International Journal of Environmental Sciences
ISSN: 2229-7359
Vol. 11 No. 6, 2025
https://theaspd.com/index.php

Modification of Orange Peel Activated Carbon for Phenol removal from Wastewater

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Abstract
The current work inquired into the modification of Orange Peel Activated Carbon (OPAC) for enhanced dismissal of phenol from wastewater. The Activated Carbons (ACs) were prepared by pyrolysis (OPAC), nitric acid (HNO_3-OPAC), and zinc chloride ($ZnCl_2$-OPAC) method. The adsorption pattern of ACs was evaluated by FTIR, BET, XRD, SEM, and EDAX studies.$ZnCl_2$-OPAC, with the highest Brunner Emmet and Teller (BET) surface area (1489 m2/g) among all, was the best for phenol adsorption with maximum efficiency (95 %) verified by the batch studies and characterization study. The non-linear isotherm models were examined in which Freundlich model turned out to be the most fitted for all three adsorbents. Consumed carbon could be achieved by using NaOH. 86.7 % Phenol removal in pharmaceutical company wastewater was attained by $ZnCl_2$-OPAC.
Keywords: *Activated Carbon, Adsorption, Wastewater, Phenol*

INTRODUCTION

The study recommended that phenol is a prime contaminant in water since its concentration greater than 1 ppb in potable water causes duress to human health [1]. Hence, the dismissal of phenol from wastewater is viewed as a crucial challenge worldwide. Adsorption is an inexpensive and systematic technology for the remediation of contaminants from wastewater. Different contaminants from water systems have been removed by various adsorbents such as commercial, inorganic, and organic [2]. Studies have shown commercial materials have high adsorption capacities and can be reused, but they are exorbitant and non-biodegradable [3]. Inorganic adsorbents such as zeolites, clay, and biochar are most abundant in nature; studies have shown they are mostly used for the dismissal of toxic metals in water systems and have low adsorption capacities [4]. Organic adsorbents, most commonly known as agricultural wastes, are derived from renewable sources. Studies have shown agricultural wastes such as rice husk[5], cotton fibers [6], coconut husk [7], banana shell [8], cane waste fiber [9], and orange peel [10] have huge potential to be used as an adsorbent because of low cost, great biomass, and reduced global warming [11]. About 60 million tons of orange have been produced annually worldwide, and orange peel waste is about 32 million tons recovered annually [12], a prime material for the remediation of contaminants. Orange peel is a suitable biosorbent for phenol [10] due to its low ingestion of water and abundance in nature. The combination of chemical elements in orange peel depends upon the growing conditions, location, and varieties [13]. The elemental analysis of orange peel is shown in Table 1, which confirms that the orange peel have the high mass percentage of carbon.

Table 1 Elemental constituents of Orange Peel

Chemical Composition	Mass % [14]	Mass % [15]	Mass % [16]	Mass % This study
Carbon	59.49	54.4	48	48.3
Oxygen	40.70	46.93	45.01	35.94
Hydrogen	7.05	5.91	5.98	2.30
Nitrogen	0.66	1.5	1.3	-

The factors that affect the adsorption inclination of activated carbon are pH, Temperature, particle size, surface area of sorbent, functional group of adsorbents, and specific pollutants in wastewater. Many studies suggested the dismissal of phenol increased with temperature and pH of the solution [17], [18]. The study by Mohammad proposed that phenol adsorption increases with a reduced particle size of sorbent [19]. Therefore, three distinct methods (pyrolysis, HNO_3 activated biochar, and $ZnCl_2$ activated biochar) were inquired in the current work. The adsorption trait of prepared ACs was evaluated by FTIR, BET, XRD, SEM, and EDAX studies. The efficiency of ACs was compared by Freundlich isotherm and Langmuir isotherm. Furthermore, the dismissal of phenol by prepared carbons was examined with pharmaceutical wastewater. The enhanced quality of water was designed by COD, BOD, TDS, and phenol values.

MATERIALS AND METHODS

The Orange was accumulated in the locality of Sitamarhi, Bihar, India. The peel was detached and crushed with one's hands. The crushed peel was put in an oven at 120 °C to dry, and then the drained peel was kept in a silica crucible for further experiment.

Synthesis of Orange Peel Activated Carbon (OPAC)

The silica crucible which contained dried peel was heated in the blast furnace at 700 °C. After reaching 700 °C, the temperature was sustained for 150 min. The material was kept inside the same to cool for the next 24 h. The material was put in a vacuum sealed container for 48 h to take off moisture. The char particles were measured and put in a tube for further adsorption studies.

Synthesis of HNO_3-OPAC

The dried peel was carbonized with a concentrated nitric acid solution in a 1:1 weight ratio. The charred solution was kept for 24 h to immerse. The excess nitric acid was pulled out with alkaline water. The saturated char was kept inside the hot air chamber to desiccate at 100 °C. The dried char was exposed to thermal decomposition in a blast furnace at 500 °C for 120 min. The material was placed inside the same to cool for the next 24 h. The material was put in a vacuum sealed container for 24 h to dismiss moisture. The char particles were measured and put in a tube for further adsorption studies.

Synthesis of $ZnCl_2$-OPAC

The dried peel was mixed with zinc chloride solution in a 1:1 mass ratio and heated for 1 h at 70 °C. The solution was kept for 24 h to immerse. The excess zinc chloride was pulled out with alkaline water. The filtered slurry was kept inside the hot air chamber to desiccate at 100 °C. The dried char was exposed to thermal decomposition in a blast furnace at 500 °C for 120 min. The material was placed inside the same to cool for the next 36 h. The material was put in a vacuum sealed container for 36 h to dismiss moisture. The char particles were measured and put in a tube for further adsorption studies.

RESULTS AND DISCUSSION

Characterization studies

BET surface area

The superficial area of ACs increased from 65.7 m^2g^{-1} to 1489 m^2g^{-1} because of the rise in the pore density on the surface. The most expanded superficial area was given by $ZnCl_2$-OPAC (1489 m^2/g) among the prepared ACs. Therefore, it shows the highest dismissal of phenol (95 %), followed by HNO_3-OPAC and OPAC (85 % and 79 %, respectively). The surface area of different ACs has been reported previously is shown in Table 2. Due to the impregnation of orange peel with $ZnCl_2$, the microporous tendency of $ZnCl_2$-OPAC increased [20]. The bond between the molecules is broken by $ZnCl_2$. This leads to the rise in spaces between char layers of $ZnCl_2$-OPAC [21]. Hence, more surface area was reported by $ZnCl_2$-OPAC as compared to HNO_3-OPAC and OPAC.

Table 2 Surface area of distinct Biochars

Biochars	Surface Area (m^2/g)
AC of lacosperma hydrochar [22]	1135
AC of sugarcane bagasse [23]	697.37
AC of $Ni(OH)_2$ nanoplate [24]	960.3
AC of clay [25]	305.5
$ZnCl_2$ activated coco peat carbon [26]	910
AC of palm tree seed [27]	577
OPAC (present study)	65.7
HNO_3-OPAC (present study)	78.6
$ZnCl_2$-OPAC (present study)	1489

SEM and EDAX analysis

The modification in surface micropattern of the orange peel can be directly observed by SEM [28]. SEM depiction of ACs detects the appearance of mini pores to large pores. The SEM prints of $ZnCl_2$-OPAC prior to and subsequent to phenol adsorption are presented in Fig.1. Prior to adsorption, $ZnCl_2$-OPAC exhibits a regular arrangement of mini pores and larger pores. In contrast, after phenol adsorption, the micropores and macropores of the adsorbent appear to be covered by the adsorbate. The superior adsorption capacity of $ZnCl_2$-OPAC for phenol is evident compared to HNO_3-OPAC and OPAC. These observations confirm the effectiveness of $ZnCl_2$ in enhancing the pore structure of AC.

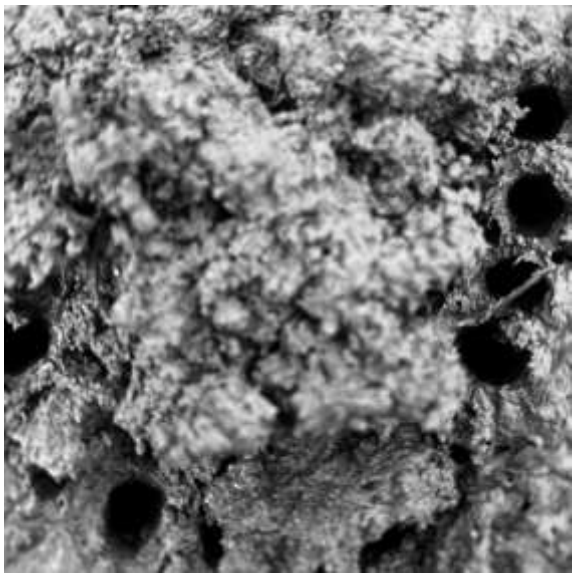
a  b 

Fig. 1 SEM print of a) $ZnCl_2$-OPAC b) $ZnCl_2$-OPAC after phenol adsorption

EDAX analysis examined the elemental combination of ACs. The weight percentage of carbon in OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC is 49.59 %, 64.95 %, and 88.77 % respectively. This report suggests $ZnCl_2$ activated char would be a superior choice than pyrolysis or acid activation method to prepare AC.

FTIR analysis

The functional groups present in the adsorbent which is accountable for adsorption are detected by FTIR spectrum. The porous structure along with the reactive groups present on the surface of AC is crucial for any adsorbents to conduct adsorption [29]. The FTIR spectra of $ZnCl_2$-OPAC pre- and post-phenol adsorption are presented in Fig. 2. The spectra indicate that some functional groups which are crucial to conduct adsorption are displaced or vanish after phenol adsorption. The broad-ranging peak at 3200 cm^{-1} before adsorption was responsible for quiver in the alcohol or phenol-containing –OH group. This peak shifted to 3384 cm^{-1} after adsorption. Hence, the -OH group was crucial for phenol adsorption on $ZnCl_2$-OPAC.

Therefore, its highest adsorption potential was confirmed by the existence of the –OH group on the surface of $ZnCl_2$-OPAC.

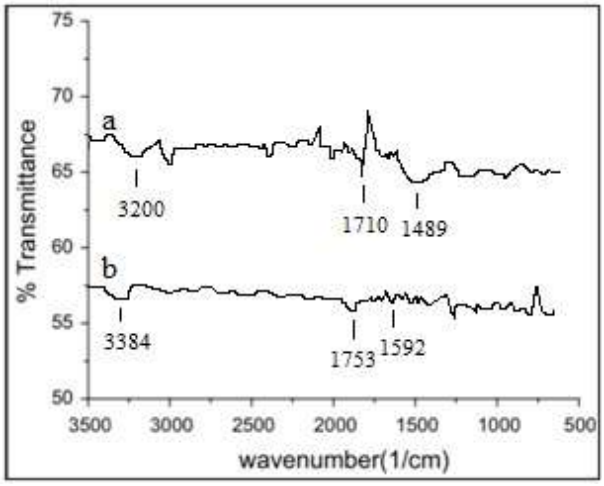


Fig. 2 FTIR spectra of $ZnCl_2$-OPAC a) before phenol adsorption and b) after phenol adsorption

X-ray diffraction- analysis

The stratum of graphite 002, 100, and 110 [30] which are linked with the three peaks (29°, 47°, and 80°) showed in the spectrum. X-ray diffraction of $ZnCl_2$-OPAC prior to and subsequent to phenol adsorption is shown in Fig. 3. The broad peak at 29° was amenable for the highest phenol adsorption due to the amorphous structure of AC. There was no deviation in the station of peaks subsequent to phenol adsorption; however, there was an elevation in the intensity of the peaks.

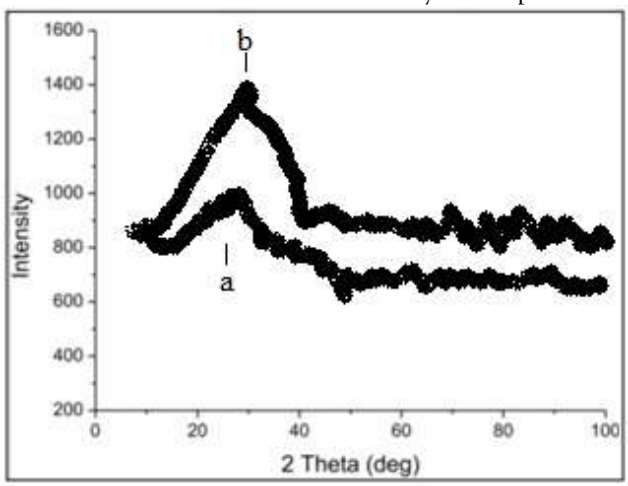


Fig. 3 XRD spectrum of $ZnCl_2$-OPAC a) before phenol adsorption b) after phenol adsorption

Batch studies

Effect of initial phenol concentration

Different standard solutions of phenol from 20 mg/L up to 150 mg/L were prepared for the study. The synthesized chars showed maximum phenol adsorption at minimal phenol concentration (10 mg/L). Adsorption decelerated at higher phenol concentrations because initially active sites adsorbed phenol, but later unoccupied active sites were not adsorbing because of the repulsive force between the molecules of phenol adsorbed on the surface and the molecules of incoming phenol [31]. Hence, the remaining studies were carried out at a minimal phenol concentration (10 mg/L).

Effect of adsorbent dosage

Various amounts of ACs from 20 mg to 450 mg were mixed with phenol solution. As the amount of adsorbent increases, consequently, phenol adsorption increases due to the availability of more active sites

[31]. For phenol adsorption, the upper limit of the adsorbent amount required by OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC were 150 mg, 150 mg, and 90 mg, respectively. To attain the highest adsorption (95%), the required dosage was lower for $ZnCl_2$-OPAC due to its highest carbon-content and surface area.

Effect of pH

The zeta potential of the adsorbent was mainly swayed by the pH of the solution [32]. For the highest phenol adsorption, the pH required by OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC were 3, 2, and 8, respectively. The consequence of pH on phenol adsorption of ACs is shown in Fig. 4. Therefore, there could be a likelihood of handling wastewater with the help of $ZnCl_2$-OPAC at neutral pH.

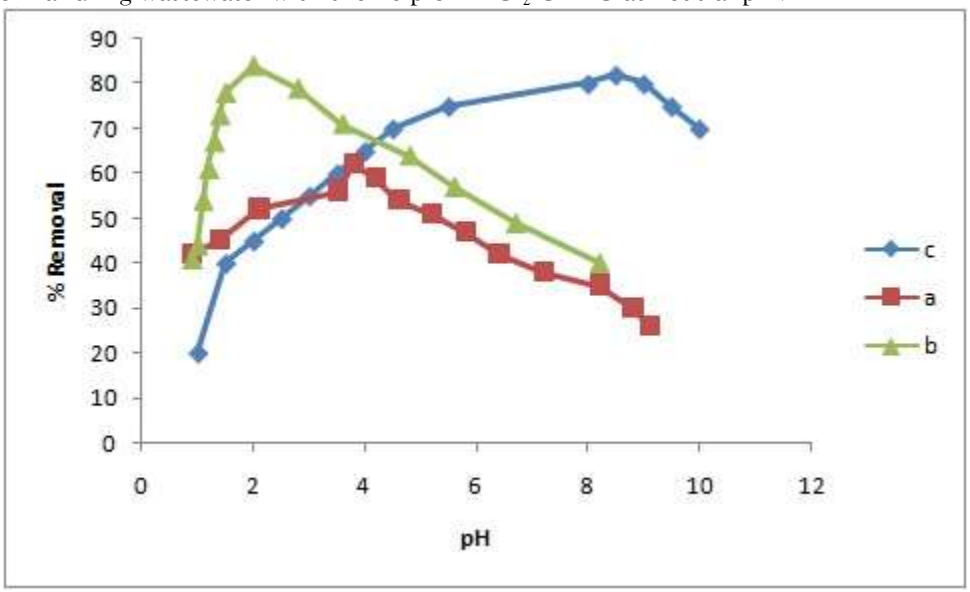


Fig. 4 Consequence of pH on phenol adsorption by a) OPAC b) HNO_3-OPAC c) $ZnCl_2$-OPAC

Effect of contact time

The equilibrium time was procured by treating the required dosage of ACs with initial phenol concentration (10 mg/L) at its most favorable pH. The speedy phenol adsorption occurred within 40 minutes because of the more active sites present on the adsorbent. The adsorption equilibrium time was observed at 90, 70, and 60 min for OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC, respectively. The phenol adsorption after equilibrium time decreases because of the occupation of adsorbate (phenol) in the active sites [33].

Adsorption isotherms

The performance of adsorption was predicted by two distinct isotherms. The frameworks of the above isotherm were obtained using nonlinear regression analysis. The mathematical expression of mean square deviation (X^2), correlation coefficient (R^2), average relative absolute error (ARAE), and normalized root-mean-square error (NMSE) are as follows [34]:

Mean square deviation (X^2)

$$= \sum_{i=1}^{n} \frac{(Q-q)^2}{q} \quad (1)$$

Normalized Root-Mean-Square-Error (NMSE)

$$= \sqrt{\frac{1}{n-2}\sum_{i=1}^{n}(q-Q)^2} \quad (2)$$

Average relative absolute error (ARAE)

$$= \frac{100}{n}\sum_{i=1}^{n}\frac{|q-Q|}{q} \quad (3)$$

Correlation coefficient (R^2)

$$= \frac{(q-Q')^2}{\sum(q-Q')^2+(q-Q)^2} \quad (4)$$

Where q (mg/g) is experimental adsorbed phenol, Q (mg/g) is calculated adsorbed phenol and Q' (mg/g) is the mean of calculated adsorbed phenol.

Freundlich isotherm

The Freundlich relationship is given by [34]

$$q' = K_F C^{\frac{1}{n}} \quad (5)$$

Where q' (mg/g) and C (mg/L) are the adsorbed phenol at equilibrium, K_F is the adsorption capability of AC (mg/g), and 1/n is the adsorption potency of AC.

The deviation measures of Freundlich isotherm for all three chars appeared to be a best fit. It was obtained that phenol uptake by OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC was multilayer adsorption. The isotherm frameworks were calculated from non-linear curve fitting graph and are shown in Table 3. The adsorption intensity of OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC was 0.4492, 0.3195, and 0.2908, respectively. The value of adsorption intensity is less than one, indicating the heterogeneous behavior of the system [35]. Therefore, among prepared carbons, OPAC revealed a high 1/n, indicates OPAC has greater heterogeneity.

Langmuir isotherm

The possibility of monolayer adsorption was decided by Langmuir isotherm. The Langmuir relationship is given by [36]

$$q' = \frac{q_m bC}{1+bC} \quad (6)$$

Where q_m is maximum adsorption capacity (mg/g), and b is Langmuir isotherm constant. Langmuir frameworks are shown in Table 3. The highest adsorption capacity (18.20 mg/g) was observed by $ZnCl_2$-OPAC as compared to OPAC = 12.79 and HNO_3-OPAC = 12.24. Langmuir model was not a better fit because of its high error functions value and low R^2 value as compared to the Freundlich model. Thus, a considerable amount of heterogeneity was detected in the system.

Table 3 Isotherm frameworks and inaccurate functions data for adsorbed phenol onto OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC by non-linear regression analysis

Isotherms			Parameters				
Freundlich Isotherm	Carbon	K_F(mg/g)	1/n	R^2	X^2	NMSE	ARAE
	OPAC	2.9872	0.4492	0.9851	0.002	0.065	0.03
	HNO_3-OPAC	3.694	0.3195	0.9801	0.002	0.085	0.124
	$ZnCl_2$-OPAC	7.4209	0.2908	0.9985	0.001	0.060	0.072
Langmuir Isotherm	Carbon	q_m (mg/g)	b (L/mg)	R^2	X^2	NMSE	ARAE
	OPAC	12.79	0.1432	0.9718	0.076	0.925	0.861
	HNO_3-OPAC	12.24	0.2884	0.9737	0.063	0.724	0.557
	$ZnCl_2$-OPAC	18.20	0.5818	0.9825	0.213	2.159	1.231

Desorption studies

Phenol-adsorbed carbon was treated with different concentrations of NaOH from 0.001 M to 0.01 M to regenerate the consumed activated carbon. The maximum desorption for OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC were 73.6 %, 84 %, and 87.3 %, respectively occurred at 0.1 M NaOH solution. This is due to NaOH reacted with the adsorbed phenol to form C_6H_5ONa; this leads to the elevation in the rate of desorption because of the anionic repulsion of AC and C_6H_5ONa [37]. This indicates that phenol adsorption onto prepared carbons was reversible. Therefore, consumed AC could be indisputably regenerated.

Treatment of pharmaceutical industry wastewater

The phenol adsorption by OPAC, HNO_3-OPAC, and $ZnCl_2$-OPAC were 74.3 %, 78.9 %, and 86.7 %, respectively. The parameters like TDS, COD, and BOD of pharmaceutical wastewater were decreased. The parameters before and after adsorption are given in Table 4. This study gives proof of the future prospects of $ZnCl_2$-OPAC in the analysis of pharmaceutical wastewater.

Table 4 Analysis of Pharmaceutical Wastewater

Factors	Pre-adsorption	Post-adsorption		
	Amount (ppm)	OPAC (mean)	HNO_3-OPAC (mean)	$ZnCl_2$-OPAC (mean)
BOD	2480	23	18	15
COD	12,420	126	108	98
TDS	2651	240	180	142
Phenol	6.54	1.4	1.1	0.6

CONCLUSION

An agricultural waste, orange peel was used for the synthesis of AC by pyrolysis, nitric acid, and zinc chloride activation processes. $ZnCl_2$-OPAC among three prepared carbons was established as the best adsorbent showed by characterization studies and batch studies. The maximum phenol adsorption by $ZnCl_2$-OPAC was 95 % at the most favorable pH of 8 and minimum AC quantity of 90 mg. Isotherm evaluation for the phenol uptake by OPAC, HNO3-OPAC, and $ZnCl_2$-OPAC showed the best fit with Freundlich isotherm, which indicates multilayer adsorption. The high desorption potential of phenol from consumed carbon indicated that the synthesized carbons can be restored. ZnCl2-OPAC was competent to remove 86.7 % phenol from pharmaceutical industry wastewater. Hence, the treatment of wastewater at a low cost can be attained by using orange peel to prepare activated carbons.

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