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Preparation and Characterization of Activated Carbon Using Pinecone (Conifer Cone) to Remove Phenol from Wastewater [†]

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Abstract: Chemical industries are generating unprecedented effluent, including toxic aromatic compounds such as phenol, which poses severe environmental risks. This study explores the acute and prolonged effects of phenol, which range from the death of animals, birds, and fish to reduced plant growth, reproductive problems, and changes in appearance and behaviour. Additionally, oral exposure to phenol can be toxic to humans. Meanwhile, the agricultural sector faces challenges in finding salvage value for increasing amounts of waste. To address this issue, our research analyzes organic materials with no market value and investigates the feasibility of achieving efficient adsorption using their char. We specifically examine pine nuts, an abundantly available waste material. Our objective is to synthesize an organic adsorbent material that meets specific criteria: organic, readily available at zero cost, derived from waste with no other utility, native to the area, abundantly accessible, possessing a large surface area, and demonstrating superior adsorption capabilities. This research employs chemical activation using four acids (nitric acid, sulfuric acid, hydrochloric acid, and orthophosphoric acid) and involves drying and heating the samples at different elevated temperatures. The selection of the optimal adsorbent is based on an analysis of the BET (Brunauer–Emmett–Teller) surface area and pore volume, ensuring its efficacy. The adsorption efficiency was also tested with the help of a UV spectrophotometer to assess its efficiency using Beer–Lambert's law. The study also goes through an ultimate analysis to measure the amount of carbon content in our adsorbent. Through this study, we aim to develop sustainable waste management practices by utilizing pine nut waste as a valuable resource for effective phenol removal.

Keywords: adsorption; wastewater; pinecone; phenol; activated carbon

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1. Introduction

Phenols, characterized by their aromatic ring with a hydroxyl group attached to it, belong to the phenolics family of organic hydroxyl compounds. These compounds could cause severe harm to various organ systems upon inhalation or skin exposure. The liver, kidneys, and central nervous system are particularly vulnerable to the short and long-term effects of phenolic compounds [1]. The toxic effects of phenols extend beyond acute harm, as absorption through the skin, inhalation of vapors, or ingestion can be poisonous, leading to morbidity and mortality [2,3].

Phenols can naturally arise in water bodies from the breakdown of organic matter in industrial discharge, domestic waste, or runoff from pesticide-treated agricultural lands. It is imperative to prevent the pollution of vital water sources and the disruption of aquatic habitats; it is also essential in scenarios where effluents require biological treatment before phenol-contaminated discharge, as phenols hinder the effectiveness of such treatment processes. Acknowledging this, governmental agencies such as the US Environmental Protection Agency (EPA) have designated phenols as criteria pollutants and imposed stringent standards on the discharge of phenol-contaminated effluents [4,5]. In recent times, the chlorination of municipal drinking water has raised concerns regarding the presence of phenols [6].

Complete phenol biodegradation often does not occur due to the absence of favorable conditions or inadequate populations of microorganisms [7]. Activated carbon (AC) has long been recognized as an effective adsorbent for removing various pollutants, including phenol, from wastewater [8]. However, its widespread application in industrial processes has been hindered by high costs and limited reusability. Exploring alternative sources of activated carbon precursors has emerged as a promising solution to address these challenges. This research paper focuses on utilizing organic domestic waste materials as a cost-effective and environmentally friendly precursor in activated carbon production.

In wastewater treatment, attention has been given to using activated carbon derived from biochar as an effective adsorbent for removing organic and inorganic pollutants [9]. Biochar, a solid material generated through the thermal conversion of biomass under oxygen-limited conditions, possesses desirable properties for wastewater treatment applications. Large macropores are typically exhibited by biochar derived from wood-based feedstock, while pore sizes ranging from 1 to 10 μm are observed in biochar derived from cellulosic straws. The surface area of biochar has been enhanced with higher impregnation temperatures by promoting the degradation of lignin and cellulose and the removal of functional groups [10].

The process of adsorption, a fundamental process in wastewater treatment, involves the transfer of substances from a liquid or gaseous phase to the surface of a solid adsorbent. The BET (Brunauer–Emmett–Teller) isotherm, commonly used for measuring the specific surface area of materials, particularly in multilayer adsorption systems, is utilized [11]. Based on some references, we discovered that pinecones are abundantly available as forest waste, compromising around 25% of forest tree mass, predominantly in India and neighboring countries. This adsorbent also has a history of removing a couple of industrial-level organics as well [12–15]. Using biochar as a cost-effective and environmentally friendly adsorbent can contribute to sustainable wastewater treatment practices while addressing economic and environmental concerns [16,17].

Adsorption capacity (mg/g)

$$\left[\frac{C_o - C_e}{m}\right] \times V \tag{1}$$

C_o—Initial concentration (mg/L), C_e—Concentration at equilibrium (mg/L), m—mass of the adsorbent (g), V—is the volume of solution containing solute or adsorbate (L)

$$\%\ \text{Removal} = \left[\frac{C_o - C_e}{C_o}\right] \times 100 \tag{2}$$

2. Materials and Methods

The raw material was procured from its natural habitats in the campus area and brought in batches for operations and experiments. The material was washed with distilled water and cleaned. It was then sun-dried for approximately two hours with proper exposure to sunlight. Later, it was kept in a beaker to heat at 100 °C in a hot air oven for 24 h. After sufficient drying and reaching appropriate brittleness, the pine seeds were crushed in two to three stages using appropriate size reduction equipment.

The crushed product was passed through a sieve shaker to collect particles smaller than 420 microns for our raw product, this being the lowest particle size available as per the standard mesh size. This raw product was stored in a beaker and kept in an oven furnace for 24 h to remove any potential moisture.

2.1. Preparation of Char

Samples of the raw product were weighed in crucibles and kept in the muffle furnace at 400 °C for 1 h. The crucible samples were cooled overnight and weighed again to check for material or mass loss. Weight reduction was observed. The cause of the weight reduction is unknown but could be due to unaccountable moisture. Slight traces of greyish ash were also observed, which had to be discarded.

2.2. Chemical Activation

Stock solutions of 1M were used for sulfuric acid, orthophosphoric acid, nitric acid, and hydrochloric acid, and the acids were shortlisted based on our preliminary experiments. Twenty-five of the four solutions were used to impregnate the char sample, and the acid was added at 1:4 (weight/weight) onto the char. The samples were then stirred at 450 rpm for 120 min using a magnetic stirrer. The time was allocated such that it was sufficient for the particles to settle and mix with the chemical agent.

The samples were kept in the oven at 70 °C for 24 h. Later, they were transferred to crucibles and kept in the muffle furnace at 500 °C for 120 min. The samples were cooled at room temperature overnight inside the furnace. The samples were then washed with distilled water to remove ash and other impurities. Washing was repeated until the sample achieved a pH of 7 in distilled water solution, as the pH level is important for the activated carbon to be maintained [18]. Once neutralized, the samples were kept in the hot air oven to dry for 24 h at 105 °C to ensure no moisture remained present [19]. The four respective samples were then transferred to airtight containers and labelled.

2.3. Adsorption Studies

In total 100 mL of a 100 ppm phenol solution was prepared and placed in a 250 mL conical flask. The solution was then supplemented with 25 mg of AC and sealed with a stopper. The setup was placed in the incubator shaker with the rpm set to 180 based on our preliminary experiments. Every 60 min, 3 mL of the sample was extracted from the flask and transferred to vials. The incubator shaker was deactivated during the sample extraction, and the flask was undisturbed for 60 s to facilitate the settling of AC particles at the bottom of the flask.

The collected samples were centrifugated at 10,000 rpm for 10 min to separate the two layers of phenol and activated carbon. Subsequently, the absorption values of the samples were determined using the UV spectrophotometer at 270 nm, as that is the region where phenol shows its peak. The absorbance values can range from zero to one and be diluted if the value exceeds one. The dilution factor was considered in subsequent calculations.

3. Results and Discussion

3.1. Preliminary Results of Adsorbent Preparation

At all elevated temperatures, including the lowest temperature of 500 °C, it is observed that all the samples impregnated with hydrochloric acid and sodium hydroxide fail, as the entire sample had transformed into grey ash. Therefore, using one acid and a base in further studies is ruled out.

3.2. Ash Content

The sulfuric acid-impregnated sample exhibited maximum ash content compared to all the other acids under investigation. The nitric acid sample displayed a lesser ash content than sulfuric acid. There was no visible ash content in the orthophosphoric acid-impregnated sample throughout the investigation. The visible ash content observed in the samples impregnated with all the acids in a 1:4 ratio is summarized as given in Table 1.

Table 1. Observation of ash content for 1:4 ratio impregnated samples.

Acid	500 °C	600 °C	700 °C	800 °C
H_2SO_4	Little ash	Little ash	Ash less than AC	Ash qty > 700 °C
HNO_3	Lesser ash	Lesser ash	Ash less than AC	Ash qty increased, but <H_2SO_4 at 800 °C
H_3PO_4	No ash	No ash	Unusual moisture on surface, remained even after 22 h drying at 105 °C. Looked like bitumen	Similar sample in 700 °C, and remained negligible, witnessed a qty decrease
HCI	Only ash	Only ash	Only ash	Only ash

3.3. Amount of Yield

The weight loss was higher in samples activated at 700 °C than at higher temperatures; hence, the yield was the lowest. Additionally, an increase in the ratio of acid added to biochar resulted in a corresponding increase in weight loss during activation. An increase in activation temperatures generally leads to an increase in the observed weight loss in the final yield of activated carbon. This trend was observed in all the samples except those impregnated with sulfuric acid, as shown in Table 2.

Table 2. Wt. Loss during activation for all 1:4 ratio samples.

1:4 Acid	Wt. Loss (g)		
	600 °C	700 °C	800 °C
SF	3.34	2.954	3.774
NTR	1.373	1.764	1.972
OPP	2.298	2.603	3.019
HDC	1.921	1.984	2.017

3.4. Neutralization of pH in the Activated Carbon

The sample activated at 700 °C exhibited a significantly lower amount of distilled water (DW) required to wash off the excess acidity than all other temperatures. On an industrial scale, washing with DW consumes a considerable amount (1–2 L) for less than 1 g of activated carbon (AC), meaning heating at 700 °C could be more economically efficient. Some samples were declared “Fail” because they were either burnt or they did not show the desired results as the others did.

The following methods were implemented to reduce DW consumption:

The AC particles were washed with DW, and the top layer of water was discarded after the particles settled at the bottom. The remaining liquid was dried in a hot air oven to remove residual acidity. This took about 10 min. The AC was then rewashed, and the steps were repeated. This method resulted in the removal of acidity after each wash. The AC was submerged in a 1M sodium bicarbonate solution overnight, followed by two to three regular washes with hot DW [20]. This method reduced the DW required for washing. The washing methods were a trial-and-error approach to minimize the usage of DW.

3.5. Adsorption Studies

During adsorption studies, samples with increased impregnation temperature resulted in an increase in the % removal. The maximum % removal was observed at 700 °C, gradually decreasing at 800 °C. The removal % was typically higher during the first hour, followed by a decrease in the second hour. However, the % removal increased again during the third hour. This trend was observed in most samples and saturated after 3 to 4 h. Further equilibrium studies determined the exact point of the highest removal %. Comparing the

1:4 (ratio of sample to the quantity of acid used) impregnated samples shows that sulfuric acid performs better in % removal compared to nitric acid and orthophosphoric acid. All the samples exhibited optimum % removal at 700 °C with a decreasing trend as the temperature exceeded 700 °C, as seen in Table 3.

Table 3. Adsorption studies of sulfuric acid (SF), nitric acid (NTR), and orthophosphoric acid (OPP) samples with 1:4 char-to acid ratios.

SF 1:4 Sample		**NTR 1:4 Sample**		**OPP 1:4 Sample**	
T (°C)	**% Removal**	**T (°C)**	**% Removal**	**T (°C)**	**% Removal**
500	24.082	500	18.106	500	22.485
600	24.76	600	23.52	600	23.106
700	35.695	700	34.097	700	0
800	34.718	800	32.633	800	0

3.6. Surface Area and Pore Volume

There was an observed increase in surface area with an increase in activation temperature. A larger pore volume was obtained when the impregnation temperature was higher. The samples impregnated with sulfuric acid exhibited the lowest surface area at the lowest temperature of 500 °C, but also showed the lowest % removal, as shown in Table 4 and Figure 1, with the sample failing at 600 °C. Samples chemically activated with nitric acid showed the lowest surface area at 500 °C and achieved the highest % removal.

Table 4. BET surface area data of sulfuric acid (SF) 1:4 ratio sample.

1:4 SF Samples	
Temperature (°C)	**Surface Area (m^2/g)**
500	539.07
600	0
700	639.65
800	849.35

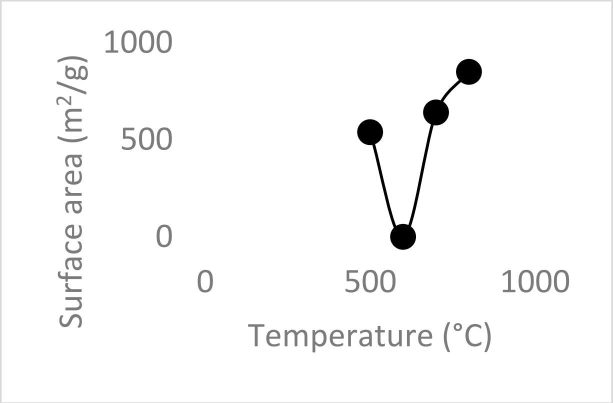

Figure 1. The plot of BET surface area of sulfuric acid 1:4 ratio sample.

The best results were observed in the sulfuric acid sample activated at 700 °C. Detailed observations of the adsorption studies are presented in Table 5.

Table 5. Adsorption studies of sulfuric acid (SF) 1:4 ratio sample.

700 °C SF 1:4 Ratio					
Time	**Co**	**Absorbance**	**Ce**	**Adsorption Capacity**	**% Removal**
1 h	100	0.347	61.6	154	38.40
2 h	100	0.371	65.9	137	34.14
3 h	100	0.376	66.7	133	33.25
4 h	100	0.355	63	148	36.98
				Avg=	35.69

Previous research papers suggest that activating lignocellulosic biomass with orthophosphoric acid results in activated carbon with high surface area and pore volume [21]. However, the conclusions from these adsorption studies indicate less than 40% removal, and it was observed that orthophosphoric acid samples failed at higher temperatures of 700 °C and 800 °C. In contrast, samples prepared with other acids still yielded some results.

Smaller ratios such as 1:1.5, 1:2.5, and 1:3.5 were tested to reduce the amount of orthophosphoric acid used during impregnation. The samples were also impregnated at different elevated temperatures, including 500 °C, 600 °C, 700 °C, and 800 °C. Only the samples impregnated at 800°C failed, specifically for the 1:2.5 and 1:3.5 ratios, while the remaining samples showed some yield, as shown in Table 6.

Table 6. Wt. Loss during activation for orthophosphoric acid (OPP) samples for 1:1.5, 1:2.5, and 1:3.5 ratio samples.

OPP Samples			**Wt. Loss (g)**	
Char:Acid Ratio	**500 °C**	**600 °C**	**700 °C**	**800 °C**
1:1.5	0.403	0.567	0.9	0.924
1:2.5	0.451	0.591	1.047	Fail
1:3.5	0.589	0.645	1.221	Fail

This sample's BET surface area and pore volume results demonstrated the highest values observed at 700 °C as shown in Table 7 and Figure 2a,b. Furthermore, the surface area and pore volume plots exhibited similar trends. The following table and plot present the detailed results.

Table 7. Surface area & pore volume data of orthophosphoric acid (OPP) 1:1.5 ratio sample.

1:1.5 OPP Samples		
Temperature (°C)	**Surface Area (m^2/g)**	**Pore Volume (cm^3/g)**
600	454.56	0.12
700	2150.99	1.0228
800	874.57	0.3671

During the adsorption studies, improved results were observed for these samples compared to the 1:4 samples. The % removal ranged from 30% to 89% as seen in Table 8. The sample with a char-to-acid ratio of 1:1.5 and impregnated at 700 °C demonstrated the best results.

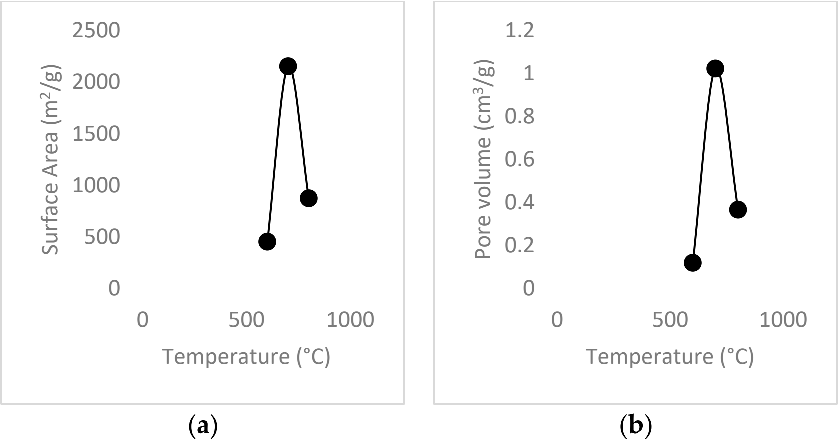

Figure 2. (**a**): Plot of BET surface area of orthophosphoric acid 1:1.5 ratio sample. (**b**): Plot of Pore vol data of orthophosphoric acid 1:1.5 ratio sample.

Table 8. Adsorption studies of orthophosphoric acid (OPP) 1:1.5 ratio sample.

700C OPP 1:1:5 Ratio					
Time	**Co**	**Absorbance**	**Ce**	**Ad. Capacity**	**% Removal**
1 h	100	0.158	10.604	178.79	89.40%
2 h	100	0.172	11.544	176.91	88.46%
3 h	100	0.3	20.134	159.73	79.87%
4 h	100	0.313	18.521	162.96	81.48%
				Avg=	84.799353

Adsorption studies were conducted under different conditions by varying the parameters. For instance, when the rpm was set at 150, the AC dosage was 25 mg, based on our preliminary studies, the phenol concentration was 50 ppm, the volume was 200 mL, the temperature was 30 °C, and the % removal was below 20%. Therefore, it can be concluded that decreasing the rpm, dosage, and pollutant concentration also leads to a decrease in the % removal capacity of the adsorbent sample. A raw sample, crushed and sieved through a 425-micron screen, underwent Ultimate analysis.

3.7. Ultimate Analysis

The result of the ultimate analysis exhibits an approximate carbon content of 50% as demonstrated in Table 9, a positive indication that the pore structure in any adsorbent is formed by carbon content. Production of a higher quality adsorbent is increased with higher carbon content.

Table 9. Results of Ultimate analysis.

Composition (%)	**N**	**C**	**S**	**H**	**Sample Weight (mg)**
Pinecone-Conifer cone	0.67	48.90	0.03	7.25	14.25

4. Conclusions

The adsorbent prepared with a 1:1.5 ratio, involving 1 g of char and 1.5 mL of orthophosphoric acid impregnated at 700 °C demonstrated the most favorable and optimal outcome. The overall percentage of removal achieved by this sample was approximately 85%. The peak efficiency in removal is attained within the initial 60 min of the adsorption study, followed by a slight decline in the third hour and a subsequent increase in the fourth hour.

The BET analysis shows that the sample's surface area was 2150.99 m^2/g, while the pore volume was 1.0228 m^3/g. These values surpass those documented in other research papers by a considerable margin. However, replicating the same level of yield quality presents a unique challenge, as a reason for white ash generation at the chemical activation stage.

In conjunction with the analysis of BET surface area and pore volume, the adsorption studies substantiate the viability of producing a superior-quality adsorbent utilizing a precursor such as a pinecone. Such an adsorbent successfully fulfils the objectives of being economically viable, locally sourced, and highly efficient in removing pollutants such as phenol. However, further studies need to be conducted on its performance thermodynamically at higher temperatures, along with more spectroscopy methods. The adsorbent performance at high concentrations of phenol is still unknown.

Pinecones being widely available in many parts of the country makes it a very diverse adsorbent, but it is very prone to contamination. We faced a major challenge in removing unwanted substances and drying because of its nature not to let go of water quickly. The presence of moisture could possibly be the reason for the formation of white ash and yield shortage. Orthophosphoric acid is relatively the weakest among our acid trials, allowing us to study the chemical activation potential of weaker acids at various concentrations. Performing kinetic studies at various intervals will also help plot absorbance levels at an optimal rpm. Using the remaining acids at lower concentrations might also be a good choice as acids such as sulfuric acid produced a significant removal of 35%, showing an excellent scope for improvement. The challenge with more vital acids was neutralizing them after chemical activation; since we were neutralizing them with water, we could have washed some of the adsorbents away.

The Pinecone (Conifer cone) derived adsorbent is predominantly carbonaceous, with significant carbon content (48.90%). Other elements, such as hydrogen and nitrogen, suggest a diverse range of functional groups that could interact with various contaminants during adsorption. This composition suggests that the material could be a potential adsorbent for specific applications, but its efficiency and specificity would need further testing and analysis.

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