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Removal of Congo red Dye from a Synthetic Wastewater Using Parthenium Carbon as a Bio-Adsorbent- An Environmental Sustainability

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Abstract: In this study analysis were conducted for synthetically prepared Congo red dye. Activated parthenium carbon derived from Parthenium hysterophorous weed employed as an adsorbent. Physical activated carbon (PAC) & chemical activated carbon (CAC) both were used for experiential under batch studies. Dye molarity 1500 mg/l, other parameters like carbon dose, pH, & time in contact were examined. Optimum adsorbent of 3g/50ml, pH 8.5, time in contact of 60 min were found effective. Column studies were carried to examine the role of dye starting concentration, rate of flow & bed height on process of sorption. The optimum pH and Flow rate was 8.5 and 10 ml/min respectively. Modern techniques like IOT was used in getting real time data of parameters. SEM, FTIR were recorded which shows good surface morphology, group of functional elements (C=O, C-O, O-H). Parthenium weed can be considered as ideal material for dye sorption, the efficiency of sorption studies observed 95-99%. This model best fits for Freundlich isotherm adsorbent is heterogeneous; overall process is economical and sustainable.

Keywords: Adsorption, Parthenium Carbon, Dye Contamination, Environmental Sustainability, IoT

1. Introduction

Among all the resources which naturally driven, water is most vital and dynamic role in the existence of all creatures. Mainly surface sources like ponds, lakes, Rivers etc. People depend on these resources for their livelihood it may be domestic, agricultural and industrial activities [1]. But due to pollution from Natural and Anthropogenic Activities these sources are getting pollute, Industries like textile, food, paints, paper, printing, leather and cosmetics [2]. Which using Dyes in their manufacturing Activities are discharging their wastewater or effluent with improper treatment to water bodies because serious environmental crisis mainly it reduces photosynthetic activity leads to reduction in penetration of sunlight which decrease Dissolved oxygen level [3]. It also cause problems like increase in biological oxygen demand, Bioaccumulation, Eutrophication growth of unwanted spices due to overloading of nutrients, Effect on Aquatic life because some dyes are Carcinogenic in nature [4]. In addition to its impact on the aesthetic appeal of water bodies [5], numerous rivers in India, such as the Yamuna, Ganga, Noyyal, Sabarmati, Bandi, and Damodar, are affected [6]. In Karnataka, rivers including Krishna,

Vrushabhavathi, Cauvery, and Shimsha are notable examples. Dye are Categorized in to Natural & Synthetic dyes based on its origin and based on its application in industries they are divided in to Direct dye-Congo red, Reactive Dye-Reactive Pink, Disperse Dye Disperse yellow, Acidic Dye-Acid orange 7, Basic dye Methylene Blue [7], Methyl orange [8], Vat dye Indigo vat blue [9] and Sulphur dyes Sulphur black. Among all industrial sectors Textile Industry is the most Water consuming industry for their Manufacturing Activity and it also discharge dye contain wastewater to water bodies in many places across the country [10]. *Parthenium Hysteroporos* is a weed, it belongs to plantae a plant kingdom, Asteraceae a plant family, parthenium which plant genus & *parthenium hysterophorus* a plant species it is famous as congress grass in our local areas. It is very much known for its allelopathic effect & chemicals which suppress crops [11].

Many researchers worked on removal of dyes using much technique like, Electro Coagulation [12], Microbial Decolorization, Fenton Technique, Ozonation, Membrane Separation, Flocculation, and Adsorption [13], bioreactor [14]. Adsorption Technique was opted in this Research work to remove synthetically prepared Congo red dye by using *Parthenium Hysteroporos*

weed as Adsorbent. The key innovations in this research include the transition from batch studies to fixed-bed column studies, enabling a more comprehensive analysis. Additionally, Parthenium carbon was developed to investigate continuous adsorption, breakthrough behaviour, and the adsorption capacity of Parthenium carbon [15].

Parthenium is highly noxious unwanted weed with less or no economic importance its rapid growth leads to problem for agriculture and biodiversity. Leads to skin disease, respiratory problems and allergic reactions to human, even cattles do not eat this grass sometimes rashes will be observed on cattles also in agriculture due to overgrowth of parthenium many crops get low yield and loss to farmers [16].

It is very difficult to treat Industrial Effluent and also in the other way it was difficult to monitor the Operation Parameters like pH, Temp, Dye Concentration and some other, According to CPCB standards for effluent discharge from Textile industry or Dye discharging sectors pH should be 6.5-8.5, Colour 150 PCU, BOD 30 mg/L. in the current research IOT (Internet of Things) and Image Processing is used to get real time data in treating industrial effluent with respect to date and time. This helps in continuous monitor of treated effluent from fixed bed column and reduce man power and also helps in maintaining record of every day [17].

Many adsorbents were used by researchers like Rice Husk [18], coconut shell [19], leaves [20], polymers [21], sugarcane baggase [22], Tea residue [23], waste ash [24] and many But cost of activated carbon and regeneration force researchers to move towards low cost bioadsorbent like parthenium, adsorbent is much important in technique adsorption, its physical and chemical characteristics, surface pores, Morphology, its functional group which is favorable adsorption and also size of the carbon is divided in to two types powdered & a granular carbon, in current research powdered activated carbon is used because it is best for lab scale study moreover granular activated carbon is suitable for pilot scale study. And also charges of adsorbent and adsorbate also very important at low pH Adsorbent poses negative charge and attract negatively charge dyes mainly anions and at higher pH adsorbent act positive charge attract positively charge cations [25].

However research on parthenium weed as an adsorbent is very low Batch studies were carried by some researchers but column studies to know its adsorption capacity and breakthrough along with IOT and image processing is considered as research gap in this field and it is an alternative way to treat Industrial Effluent which contains dye [26].

Sustainability Goals (SDG): This research mapped to SDG 3, 6, 9,13,14,15.

2. Materials and Methods

2.1 Adsorbent

Parthenium hysteroporous unwanted plant/weed utilized in the form of adsorbent, a well grown complete weed was harvested & cleaned to eliminate soil and dust, pollen grains, leaves then it is dried on terrace under light of sun for 72 hrs. Only well grown stem taken in to account. It was smashed to reduction in size of particle then sieved in 300u. The powdered form of parthenium took for the activation process. Activation is done to promote the strength of adsorption and adsorbent material it is done via physical activation heating adsorbent material at 250-300 C in muffle furnace. Chemical activation is done by using Concentrated acid in that H₂SO₄ 1.2 (W:V) & washed in remove acidic content till it reach the ph 6.5-7 then soaked with sodium bi carbonate for removal of additional acids to kept in oven for 20 hrs at warm temperature to eliminate moistures and it was used as adsorbent [27]. Parthenium Toxicology: parthenin is thought to be the main toxin and allelochemical that causes most of the ill effects that the plant, people, animals, and the ecology. A number of harmful allelochemicals are released by the very invasive plant Parthenium hysterophorus, however the most noticeable and prevalent one is Sesquiterpene lactone, Phenolic acids, toxicity analysis were done for treated water using HPLC found toxins less than 0.05%.

2.2 Adsorbate

A Congo Red dye (CR) used as an adsorbate, where it belongs to group of Direct dye based on the application in textile industrial sector, Molecular formula: C₃₂H₂₂N₆Na₂O₆S₂, shows in figure 1 and possess negative charge Anionic nature [28].

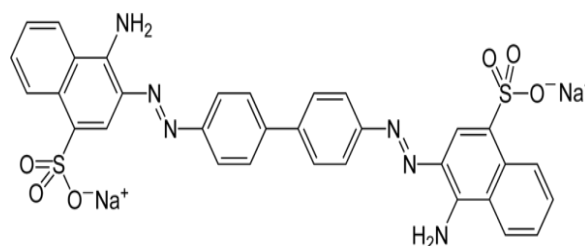


Figure 1. Structure of Congo red Dye

Wavelength (λ max) at around 497–498 nm, and its molar mass is 696.665 g/mol. A precisely weighed powdered CR dye was dissolved in RO water [29].

2.3 Sorption studies

In a 250 ml conical flask; the effects of variables were examined, including pH, time in contact, adsorbent weight & adsorbate concentration as shown Figure 2. 1.5g of powdered Congo red dye was added to 1.0L of water/solvent to create the dye stock aqueous solution,

which had a concentration of 1500 PPM. Additionally, a synthetic sample was made, 50 ml of working solution were taken for analysis, and various concentrations were created by a series of dilutions using RO water [30]. All of the flasks were kept in the shaker at 150 rpm and 25°C for a predetermined period of time, which ranged from 20 to 140 minutes, at 20-minute intervals. Varying quantities of adsorbent (0.5–4.0 gm) were added to each flask shown in figure 2, Adsorbate initial concentration was varied. Each 250 ml flask contained 50 ml Congo red dye, which ranged in conc. from 10 - 5000 mg/L. A pH meter and a diluted 0.1 M HCl and 0.1 M NaOH were added to balance the pH of each flask between 2 and 12. The absorbance of the supernatant water then measured after the solution filtered and the flasks were taken out. For both Physically activated Carbon (PAC) and chemically activated Carbon (CAC) all the experiments were carried out in three trails and two consecutive results were considered for % removal calculation [31].

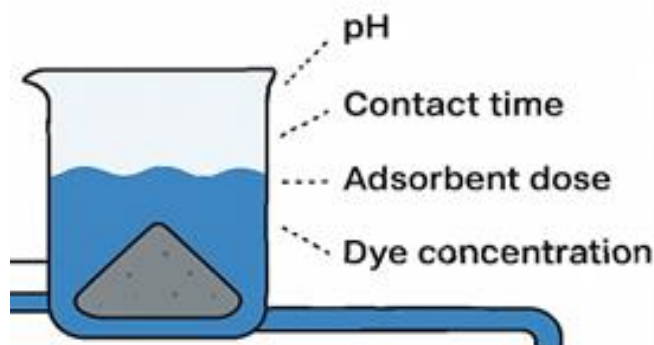


Figure 2. Batch Studies with Operating Parameters

$$\text{Adsorption \%} = (C_0 - C_e) / C_0 * 100$$

Where C_e = CR dye's final molarity (mg/L) and C_0 = beginning molarity (mg/L).

Parthenium hysterophorus's adsorption capacity, which was determined using the mass balance technique

$$Q_e = (C_0 - C_e) * V / W, \quad Q_e = \text{Parthenium hysterophores is adsorption capacity (mg/g).}$$

V & m volume (L) & adsorbent's mass (g) respectively.

2.4 Fixed Bed Column Studies (FBC)

Glass column/ jar were taken to carry continuous-flow fixed-bed adsorption test shown in figure 3. To support the intake and outlet pipes, a 2 cm rubber cork was positioned at both the ends of carbon in a column. In order to achieve a bed height of 10 cm, column was filled with 5 cm of pebbles at both the top and bottom, followed by 1.7 cm glassed wool, glass beads, and mesh to prevent carbon from floating. The

adsorbent was packed individually in the column. For this investigation, an adsorbent (10 cm)-filled column of 5.5cm= dia & 38cm=height was used. A synthetic dye solute with an initial conc. of 1000 mg/l was taken to charge the column, which was then operated at an average flow rate = 10 ml/min. A double-beam UV/visible spectrophotometer were used to detect the remaining dye concentration after samples were taken at predetermined intervals of time [32]. Up-flow mode was used to operate the column, Column studies were carried for Congo red dye in a Fixed bed column with height 10cm, mass of carbon 50g, Bulk density=0.255 g/cm³, porosity=0.40, Hydraulic retention time 7.85 min. a physically activated carbon is used as adsorbent [33].

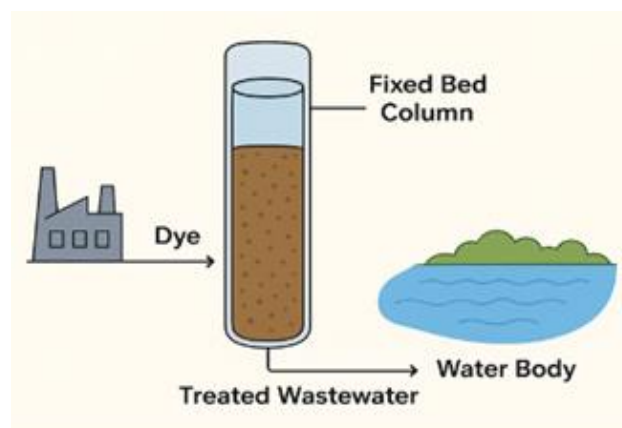


Figure 3. Treatment of dye using fixed bed column

To monitor Treatment of dyes and to record its initial and final concentration IOT (Internet of Things) was used shown in figure 4. Also it monitors pH at initial and final outlet. for this technique pH sensor and Colour sensor –(TCS 34725 with RGB sensor) was used with Signal conditioning board which convert analog to digital and microcontroller device Aurdino UNO and IR (Inferred Sensors were used to detect the object as shown in figure 4 [34].

3. Result and Discussions

3.1 Effect of Initial dye concentration

By altering the carbon content (10–5000 mg/L), the adsorption of dyes on carbon was investigated. However, it steadily declines when fewer adsorption sites become accessible, given the inhibitory effects of high dye concentrations demonstrated in this experiment, a lower percentage of decolorisation was seen at high dye concentrations. This implies that the initial dye concentration influenced the percentages of dye decolorisation since it offers a strong driving force to overcome all of the dye's mass transfer resistances between the aqueous and solid phases [35].

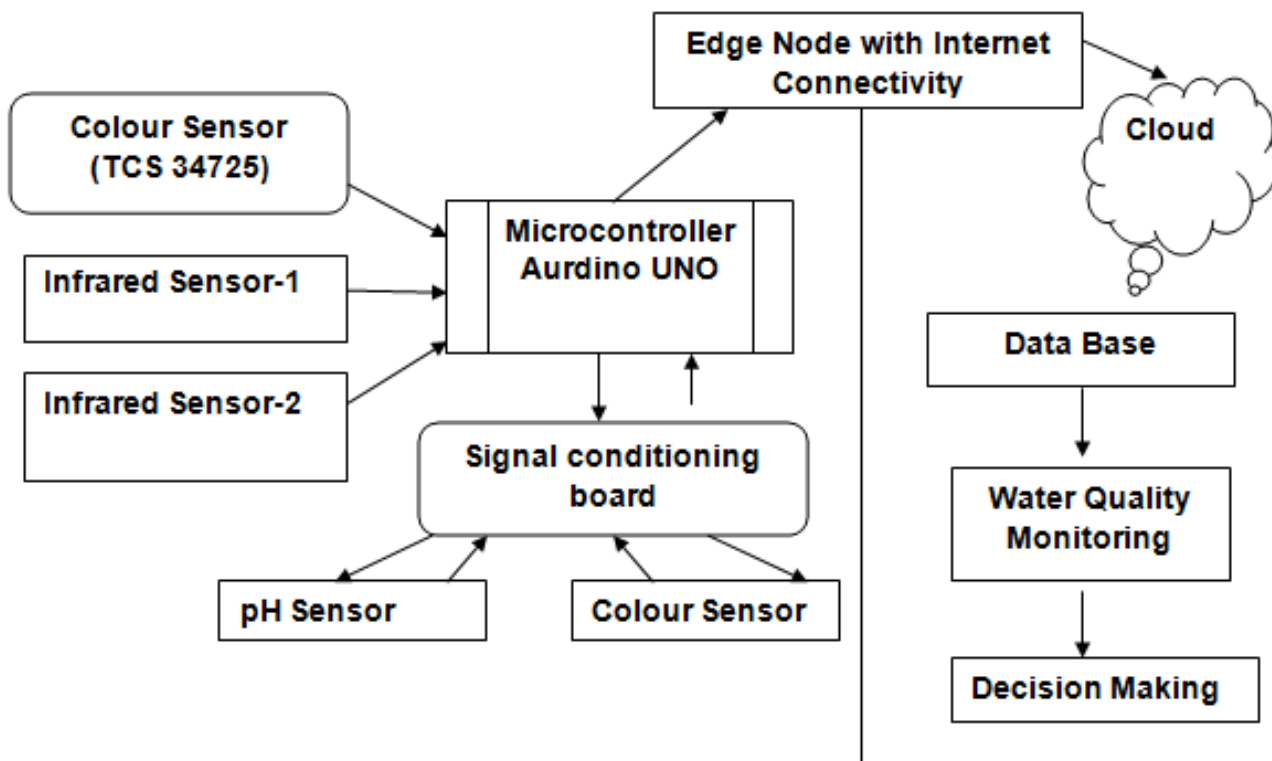


Figure 4. Shows Components of IOT system

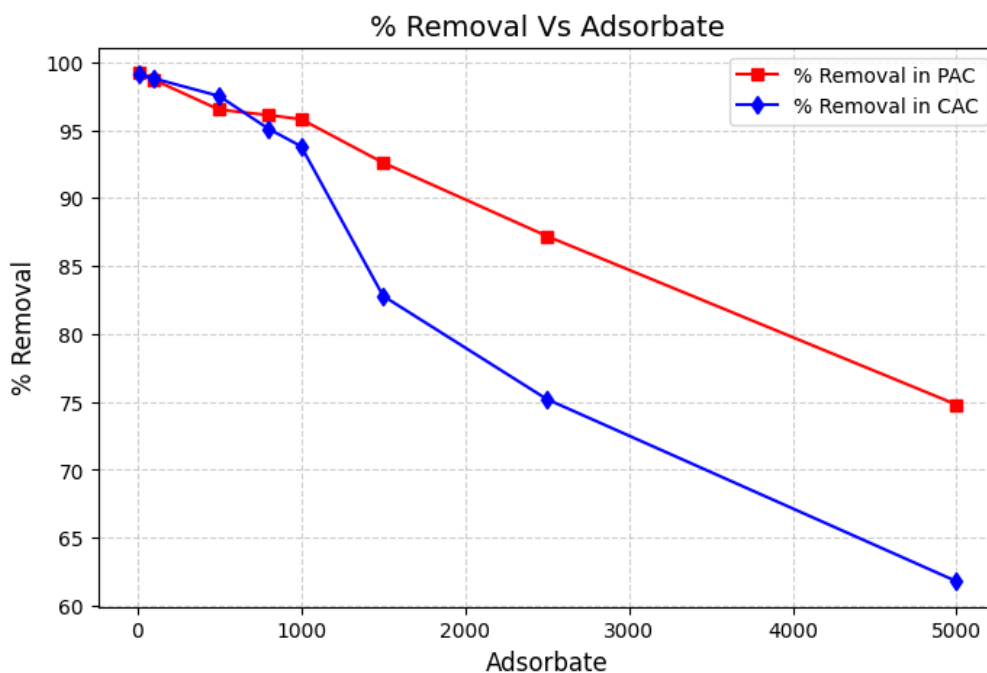


Figure 5. Graph shows Adsorbate vs % Removal at constant pH=8.5, Adsorbent=2.0 g, Contact time = 60 min).

3.2 Effect of Adsorbent Dosage

By adjusting the adsorbent dosage from 0.5 g to 4.0 g to dye conc. of 50 mg/L, the percentage of dyes eliminated been investigated. The impact of adsorbent dose was examined in an effort to improve CR dye removal. As the amount of adsorbent rises, figure 5

shows clearly show that the clearance % of CR dyes increases prior to stabilization. Up to a maximum efficiency (~99%), the removal rose as the adsorbent dosage grew; beyond that, the dye removal did not improve further, suggesting that even with the use of huge amounts of adsorbents, a complete dye removal was not possible. 3.0 g of adsorbent was employed for

the subsequent equilibrium investigations. The adsorption potential reduced as the adsorbent dose increased when an excessive amount was added to the dye solution. The adsorption capacity for PAC & CAC decreased as the adsorbent carbon weight was increased from 0.5 to 4g. This might be due to overlapped adsorption sites. Because dye ion transit to the active adsorption sites is likewise restricted, overcrowding of adsorbent particles reduces adsorption effectiveness [36].

3.3 Effect of time

Figure 6 shows how contact time affects the elimination of unmodified *Parthenium hysterophorus* at various equilibration times. It suggests that as contact duration rose, so did the rate of colour loss. The adsorbent's % removal of CR dye was fast for the first 60 minutes, following which it progressed more slowly and eventually reached saturation at various contact

times for varying dye starting concentrations. Because of the adsorbent's wide surface area accessible to adsorption in dye ions, the rate of adsorbate removal is initially greater. Because there were so few active sites on the surface of the sorbent, the amount of dye absorption increased relatively little over time [37]. The relationship between contact time and percentage removal for a fixed adsorbate concentration is further illustrated in Figure 7.

3.4 Effect of pH

Over a pH range of 2 to 12, the impact of starting pH on the bio-adsorption percentage of CR dye was investigated; the results showed that the dye removal was lowest at starting pH 2. As the pH was raised from 2 to 12, more dye was adsorbed (Figure 8). After pH 8 it's not substantial. Because of this, pH 8.5–9.0 was chosen for next equilibrium research.

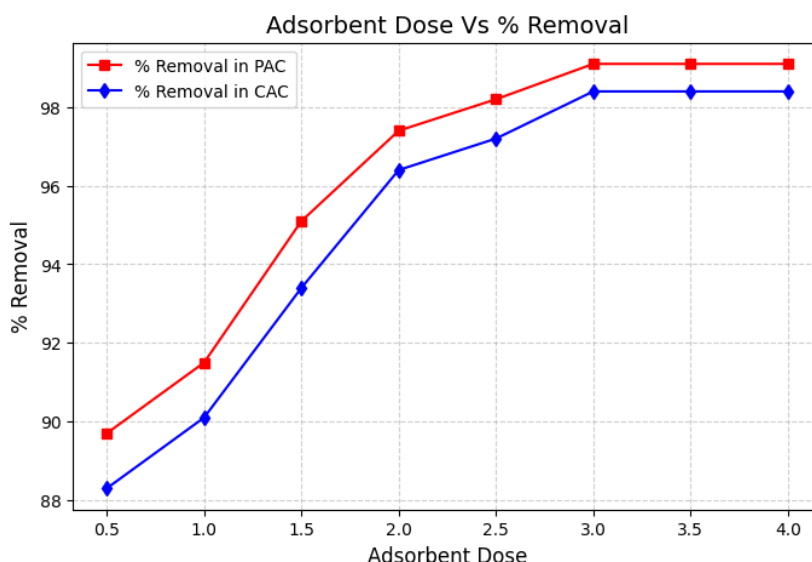


Figure 6. Adsorbent vs % Removal, at constant pH=8.5, Adsorbate = 1500 PPM, Contact time = 60 min)

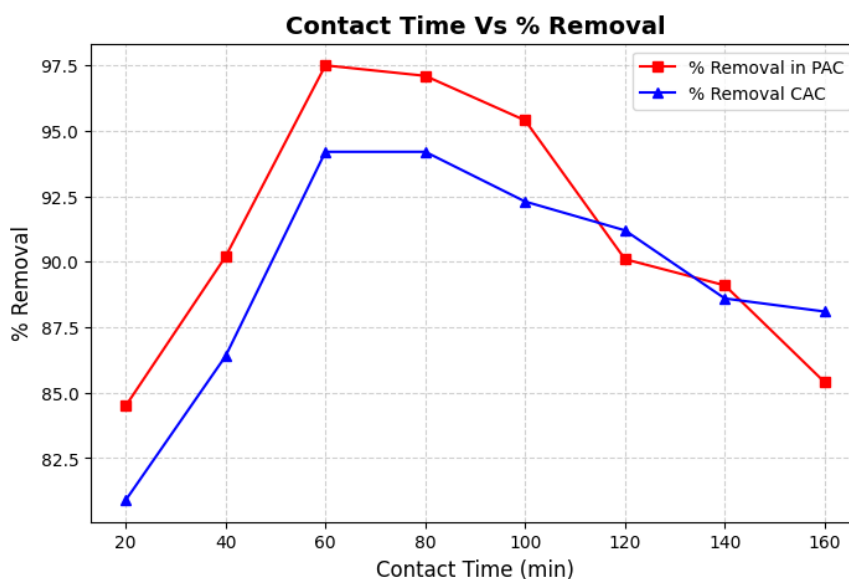


Figure 7. Contact Time vs % Removal at constant pH=8.5, Adsorbate concentration = 1500PPM, Adsorbent = 2.0g)

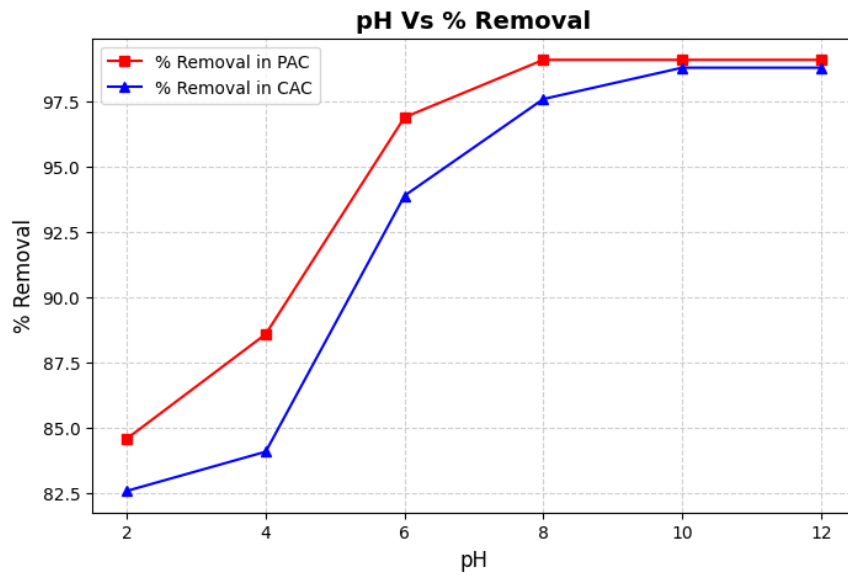


Figure 8. Graph shows pH vs % Removal, where at constant Time= 60 min, Adsorbate concentration =1500PPM,

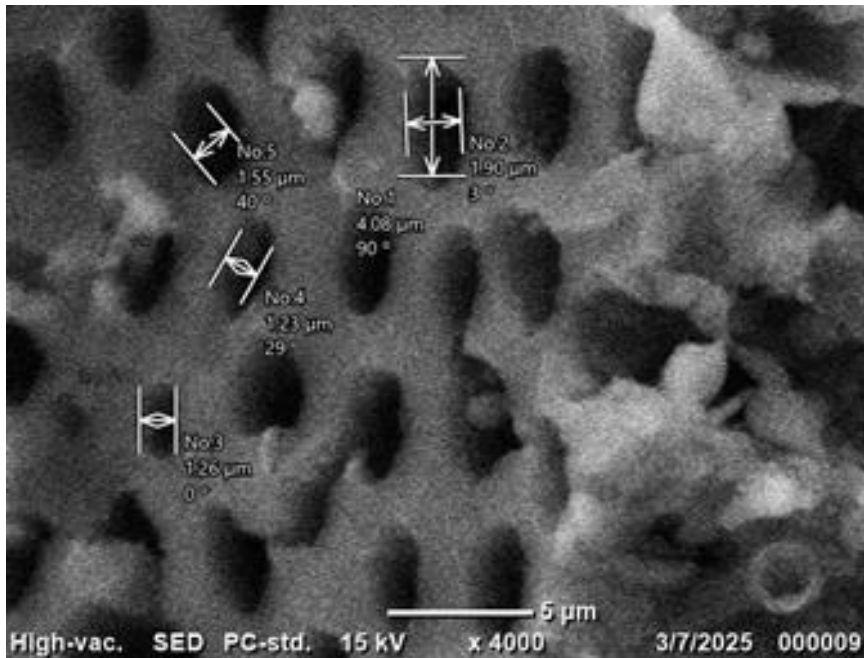


Figure 9. SEM analysis of PAC with macropores Adsorbent=2.0g)

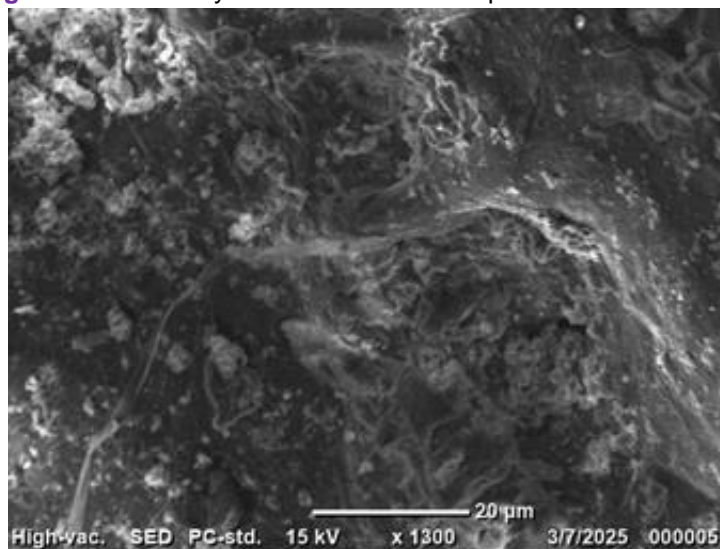


Figure 10. SEM analysis of CAC with micropores & mesopores

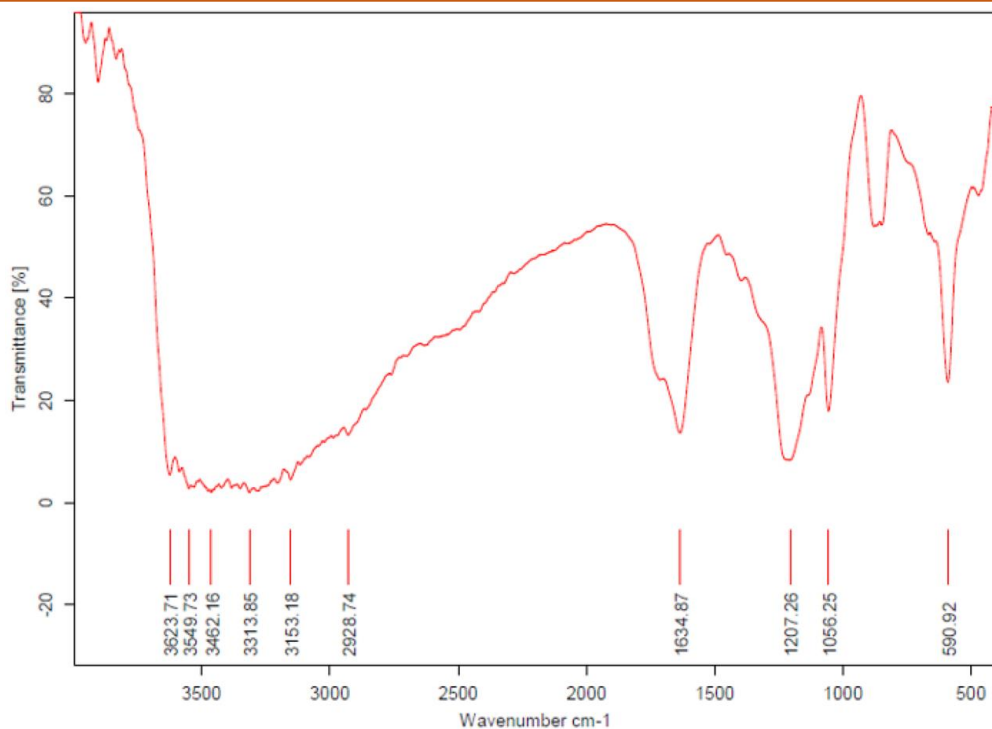


Figure 11. FTIR analysis of PAC with wavenumber

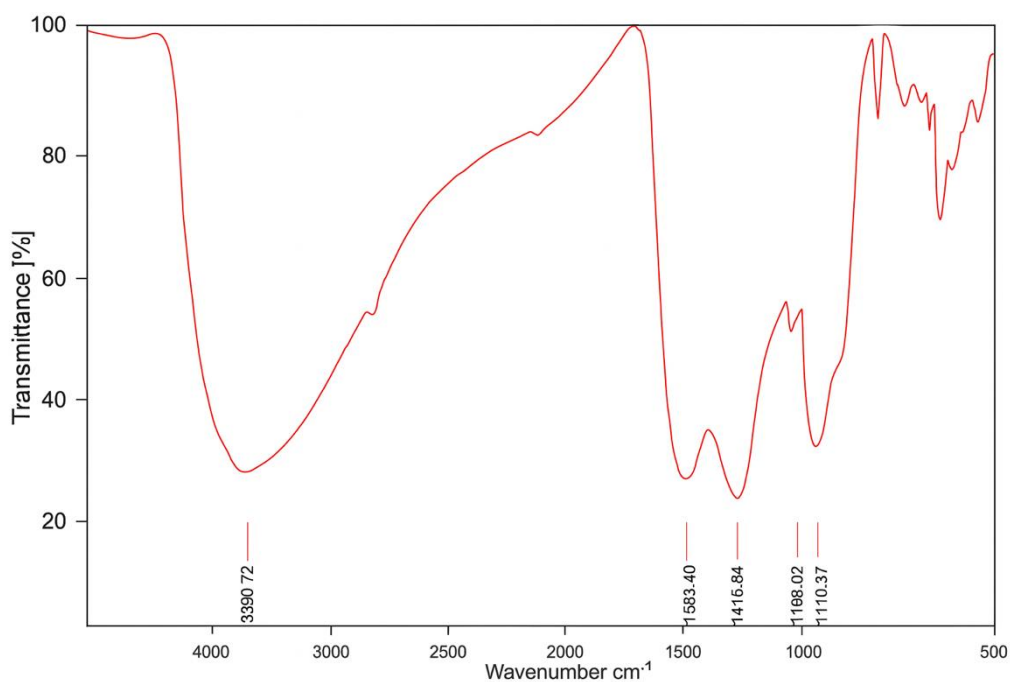


Figure 12. FTIR analysis of CAC with wavenumber

The dyes get protonated at low pH, and the electrostatic repulsion in protonated dyes and +^{ve} charged adsorbent sites leads to lower adsorption, according to adsorption tests. pH not only act in adsorption it also play vital role in Human health, aquatic and other Environmental sustainability concerns [38].

Scanning Electron Microscope (SEM) analysis was used to see how the carbon's shape changed. The interaction b/w adsorbent & adsorbate can be linked to the porosity in the biosorbent structure shown in figure 9 and 10. SEM analysis was performed both for dye loaded and unloaded carbon where Micro, Meso and

Macro Pores were observed which make dye molecule to adsorb in larger quantity makes adsorption favorable, where it shows heterogeneous and Amorphous in nature.

Fourier Transform Infrared Spectroscopy (FTIR) Spectra of the Parthenium Carbon was analyzed before and after Adsorption to know the Functional Group and also know stretching and bending based on position of the peak in the figure 11 & 12 it shows FT IR analysis of PAC where peak 3623-3462 cm^{-1} shows presence of –OH hydroxyl group, 3314 cm^{-1} shows Amine (-NH), 3153 cm^{-1} = C-H Aromatic, where 1635 cm^{-1} C=O carbonyl

which are favorable for adsorption. Same in CAC FT IR 3380 cm^{-1} show -OH group, C=C at 1583 cm^{-1} and C-H, C-O at 1416-1196 cm^{-1}

C_t in the interval of every 60 min, breakthrough was attained at 249 min where C_t/C_o is 0.05 and bed reached saturation at 813 min where C_t/C_o is 0.95 shown in table 1, total 8.8L of dye treated and 5.6g of dye removed and breakthrough was shown in figure 13.

4. Results of FBC & IOT

The adsorption with FBC was carried out from 1 to 880 min (Figure 13), with a continuous flow at flow rate 10ml/min, with initial conc. of dye 1000 mg/L (C_o), the conc. of dye at outlet with respect to time t is taken as

Table 2 shows data obtained by IOT tool where IR sensors and colour sensor were placed near the inlet & exit point of FBC which recorded in a programmed mail id which is helpful to identify the dye concentration at both inlet & outlet also documented without manpower [39].

Table 1. Results of Column Studies with Breakthrough

SI No	Time(min)	C_t (mg/L)	C_t/C_o	% Removal
1	60	0.04	4e-05	99.996
2	120	0.08	8e-05	99.992
3	180	1.66	0.00166	99.834
4	240	30.44	0.03044	96.956
4.5*	249.14	50	0.05	95
5	300	158.81	0.15881	84.119
6	360	197.6	0.1976	80.239
7	400	288.4	0.2884	71.16
8	460	386.8	0.3868	61.32
9	520	465.1	0.4651	53.49
10	580	598.4	0.5984	40.16
11	640	746.8	0.7468	25.32
12	700	856.6	0.8566	14.34
13	760	916.4	0.9164	8.36
13.5*	813.33	950	0.95	5.0
14	820	954.2	0.9542	4.58
15	880	954.7	0.9547	4.53

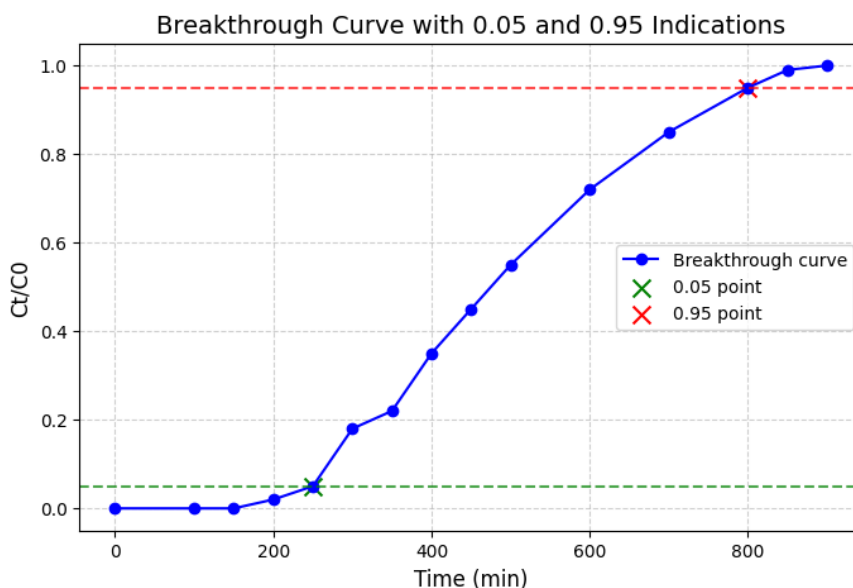


Figure 13. Breakthrough curve for fixed bed column studies from Time 1 to 880 min

Table 2. Results obtained from IOT with RGB values

Date	Time	Initial Conc. In PPM	Final Conc. In PPM	RGB values
24/7/25	06:23 PM	10	0.0233	(26.0, 21.69, 17.18)
24/7/25	06:27 PM	100	0.0064	(16.0, 31.49, 19.16)
24/7/25	06:27 PM	500	0.02865	(21.0, 18.69, 30.32)
24/7/25	06:27 PM	1000	0.07958	(23.1, 26.49, 19.18)
24/7/25	06:29 PM	1500	0.04880	(22.0, 26.69, 15.18)

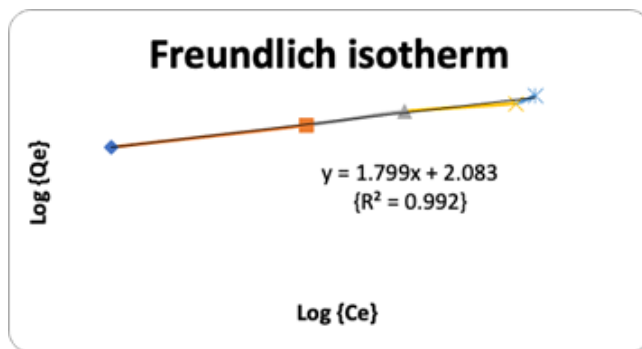


Figure 14. Freundlich isotherm with Regression equation shows adsorption is heterogeneous

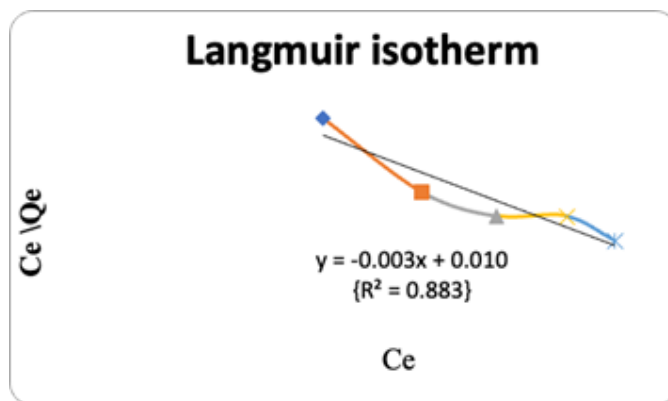


Figure 15. Langmuir isotherm with Regression equation

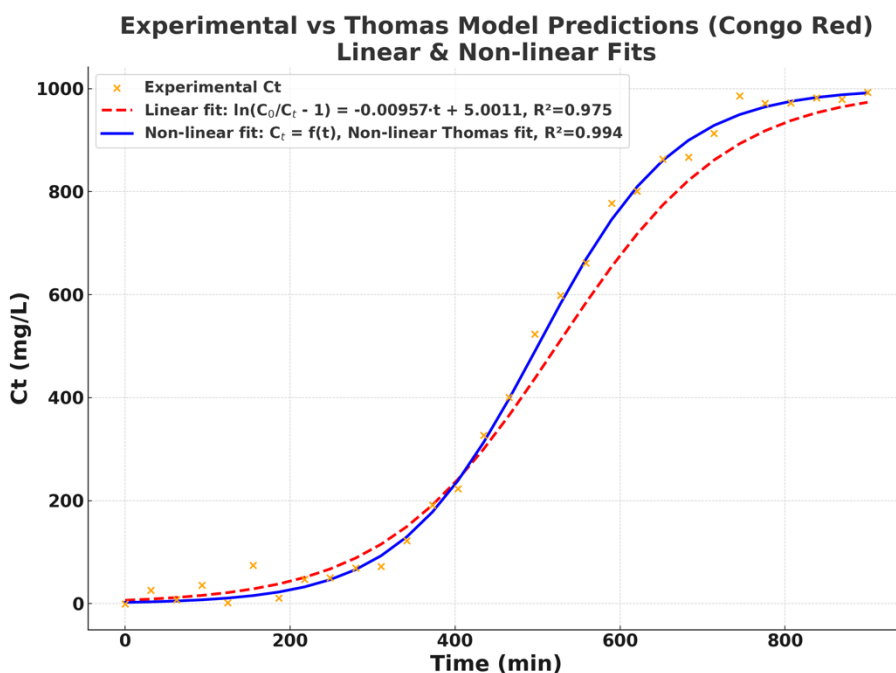


Figure 16. Thomas model with Regression equation for Column studies

By recording the Ce mg/L the final conc. of Column studies Adsorption isotherm was Fitted in to both Freundlich and Laungmuir isotherm figure 14 & 15. This shows the interconnection b/w the quantum of adsorbate adsorbed per unit mass of adsorbent. Langmuir isotherm innumerate monolayer adsorption onto homogeneous surface and freundlich isotherm is for multilayer adsorption on to heterogeneous layer. According to regression Freundlich isotherm shows best fit $R^2 = 0.992$, shows Adsorbent is Multilayer.

The figure 16 shows the experimental Ct values compared with the Thomas model predictions for both linearized and non-linear regression fits for Congo Red adsorption in an FBC. Non-linear regression parameters: $k_{th} = 9.094688e-06$ L/(mg/min), $q_0 = 104.05$ mg/g; R^2 (Ct) = 0.9943. Thomas Model Linear Fit for Congo Red The Thomas model was fitted in its linearized form, using $\log(C_0/C_t - 1)$ vs T, regression yielded following equation and fit parameters: $\ln(C_0/C_t - 1) = (-0.009569) \cdot t + (5.001124)$, $R^2 = 0.9749$, hence the breakthrough results obtained from column studies were validated [40, 41].

5. Conclusion

To achieve the objective of current study the batch studies and column studies were initiated on basis of dynamic experimental conditions such as adsorbent dosage, time, pH & adsorbate conc. the adsorption of dyes is pH dependent and this study showed that the adsorbent adsorbed better in in alkaline medium, 8.5 pH considered as optimum. The both adsorbent dosage & contact time have influence over adsorption efficiency this was effectively observed, Increase in the adsorbent dose increase the percentage removal up-to 3g for 1000ppm. if more adsorbent is added to solution decrease in removal maybe because of overlapping adsorption sites brought on min considered as optimum contact time, Main point in this study was rise in the % removal turns to reduce in Q_e (mg/g) (dye adsorbed on AC) & vise-versa. Column studies shows 90-95% of removal for 1000ppm concentration at flow rate 100 ml/min. best suit for Freundlich isotherm where $R^2 = 0.992$. Breakthrough was achieved at 240 min. Hence parthenium was the ideal and cheaply available adsorbent with high removal capacity. Limitations observed in this study was pH should be maintained with respect to nature of dye to achieve max adsorption and parthenium is allergic weed need proper precautions while extraction and activation, with proper care and balance in pH parthenium weed will be the best low cost and widely available adsorbent for dye removal.

6. Scope for future work

Work may extend to Different Categories of dyes like Basic, Direct, Reactive Etc. using Parthenium weed carbon. Industrial sector can adopt the methodology of

physically activated parthenium carbon for dye removal not only in textile it's applicable to many industries which use dyes in manufacturing activities. Recycle and reuse for gardening & other activities after tertiary treatment makes environment sustainable.

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Authors Contribution Statement

M. Rakesh: Methodology, Writing - Original Draft, Investigation, Formal Analysis, Writing - Review & Editing, B. H. Manjunath: Validation, Supervision, Resources, S. Kishor Kumar: Conceptualization, R. Shruthi: Formal Analysis & Editing. All the authors read and approved the final version of the manuscript.

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Competing Interests

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Has this article screened for similarity?

Yes

Data Availability

The data supporting the findings of this study can be obtained from the corresponding author upon reasonable request.

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