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REMOVAL OF METHYLENE BLUE DYE FROM A SYNTHETIC WASTEWATER USING ACID ACTIVATED PARTHENIUM WEED CARBON-ENVIRONMENTAL SUSTAINABILITY

M. Rakesh[1,✉], B. H. Manjunath[1], S. Kishor Kumar[2], and P. Bhoomika[3]
[1]Department of Civil Engineering, Sri Siddhartha Institute of Technology, Sri Siddhartha Academy of Higher Education (SSAHE), Tumakuru, Karnataka, India
[2]Department of Civil Engineering, Malnad College of Engineering, Hassan, Karnataka, India
[3]Department of Computer Science and Engineering, Sri Siddhartha Institute of Technology, Sri Siddhartha Academy of Higher Education (SSAHE), Tumakuru, Karnataka, India
✉Corresponding author: rakeshm@ssit.edu.in

ABSTRACT
When textile wastewater is discharged into an aquatic environment, persistent dyes pose a significant environmental risk. This reduces photosynthetic activity; some dyes are carcinogenic and cause serious threats to human health. In this study, methylene blue dye was adsorbed from a synthetic wastewater using activated carbon derived from the *parthenium hysterophorus* weed as a bio-adsorbent. Sulfuric acid was used for the activation process; both batch studies and response surface methodology (RSM) were employed. A 3-level, 3-factor box-behnken design was used for the study. Variation in dynamic parameters like adsorbate (100-1500 mg/l) and adsorbent (0.5-4.0 g), along with pH (4.5-11.5) and contact time (30-120 min) to determine the optimum values. This model best fits the Langmuir isotherm (R^2 = 0.99). The adsorption efficiency was found to be between 95% and 99%, with experimental results showing good agreement with predicted values. The regression model was found to be significant, as indicated by the analysis of variance, which showed a high coefficient of determination (R^2 = 0.98). SEM, FTIR, and XRD were recorded, which show good surface morphology. The findings from batch studies help future studies, such as continuous fixed-bed adsorption and real-time monitoring using IoT. The study shows the significance of parthenium in dye removal as a huge bio-adsorbent for practical industrial application because of its large biomass. Hence, the bio-adsorbents are beneficially used to treat industrial wastewater for a sustainable ecology and environment. Utilization of unwanted weeds as a useful product in industrial applications makes them more sustainable.

Keywords: Adsorption, Activated Carbon, Methylene Blue, *Parthenium Hysterophorus*, Response Surface Methodology.

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INTRODUCTION

The textile industry primarily uses different kinds of colours or dyes for different production activities. Due to overpopulation and rapid industrialisation, many industries are directly and indirectly responsible for the pollution of water bodies.[1] The production of paper and pulp, fabric dyeing, leather treatment, printing, cosmetics, and other processes are some common uses for these chemicals.[2] Discharging effluent into waterways impacts their aesthetic appearance and impedes the ability of sunlight to reach the waterways. Some dyes are harmful to human health, and some are dangerous to aquatic life (SDG 14 and 15 life below water and life on land).[4-8]Methylene blue is a basic cationic dye mainly used in the textile sector. Recent studies have indicated that bio-sorbents are the most advantageous because of their high efficiency and low production of chemical and biological sludge (SDG-12, responsible consumption and production).[3] One of the most promising methods for removing dyes is adsorption. Parthenium is an unwanted weed widely available all over the world. Present techniques for eliminating colour from wastewater that are commonly used are electrocoagulation, USB bioreactors, ultrafiltration, advanced oxidation, bioremediation, Fenton's oxidation, and adsorption.[9] Some other technologies applied in the field of dye removal were photocatalytic, ozonation, membrane separation, flocculation, precipitation, etc.

Recently, several economic adsorbents are used for the removal of dyes: coconut shell, rice husk, polymer, sugarcane baggasse, tea residue, natural leaf, waste ash, orange peel, and nano-composite materials, along with these natural materials like natural clay, water hyacinth, coconut coir pith, Delonix regia seed, sewage sludge, banana peel, sawdust, agro waste, Fe(III)–montmorillonite, fruit waste, sludge, areca, waste brick, red mud clay, etc. The above adsorbents were used to remove natural and synthetic dyes. Parthenium weed is a novel adsorbent with a wide scope.[10] nowadays; the main aim is the removal of noxious weeds and indirectly limiting their environmental expansion to a certain degree. Because customers and dye manufacturers are concerned about the stability and fastness of colours, they are creating dyes that are more challenging to break down after usage.[11] During the dyeing process, an estimated 10–15% of dyes were lost and expelled with the effluent.[12-15] Large-scale biomaterials might potentially be employed as inexpensive adsorbents, as they are abundant, readily available, and ecologically benign resources.[16-20] Activated carbon is now widely utilised in various industries in the form of a sorbent agent to remove dyes (SDG 9 industry, innovation and infrastructure).A research gap was identified by a literature study. It was observed that many researchers have worked on low-cost adsorbents, but they are feasible only in lab-scale studies and are not the best fit for industrial scale. Novelty in current research is introducing new perspectives in the field of bio-adsorption by opting for Parthenium, a widely available unwanted weed. In this study, analyses were carried out to find optimum results by varying dynamic parameters that influence adsorption using batch studies. This work directly supports Sustainable Development Goals-6, 9, 12, 14, and 15. The removal of harmful dyes from wastewater before discharge into aquatic systems promotes affordable and sustainable water treatment by using *parthenium hysterophorus,* a widely available weed, as a low-cost bio-adsorbent. This species exhibits rapid growth, typically flowering within 4–6 weeks after germination and producing up to 25,000 seeds per plant, contributing to its aggressive spread. Morphologically, it is characterised by an erect stem, a deeply penetrating taproot system, pale green dissected leaves, and small white capitulum flowers borne at the terminal ends. Under favourable conditions, it can attain a height of up to 2 meters. The principal toxin and allelochemical parthenin are thought to be responsible for most of the plant's harmful effects on people, animals, and the environment. Sesquiterpene lactone and phenolic acids are the most well-known and prevalent toxic allelochemicals released by the very invasive plant *parthenium hysterophorus* and join water bodies, which cause toxicity and eutrofication. (SDG-6: Clean Water and Sanitation), this study explores how nature's nuisance can become nature's cleaner.

EXPERIMENTAL

Adsorbate

Methylene blue (MB) is a cationic and basic dye. The compound is a heterocyclic aromatic compound with a molecular formula of C16H18N3SCl (Fig.-1), a molecular weight of 319.85 g/mol, and a maximum absorption wavelength of 665 nm. German scientist Heinrich Caro created MB for the first time in 1876 as a textile dye made from aniline. Extensively utilized for dyeing processes in the paper, leather, and textile industries (SDG 9, Industry, and Innovation and Infrastructure).

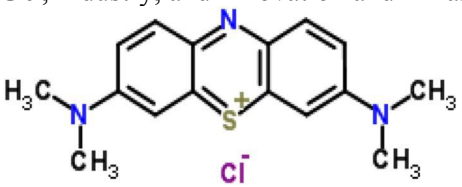

Fig.-1: Structure of Methylene Blue Dye

A calculated weighed powdered MB dye was dissolved in distilled water. Using the connection N1V1=N2V2, a series of dilutions produced an experimental solution with the required concentration.[21]

Adsorbent

To get rid of the dirt and soil, a healthy, complete parthenium weed was gathered and cleaned. After being sun-dried for 70 hours, converted into a powder and sieved through different micron sieves shown in Fig.-2(b). After adding concentrated sulfuric acid to the powder parthenium 1:1.6 (W/V), washed till it reached pH 6, and then dried for 12 hours at 120°C in an Oven. After repeatedly washing the carbonized

material to get rid of any free acids, it was overnight-soaked with 1.5% sodium bicarbonate solution to get rid of any remaining acidic content. The same material was cleaned and dried for 20 hours at 103 degrees Celsius in a hot oven and used as adsorbent (Chemically activated carbon) CAC, shown in Fig.-2(c). Size of pore = 0.3–1.0 mm, acid solubilitiness (%) = 4.0, Bulk density = 0.56 g/ml, water=2.86, moisture content (%) = 0.15. Chemical testing verified the existence of phenol and carboxylic groups. pH = 7.2, O (%) by difference = 44.58, C (%) = 49.40, N (%) = 2.31, H (%) = 3.71, and were the general characteristics for future usage. The substance was stored in sealed plastic containers.[22]

Fig.-2: Shows (a) Vat Dye Prepared for Analysis, (b) Raw Parthenium Powder, (c) Activated Powder, (d) Agitation

Activated Carbon Treated with Sulfuric Acid

A batch adsorption study was carried out in a conically shaped flask of 250 or 300 ml, taking a 50 ml working solution from a prepared stock solution (2a). Cling film was used to cover the flask of Parthenium that was applied in accordance with the design. The mixture was shaken at 26°C and 150 revolutions per minute (Fig.-2(d)). After that, it was filtered and examined at a wavelength of 665 nm in a spectrophotometer. In order to determine the impact of the adsorbent, batch tests were conducted by adjusting the dose of AC from 0.5g to 4.0g at various levels while maintaining time and pH. Variations in sample concentration and optical density, as well as experimental conditions, were also noted. Additionally, the effect of CT was examined by altering the duration from 30 to 120 minutes. The effect of pH was examined while maintaining the adsorbent and period constant by adjusting the pH from 4.5 to 11.5 using 0.02 N HCl and 0.02 N NaOH. Additionally, the impact of the adsorbate was investigated by varying its concentration while maintaining the same levels of other components using a UV spectrophotometer at a wavelength of 665 nm. The experiment was further carried out according to the box-behnken design. RSM is an effective statistical tool in Minitab and a mathematical method that is useful for building optimization procedures. It provides benefits over other approaches that are commonly used for handling large variables. In addition, it's a really helpful, economical, and practical instrument.[23] Using a formula, removal efficiency was determined.

$$\text{Adsorption \%} = (C0-Ce) / C0*100, \qquad Qe = (C0-Ce)\, V/m$$

This formula uses V to represent the volume of the solution in liters and W to represent the weight of the dried adsorbent in grams (g). For Co and Ce, the dye's initial and equilibrium time solution concentrations were given in milligrams per liter, respectively. In this work, a spectrophotometer was used to evaluate the optical density and concentration of materials after filtration at a wavelength of 665 nm. A typical graph for different MB concentrations is shown, with a coefficient of correlation (R^2) of 0.973.

Design of Experiment

According to the runs given by the box–behnken design, 3 trials were conducted to determine the percentage removal of dye by an adsorbent. The values are analyzed to check the correlation between the experimental and predicted adsorption, which, as mentioned in Table-2, experimental values we get after experimenting in the laboratory and the predicted values we got from the quadratic equation from minitab. The obtained points of both the data were plotted, which shows they are very close to a straight line. This shows excellent correlation between the response and predicted values, and the results show the quadratic model is ideal and significant.

Table-1: Design of Runs at Different Ranges for Each Factor

Runs	blocks	Adsorbate conc. (mg/l)	Absorbent dose (mg)	pH	Contact time (min)	Agitation (RPM)
1	1	100	0.3	9.5	30	150
2	1	1500	0.3	9.5	60	150
3	1	100	2	9.5	90	150
4	1	1500	2	9.5	120	150
5	1	100	1.15	7.0	30	150
6	1	1500	1.15	7.0	60	150
7	1	100	1.15	12	90	150
8	1	1500	1.15	12	120	150
9	1	800	0.3	7.0	30	150
10	1	800	2	7.0	60	150
11	1	800	0.3	12	90	150
12	1	800	2	12	120	150
13	1	800	1.15	9.5	30	150

A single centre point is used since the box-behnken design is a rotatable design that requires three levels of each factor—low, medium, and high. Minitab was used for BBD with three independent variables, and BBD under RSM was employed to maximize dye adsorption. 13 of the specified runs were carried out as shown in (Table-1). RSM is a strong statistical tool for optimization in Minitab. It is a mathematical approach that can be used to construct optimization processes and has an advantage over other methods that are typically employed when dealing with large variables. It is also a very practical, cost-effective, and helpful tool.

RESULTS AND DISCUSSION

Effect of Initial Dye Concentration

It was examined by varying the dye's concentration between 100 and 1500 parts per million. The experiments were carried out in a test sample with a constant adsorbent dose of 1 g/50 ml, a pH of 8, and a contact period of 60 minutes. As the dye concentration rises, it was shown that the percentage clearance reduces (Fig.-3) and that MB's equilibrium adsorption capacity, Qe (mg/g), on carbon rises. Qe rises as a result of greater mass transfer driving force and higher dye loading onto the adsorbent, but % removal falls as a result of saturation of constrained adsorption sites at higher concentrations. Increased concentration encourages the diffusion of dye molecules onto the adsorbent and permits more dye to accumulate per gram of carbon by generating a greater driving force for mass transfer (i.e., a larger concentration gradient between the bulk solution and adsorbent surface).[24]

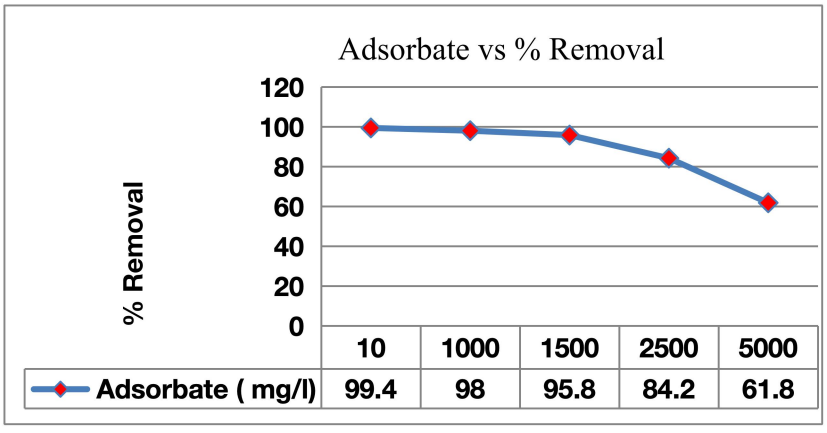

Fig.-3: Impact of Initial Dye Concentration on Methylene Blue Adsorption onto Partenium Carbon (Adsorbent Dose = 1g/50ml; Contact Time = 1hr; Ph=8)

Effect of Adsorbent

By adjusting the dose of the adsorbent between 0.5g and 4.0g, its effects were investigated. The trials were conducted with a pH of 8, concentrations of 100 and 1500 ppm, and a contact period of 60 minutes. The percentage removal climbs and exceeds 95% as the adsorbent dosage increases (Fig.-4).

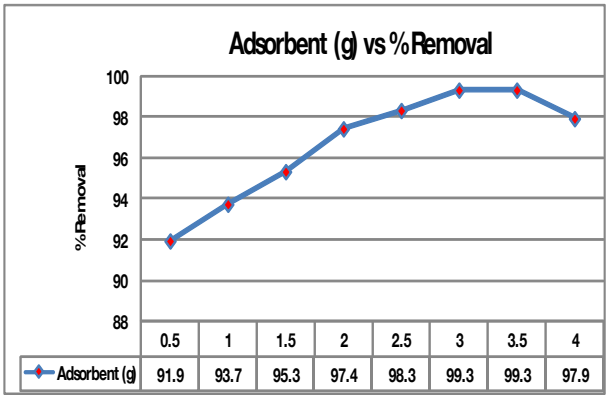

Fig.-4: Effect of Adsorbent Mass on the Adsorption of Methylene Blue onto Parthenium Carbon (dye concentration = 800 and 1500 ppm; contact time=1hr; ph=8)

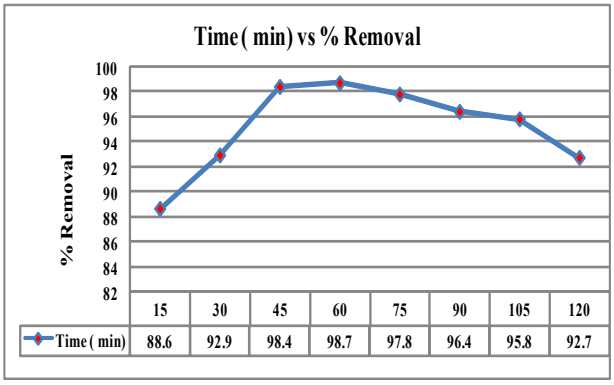

Fig.-5: Contact Time's Impact on Adsorption of Methylene Blue onto Parthenium Carbon (dye concentration = 100 and 1500 ppm; adsorbent mass=1g/50ml; pH=8)

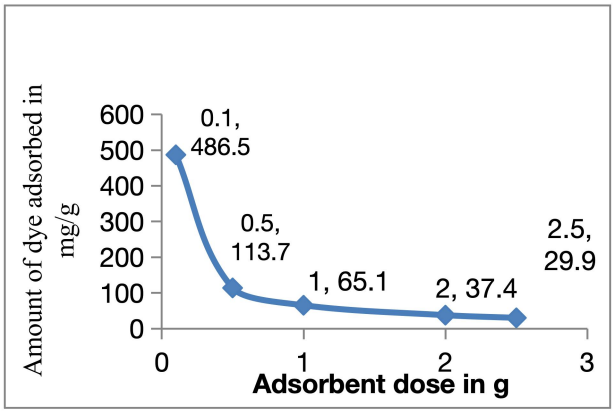

Fig.-6: Demonstrates a Reduction in the Qe quantity of MB Dye Adsorbed on Adsorbent (mg/g) as Adsorbent Mass Rises (dye concentration = 1500 ppm; contact duration = 1 hour; pH = 8)

Milligrams per gram were used to indicate the mass of MB dye that settled on the adsorbent Qe droplets. The same amount of dye covers a larger mass of adsorbent. Many unsaturated or unused adsorption sites remain. The effective surface area available for adsorption may also be decreased by particle aggregation or adsorption site overlap. The quantity of dye adsorbed per gram of adsorbent (Qe) decreases (Fig.-6) even while the total amount of dye recovered from solution increases.[25] FTIR (Fourier Transform Infrared

Spectroscopy) Spectra of the Parthenium Carbon was observed in Adsorption to know the Functional Group and also know stretching and bending based on position of the peak, number of peak, Width of the peak it was observed the presence of O-H, C-O and C=O FTIR (Fourier Transform Infrared Spectroscopy), the functional groups C-H, O–H, C–O, and C=O give characteristic absorption bands due to vibration transitions. Indicating the presence of carbonyl groups or aromatic structures, the absorption band associated with C–O stretching vibrations, which are characteristic of alcohols, esters, or ethers. Additionally, the peak assigned to C–O or C–N stretching vibrations suggests the presence of alcohols, amines, or polysaccharide structures, as shown in (Fig.-8).

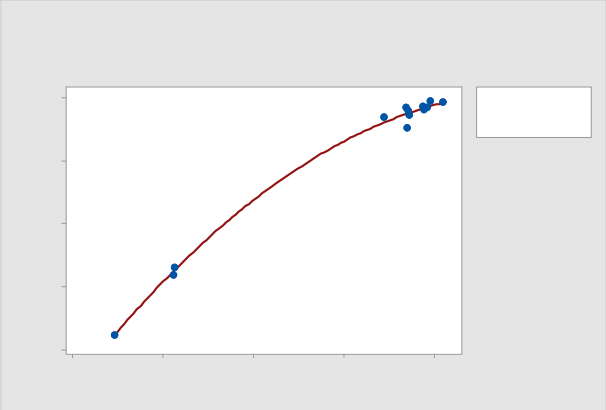

Fig.-7: Response vs Predicted Values

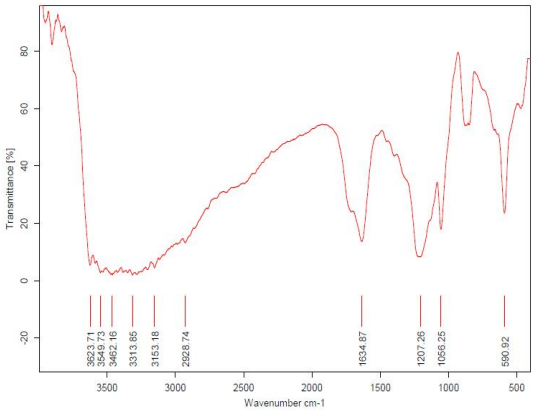

Fig.-8: FTIR Analysis of Activated Carbon

Effect of Time and pH

The proportion of removal rises with the length of interaction over the first 60 minutes (Fig.-5). Since there are more active sites and the process proceeds more slowly, 60 minutes is considered the optimal contact period for dye removal. Beyond this, the rate is controlled by diffusion resistance and reduced concentration gradient, so no significant increase in uptake occurs, consistent with adsorption kinetics. The pH of the medium affects how strong the electrostatic charges are. A higher pH results in a higher clearance percentage since MB is a basic dye. When a basic dye dissolves in water, a positive charge is generated; the adsorbent's positive charge surface frequently inhibits adsorption in the acidic area. The pH range of 6 to 12 is used for the experimental inquiry.[26] Therefore; it is believed that a pH of 8 is optimal for further investigation. At low pH, excess H^+ protonates the surface, producing a positive charge and intense competition with MB^+, which prevents adsorption; at high pH, the adsorbent surface becomes more negatively charged (deprotonation of surface functional groups), increasing electrostatic attraction toward the cationic dye.[27] According to the design, 13 runs were carried out for three factors at different ranges (Table-1). Experimental values, which are tabulated by the conducting the runs, and predicted values obtained by the software. There is not much difference between them, so the model is significant and good.

Table-2: Outcomes of the Box-Behnken Design Experiment

Runs	Adsorbate Conc (ppm)	Adsorbent Dose (g)	pH	Predicted values	Experimental values removal in %
1	100	0.3	9.5	98.563	98.6
2	1500	0.3	9.5	82.313	81.1
3	100	2	9.5	97.18	98.4
4	1500	2	9.5	99.738	99.7
5	100	1.15	7.0	99.387	99.0
6	1500	1.15	7.0	85.63	86.5
7	100	1.15	12	98.462	97.6
8	1500	1.15	12	98.51	98.9
9	800	0.3	7.0	85.55	85.9
10	800	2	7.0	100.4	99.6
11	800	0.3	12	98.37	99.2
12	800	2	12	99.55	99.2
13	800	1.15	9.5	99.30	99.3

It indicates that adsorbent and pH were very important factors; an increase in the adsorbent and pH tends to increase the % removal. The fitted line plot represents the interrelation between response and predicted values (Fig.-7). Because the values on the graph are so close to one another, the design is satisfactory. At low levels, removal efficacy increases with adsorbent dose and pH but decreases with increasing initial dye concentration. The optimal settings were determined to be around pH 8–9.5, moderate-to-high adsorbent dosage (≥1.15 g), and 60 min contact period in order to obtain removal efficiencies of up to ~99–100%. The model's predictive power is demonstrated by the high degree of agreement between predicted and experimental results (Fig. 7). The availability of surface sites, electrostatic interactions, and equilibrium constraints controls the adsorption process, indicating that the adsorbent is a useful option for successfully removing cationic dyes from aqueous solutions.

Regression Equation in Uncoded Units

Response = 67.2 -0.02401 Adsorbate + 20.68 Adsorbent + 4.93 ph – 0.000005 Adsorbate*Adsorbate – 3.03 Adsorbent * Adsorbent – 0.182 pH.*pH + 0.00790 Adsorbate*adsorbent + 0.001971 Adsorbate*pH – 1.612 adsorbent*pH.

The quadratic regression equation, which incorporates the linear, interaction, and curvature effects of pH (C), adsorbent dose (B), and adsorbate concentration (A) on removal efficiency, represents a second-order response surface model. An equation that fitted the response function and described the statistical link between the predictor factors and the response variable was produced by the regression analysis. Overall, the model demonstrates how electrostatic interactions, saturation behavior, and surface site availability all cooperate to regulate adsorption, which is consistent with the response surface approach and adsorption theory ideas like the Langmuir isotherm. S = 1.45344, R-sq 98.74%, R-sq(adj) 94.96, R-sq (pred) 85.91%. S is a metric for how well the answer is described by the model. This model's S value is 1.45344; the lower the S value, the better. R-sq, which shows the reaction's variability. Strong agreement exists between the expected R-squared of 85.91% and the revised R-squared of 94.96%. The closer the value R is to 1, the greater the correlation between the experimental and predicted values; in this case, 98.74 (0.987) is closer to 1 (Fig.-7), indicating that the model is adequate. Strong model robustness and excellent external predictive ability are indicated by the relatively good agreement between the predicted R^2 (85.91%) and the adjusted R^2 (difference <10%). This closeness implies that the model could correctly forecast responses for new experimental scenarios within the studied range.

Table-3: Analysis of Variance (ANOVA) For Response Surface Quadratic Model

Source	DF	Adj SS	Adj MS	F-Value	P-Value	Significance
Model	9	496.519	55.169	26.12	0.011	Significant
Linear	3	294.048	98.016	46.40	0.005	Significant
Adsorbate	1	93.845	93.845	44.42	0.007	Significant

adsorbent	1	128.801	128.801	60.97	0.004	Significant
ph	1	71.401	71.401	33.80	0.010	Significant
Square	3	19.579	6.526	3.09	0.189	
Adsorbate*Adsorbate	1	16.203	16.203	7.67	0.070	
adsorbent*adsorbent	1	10.937	10.937	5.18	0.107	
ph*ph	1	2.957	2.957	1.40	0.322	
2-Way Interaction	3	182.892	60.964	28.86	0.010	Significant
Adsorbate*adsorbent	1	88.360	88.360	41.83	0.008	Significant
Adsorbate*ph	1	47.610	47.610	22.54	0.018	Significant
adsorbent*ph	1	46.922	46.922	22.21	0.018	Significant
Error	3	6.338	2.113			
Total	12	502.857				

ANOVA is a statistical technique that is used for one or more factors by comparing the response variable means at different factor levels. If the F is higher and the P is lower, then it is more important. If P is lower than 0.05, then the model is statistically significant (Table-3). Where P is the probability of getting a result, the F value is the residual variance in a model. There is only 0.01% chance that the F value will show higher. In summary, high F + very low p-value → strong model significance, confirming that the regression captures true system behavior rather than random variation.

The form of the carbon was examined via SEM (Scanned Electronic microscopy) examination.[28-30] the porosity in the bio-sorbent structure can be connected to the relation between the adsorbent and the adsorbate. Both dye-loaded and unloaded carbon was subjected to SEM examination, showing good Surface morphology favorable for adsorption (Fig.-9).XRD (X-ray diffraction) shows carbon is Amorphous in nature (Fig.-10).

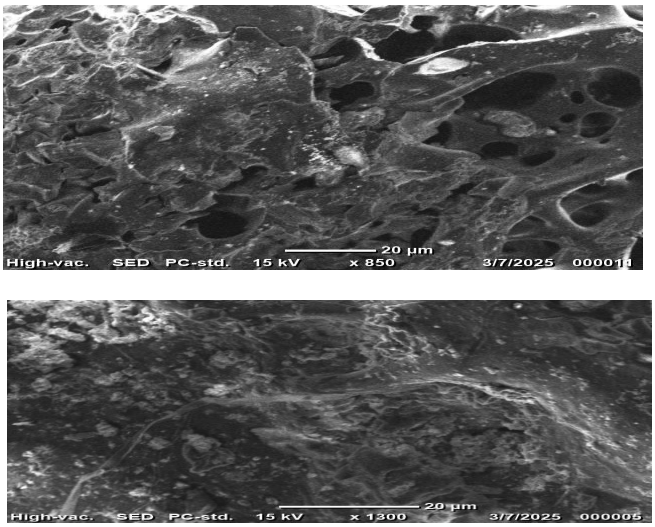

Fig.-9: SEM after and before Adsorption

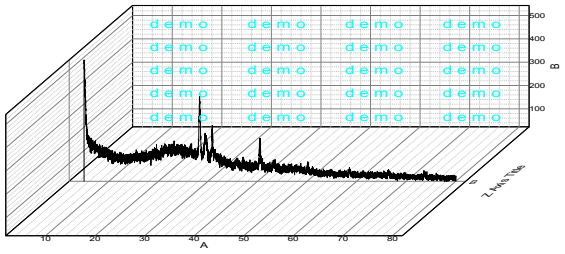

Fig.-10: XRD of Activated Carbon

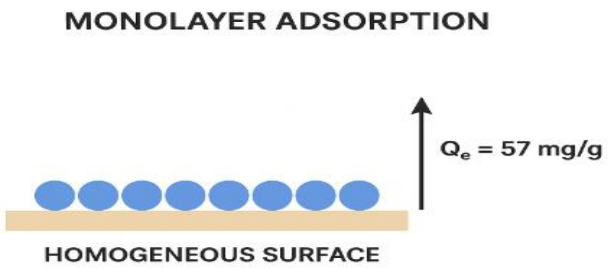

Fig.-11: Langmuir Isotherm Model of Adsorbent Surface

Table-4: Results Show Adsorption Isotherm Models

Parameter (Langmuir)	Value	Parameter (Freundlich)	Value
Slope (1/Qmax)	0.017618	Slope (1/n)	0.082410
Intercept (1/(Qmax·KL	0.086415	Intercept (ln Kf)	3.551200
Qmax (mg/g)	57.012	N	12.134
KL (L/mg)	0.2039	Kf	34.855
Linear fit slope	0.017618	Linear fit slope	0.082414
Linear fit intercept	0.086415	Linear fit intercept	3.551156
R^2 (Langmuir)	0.99794	R^2 (Freundlich)	0.94678

The (Fig.-11) describes the monolayer adsorption, where the dye molecules in blue shows monolayer on the surface of the adsorbent, here the surface is homogeneous where uniform, single layer deposit on the surface and all have equal energy and affinity, each void can occupy one dye molecule after all voids are filled no more adsorption takes place, this theory described by langmuir isotherm R^2= 0.99794 (Table-4), removal of dye and dye uptake efficiency, enabling effective removal of pollutants from wastewater, supporting SDG-6, Optimizing these parameters using RSM reduces chemical and material wastage, aligning with SDG-12.

CONCLUSION

With the high removals of 95-99%, the study demonstrates that acid-activated *parthenium hysterophorus* carbon is an effective, low-cost, and sustainable bio-adsorbent for the removal of methylene blue dye from wastewater. Response surface-based process parameter optimization ensures efficient resource utilization while maintaining high efficiency. The adsorption process follows the Langmuir isotherm, indicating monolayer adsorption on a homogenous surface. Among the various factors, pH, adsorbent dose, and contact time significantly affect the removal efficacy. Most importantly, the employment of an invasive weed as a commodity with value addition will solve the environmental problems caused by its proliferation and promote a turn of waste into wealth. With the enhancement of water purification and a decrease in the pollution by dangerous dyes, our approach strongly supports SDG-6. Further, by recommending scalable and innovative wastewater treatment approaches, it also supports SDG-9 and 12 through sustainable material usage and responsible consumption. Moreover, the study also supports more general environmental goals, including SDG-13 (climate action), SDG-14 (life below water), and SDG-15 (life on land) by promoting ecological conservation and sustainable environmental management. Scope for future work: The results of batch studies can be optimized, and continuous fixed-bed column studies can be conducted to determine the flow rate, adsorption capacity, breakthrough, and saturation of carbon. Furthermore, real-time data in the industrial application can be checked by IoT (Internet of Things) and image processing.

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CONFLICT OF INTERESTS

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation.* All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

M. Rakesh https://orcid.org/0009-0008-0477-4384
B. H. Manjunath https://orcid.org/0009-0005-0450-1640
S. Kishor Kumar https://orcid.org/0000-0002-6801-149X
P. Bhoomika https://orcid.org/0009-0005-0461-6050

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