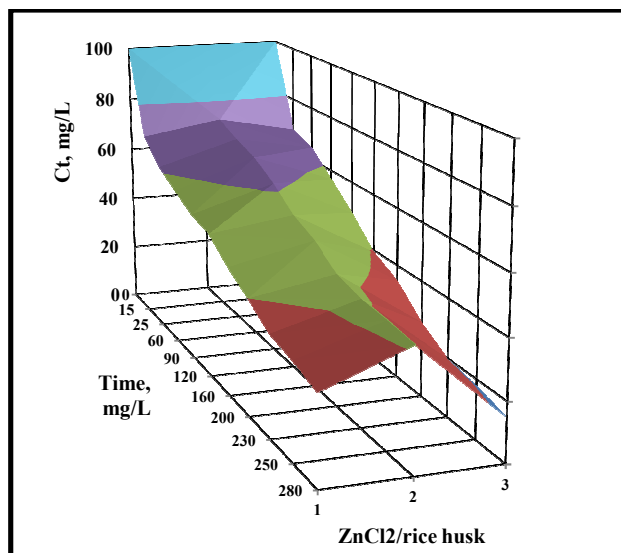


Fig. (21). Parachlorophenol concentration versus time for various Impregnation ratio, IR, (AT)=3hour, $T=600^\circ\text{C}$.

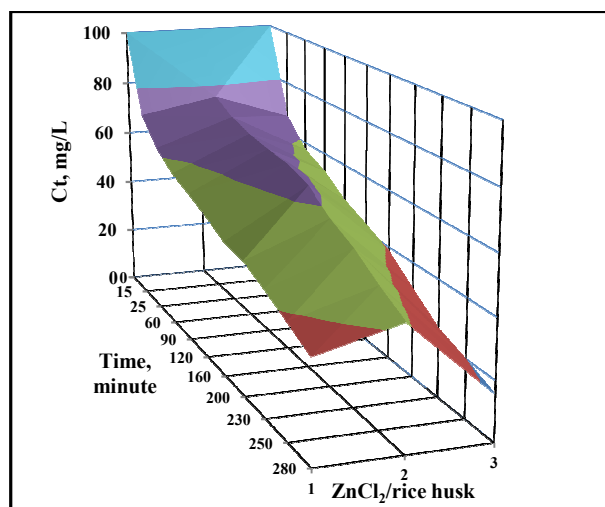


Fig. (22). Phenol concentration versus time for various Impregnation ratio, IR, (AT)=3hour, $T=700^\circ\text{C}$.

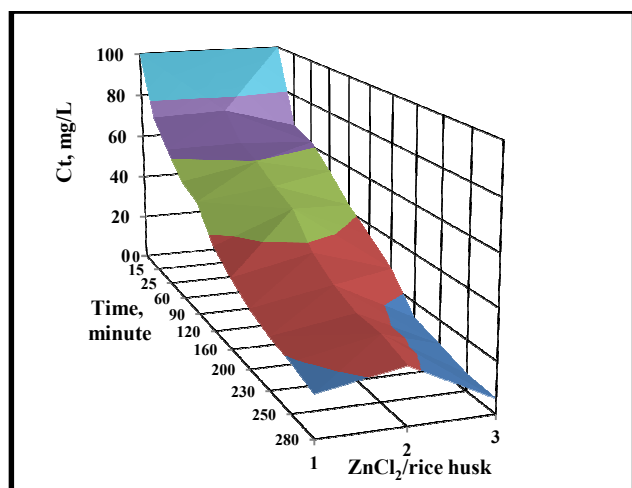


Fig. (23). Paraclorophenol concentration versus time for various Impregnation ratio, IR, (AT)=3hour; $T=700^\circ\text{C}$.

4.6. Applicability of Adsorption Isotherms

Adsorption data are analyzed using four different models: Freundlich, Langmuir, Temkin, and BET. The ranges of the coefficients of determinations (R^2) for these models are listed in Table (2). From this table, it seems that Temkin model gives the highest range of R^2 for phenol and parachlorophenol. This means that this model fairly fits experimental data. Langmuir and Freundlich models have close values of R^2 ranges which are lower than that of Temkin model while BET model gives the lowest R^2 ranges.

The high fitness of the Langmuir model for the adsorption process of activated rice husk indicates the monolayer concealment of phenol and parachlorophenol on the outer surface of activated rice husk, in which the adsorption occurs uniformly on the active part of the surface (Abdel Wahab et al., 2005). The adsorption capacity for Phenol and Parachlorophenol using Langmuir model are 39 and 44.64 mg/g respectively. The corresponding values for Temkin model are 38.82 and 44.94 mg/g.

Table (2). Ranges of R^2 of the used adsorption models.

Model	R^2 range for:	
	phenol	parachlorophenol
Freundlich	0.9090-0.9817	0.9074-0.9899
Langmuir	0.9032-0.9876	0.9035-0.9887
Temkin	0.9624-0.9967	0.9269-0.9977
BET	0.7642-0.9638	0.7461-0.9101

5. Conclusions

Chemical activation method is found to be superior than physical activation method for all raw materials used (RH, PF and DS) and the best raw material is found to be RH which gives maximum removal of 84.38% and 94.06% for phenol and parachlorophenol respectively under activation temperature of 700°C , activation time 3 hours, and impregnation ratio 3:1.

The removal of Phenol and Parachlorophenol is increased with the increase of activation temperature, activation time, and impregnation ratio and the experimental data is found to agree Temkin model better than other adsorption models. Maximum adsorption capacity for RH calculated by Langmuir model is 39 and 44.64 mg/g for Phenol and Parachlorophenol respectively.

Nomenclature

- C_t Concentration of solute in solution at any time, (mg/l)
 C_0 Initial concentration of adsorbate (mg/l)
 C_e Concentration of solute in solution at equilibrium, (mg/l)
 q_e Adsorption capacity, (mg/g)
 R^2 Correlation coefficient
 t Time, (minute)
 V Volume of solution, (L)
 W Weight of adsorbent, (gm)

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