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Adsorption of Direct and Reactive Dyes by Chemically Activated Carbons Derived from Caesalpinia *pulcherrima* Pod

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ABSTRACT

In this paper, the adsorption of direct red 81 (DR-81) and reactive yellow 145 (RY-145) onto activated carbon was prepared from *Caesalpinia pulcherrima* pod (CPP) in an aqueous solution was investigated. Activated carbon samples were prepared using three different acids and were examined for their Physicochemical characteristics. Visualized the surface porosity of activated carbon from the *C. pulcherrima* pod using scanning electron microscopy (SEM) analysis. Energy Dispersive X-ray Analysis EDX and Fourier transformation infrared (FTIR) spectroscopy techniques were used to detect surface functional elements. The study finds that *C. pulcherrima* pod-derived activated carbon is an effective and low-cost biosorbent for removing reactive yellow and direct red dyes from wastewater. It could also be used as an eco-friendly and cost-effective adsorbent in industrial wastewater treatment.

Keywords: *Caesalpinia pulcherrima,* Activated carbon, Surface area, Dye Adsorption, Textile Dyes, Reactive Yellow, Direct Red

INTRODUCTION

Activated carbons are exclusive and adaptable adsorbents due to their wide interparticle surface area, microporous structure, high adsorption capacity, and strong surface reactivity. The adsorption quality of activated carbon varies according to the functional groups and ions present on its surface [1]. Their important applications include the adsorptive removal of colour, smell and taste, as well as other unwanted organic and inorganic pollutants from drinking water, the treatment of industrial wastewater, air purification in inhabited spaces such as chemical

industries, food processing, restaurants and the purification of many chemical, food, and pharmaceutical products [1-4].

Activated carbon is produced by a regulated process of dehydration, carbonization, and oxidation of organic molecules [5]. It may be made from a variety of materials and used in laboratory research. The frequently used materials in commercial practice are industrial and agricultural by-products and forest waste, including coconut shells [6], sugar beet bagasse [7], rice straw [8], bamboo [9], rattan sawdust [10], molasses [11], rubber wood sawdust [12], oil palm fiber [13], waste apricot [14], and coconut husk [15]. Carbonization is a thermal treatment performed at 400-800°C to convert raw materials to carbon. This strengthens the material and forms an initial porous structure, which is required for the carbon to be activated. Adjusting the carbonization conditions can have a substantial impact on the end product.

At higher carbonization temperatures, the material's condensation increases, resulting in a decrease in the number of holes due to the enhanced mechanical strength. To select the appropriate process temperature for carbonization, it becomes critical that you do so based on the intended product. [16]. During the process of carbonization, a porous structure is initially created in the raw material. However, this structure is further refined during the activated carbon process. This process results in the conversion of the carbonized raw material into a form that contains an even greater number of pores, each randomly distributed in various sizes and shapes. The end product has an incredibly high surface area, making it highly efficient for various applications. The activated carbon process enhances the properties of the raw material, creating a product that can effectively purify air and water, remove impurities, and much more [17, 18].

The most extensively used methods for removing dyes from water and industrial wastewater include filtration, ozonation, flocculation, reverse osmosis, adsorption, electrochemical and biological degradation, chemical oxidation, and photocatalytic degradation. Even though such approaches exist, attempts to develop acceptable solutions with high productivity, low cost, and accessibility are rare [19, 20]. Adsorption is one of the approaches that has gained a lot of attention because of its benefits, such as low cost, process flexibility with no sludge generation, process simplicity, efficiency, and high speed [21, 22].

Activated carbon from locally available biowaste (*Caesalpinia pulcherrima* pod) is applied for the first time in batch adsorption of DR-81 and RY-145 dye with three different acids (Sulphuric acid, Phosphoric acid, and Nitric acid) as an activating agent. The outcome

of various conditions such as pH, adsorbent concentration, and contact time on the performance of the method was assessed.

MATERIALS AND METHODS

Caesalpinia pulcherrima pods are utilized as a precursor to the production of activated carbon. The pod was rinsed with distilled water, sun-dried for 5 days, and then crushed into little pieces to be utilized in the chemical process of producing activated carbon. The carbonized material was finely pulverized and analyzed utilizing physicochemical techniques and surface analysis.

Carbonization procedures

The carbonized pod material is impregnated with a boiling solution of 10% $H2SO_4$, HNO_3, and H_3PO_4 for an hour before being soaked in the same solution for one day. After 24 hours, the remaining solution was transferred and allowed to air dry. The material was then carbonized in a muffle furnace at 130-140 degrees Celsius. The dry material was crushed and activated in a muffle furnace at 800°C for 1 hour. The material was then rinsed with a large volume of water to remove the remaining acid.

Acid process

The dried material was carefully soaked in an excess of the appropriate acid solution for 24 hours. Charring of the substance occurs instantly, followed by the release of heat and fumes. After 24 hours, the surplus sulphuric acid was decanted and allowed to air dry. After the reaction had ceased, the mixture was carbonized in a muffle furnace at 150-160 °C. After this period, the product was rinsed with a considerable volume of water to remove free acid, dried at 120°C, and activated at 800°C.

Characterization of the Activated Carbon

Studying the physiochemical characteristics of activated carbon samples from *Caesalpinia pulcherrima* pods involves a range of analyses to determine their properties and suitability for various applications. Some common physiochemical characteristics that might be studied.

FTIR spectra and SEM

The FTIR 1725x (Perkin-Elmer) Spectrometer was utilized to analyze the electronic structure of carbon samples. Measurements were conducted across the spectrum from 4000 to 400 cm-1. Carbon samples containing 0.33 wt% were mixed with dry KBr (Merk, spectroscopy grade) and then compressed to produce suitable tablets. The Scanning Electron Microscope

(SEM) (Make: Philips SIRON with EDX facility at IISc Bangalore) was used to investigate the morphological characteristics of the carbon samples.

Fourier transform infrared spectroscopy (FTIR)

FTIR is a highly effective method for identifying chemical bonds in a molecule by analyzing the absorption spectrum of light, with characteristic wavelengths for each bond type. Activated carbon samples were analyzed using a Thermo Nicolet Avatar 370 spectrometer, where 0.1 g of sample was mixed with KBr and compressed at 15 kPa cm-2 pressure. The FTIR chromatogram shows the wavelengths of different functional groups, identified by comparing values with those in the library.

Scanning electron microscopy (SEM)

The Scanning Electron Microscopic (SEM) is a fascinating electron microscope that captures images of a sample by scanning it with a powerful beam of electrons in a precise raster scan pattern. The morphology, size, and structures of the particle were analyzed utilizing scanning electron microscopy (Model 6093, JEOL).

Energy Dispersive X-ray Analysis (EDX)

The Energy Dispersive X-ray Analysis (EDX) method, utilized alongside Scanning Electron Microscopy (SEM), is employed to analyze the elemental composition and chemical properties of a sample. By directing an electron beam onto the surface of a conductive sample, X-rays are emitted due to the impact of the material. The material influences the energy of emitted X-rays, with a penetration depth of about 2 microns. Despite the time-consuming nature of acquiring images due to low X-ray intensity, this technique may struggle to detect elements with low atomic numbers.

Batch adsorption studies

A series of 250 ml flasks holding 200 ml of solutions of Direct Red (DR-81) and Reactive Yellow (RY-145) at varying concentrations were filled with a known amount of activated carbon sample. After a full hour, the flasks were shaken at a steady temperature until they reached equilibrium. Centrifugation was used to remove the carbon compounds for 10 minutes at 10,000 rpm. By employing a calibration curve, the UV-vis Spectrophotometer was used to determine the adsorbate concentrations in the filtrates at wavelength lmax = 650 nm. The equilibrium EB uptake, qe (mg/g), was computed.

Results and Discussion

Table 1 lists the attributes of activated carbon made from Caesalpinia pulcherrima pod waste using different techniques. LSAC1, LSAC2, and LSAC3 carbon produced by acid processes had acidic pH values. Acidic groups that have been added to the surface of activated

carbon could cause this. Most commercially activated carbons are basic because the carbon still contains some residual salts. There are not many differences in conductivity levels. This is because the conductance may be caused by the anions in basic carbons and the cations in anionic carbons [23, 24].

Table -1: Physio-chemical characteristics of *Caesalpinia pulcherrima* pod wasteactivated carbon

S. No	Carbon Properties	CPPAC1	CPPAC2	CPPAC3
1	pH	7.16	9.64	8.24
2	Moisture Content, %	5.9	2.8	6.4
3	Ash Content, %	8.82	18.54	16.22
4	Volatile matter, %	23.5	20.6	18.1
5	Conductivity, ms/cm	0.43	0.72	1.68
6	Specific Gravity, S	1.24	1.35	0.716
7	Bulk Density, g/cm3	0.487	0.362	0.315
8	Porosity, %	58.64	76.3	72.5
9	Matter soluble in water, %	4.84	3.48	6.14
10	Matter soluble in Acid, %	6.2	14.9	16.2
11	Iodine Number, mg/g	164	325	512
12	Surface area, m2/g	143.25	267.51	432.81
13	Fixed Carbon, %	66	59.5	61
14	Yield, %	45.2	54.8	62.5
15	Process	H2SO4	HNO3	H3PO4

A respectable percentage of carbon was found and data was shown in Table 1. The number of contaminants in carbon revealed by acid-soluble materials has an impact on the quality of the water. However, current research indicates that all carbons have very little impurity. The key factor that increases an activated carbon's adsorption power is porosity. The bulk density and specific gravity of the activated carbon are correlated with their porosity. In comparison to other activated carbons, LSAC3 has a larger surface area and porous carbon. The iodine number characterizes the performance of activated carbon. It's a gauge of activity level, where a larger number indicates a higher level of activation [25, 26].

Fourier Transform Infrared Spectroscopy (FTIR)

Non-destructive, quantitative, and qualitative analyses are now feasible with great accuracy and precision because of technological advancements in instruments. In the past, inferential reconstruction has mostly been employed in qualitative analysis to acquire structural data. The functional groups involved in adsorption may be identified thanks to the band shift and variations in signal strength.

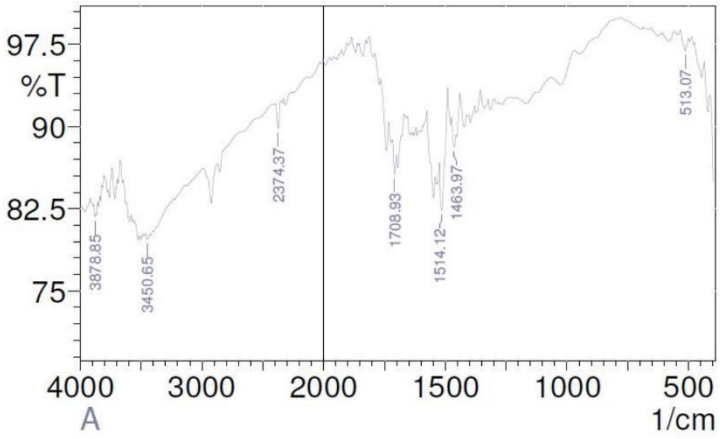


Figure 2. FT-IR spectrum for activated carbon from *Caesalpinia pulcherrima* pod processed by H2SO4

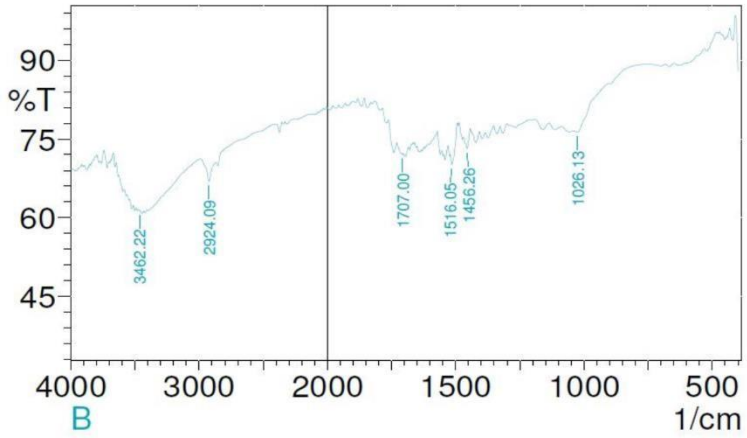


Figure 3. FT-IR spectrum for activated carbon from *Caesalpinia pulcherrima* pod processed by HNO3

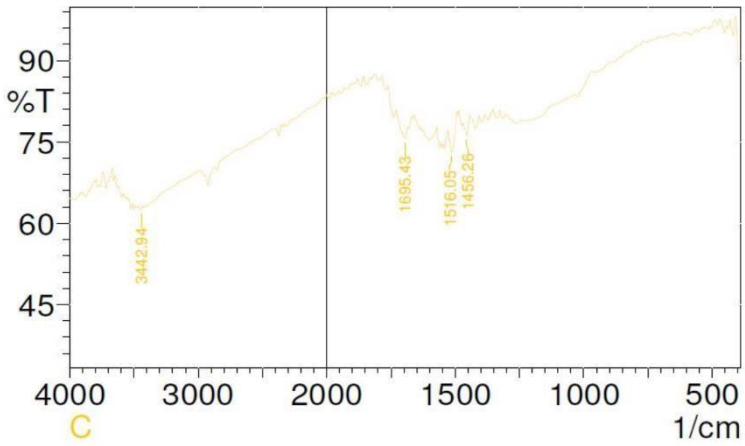


Figure 4. FT-IR spectrum for activated carbon from *Caesalpinia pulcherrima* pod processed by H3PO4

Figures 2, 3, and 4 display FT-IR spectra of activated carbon of Caesalpinia pulcherrima pod samples that were activated by various acids. The Caesalpinia pulcherrima pod's spectrum showed two bands: one at 2924 cm-1, which was only visible in sample B, and the other at 3878, 3462, 3450, and 3442 cm-1, which was attributed to the O–H stretching vibration of hydroxyl groups and intrinsic moisture.

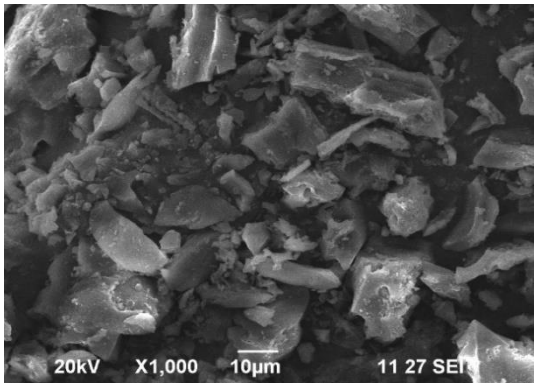
The band 2374 cm-1, shows that CO2 is present in the camber at sample A only. All three samples showed bands at 1708, 1707, and 1695 that may correlate to C=O stretching vibrations. The vibrations at 1516, 1514, 1463, and 1456 cm-1 may be associated with the aromatic ring stretching vibration of lignin, the C–H deformation vibration in lignin, and the C–C stretching vibration of carbohydrates in the lignocellulose texture, in that order. Furthermore, a band that appeared at 514 cm-1 indicated that sample A was the only one with the P=O stretching vibration. During heat treatment, the aromatization of the structure is facilitated by the activating reagents [27, 28].

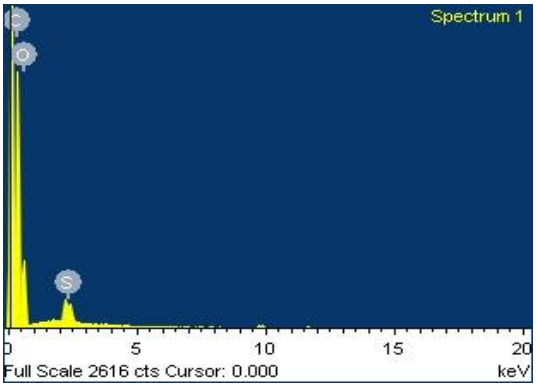
Table 2: FTIR absorption bands and corresponding possible functional groups of activated carbon of *Caesalpinia pulcherrima* pod

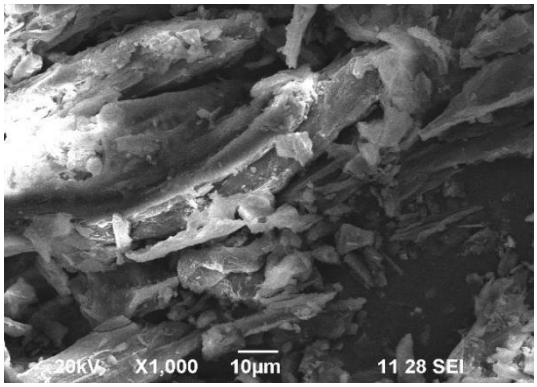
S. No	Sample	Band Position (cm-1)	Possible Assignment
1	**A**	513	P=O Stretching Vibration
		1464, 1514	C=C Stretching Vibration
		1709	C=O Stretching Vibration
		2374	CO2 Present in Chamber
		3451, 3879	O-H Stretching
2	**B**	1026	P=O Stretching Vibration
		1456, 1516	C=C Stretching Vibration
		1707	C=O Stretching Vibration
		2924	C-H Stretching
		3462	O-H Stretching
3	**C**	1456, 1516	C=C Stretching Vibration
		1695	C=O Stretching Vibration
		3443	O-H Stretching

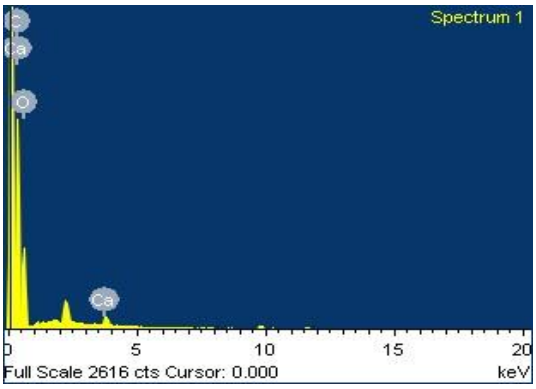
Microanalysis of structural and elemental composition of *Caesalpinia pulcherrima* pod using SEM-EDX technique

Caesalpinia pulcherrima pod microstructure and elemental composition were ascertained by scanning electron microscopy and energy-dispersive X-ray microanalysis (SEM-EDX). The surface physical morphology of activated carbon samples and Caesalpinia pulcherrima pods were observed using the SEM method. In Figure 5, the micrographs are displayed. The activated carbon surface morphology of Caesalpinia pulcherrima pods treated with various acids varies significantly. Following chemical activation, porous structures were formed, resulting in a surface full of voids in the activated carbon samples. On the surfaces of activated carbon, cracks, fissures, and some grains of different sizes developed in huge holes.









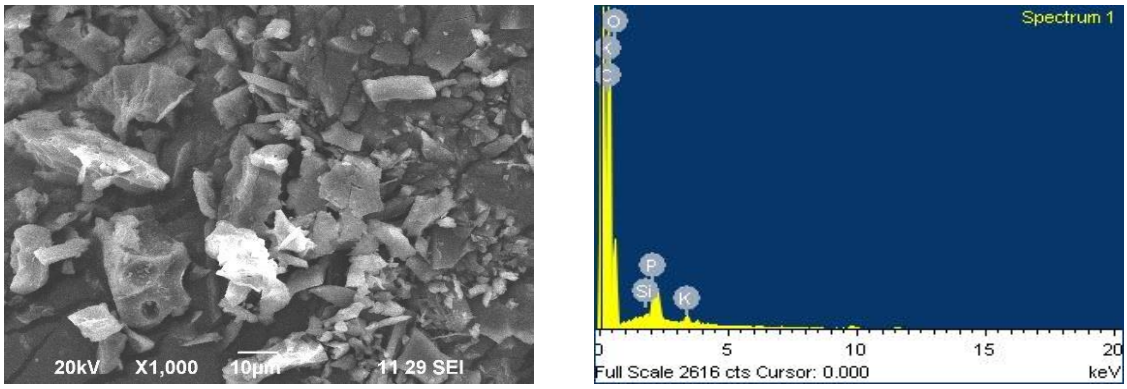


Figure 5: SEM-EDX image of *Caesalpinia pulcherrima* pod powder

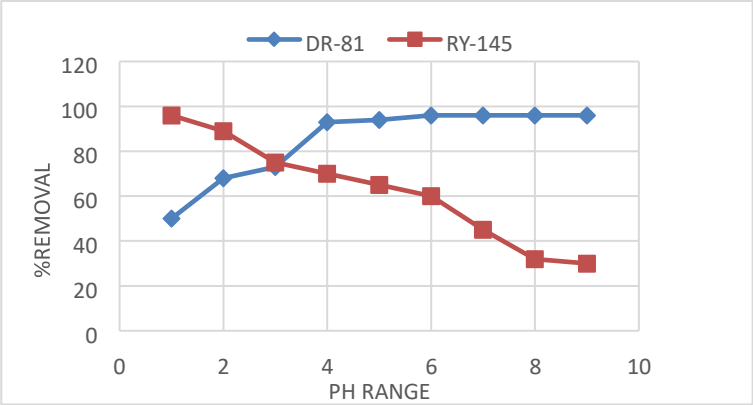
Table 3: EDX Elemental Microanalysis of activated carbon of *Caesalpinia pulcherrima* pod by using different acids

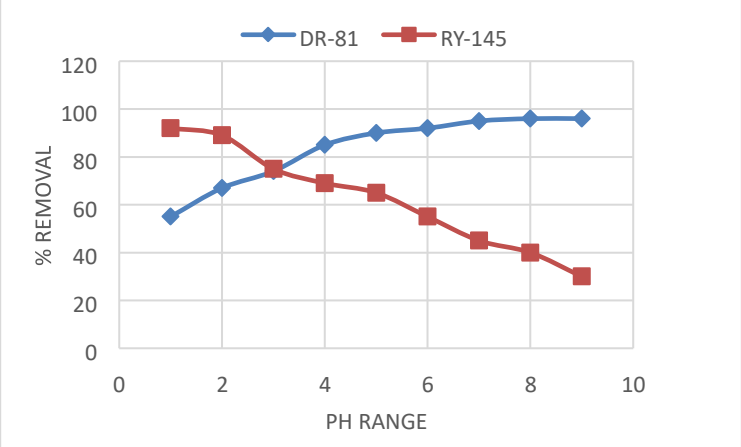
Sample	Element	Weight%	Atomic%
A	C K	57.32	67.35
	O K	41.81	33.32
	S K	0.56	028
B	C K	47.54	53.51
	O K	48.37	42.31
	Ca K	1.84	0.54
C	C K	70.96	76.51
	O K	24.63	22.22
	Si K	0.24	0.08
	P K	0.67	0.29
	K K	0.37	0.16

According to EDX, the carbon samples are mostly made up of different amounts of carbon and oxygen. Samples A and B (H2SO4 and HNO3) have larger carbon and oxygen contents than Sample C (H3PO4). In the sample C, silicon, phosphorus, and potassium were also found. The samples' EDX examination essentially reveals neither the presence of phosphorus nor nitrogen, which might account for the remarkably strong adsorbent qualities noted, especially for this activated carbon [29].

Effect of PH:

pH is the most crucial factor in dye adsorption investigations because it regulates the adsorbentadsorbate interaction, which causes fluctuations in pH values. Establishing the optimal pH levels is very important. The optimal activity of activated carbon on dye removal was determined by adjusting the reactive yellow 145 and direct red 81 solution's pH from 2 to 10.





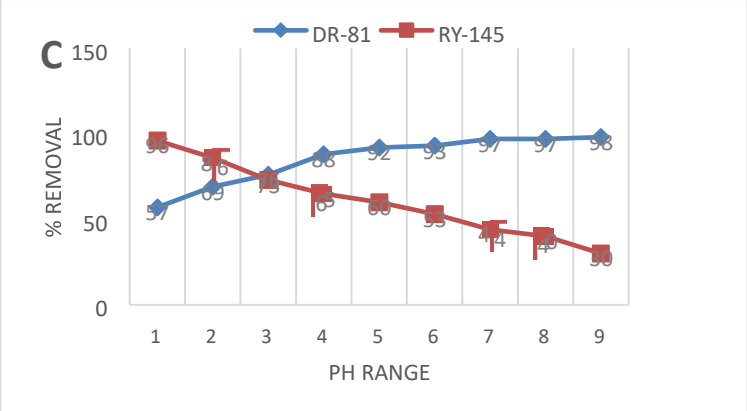


Figure 6: Effect of PH on dye removal of Activated carbon synthesized by using a) Sulphuric acid b) Nitric acid C) Phosphoric Acid

The removal efficiency of the Reactive yellow 145 and Direct red 81 dyes on synthesized activated carbon at different PH levels is shown in Figure 6(a, b, c). The graph demonstrates that the highest RY-145 dye removal efficiencies from activated carbon were noted at pH 3, and efficiency sharply declines when the dye solution's pH rises. More than 30% of synthesized activated carbon was lost at the maximum pH value of 10.0. On the other hand, the DR-81 dye graph shows that removal capacity increased when the pH value increased, reaching its maximum removal capability at pH 5. This observable fact may be explained by the adsorbent's anionic and cationic properties, as well as the transmission and removal of protons from its surface effective groups [30, 31].

Effects of adsorbent dosage and contact time:

The adsorbent concentration varies from 0.01-0.1g, which affects removal efficacy. Equilibrium and maximum adsorption efficiency were reached between 0.05 and 0.1g. Therefore, the adsorbent mass concentration within the range of 0.05-0.09g/l at a certain contact period is selected to assess the adsorbent mass of the manufactured activated carbon. As the adsorbent dosage was increased from 0.05 to 0.09 g/L, the adsorption quantity reached equilibrium (qe) of the activated charcoal, with almost identical values for all samples (Fig. 7 a,b,c). This is because less active sites are produced as a result of limited aggregation, which appears at higher adsorbent concentrations [32, 33].

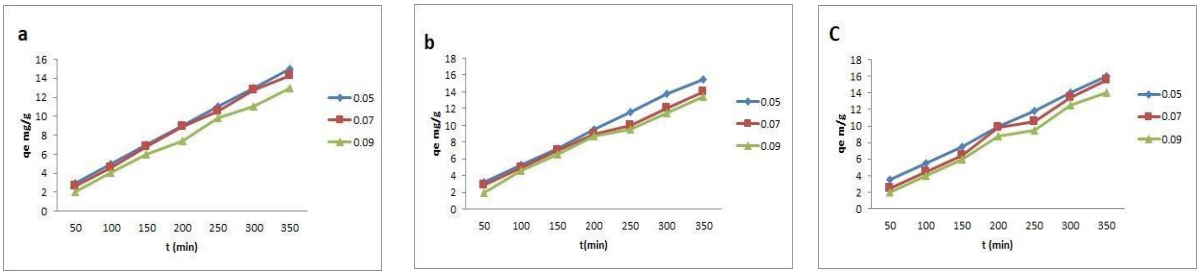


Figure 7: Effect of Adsorbent dosage and contact time on the adsorptive uptake of biowaste biowaste-activated charcoal synthesized by a) Sulphuric Acid b) Nitric Acid C) Phosphoric Acid

Effect of the temperature:

The impact of temperature on adsorption was assessed at the optimal concentration of adsorbent. The temperature dependence of the dye was found to be best at an adsorbent dosage of 0.05 g/L. Figures 8a, b, and c illustrate the dye's capacity to adsorb onto activated charcoal samples at temperatures between 30 and 50 °C. The results show that when the solution temperature rose from 30 to 50 °C, the dye's equilibrium adsorption capacity increased. The increase in adsorption capacity may be described by strong interactions between dye molecules and the activated charcoal's synthetic active sites. Direct red and reactive yellow show comparable behaviours when adsorbed.

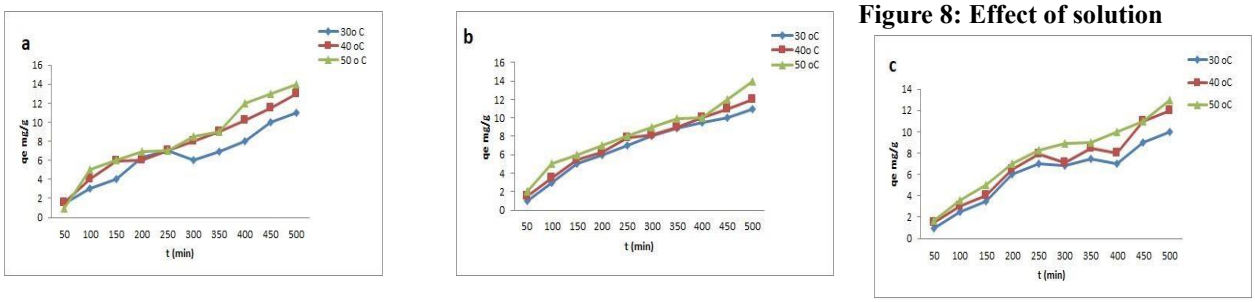


Figure 8: Effect of solution temperature on the adsorption of the dye onto AC synthesized by a) Sulphuric acid b) Nitric Acid c) Phosphoric acid (conditions: W=0.05 g/L; C0=10 mg/L).

As evidence, several research findings show that food dye adsorption rose with temperature, perhaps speeding up the reaction at the optimal temperature. The most plausible explanation is that when temperatures rise, the surface area and pore depth increase, increasing the probability that the dye will pass through the outer border layer. This supports the results of earlier studies [34-35].

Conclusion

By chemically activating the *Caesalpinia pulcherrima* pod in phosphoric, nitric, and sulfuric acids, activated carbon was produced. The conditions of preparation affect the carbon's quality. To compare the three activated carbons, many factors have been found to identify the quality of the activated carbon such as pH, density, iodine number, surface functional groups, and surface characterization by SEM and EDX. H_3PO_4 treated Caesalpinia *pulcherrima* pod produces superior activated carbon due to its greater carbon content and lower oxygen concentration, as supported by EDX experiments. These activated carbons are employed in adsorption processes to remove organic colors from aqueous phases, such as direct red.

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