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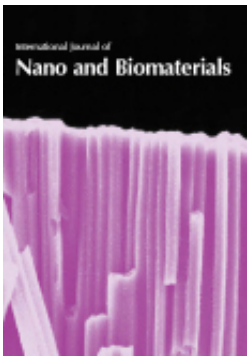

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Synthesis and characterisation of nano-adsorbents from chemically modified Parthenium hysterophorus for the treatment of industrial wastewater

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Abstract: Chemical modification of locally grown weed *Parthenium hysterophorus* (PH) has been carried out to increase the adsorption capacity, as well as the capacity to remove the soluble organic compounds and colour. Four different samples were prepared by treatment with two acids: a) H_2SO_4; b) HCl, and two bases; c) KOH; and d) NaOH. This nano-adsorbent was then characterised using FTIR, SEM, and XRD to identify the functional groups as well as surface morphology. The application of this nano-adsorbent has been explored for the removal of dye from textile industry wastewater. Batch Experiments with different samples have been performed to study the removal efficiency of synthesised nano-adsorbent. The effect of different variables like (nano-adsorbent dosage, dye concentration, pH of wastewater, and contact time was studied to optimise the parameters. The KOH-modified nano-adsorbent has shown the best removal efficiency at an optimum set of parameters.

Keywords: nano-adsorbent; dye removal; adsorption; wastewater; water treatment.

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Pardeep Rao has done MSc and MTech (Environmental Science and Engineering) with Ist Division from GJUST, Hisar, Haryana, India. He has completed an Mtech thesis entitled 'Removal of dye from Industrial wastewater using nano adsorbent prepared from Parthenium hysterophorus'. His area of interest includes wastewater treatment and environmental remediation of organic pollutants.

1 Introduction

Developmental activities around the world have increased rapidly in the 21st century, leading to increased industrial activities (Gupta, 2022). These activities consume a lot of water and release many inorganic and organic pollutants. The industrial effluents from many industries also contain a significant amount of synthetic organic dyes, which are a serious concern to esthetics and the ecosystem. Industries such as textile, plastic, dyeing, paper, and colouring are contributing a lot in the release of such effluents. The textile industry uses a number of dyes to colour their products and correspondingly releases a lot of dyestuff in the wastewater discharge. Wastewater released by different industries contaminates water resources. This has many consequences on aquatic as well as terrestrial life as textile dyes are mutagenic as well as carcinogenic to the aquatic life (Patanjali et al., 2022).

Worldwide, more than 10,000 types of dyes are available. Synthetic dyes find extensive use in the textile industry (Patanjali et al., 2022). In recent time, issues related to the use of dyes in production has gotten special attention as it may lead to acute health problems. Dysfunction of organs like the kidney, brain, and CNS has been reported due to the use of dyes of a basic nature (Soliman and Moustafa, 2020). Similarly, Cationic dyes like methylene blue (MB) are being used in dyeing cotton, silk, and wood in many applications around the world. It may contaminate the water resources and may cause health hazards to aquatic as well as terrestrial food chains. This may lead to health problems like diarrhoea, jaundice, vomiting, necrosis, and cyanosis (Saini et al., 2018). High concentrations of MB dye can even lead to chronic effects like cancer (Elgarahy et al., 2021). Being a toxic dye, it affects the living life in the water adversely due to high solubility in the water (Mehra et al., 2021). So, awareness about the removal of these dyes by employing various natural adsorbents is very much important for the wastewater treatment.

Different methods like Membrane filtration, Ion exchange, ozonisation, and coagulation/flocculation can be deployed for the decontamination of dyes from wastewater. However, the suitability and efficiency of the specific manufacturing process is a great concern. Adsorption is a very effective method of removing dye from wastewater (Sivamani and Leena, 2009), as it offers a simple, flexible, and low-cost process to achieve complete decontamination. Also, a number of agricultural waste and industrial waste materials can be used for synthesising the adsorbents (Sardar et al., 2021; Demirbas, 2009; Eletta et al., 2018; Yagub et al., 2014; Gimbert et al., 2008). The factors that influence the removal process may include the adsorbent surface area, interaction between adsorbent and adsorbate, particle size of adsorbent, pH, temperature, and contact time in the reaction phase (Allen and Koumanova, 2005; Crini, 2006).

In the present work, Parthenium Hysterophrous (PH) has been taken for preparation of adsorbent, which is a flowering plant belonging to the family '*Asteranceae*' (Kadhom et al., 2020; Lata et al., 2007; Bharathi and Ramesh, 2013; Joshi et al., 2016). It is available in different subtropical and tropical regions of the world (Nyasembe et al., 2015). In recent times, PH has been available all around India, growing in land of approximately 35 million hectares (Lalita and Kumar, 2018). It is noticed around very common areas like irrigation and drainage canals, sides of roads, residential colonies, railway tracks, etc. (Meena et al., 2017). The plant is a weed, and its growth rate is also very high. It is also used for various purposes like treating allergies, diseases, soreness, and anaemia, and is also used as a vermifuge, blood purifier, and insecticide (Khaliq and Chaudhary, 2016). There are a lot of methods to convert PH to activated carbon adsorbents, and carbonisation is regarded as one of the most favourable processes for conversion. It is a slow pyrolysis process in which PH is heated under controlled parameters at temperatures ranging from 400°C to 600°C to convert into activated carbon.

2 Experimental

2.1 Synthesis of nano-adsorbent

Parthenium hysterophrous (PH) weed was collected from Milkpur, Bhiwani District of Haryana State of India. It was washed with double distilled water to remove all dust and impurities and then dried in sunlight for 10 days to remove all moisture. Dried PH was crushed in a grinder, and then the grinded material was sieved using a micropore size sieve. The material was stored in an airtight container to avoid moisture. The utilisation of chemical activation has gained significant popularity due to its ability to operate at lower activation temperatures and provide high yields of carbon products. Activation of agricultural waste facilitate the process of breaking chemical bonds and creating new functional group connections within the precursor structure. It also reduces porosity of samples (Koubaissy et al., 2014, Kunusa et al., 2021)

Dried weed was chemically modified with two acids and two bases (1N KOH, 1N NaOH, 1N HCl, and 1N H_2SO_4) for increasing adsorption capacity, removal of organic compounds (soluble), and elimination of colour from aqueous solution (Lata et al., 2007) Sulphuric acid has given maximum yield of activated carbon and Hydrocholoric acid has shown near minimum yield of activated carbon (Sivaraj et al., 2010). So one active acid and one inactive acid was selected for study. NaOH and KOH bases has been selected for activation of Parthenium *Hysterophrous* on basis of literature given by Ukanwa et al. (2019)

For chemical modification with 1N H_2SO_4, 12 grams of homogenised waste was mixed with 250 ml of 1N H_2SO_4 and kept overnight. Then, the solution was filtrated with Whatman filter paper, and solid residues were washed with double distilled water. The residue was dried in a hot air oven and was carbonised in a muffle furnace at 500°C for 3 h. The resulting material was again ground and stored in an airtight container to avoid moisture. The prepared carbonised adsorbent has been labelled as H_2SO_4 treated PHAC nano-adsorbent (S1). A similar method was used for the preparation of HCl-treated PHAC nano-adsorbent (S2), KOH-treated PHAC nano-adsorbent (S3), and

NaOH-treated PHAC nano-adsorbent (S4). Nomenclature of the prepared samples has been given in Table 1.

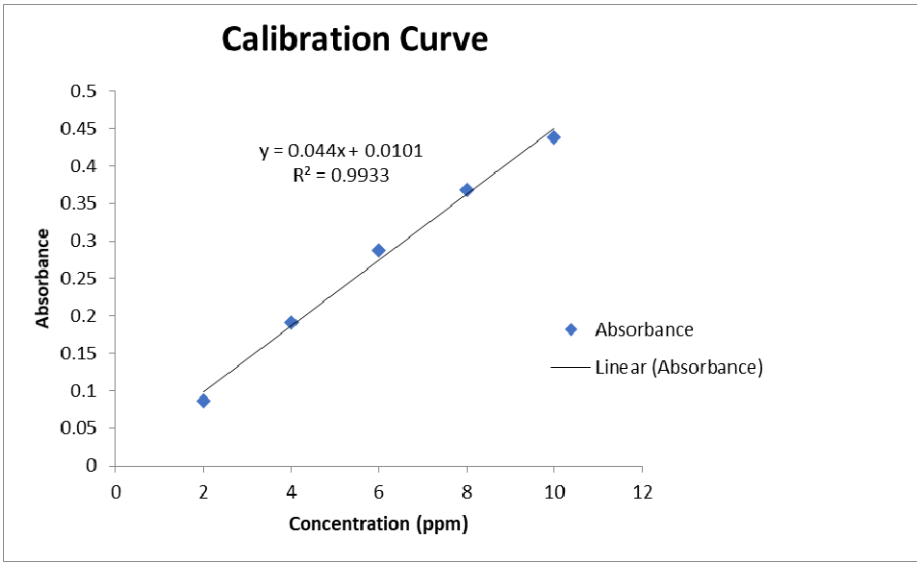
Table 1 Nomenclature of samples

Sample	*Description*
S1	H_2SO_4 treated PHAC nano-adsorbent
S2	HCl-treated PHAC nano-adsorbent
S3	KOH-treated PHAC nano-adsorbent
S4	NaOH-treated PHAC nano-adsorbent

2.2 Dye solution preparation

In order to explore the applications of prepared samples for the removal of dye, Methylene blue dye was used to prepare the simulated wastewater. Stock solution of dye was prepared at a concentration of 1000 mg/L by dissolving dye (Analytical Reagent Grade) in double distilled water. The desired MB concentrations were prepared from the stock solution by dilution method. All the experiments were conducted by diluting the stock solution in appropriate concentrations between 10 mg/L and 50 mg/L (Yaseen and Scholz, 2019). The Concentration of dye in solution before and after Adsorption was calculated from the calibration curve using a UV-VIS spectrophotometer (UV 1800 SHIMADZU) at a wavelength (λ_{max}) of 670 nm at Figure 1.

Figure 1 Calibration curve of absorbance vs. concentration of methylene blue (see online version for colours)



2.3 Adsorption study

In the batch adsorption process, 100 ml dye with fixed pH and Concentration was added to 200 mg adsorbent at room temperature in a 250 mL Erlenmeyer flask. The mixture was

stirred at 120 rpm for a fixed time period on the rotatory shaker and removed. This mixture was then centrifuged at 4000 rpm for 5 min to separate the adsorbent from the dye solution. The Concentration of the residual dye was used for estimating the absorbance of the supernatant. The Concentration of the residual dye has been estimated by absorbance values taken with a spectrophotometer (617 nm) before and after treatment. The experiments were carried out by changing adsorbent concentration (0.5–2 gm/L), dye solution concentration (10–50 mg/l) as well as pH (4–12) for different time periods. pH has been adjusted by adding appropriate amounts of 1N HNO_3 or 1N NaOH solution. The removal percentage of methylene blue was calculated for each run by the following expression:

$$\text{Removal \%} = (C_0 - C_e)/C_0) \times 100 \tag{1}$$

where

C_0: adsorbate initial conc. (mg/L)

C_e: adsorbate conc. at equilibrium (mg/L).

Adsorption capacity, q_e, is defined as the amounts of adsorbed dye at equilibrium onto adsorbent material and was calculated by using the equation-

$$\text{Adsorption capacity } (q_e) = (C_o - C_e) \times V \tag{2}$$

where

q_e: at equilibrium, adsorbed dye molecules per unit adsorbent (mg/g)

V: volume (ltr.)

3 Material characterisation

FTIR analysis of all samples of nano adsorbent was recorded with the help of an FTIR spectrophotometer for studying the functional group and their binding site. XRD analysis has been used to investigate the crystalline and amorphous nature of prepared nano-adsorbent. SEM (LEO 435 VP) has been used at 15 kV to analyse the surface morphology of the adsorbent with background subtraction.

3.1 Fourier transforms infrared spectroscopy

FTIR spectra of the samples have been shown in Figure 2(a)–(d) simultaneously. They indicate various functional groups on the prepared nano adsorbent. Different functional groups are present on the surface of the PHAC nano-adsorbent that is mainly responsible for removing dye from the wastewater. The functional group on the surface of the adsorbent helps in the removal of a great amount of organic dye from the wastewater (Bedada et al., 2020). Various functional groups present on the adsorbent surface containing carboxylic, phenolic, and carbonyls were responsible for the removal of organic contamination from the wastewater (Jain et al., 2016). FTIR spectra of untreated Parthenium hysterophorus has shown peaks between 3500–3000 cm^{-1}, 2395–2235 cm^{-1}, 2200–2000 cm^{-1}, 1738 cm^{-1}, 1402 cm^{-1}, 1378 cm^{-1}, 1170–1082 cm^{-1}, 1059 cm^{-1} (Lanjewar et al., 2020)

A spectrum of these adsorbents has been recorded in the range of 400–3500 cm^{-1}.

FTIR spectrum in Figure 2(a) shows five broad peaks in S1 sample. The peak at 3429.75 cm^{-1} represents –OH stretching. This wide peak in the range 3500–3000 cm^{-1} can be attributed to the existence of free and intermolecular fused –OH group that shows the presence of lignin and carbohydrates (Lanjewar et al., 2020). This functional group is attached to the phenols and organic acid (Fito and Van Hulle, 2021). The peak observed around 2320, 1619.17, and 1120 cm^{-1} corresponds to –CH, C = C, and C-O stretching simultaneously. Moreover, the peaks at 675.71 and 616.30 cm^{-1} are due to aromatic compounds and their bending modes.

Figure 2(a) FTIR spectrum of H_2SO_4 treated PHAC (S1) (see online version for colours)

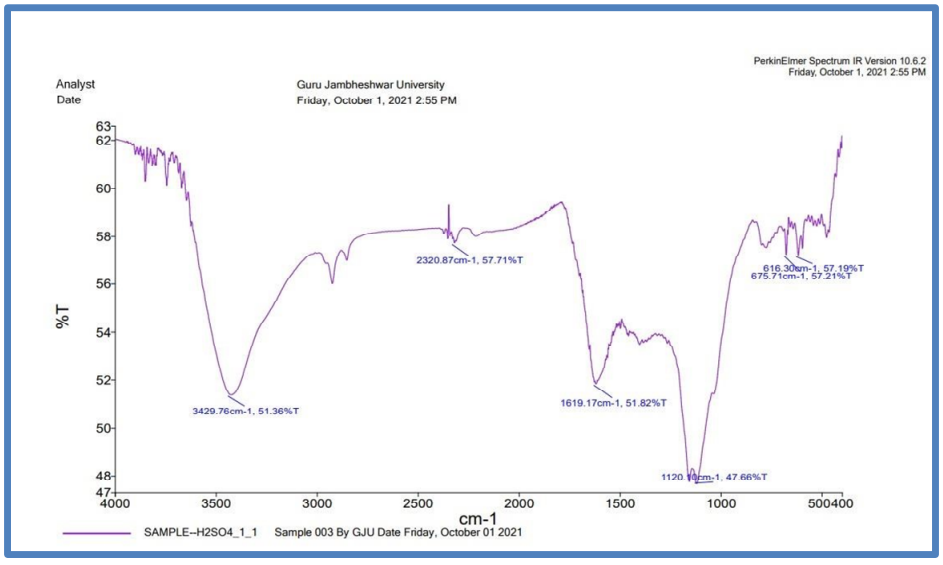
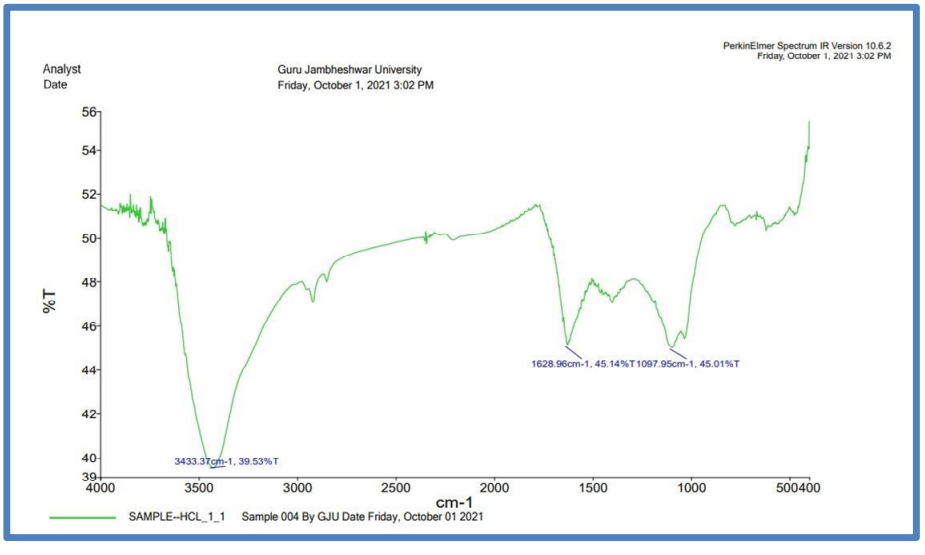


Figure 2(b) FTIR spectrum of HCl treated PHAC (S2) (see online version for colours)



In the FTIR spectra of Sample S2 (Figure 2(b), The absorption peak due to OH bonding is shifted to 3433.37 cm^{-1} in Sample S2. The other prominent peaks, 1628 and 1097.95 cm^{-1}, are due to C=C and C–O, respectively, groups.

Figure 2(c) shows the FTIR spectrum of sample S3. The first peak obtained at 3786.73 cm^{-1} and 3427 cm^{-1} represent O–H stretching. The peak at 1633.23 cm^{-1} is linked with the C=C bond. The peak at 1032.58 cm^{-1} contains in the range of C–O single bond.

Figure 2(c) FTIR spectrum of KOH treated PHAC (S3) (see online version for colours)

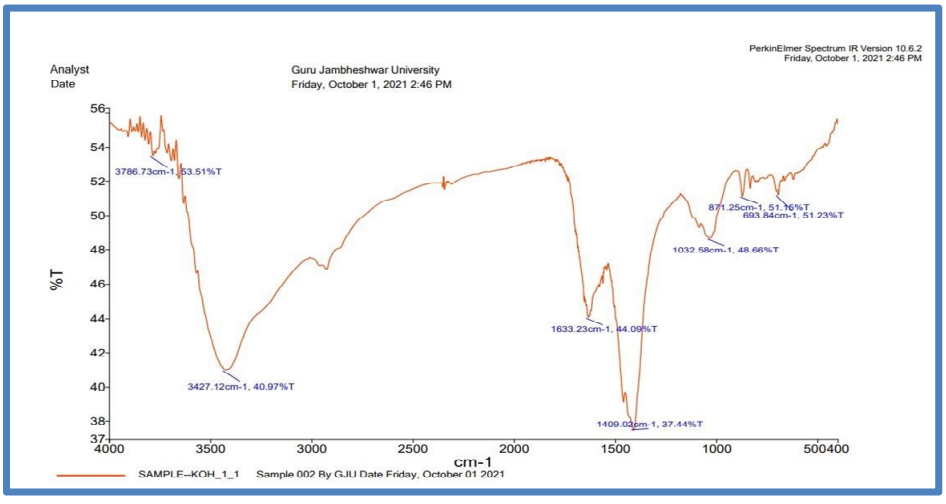
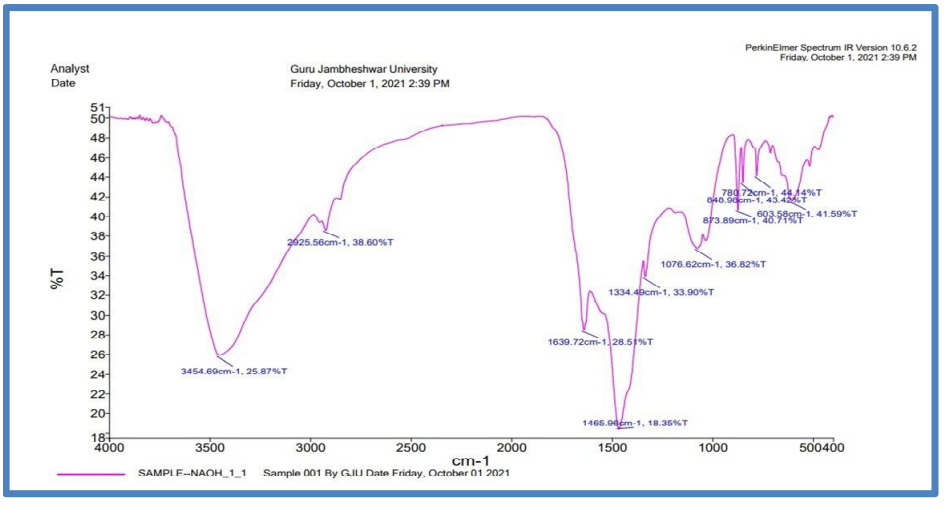


Figure 2(d) FTIR spectra of NaOH treated PHAC (S4) (see online version for colours)



FTIR spectra of Sample S4 show six prominent peaks (Figure 2(d)). The prominent peaks are at 3454 cm^{-1}, 2925.56 cm^{-1}, 1639.12 cm^{-1}, 1465.96 cm^{-1}, 1465.96 cm^{-1} and 1334.49 cm^{-1}. The peak obtained at 3454 cm^{-1} is attributed to the intermolecular bonded and free OH group present. The adsorption band at 2925.56 cm^{-1} indicates asymmetric

stretching of C–H in the (–CH2) and (–CH3) groups. The small peak at 1639.72 cm^{-1} indicates ketone stretching vibration. Peak intensity is weak, indicating an amount of carboxyl group. A peak at 1465.96 cm^{-1} represents –C=C–C and aromatic C=C group. Lastly, 1334.49 cm^{-1} shows bending vibration of C–H2 as well as C–H3

3.2 SEM analysis

a *EM/EDAX analysis of H_2SO_4 treated PHAC*

Figure 3(a)–(d) shows SEM Images of S1–S4 respectively. The analysis has shown that the sample consists of an irregular distribution of adsorbents ranging from 60.1–246 nm diameter size (S1), 55.9–176 nm diameter size for S2, 60–294 nm diameter size for S3, 44.4–329 nm diameter size for S4.

Figure 3 SEM of: (a) S1; (b) S2; (c) S3 and (d) S4

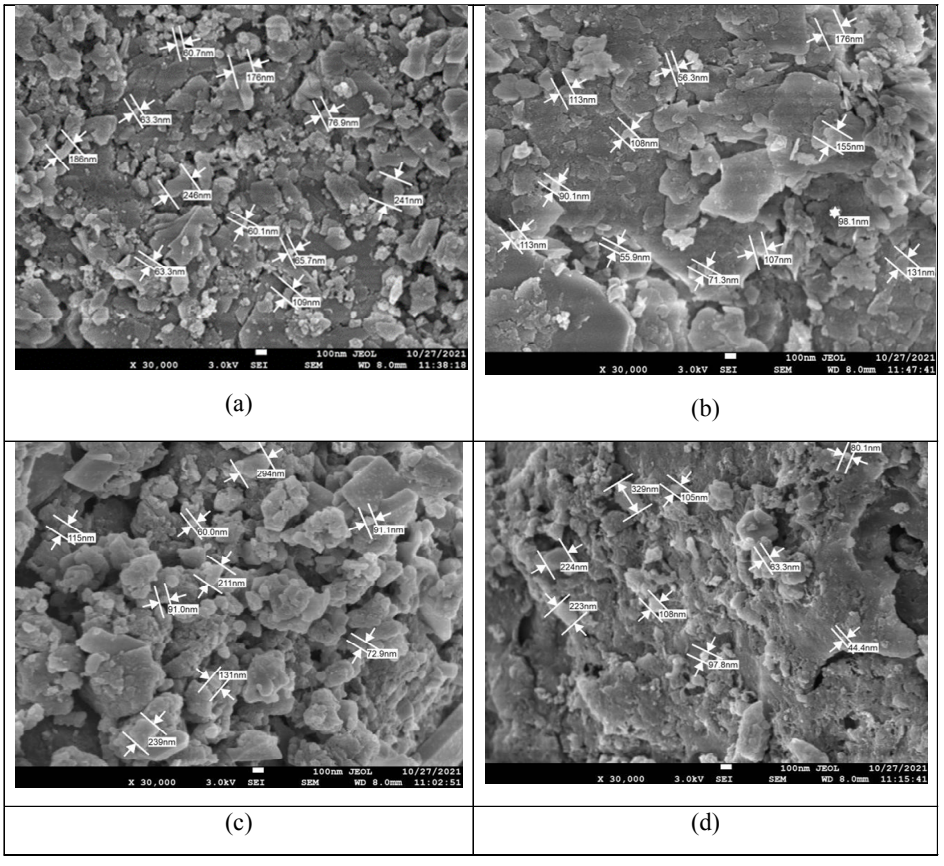


Figure 4(a)–(d) shows elemental analysis of all samples. All samples contain Si, C, Na, Al, K, Cu, Ca, and Mg in varying percentage by weight but carbon is missing in S1. It may be due to reaction of parthenium Hysterophrous with sulphuric acid during carnonzation of sample. In Sample S1, Oxygen was present by weight at 51.48%, Si was present at 32.45% by weight, and S was present at 3.49% by weight. Some other

elements like Na, Al, K, Cu, Ca, and Mg were also found with a minor weight percentage.. In sample S2, Carbon was present by weight at 61.16%, oxygen at 20.24% by weight, K at 4.74% by weight, P at 3.80% by weight, and Ca at 3.06% by weight. Some other elements like Si, Na, K, Cl, and Mg were also found with a minor weight percentage. In sample S3, Carbon was present by weight as 29.54%; oxygen was present at 38.46% by weight, K was present at 14.86% by weight, Ca was present at 8.55% by weight, and Mg was present at 2.21% by weight. Some other elements like P, Na, Si, Cu, and Cl were also found with minor weight percentages. In sample S4, Carbon was present by weight at 35.53%, oxygen was present at 38.48% by weight, Na was present at 16.93% by weight, Mg was present at 1.18% by weight, and Si was present at 1.01% by weight. Some other elements like Al, Si, P, S, K, Ca, Cu, and Cl were also found with minor weight percentages.

Figure 4(a) EDAX spectrum of H_2SO_4 treated PHAC (S1) (see online version for colours)

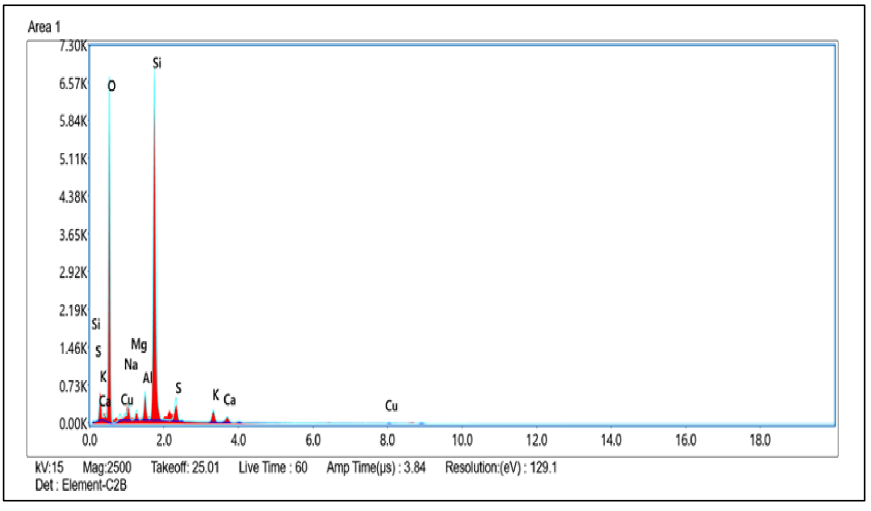


Figure 4(b) EDAX spectrum of HCl treated PHAC(S2) (see online version for colours)

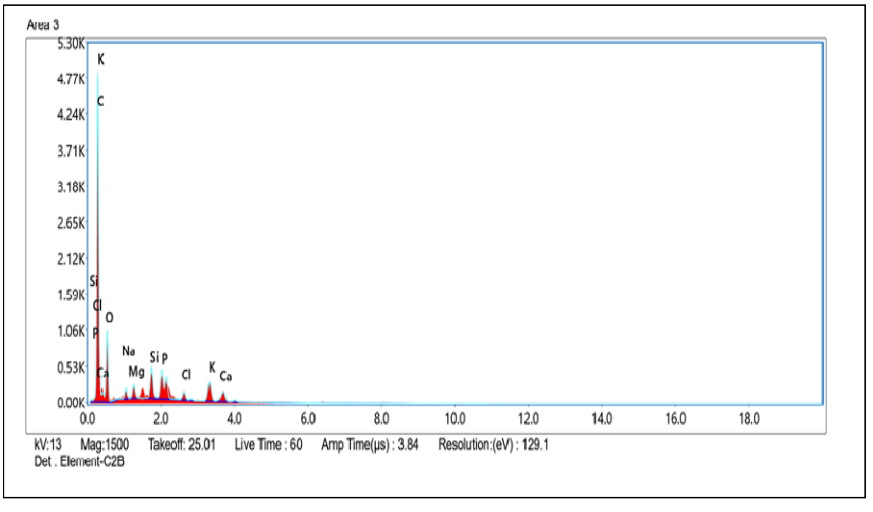


Figure 4(c) EDAX spectrum of KOH treated PHAC(S3) (see online version for colours)

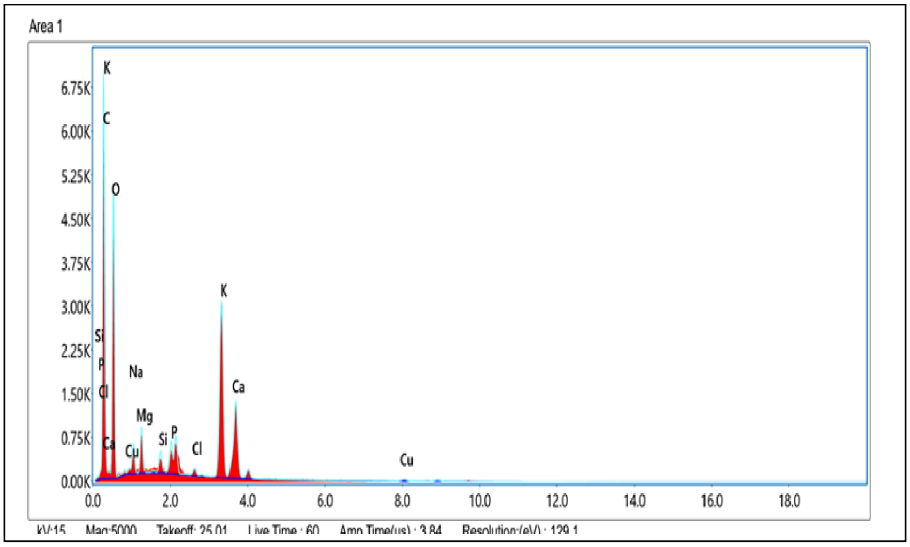
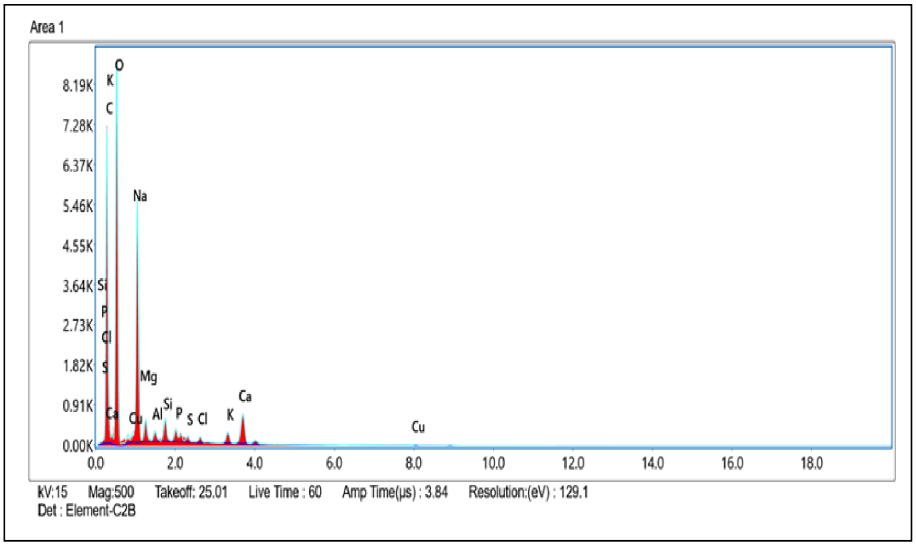


Figure 4(d) EDAX spectrum of NaOH treated PHAC(S4) (see online version for colours)



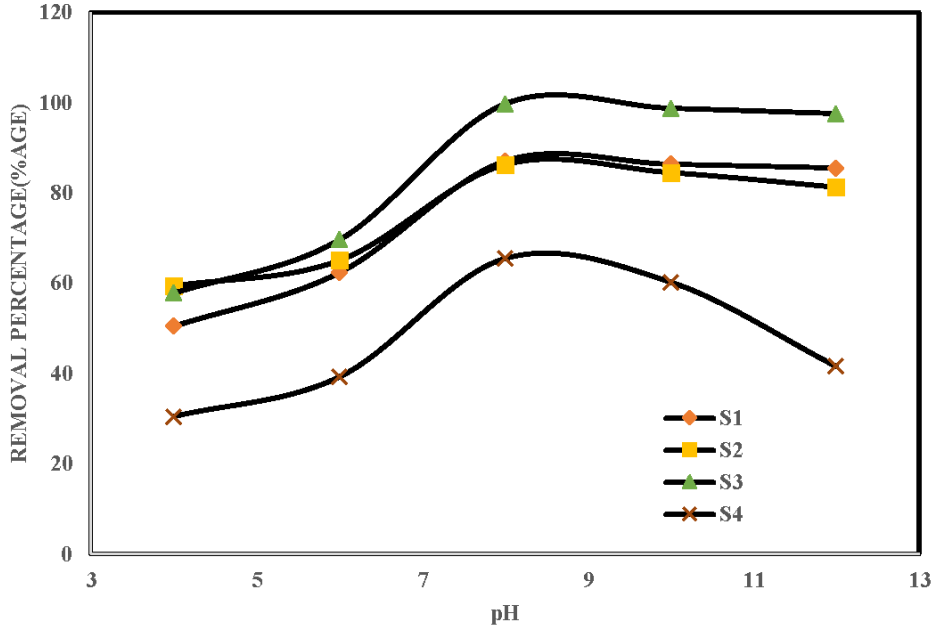
4 Adsorption analysis

a *Effect of pH*

The pH value was varied from 4 to 12 to study its effect on dye removal by keeping all other parameters (MB concentration 30 ppm, the adsorbent dosage 0.1 g/100 ml, 60 min contact timing) constant. The effect of pH in dye removal for different samples has been shown in Figure 5. Generally, when pH increases, the adsorbent surface will attain negative sites, and it will attract cationic adsorbates, which are positively charged.

Whereas on decreasing pH, the adsorbent surface will attain positive sites, it will attract anionic adsorbates, which are negatively charged. It signifies that a high pH value is suitable for removing dye, which is a cationic dye, from the waste water of industries. pH value of the solution will influence the chemistry of the surface as well as adsorbent-adsorbate reactions, and it further enhances the adsorption removal efficiency. But in this case, two samples (S1 & S2) are acid-modified adsorbents, and two samples(S3 & S4) are basic modified adsorbents. In all samples, percentage removal will increase from pH 4 to 8, and then it starts decreasing up to pH 12. The percentage removal of Methylene Blue increased from 50.42 to 87.04 and decreased to 85.54 for S1, from 59.36 to 86.27 and decreased to 81.27 for S2, from 57.77 to 99.72 and decreased to 97.59 for S3, from 45.1 to 65.45 and decreased to 41.5 for S4 on increasing pH from 4-12. Maximum removal is 99.77% for sample 3. The change in trend is due to a change in the chemical structure of PH when it is treated with acid and bases. Both the bases can show different effect as KOH is more effective at creating micropores due to the evidence of an interaction between layers, while NaOH is extremely active for disordered carbon materials. We have done these experiments twice and we have taken average of these two reading. Sample 3 is itself more basic in nature and it will lose –OH ions more readily than NaOH. Further, the Concentration of hydroxyl ions increases with increasing pH of the solution. More hydroxyl ions, more MB will be adsorbed on the surface of the adsorbent. Removal of MB may be due to electrostatic attractions between MB and adsorbate and the formation of the covalent bond between adsorbate and adsorbent.

Figure 5 Effect of pH effect on methylene blue dye removal (see online version for colours)

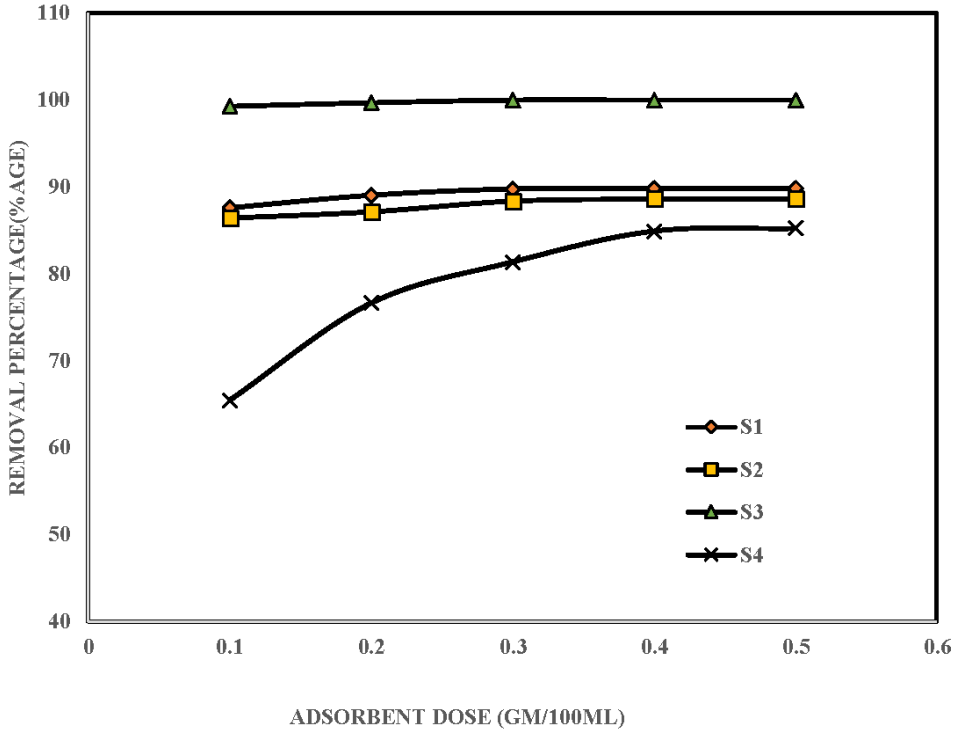


b *Effect of adsorbent dosage*

To study the effect of the Dose of adsorbent on the removal of methylene blue, a batch experiment for Adsorption was performed by changing the adsorbent dosage

(0.1–0.5 g/100 ml) in the test solution while keeping the Concentration of the initial dye (30 ppm) at room temp and pH 8 for 1 h. Dye removal increased linearly with an increase in adsorbent Dose in all samples. The optimum degradation was 89.82% at 0.4 g of the adsorbent Dose for S1, 88.63% at 0.5 g of the adsorbent Dose for S2, 100% at 0.5 g of the adsorbent Dose for S3, and 85.22% at 0.5 g of adsorbent Dose for S4. 100% percentage removal was observed at 0.5 g adsorbent dose for S3. The addition of adsorbent has increased the adsorbent surface area and availability of more adsorption sites, which enhances the removal of dye from the solution (Deng et al., 2011). Figure 6 shows the effect of nano-adsorbent Dose on methylene blue dye removal.

Figure 6 Effect of nano-adsorbent dose on methylene blue dye removal (see online version for colours)

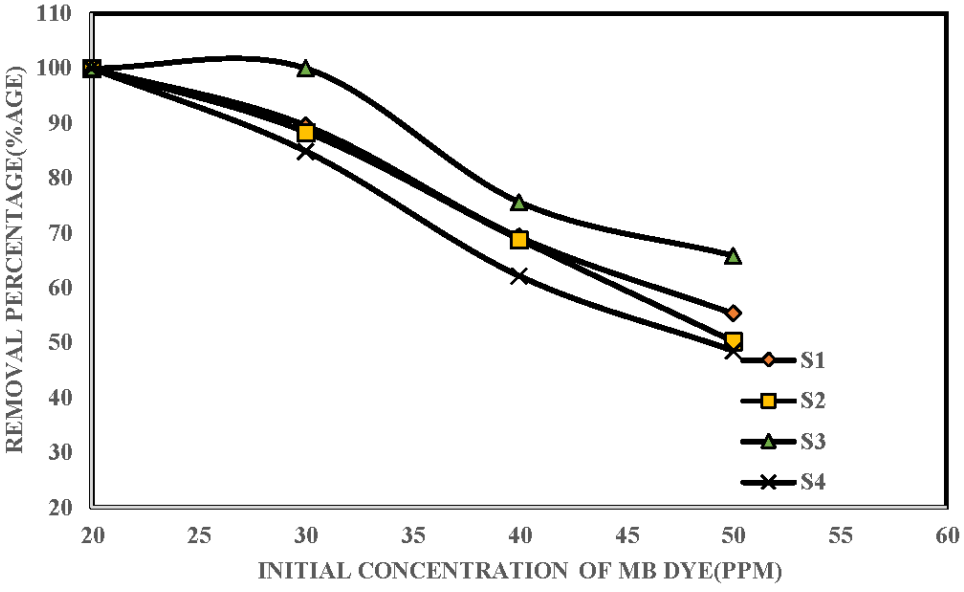


c *Effect of initial concentration of dye*

Figure 7 shows the effect of methylene blue dye in solution on removal percentage, keeping all other parameters constant (Dose of adsorbent as 0.4 g/100 ml, room temperature, pH 8). The percentage removal of methylene dye decreases from 100% to 55.32% in S1, from 100% to 50.28% in S2, from 100% to 65.88% in S3, and from 100% to 48.59% in S4 with increasing Concentration of methylene blue. The maximum decrease in removal percentage is in S4. From the results, it's observed that percentage removal drops with the additional rise in the dye concentration. The removal percentage of adsorbate depends on active sites present on the adsorbent. When these active sites

reach the saturation point, a decreasing trend will follow (Sun et al., 2010; Chiou and Li, 2002). On the other hand, adsorption capacity increases with an increase in dye concentration. It is observed that adsorption capacity increases from 20 to 27.7 in S1, 20 to 27.53 in S2, 20 to 30.6 in S3, and 20 to 24.87 in S4 on increasing initial dye concentration. Although percentage removal decreases, adsorption capacity increases with an increase in initial dye concentration up to 40mg/l in the test solution. Maximum removal percentage of S1, S2, S3, S4 is observed at 20 ppm initial dye concentration.

Figure 7 Effect of initial concentration on methylene blue dye removal (see online version for colours)



d *Effect of time of contact*

Figure 8 shows the Effects of time of contact on MB removal, keeping all other parameters (pH 8, initial MB concentration 30 ppm, the adsorbent dosage of 0.4 g/ 100 mL) constant. The efficiency of Adsorption increased sharply till time reached 90 min. Maximum percentage removal was 70.75% (S1), 87.04% (S2), 88.51%(S3), 70.3% (S4) at 90 min. The diffusion of adsorbate in aqueous solution towards the adsorbent determines adsorption efficiency and time consumed by adsorbate. The optimum value of time was 90 min for the maximum removal percentage.

e *Effect of surface modification of adsorbent*

Experiments have been done with S1, S2, S3, and S4 as adsorbents with different Concentration of dye (50–500 mg/l), different contact time (30–90 min), different adsorbent Dose (0.1–0.5 g/100 ml). It was observed that in all samples, the S3 sample showed the best results. The best results for all samples have been presented in Table 2.

Figure 8 Effect of contact time on the degradation of methylene blue dye (see online version for colours)

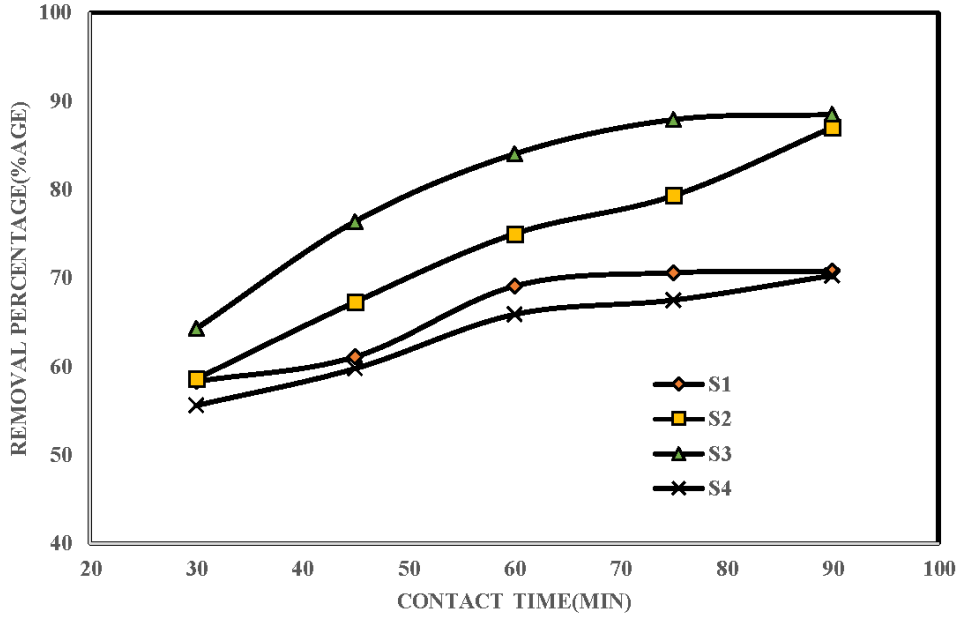


Table 2 Optimum conditions found during experimental results of synthetic solution

S. no	*Sample*	*pH*	*Adsorbent dose* (gm/1000 ml)	*MB dye concentration* (ppm)	*Contact time* (min)
1	S1	8	0.4	40	90
2	S2	8	0.5	40	90
3	S3	8	0.3	50	90
4	S4	8	0.5	30	75

5 Conclusion

The Nano Adsorbents prepared from chemically treated Parthenium Weed have shown good capability for the removal of Methylene Blue from wastewater of the textile industry. KOH-treated PHAC sample was found to have the highest efficiency in the removal of the methylene blue. Also, the maximum dye was removed within 90 min of the start of every experiment, and a pH value of 8.0 was found to be optimum for the maximum for all the adsorbents. However, the surface morphology has shown an irregular distribution of samples. Thus, the nano adsorbents synthesised from the chemically modified Parthenium Weed offer a very good opportunity for application in textile industry wastewater treatment.

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