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Application of Taguchi Methodology in adsorption of Malachite Green dye using chemically enhanced *Bambusa Tulda* (Indian Timber Bamboo)

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ABSTRACT

The capacity of *Bambusa Tulda* (BT) with chemical improvements to remove Malachite Green (MG) from water bodies is the main topic of this research. These improvements were achieved by employing an idealised Taguchi L16 orthogonal array design. The pH, starting dye concentration, bioadsorbent dose, and contact duration were all elements considered during the treatment process. Each one was found to have an impact. According to the findings, pH, preliminary dye concentration, contact duration, and bio-adsorbent dose in that order were the major elements in the elimination of MG dye. The optimal specific nitrification rate (SNR) conditions were determined to be pH-3, an initial dye concentration of 100 mg/L, a bio-adsorbent dose of 0.20 g/100 mL, and a contact time of 90 minutes. Contact time and bio-adsorbent dose were shown to have an effect on the results, while pH was the most relevant factor overall. This was confirmed using analysis of variance (ANOVA). Adsorption occurred in a monolayer in the Langmuir isotherm model. The chemical may include hydroxyl and carboxyl groups, according to the Fourier transform infrared spectroscopy (FTIR) data. Scanning electron microscopy was used to examine the exterior morphology of the *Bambusa Tulda*. Malachite Green dye may be removed from wastewater using a bio-adsorbent made from chemically enhanced *Bambusa Tulda*.

1. Introduction

With increased urbanisation and the industrialisation of textile and paint industries, there is a greater usage of dye in everyday life. This has increased the dye waste in the effluent water from such industries [1]. As one of the world's biggest environmental challenges, water pollution must be addressed. Toxic compounds and a wide range of various dye chemicals are found in wastewater from the textiles industry, as well as from paper and plastics production. These pollutants may harm humans and fish populations, as well as the microorganisms that live there. The

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use of dyes for its goods necessitates a significant amount of water use. Therefore, they release a significant quantity of dye effluent [2]. The first indicator of contamination in wastewater is its color. Because dyes are ubiquitous in the natural world, they efficiently absorb sunlight, decreasing photosynthesis and dissolving oxygen levels in bodies of water. As a result, hydrophytes and phytoplankton absorb less light, causing COD concentrations to rise. Therefore, it is important for ecology to remove these contaminants from water used for aquatic purposes [3]. Dye effluents include toxic and carcinogenic compounds in vast quantities that are cumbersome to manage and dispose of. Dyes are particularly challenging to remove from effluent because their refractory organic molecules are resistant to aerobic digestion, light, heat, and oxidizing agents [4]. Every year, manufacturers worldwide produce more than 7 x 10^5 dyes, with over 100,000 available for commercial use. Effluent color molecules have been removed via a variety of processes, including adsorption, coagulation, flocculation, biodegradation, and others. However, the efficacy, expense, and ecological impact of various alternatives vary widely [5-6]. Biological, chemical, and physical are the three primary types of color removal techniques. There are good and bad aspects to each subgroup. Due to their high cost and the inefficient waste management associated with large-scale removal procedures, they are seldom employed in the industry. Oxygen may alter its visual appearance via a chemical event known as oxidation [7]. The oxidation process is controlled by the concentration of oxygen in the air and the chemical nature of the material it comes into contact with. Real oxidation occurs only at the molecular level. A membrane is a thin layer of semi-permeable material that works as a barrier to separate things when a driving force is applied across its surface [8]. When disinfectants react with water, disinfection by-products are produced along with the intended bacteria and natural organic material. Multiple advancements have reduced the production and operating costs of membranes. Processes such as reverse osmosis (RO), nanofiltration (NF), and microfiltration (MF) are discussed. Industrial dye effluent is a significant source of wastewater. Water-soluble synthetic dyes degrade at a slower pace and contain a more persistent aromatic component than natural colours. However, the expense of such processes is significant, and the elimination rate is low. Membrane filtration is a technique that may continually clear, concentrate, and extract color from wastewater. Special properties like its resistance to heat, chemicals, and microbes set it apart from competing approaches. If the effluent includes a low concentration of colours, this filtering technology may be used for water recycling inside a textile dye facility; however, since it cannot minimize the dissolved solid content, water reuse is a challenging undertaking. Dye-laden water has also been treated well using membrane separation technologies, including reverse osmosis (RO), nanofiltration (NF), and microfiltration (MF) (greater than or approximately 90% removal efficiency). Pore clogging and membrane fouling are still persistent problems in these processes. Ion exchange, the removal of soluble dyes, is only one of the many benefits of this approach, along with the fact that no adsorbent is wasted during the regeneration process. The high price tag is a key drawback of this approach. The ion exchange process is not particularly efficient for dispersing pigments, and organic solvents are costly. The dye in the solute has a static charge, which affects the optimal coagulant concentration, and removing the sludge created after coagulation is challenging. The high cost of disposal is directly related to the volume of sludge produced. Compared with other methods, adsorption is a novel way of restoration with little operational sludge process. Adsorption beats all other pigment management approaches simply because it offers superior performance, lower operating costs, less toxicity, and larger surface area; in addition, it is easier to maintain and utilise. It is also simple to isolate, making it ideal for wastewater treatment [9-13]. Adsorption, in chemistry, is the process by which molecules of a gas, liquid, or dissolved solid attach to a surface. A wide range of conditions are suitable for adsorption [14]. An adsorbate film is created as the adsorbate molecules or atoms are deposited onto the adsorbent's surface. Using bio-adsorbents allows the adsorption process to be both cost-effective and environmentally friendly. Adsorption is one of the treatment strategies that may be used to get rid of different components of the color. Because the whole dye molecule is removed by adsorption, there are no leftover dye fragments in the wastewater [15]. Activated carbon is the most generally used adsorbent for dye removal from effluent, but activated carbon is expensive, and this limits its application on a large industrial scale. Despite its prolific use in industries, activated carbon remains an expensive material; therefore, cost-effective materials are needed [16-19]. However, activated carbon's universality as an adsorbent for a wide spectrum of pollutants in water might also restrict its wider use due to its high cost. In an attempt to reduce the cost of adsorption, low-cost adsorbents have been created. These may be broken down into two broad categories: 1. natural materials like wood, peat, and coal and 2. industrial, agricultural, residential wastes or by-products like slag and sludge. Anything can be made from either inorganic or organic components [20-23]. In recent publications, researchers have used a wide variety of adsorbents: barley straw, used tea leaves, used sago, peanut hulls, hazelnut shells, sawdust, neem bark, ash from thermally treated rice chips, used banana peels, seaweed, rice husk, rice husk ash (RHA), used orange peels, used cocoa shells, used tree fern, coffee residue, palm kernel fiber, seed shell, olive stone waste, grape stalk, bagasse, fly ash, wheat bran, carrot juice pulp, carrot juice, carrot cabbage pulp. Using plant wastes to treat wastewater has several advantages, including ease of use, high adsorption capacity, low cost, high selectivity for heavy metal ions, and straightforward regeneration [24-42]. However, because the soluble organic components of the plant materials are released during the adsorption process, using untreated plant waste as an adsorbent may result in a variety of problems. The effectiveness of chlorine disinfection might be enhanced by treating plant wastes in advance. Pre-treatment techniques may include the application of adaptation agents such as sodium hydroxide, calcium hydroxide, sulfuric acid, thioglycolic acid, hydrochloric acid, tartaric acid, formaldehyde, epichlorohydrin, etc. [43-44]. It was determined that bamboo biochar had a very high adsorption ability for the removal of the metal complex dye acid black 172. In the adsorption process for dye removal, the temperature was shown to be the most relevant factor, with a relative relevance of 29%, followed by time, ionic strength, pH, and dye concentration. High-surface-area activated carbons were obtained by thermal activation with phosphoric acid of waste bamboo scaffolding. The adsorption capacity of the produced carbons was evaluated using Acid Yellow 117 (AY117) and Acid Blue 25 (AB25) dyes. While AB 25 and other smaller-molecule dyes were quickly absorbed by commercially available carbon, larger-molecule dyes like AY117 showed almost no adsorption on the produced carbon [45-47]. Bio-material adsorbent is essential to reduce the impact of dye pollution in water systems, and it should be locally available in abundance. Bamboo (*Bambusa Tulda*, a kind of bamboo plant) has been employed as a bio-adsorbent for the removal of Malachite Green (MG) dye from water environments as part of ongoing research into the use of low-cost material as an adsorbent for the removal of pollutants from wastewater. It is highly robust and eco-friendly and contains anti-bacterial qualities; the key elements of *Bambusa Tulda are* cellulose, hemicellulose, lignin, etc. The adsorption capability of *Bambusa Tulda* was enhanced by simple and inexpensive chemical changes. The primary goal of this research was to develop a strategy for reusing *Bambusa Tulda* waste biomaterial in wastewater treatment by evaluating the performance capacity of the modified *Bambusa Tulda* via adsorption technology and the Taguchi technique.

2. Materials and methods

2.1. Modification and characterization of Bio-adsorbent

The bamboo samples were locally purchased and made into chips and sieved (505 microns); afterward, they were washed with distilled water to remove foreign particles and then dried at 60 °C for 24 hours. The washed bamboo chips were treated with a 0.1 M Na_2CO_3 solution at room temperature for around four hours. The excess Na_2CO_3 was removed by washing the bamboo chips numerous times with distilled water; then, the pH was reduced to between 7.0 to 7.5, and the material was again dried at 60 °C for 24 hours. The Na_2CO_3 treated bamboo chips were named NCBT.

2.2. Preparation of adsorbate

MG dye is widely used as a coloring dye in the textile, leather, and pharmaceutical industries. It is also used as a coloring agent in a variety of foods and food additives. An overview of MG dye is shown in Table 1.

For experimental purposes, a standard stock solution was prepared at a concentration of 1000 mg/L. Subsequently, the required amount of dye (Merck, India) concentration was prepared per the requirements by diluting it with double distilled water. MG is a complex and widely used industrial dye [14]. MG dye is a well-known micropollutant common in industrial wastewater, making green plants vulnerable. It is a non-biodegradable chemical that lingers in the environment and enters the food chain. It can cause genotoxic, mutagenic, and carcinogenic effects on biological organisms. Presently, global societies are pushing for the use of effective and innovative wastewater treatment technology that does not harm the environment [48].

2.3. Taguchi methodology

The key purpose behind using Taguchi methodology is the orthogonal array and signal-to-noise ratio (SNR). The orthogonal array consists of factors with different levels forming a different combination of experiments [49-54]. After the preliminary feasibility study, it was found that pH, Initial MG Concentration (mg/L), NCBT Dose (g/100 mL), and Contact Time (mins) were the most important factors, represented by A, B, C, and D, respectively. All the factors were tested in four situations (Level 1, 2, 3, and 4) and are shown in Table 2. Using the information in Table 2, an orthogonal array of 16 experiments was generated, as shown in Table 3.

Table 1. Overview of MG dye.

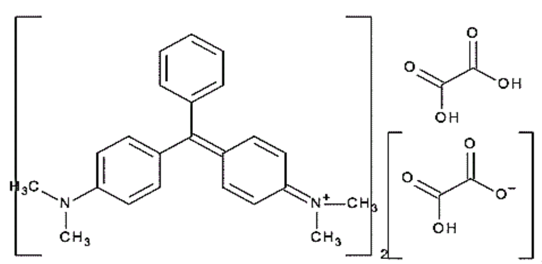
Absorption maximum λ max (water)	616 - 620 nm
Bulk density	400 - 500 kg/m^3
CAS number	2437-29-8
Chemical formula	$C_{46}H_{50}N_4$ * 2 C_2HO_4 * $C_2H_2O_4$
Hill Formula	$C_{52}H_{54}N_4O_{12}$
Melting Point	159 °C
Molar Mass	927.01 g/mol
pH value	2.4 (10 g/l, H_2O, 24 °C)
Solubility	110 g/l
Structure formula Image	

Table 2. Factors and levels for Design of Experiment (Taguchi Methodology).

Factor	Name	Level Value			
		1	2	3	4
A	pH	3	5	7	9
B	Initial Dye Concentration (mg/L)	50	100	150	150
C	Bio-adsorbent dose (g/100 ml)	0.20	0.30	0.40	0.50
D	Time (mins)	30	60	90	120

Table 3. L16 orthogonal array.

Exp. No.	A	B	C	D	pH	Initial MG Concentration in mg/L	NCBT dose in g/100 mL	Time (mins)
1	1	1	1	1	3	50	0.2	30
2	1	2	2	2	3	100	0.3	60
3	1	3	3	3	3	150	0.4	90
4	1	4	4	4	3	200	0.5	120
5	2	1	2	3	5	50	0.3	90
6	2	2	1	4	5	100	0.2	120
7	2	3	4	1	5	150	0.5	30
8	2	4	3	2	5	200	0.4	60
9	3	1	3	4	7	50	0.4	120
10	3	2	4	3	7	100	0.5	90
11	3	3	1	2	7	150	0.2	60
12	3	4	2	1	7	200	0.3	30
13	4	1	4	2	9	50	0.5	60
14	4	2	3	1	9	100	0.4	30
15	4	3	2	4	9	150	0.3	120
16	4	4	1	3	9	200	0.2	90

As per Taguchi's methodology, the potential parameters are considered in four different categories (pH, Initial MG Concentration in mg/L, NCBT dose in g/100 mL and Time in mins): largely considered as improved, whereas nominal is considered as most excellent (1), nominal is best (2), and small is improved. The objective of the research was to remove MG dye from wastewater using a NCBT bio-adsorbent. The superiority feature selected was large and better for MG Dye removal, and it is defined in Equation 1 [55-56]. The SNR with higher superiority features is given in Equation 2.

$$\text{Removal (\%)} = \frac{(C_o - C_e)}{C_o} x100 \quad (1)$$

$$\text{SNR} = -10 \log \frac{\sum_{i=0}^{n} \frac{1}{y_i^2}}{n} \quad (2)$$

Batch experiments were performed in a 250 mL Erlenmeyer flask at 150 rpm (agitation speed) at 30°C in a temperature-controlled orbital shaker. 0.1 M-HCl and 0.1 M-NaOH solutions were utilized in the experiment to maintain the pH level. Before measuring the equilibrium concentration of the MG Dye, the adsorbent was allowed to settle for a while and then measured by a UV- VIS spectrophotometer.

2.4. Characterization study

The functional group active in the adsorbent for dye removal was identified using FTIR analysis, which has a range of 450 cm^{-1} to 7500 cm^{-1}. The functional groups of the adsorbent can be identified using infrared light. An infrared frequency modification is assimilated by a particle. It is important to note that the particle's fundamental frequency is in the infrared. Infrared light can only be absorbed by a particle if it has a dipole moment that matches the frequency range of IR light. There is no change in the dipole moment or bending in the bonds with N_2 and H_2 atoms, hence they do not show IR radiation. A high-resolution picture of the adsorbent was obtained using scanning electron microscope (SEM) analysis, with magnification powers ranging from 8x to 300,000x. The primary goal of the SEM analysis was to discover the adsorbent's surface roughness and shape. As a high-definition imaging tool, SEM is an excellent choice. Images are arranged in the SEM using either low-energy auxiliary electrons (SEI) from the sample surface or high-energy backscattered electrons (BEI). Although data on 50 to 150 3-mm thicknesses of the sample may be gleaned from SEI images, surface highlights from greater depths can be uncovered by BEI images. When compared to light microscopy, SEM offers superior amplification, resolution, and field depth.

3. Results and discussion

3.1. Optimization by Taguchi methodology

All 16 experiments are presented in Table 4 showing SNR and the Mean. SNR was calculated using Equation 2, and the Mean is the average of the experimental values conducted thrice for the best minimum error. The removal rate varied from 91.38% to 99.73% based on the combination of four factors. From these, the related SRN of the single factors with a single-level entry is shown in Figure 1; it also showed that the largest SNR variation occurred with pH, Initial Dye Concentration, Contact Time, and Adsorbent Dose. In other words, the most important factor was pH, while the least important one was the Adsorbent Dose. The descending position was pH > Initial Dye Concentration > Contact Time > Adsorbent Dose. It was again verified by ANOVA (Abbasi et al., 2017). The outcome of ANOVA represents the same position of impact for MG Dye removal as exemplified in Figure 2 of the percentage contribution chart, the highest to lowest was pH > initial dye concentration > equilibrium time > bio-adsorbent dose. Hence, the most favorable condition for the Response Table for SNR in Table 5 was found to be A1, B2, C1, and D3 namely pH- 3, Initial MG Concentration of 100 mg/L, bio adsorbent dose-0.20 g/100 mL, equilibrium contact-time- 90 mins. The different parameters such as pH, initial dye concentration, and contact time play a dominant role in the adsorption techniques and are responsible for the removal of dye contamination from water or wastewater bodies [57- 59].

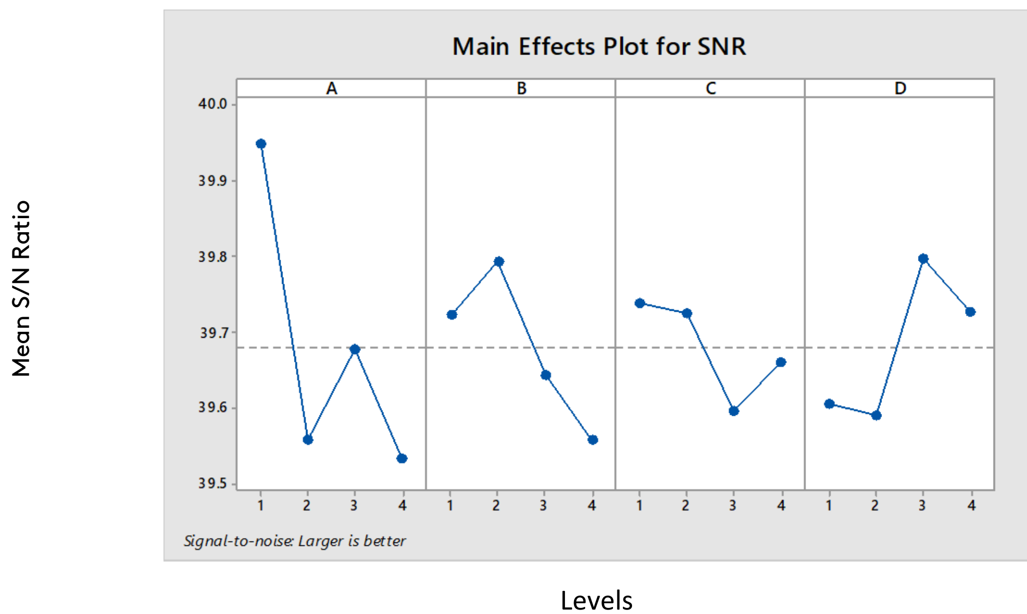


Fig. 1. Main Effects Plot for SNR.

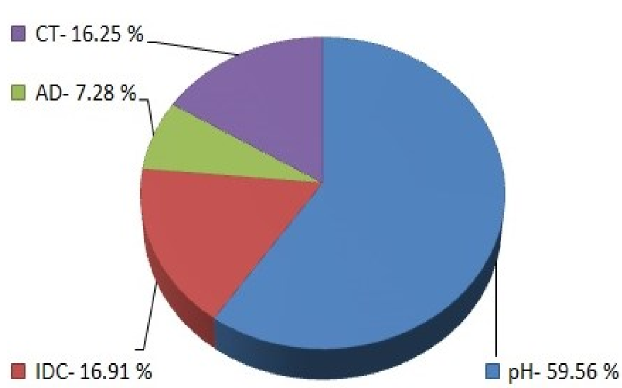


Fig. 2. ANOVA percentage of contribution chart.

Table 4. L16 orthogonal array along with results.

Exp. No.	pH	Initial MG Concentration (mg/L)	NCBT dose (g/100mL)	Equilibrium contact time (mins)	Removal (%)			SNR	Mean
					Run 1	Run 2	Run 3		
1	1	1	1	1	99.22	99.28	99.03	39.93	99.18
2	1	2	2	2	99.37	99.33	99.21	39.94	99.30
3	1	3	3	3	99.49	99.44	99.36	39.95	99.43
4	1	4	4	4	99.75	99.75	99.69	39.98	99.73
5	2	1	2	3	98.82	98.77	98.53	39.89	98.71
6	2	2	1	4	97.86	97.4	97.32	39.78	97.53
7	2	3	4	1	93.85	92.24	92.32	39.35	92.80
8	2	4	3	2	92.65	90.67	90.81	39.22	91.38
9	3	1	3	4	96.13	95.32	95.16	39.60	95.54
10	3	2	4	3	98.41	98.1	98.01	39.84	98.17
11	3	3	1	2	97.41	96.78	96.76	39.73	96.98
12	3	4	2	1	95.54	94.36	94.42	39.53	94.77
13	4	1	4	2	94.91	93.76	93.63	39.47	94.10
14	4	2	3	1	96.25	95.34	95.31	39.61	95.63
15	4	3	2	4	95.62	94.49	94.52	39.54	94.88
16	4	4	1	3	95.33	94.09	94.16	39.51	94.53

Table 5. Response Table for SNR.

Factor / Level	pH	Initial MG Concentration in mg/L	NCBT dose in g/100 mL	Time (mins)
1	**39.95**	39.72	**39.74**	39.61
2	39.56	**39.79**	39.73	39.59
3	39.68	39.64	39.60	**39.80**
4	39.53	39.56	39.66	39.73
Delta	0.41	0.23	0.14	0.21
Rank	1	2	4	3

3.2. Adsorption isotherms

Isotherm studies were conducted by varying the adsorbent dose and keeping the other parameters, viz., Initial Dye Concentration (mg/L)-100 mg/L, Adsorbent Size-505 microns, Agitation Speed-200 rpm, Temperature-*298 K,* and pH-7 constant. The Langmuir isotherm and Freundlich isotherm were applied in the present study. The Langmuir adsorption model can be expressed in the following form:

$$q_e = \frac{Q_{max} K_L C_e}{1 + K_L C_e} \quad (3)$$

where q_e = equilibrium loading on the adsorbent (mg/g), Q_{max} = ultimate adsorption ability of the adsorbent (mg/g), K_L = relative energy of adsorption, also known as the equilibrium constant, and C_e=equilibrium concentration in the waste stream (mg/ l).

The Freundlich adsorption model can be expressed in the following form:

$$q_e = K_F C_e^{1/n} \quad (4)$$

where q_e = optimum load on bio-adsorbent (mg/g), K_F = adsorption ability at a unit concentration (mg/g) (l/mg), 1/n = adsorption intensity β typically empirically determined (dimensionless), C_e = equilibrium concentration in the wastewater (mg/L), and K_F and n are constants [60-65].

The experimental value trends matched the Langmuir isotherm model in comparison to the Freundlich model, signifying that the adsorption was a monolayer type. The Q_{max} value was 142.85 mg/g, b=0.020 l/mg, the K_F value was 1.621 mg/g (l/mg), and n = 0.839, as shown in Figures 3 and 4.

The value of R_L was found to be 0.0613, which signifies that the adsorption was a favorable process.

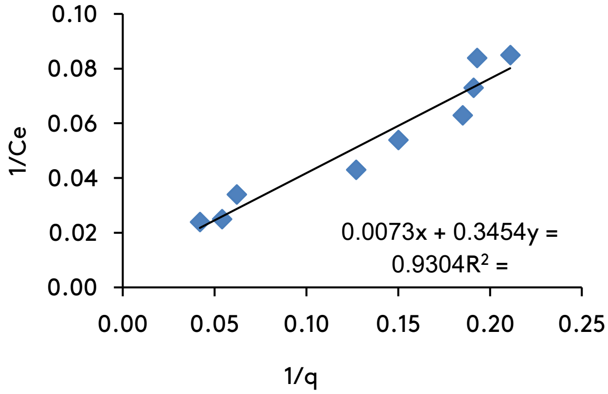


Fig. 3. Langmuir Adsorption Isotherm.

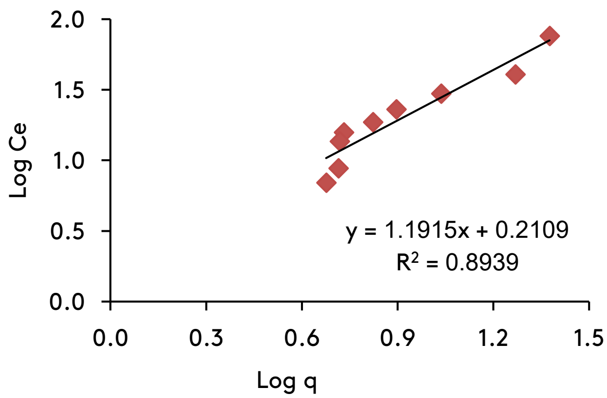


Fig.4. Freundlich Adsorption Isotherm.

3.3. *Characterization analysis*

The chemical structure and functional group of *Bambusa Tulda* were determined using FTIR analysis. As can be seen in Figure 5, the FTIR spectrum was measured between 4000 and 400 cm^{-1}. Observing a sharp peak at 3451.35 cm^{-1} is indicative of the hydroxyl (O-H) group, whereas peaks at 2920.09 cm^{-1} are characteristic of the CH_3 group (alkane group). At 1735.59 cm^{-1}, a carboxyl group was detected. The alkenyl bond is shown by the peak at 1635.71 cm^{-1}. NCBT was shown to be effective for removing MG dye from wastewater bodies due to the presence of hydroxyl and carboxyl groups on its surface. SEM analysis was conducted to understand the exterior morphology of *Bambusa Tulda*. When the pores were chemically modified, they became much larger and more active for adsorption, as shown in Figure 6. The MG dye molecules filled in the pores structure sites on the NCBT's surface, leaving a smooth surface over the pores cavity, as shown in Figure 7.

4. Comparative study assessment

Comparative research was performed to understand the different adsorbent and dye behaviour in response to optimum parameters like pH and contact time (Table 6).

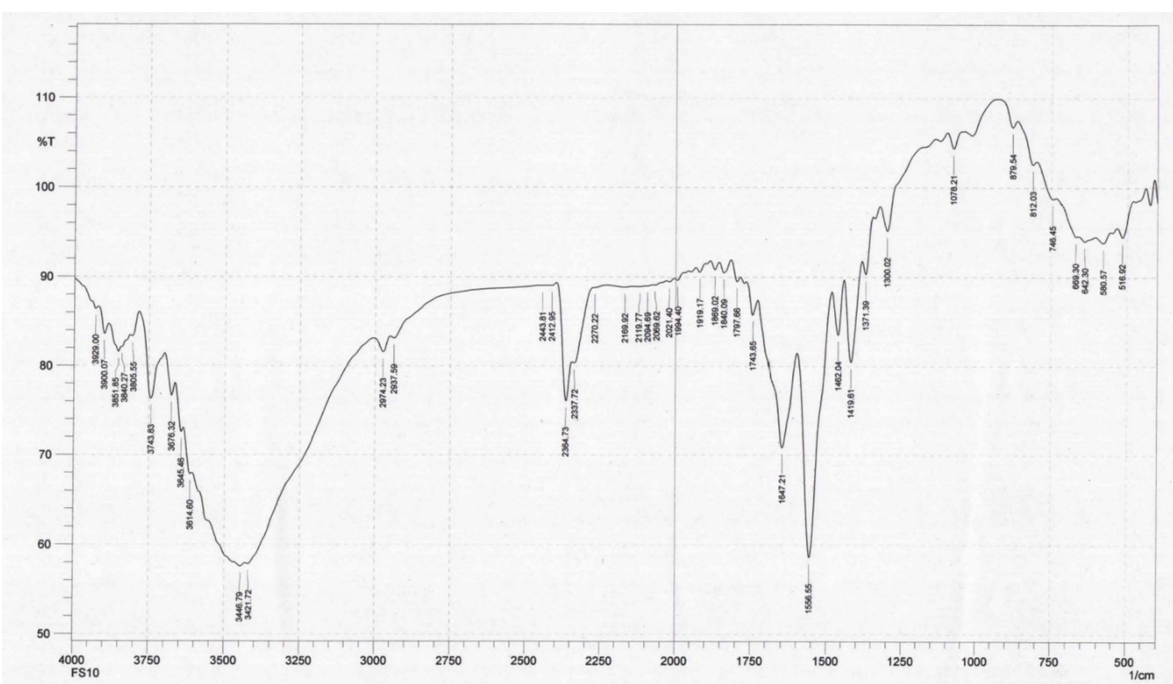


Fig. 5. FTIR analyses of NCBT adsorbed MG Dye.

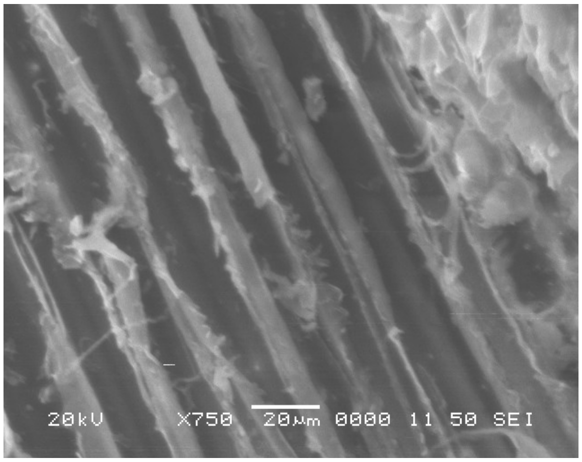


Fig. 6. SEM image of NCBT.

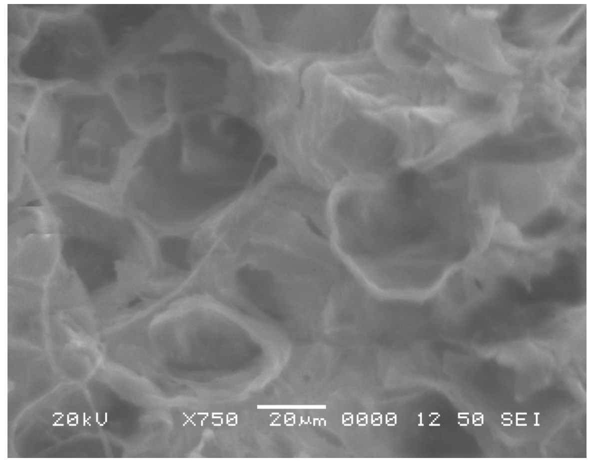


Fig. 7. SEM image of NCBT adsorbed MG Dye.

Table 6. Comparative study.

Optimum Parameters	Dye	Adsorbent	References
pH-7	Methyl Orange	decorated ZnO nanoparticles with SnO_2 nanoparticles	[23]
pH = 4	Methyl Orange	decorated MWCNTs (MWCNTs-ZnO)	[24]
pH=3	Methyl Orange	modied MWCNTs	[25]
pH-3	Malachite Green	Na_2CO_3 treated bamboo chips	Present research
Contact Time: 25 minutes	Methyl Orange	decorated multi-walled carbon nanotubes (MWCNTs) with different amount of ZnO nanoparticles	[32]
ranging from 5 to 30 min	Methyl Orange	modified MWCNTs with different amount of ZnO nanoparticles	[55]
From 5 to 35 min	Methyl Orange	Using MWCNTs@TiO_2 as Photocatalyst	[11]
Contact Time: 90 minutes	Malachite Green	Na_2CO_3 treated bamboo chips	Present research

4. Conclusions

Intensive research was done to identify the performance of NCBT for the removal of MG dye from wastewater. Taguchi's methodology was optimized to understand the optimum adsorption parameters. The results of the set of 16 experiments showed a higher removal rate, which varied from 91.38% to 99.73%, depending on some major factor combinations. The prominent parameters displayed were pH, initial dye concentration, contact time, and absorbent dose. The most favorable conditions for the response table as measured by SNR in Table 5 were found to be A1, B2, C1, and D3, namely pH-3, initial dye concentration of 100 mg/L, bio adsorbent dose of 0.20 g/100 mL, and contact time of 90 mins. The adsorption model follows the Langmuir isotherm, which signifies monolayer adsorption techniques with a Q_{max} value of 142.85 mg/g and R^2 = 0.930. The value of R_L was found to be 0.0613, indicating that the adsorption process was favorable. The Fourier transform infrared spectroscopy displayed hydroxyl and carboxyl groups. The external morphology of *Bambusa Tulda* was studied using SEM analysis. Thus, it could be concluded that NCBT is a good bio-adsorbent for the removal of Malachite Green dye from wastewater.

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