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Ricinus communis Activated Charcoal preparation, Characterization and application for Methyl Red Adsorptive Removal

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ABSTRACT

The phosphoric acid activated *Ricinus communis* stem carbon (PRCS) is used to degrade methyl red dye from aqueous solution under solar light irradiation The studied Physiochemical properties, surface morphology, elemental composition and crystalline nature of PRCS are reported. The optimum experimental conditions are fixed by optimizing the experimental parameter such as contact time, pH, carbon doses and dye concentration. The maximum degradation efficiency of MR by UV spectroscopy found to be 82.79% at pH 3, 0.25 g of PRCS, 90 min irradiation time and dye concentration of 20 ppm. Adsorptive and photocatalytic degradation of dye explained by isotherm and kinetic studies. From the results it is clearly evident that PRCS could be used as an ecofriendly photocatalyst for the removal of dyes from waste water.

Keywords: Degradation, Methyl red dye, Activated charcoal, *Ricinus communis*, PRCS.

INTRODUCTION

Textile effluent contain large amount of synthetic dyes. These dyes are mixed with soil and water resource that are carcinogenic, toxic and neurotoxic in nature[1]. Discarding the dyes in water source is a difficult one because they are very sturdy and they are very soluble in water. If the waste waters are not managed properly, they will harmful to the environment. Dye effluent containing variety of dyes. Methyl red used in textile dyeing, paper printing and as an indicator in acid base titrations. Discharge of methyl red from various industries cause harmful effects to human beings, plants, animals and the environment. It causes skin irritation and skin sensitization. If inhaled it damages the central nervous system, digestive path irritation, kidney failure and severe depression. In animal it causes severe effect in reproductive and fetal.

The methods for removal of dyes are oxidation/precipitation, nano filteration, electrocatalytic separation, photochemical degradation, degradation using solar light and adsorption[2-4]. Among these photo degradation is the efficient, sustainable method for the removal of dyes from waste water. The solar energy is the potential energy source which is utilized for the degradation of dyes. Nowadays, a several number of adsorbents and photocatalysts have been used for dye degradation. Activated charcoal is more effective because of their surface area, and high capacity[5]. In recent years the agricultural waste is used for preparing activated charcoal and used as a photocatalyst for the removal of dyes. Some examples for agricultural waste was employed for adsorption of dyes such as waste orange peel[6], wheat straw[7], saw dust[8], barely husk[9], and water melon rinds[10], lime peel[11]. In this study *Ricinus communis* [12]stem is used and as a photocatalyst. *Ricinus communis* has the potential for phytoremediation of distillery waste[13]. The endemic of the plant is India and planted throughout the country in gardens and fields and also breed wild in waste place[14-15].

The activation process of a photocatalyst is carried out by either physical or chemical activation process. The properties of the activated carbon can be differed by the precursor, the procedure and process condition. These can perturb the pore sizes, the shape of the pores and their surface[16]. The carbon yield is too and the temperature employed in chemical activation is lesser than that of physical activation[17]. In this work Activated charcoal is prepared from *Ricinus communis* stem (PRCS) by chemical activation method. The chemical activation is processed by using phosphoric acid. The bio adsorbent is used to degrade methyl red dye from aqueous medium and textile waste water under solar light irradiation. The physiochemical properties of adsorbent were determined[18]. The kinetic studies were performed. Freunlich, Langmuir, Tempkin, D-R isotherm were investigated. Column studies, UV spectroscopy are used to know MR degradation on PRCS in textile waste water. The surface morphology of before degradation of Methyl red dye, after degradation of methyl red dye on PCRS activated carbon was reported by EDAX, XRD, SEM. The functional group analysis of *Ricinus communis* stem was identified by FTIR spectroscopy. The importance of this present work is to investigate the Photo catalytic degradation efficiency of PCRS along with isotherm and kinetic models.

EXPERIMENTAL

MATERIAL AND METHODS

Raw material and chemicals

Ricinus communis stem was collected near Saravanampatti, Coimbatore, Tamilnadu. It was washed with water thoroughly. It was dried, powered and stored in air tight container. NaOH, H_3PO_4, Methyl red were purchased from Merck Company.

Preparation of Phosphoric acid activated Ricinus communis stem (PRCS)

6.0 g of *Ricinus communis* stem powder mixed with 12 mL of phosphoric acid thoroughly and kept in a magnetic stirrer for 24 h agitation. The resultant mixture is heated in a hot air oven for 4 h, after 0.5M NaOH is added in a room temperature and the whole mixture is kept for 24 hours. Finally the product is washed, filtered and dried in a hot air oven at 100°C for 5 hours.

Characterization

The physiochemical characteristics are reported. The surface analysis of the adsorbent was determined by SEM, EDX. FTIR spectroscopy was used to find out the functional group where present in the adsorbent. UV spectroscopy was employed to detect the biodegradation of the adsorbent. XRD spectroscopy was applied to find out elemental composition of the adsorbent.

Photo catalytic studies

The photocatalytic degradation of methyl red onto PRCS is studied by taking 50 mL of different dye concentration in a series of 250 mL conical flasks with fixed PRCS dose, the mixture is stirred and agitated in a bench shaker for 10-15 min for the equilibrium between PRCS and adsorbate solution. After equilibrium, the whole content is exposed to sunlight irradiation. The absorbance is measured at regular interval using UV-VIS spectrophotometer 119. The percentage of degradation efficiency is calculated by the following equation.

$$\eta = \frac{Ao\text{-}At}{Ao}$$

Where A_0 is the initial absorbance of the dye and A_t is the absorbance of dye after degradation[19].

RESULTS AND DISCUSSION

Physiochemical Characterization

The physico chemical properties of PRCS were given in Table 1. The higher yield and iodine value indicated that increasing the adsorption ability of the adsorbents[20]. Acidity of adsorbent surface and basicity of adsorbrnt surface was 4.30 mmol/g, 2.95 mmol/g. Acidity and basicity were calculated by Boehm titration method[21]. Boehm titration investigated about the basic and oxygenated acidic surface groups on activated carbon[21]. From this method we conclude that the activated carbon containing phenyl carboxyl groups. This is confirmed by the FTIR analysis results. From the above results t the adsorbent (PRCS) could be employed as an efficient material for the degradation of dyes from waste water.

Table 1: Physiochemical Characterization of PRCS

Parameter	PRCS
Yield	70%
Moisture content	5.81%
Ph	4.82
Acidity of the Surface	4.30 mmoles/gm
Basicity of the surface	2.95 mmoles/gm
pH_{zpc}	3.6
Boehm titration Basic sites	2.30m.eq/gm
Phenolic and Carboxylic groups	1.13
Carboxyl groups	0.73
Iodine value	1876.25 mg/g

Surface Characteristics

Scanning electron microscopic study of PRCS

The physical morphology of the *Ricinus communis* stem was observed at 10μm magnification. The SEM analysis was done by using the instrument JSM 6390 model. The micrograph (Fig. 1) showed that during carbonisation of the *Ricinus communis* stem permeated with H_3PO_4, the evaporative matter develops high pressure, which detonates the integral structure of the particle and generates holes on the surface of the carbon, and also causes the evaporation of acids. During carbonization leaving the area free that was formally occupied by the acids[22].

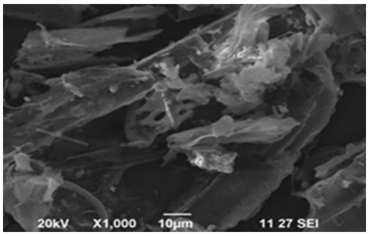

Fig. 1. SEM image of PRCS

Energy dispersive X-ray Spectra (EDX)

In this, EDX spectrum was utilized to confirm the presence of O,Na,C,Ca,K,Zn,P,Cl,Al,S and Si in the adsorbent. The presence of phosphorous in PRCS was due to the impregnation of *Ricinus communis* stem with H_3PO_4[23]. The composition of PRCS is given in the Table 2. EDAX spectra of PRCS are displayed in the Figure 2.

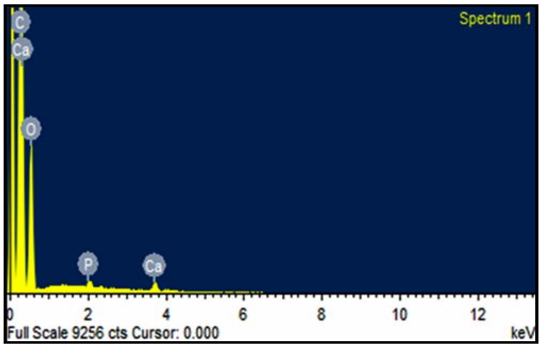

Fig. 2. EDX spectrum of PRCS

Table 2 : Composition of PRCS

Elements	PRCS (Mass%)
C	72.62
O	26.21
P	1.06
Ca	0.11
Na	---
Al	---
K	---

Cl	

X-ray Refractory Diffraction (XRD)

The XRD pattern of PRCS was shown in Fig. 3. The resultant peaks only have broad peaks and no sharp peak. So this study confirmed the amorphous structure of the adsorbent material PRCS[24].

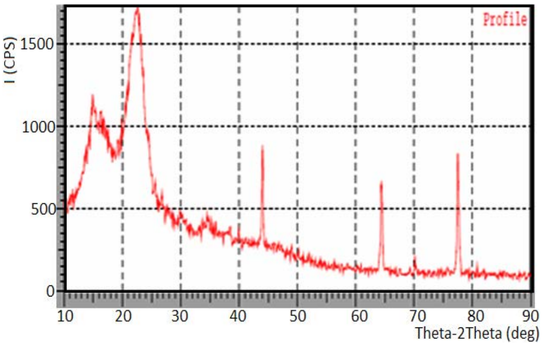

Fig. 3. XRD spectra of PRCS

Fourier Transform and Infrared Spectroscopy

Functional groups in PRCS was analysed

by using FTIR shown in Fig. 4. Using Jasco 460 plus spetrophotometer in the spectral range of 4000-400 cm^{-1} for this studies. The frequency range at 3773.89 cm^{-1} indicated that the appearance of -OH stretching of phenols and alcohols. The peak at 3524.10 cm^{-1}, 3332.17 cm^{-1} confirmed that the existence of hydroxyl ion, -OH stretching frequency. The frequency at 1713.83 cm^{-1}, 1510.35 cm^{-1} shows the presence of C=O stretching, C-O stretching in carbonyl, carboxylic acid and lactones. The frequency around1325.15 cm^{-1}, 1051.25 cm^{-1}, 589.28 cm^{-1} confirmed the C-O vibration, stretching vibration of -OH groups, halide groups.

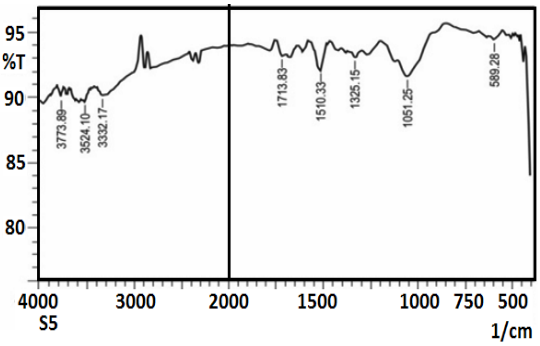

Fig. 4 : FTIR image of PRCS

Photocatalytic studies for the degradation of MR onto PRCS

Effect of contact time

The percentage degradation was found to increase with increasing contact time and then it becomes stable after attainment of equilibrium time as so the maximum degradation achieved at 120 min for PRCS. This may be due to the fact that initially surface of adsorbent sites were highly vacant and solute concentration was high, and after the equilibrium time there was no change in the degradation of MR onto PRCS adsorbent, because the active sites are lesser in the surface of the adsorbent at this stage. The maximum degradation efficiency of MR onto PRCS was 82.79%. The effect of contact time for the degradation of MR onto PRCS is shown in the Figure 5.

Effect of solution pH

For this work, 50 mL of 20 ppm adsorbate solution, 0.2 g of PRCS was taken in the series of conical flask with pH range varying from 2-9 and the whole content kept in a bench shaker for 10 min to attain the equilibrium condition[25]. After the attainment of equilibrium the mixture was exposed to direct sunlight irradiation for the degradation of MR. The absorbance were taken with regular time interval. The degradation efficiency of MR onto PRCS was given in Fig. 6. Maximum degradation of MR dye (82.79%) occurs at the pH of 3, when increasing the solution pH degradation of dye degreases gradually. This is due to the fact that the effect of pH depends on zero point charge of the adsorbent PRCS. The zero point charge of PRCS was 3.6, if the solution pH was below the zero point charge if favours anionic dye degradation since the charge of the composite was positively charged(H$^+$) and if the solution pH was below the zero point charge if favours cationic dye degradation because PRCS was negatively charged(OH-).[23]

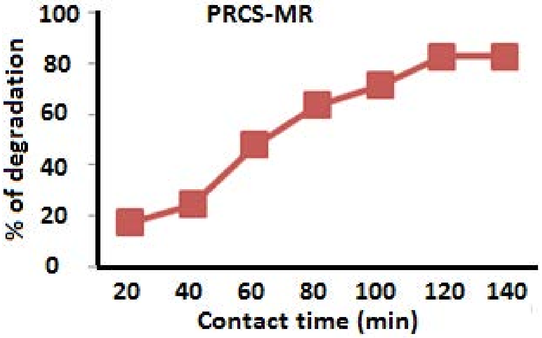

Fig. 5. Effect of contact time for the degradation of MR onto PRCS

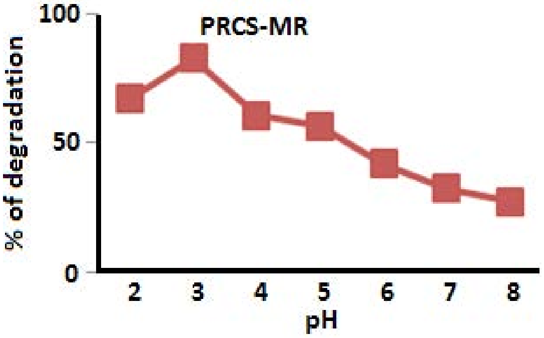

Fig. 6. Effect of pH for the degradation of MR onto PRCS

Effect of adsorbent dose

The effect of adsorbent dose on the degradation of MR dye was investigated and shown in the Fig. 7. There was an increase in degradation efficiency of MR dye while increasing for the dose of PRCS from 0.05 g to 0.25 g at pH 3 and the contact time of 120 minutes. Furthermore addition of adsorbent dose did not show appreciable change in the degradation efficiency of MR. It was confirmed with Figure 7.

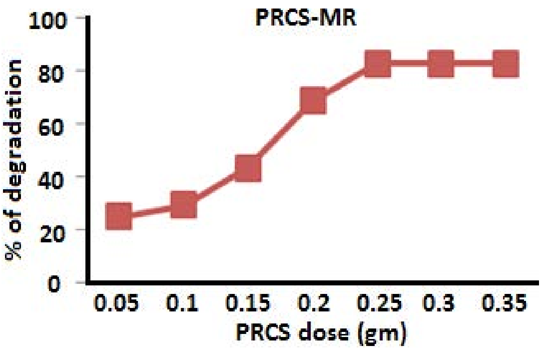

Fig. 7. Effect of PRCS dose for the degradation of MR

Effect of MR dye concentration

The effect of initial dye concentration of MR onto PRCS was studied using initial dye concentration ranges from 20 ppm-140 ppm at pH 3 and the contact time is 120 minutes. The degradation efficiency of MR onto PRCS was decreased from 82.79% to 23.14%. The result was given in the Fig. 8, which indicates the reduce in dye degradation with increase in dye concentration.

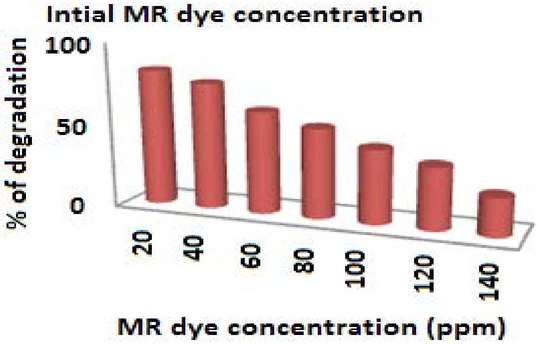

Fig. 8. Effect of initial dye concentration of MR onto PRCS

Conformation analysis for the degradation of MR onto PRCS adsorbent

The Ultraviolet-Visible(UV-Vis) spectra for the decay of MR onto PRCS were shown in Figs.9 and 10. The percentage of degradation of MR was 82.79% onto PRCS. The decolourization and the degradation of MR were confirmed by using UV-Visible spectral analysis. Examination of spectral lines of dyes indicated that the absorbance peaks at initial of the dyes drastically reduced when the contact time increases. The effectiveness of the dye decolourization was connected with the dye chemical structure, molecular weight and the presence of functional groups. Results obtained reveal colour removal by the analysis isolate due to the biodegradation of the dyes rather than adsorption.

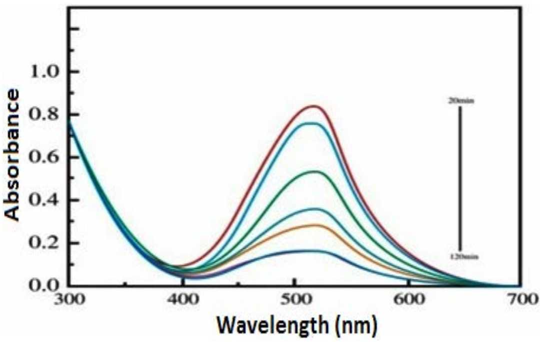

Fig. 9. UV-Spectra of MR onto PRCS

Fig. 10. Before and after treatment of MR onto PRCS

Degradation of Textile effluent using PRCS and charcoal

The degradation process was carried out using PRCS and charcoal for the textile effluent which was collected from Mangalam, Tirupur District. The degradation process was investigated under direct sunlight irradiation at optimum conditions. The degradation efficiency of textile effluent was found to be 24.65% for PRCS and 53.99% for charcoal at a contact time of 120 min, because the increased surface area of the charcoal decays the maximum percentage of organic pollutant than the prepared adsorbents. The comparison of degradation capacities of the textile effluent onto PRCS and charcoal was given in the Table. The UV-absorbance spectrums was also shown in the Figures 11 and 12.

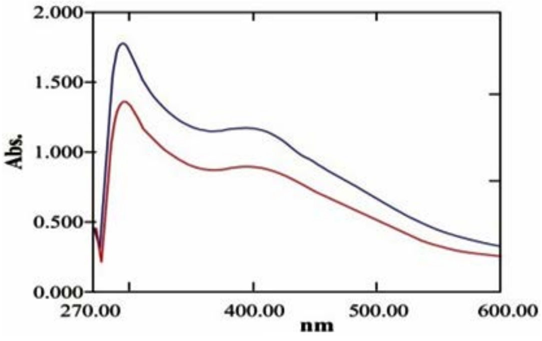

Fig. 11. UV spectra of textile effluent onto PRCS

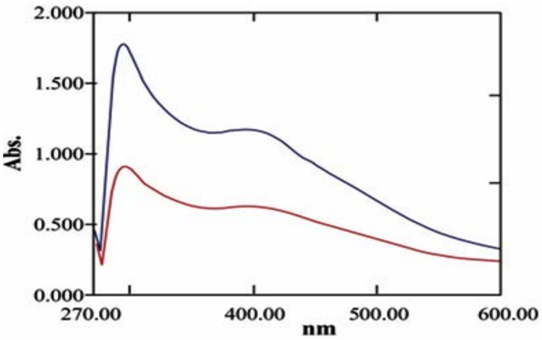

Fig. 12. UV spectra of textile effluent onto Charcoal

Table 3: Degradation capacities of the textile effluent onto PRCS and charcoal

Adsorbent	Stimulated waste water		Effluent	
	Time(min)	Degradation	Time(min)	Degradation
PCRS	120	82.79%	120	24.65%
Charcoal	-	-	120	53.99%

Column studies for the Textile effluent onto PRCS

For adsorption purpose mini-columns was used, each column was packed with a layer of cotton, above the cotton 1 g PRCS was filled with bed height around 20mm. Flow rate of dye was 2 mL per minute throughout the experiments. The concentration of the effluent was detected by measuring their absorbance using UV spectrophotometer. The contact time with a given part of the adsorbent was limited, a true equilibrium was never reached, which was shown in Figure 13.

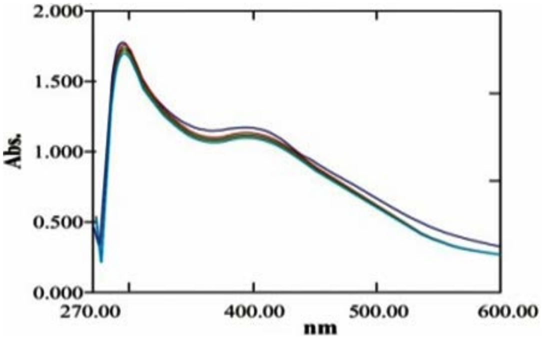

Fig. 13. Column studies for the Textile effluent onto PRCS

Kinetic Studies

The photocatalytic degradation of methyl red dye by PRCS adsorbent is explained by the kinetic studies. The kinetics was analysed by using pseudo I order and pseudo II order models. The correlation regression coefficient R^2 and maximum adsorption capacity qe of the adsorption of MR dye on PRCS are 0.9939 and 18.93(mg/g minute). This is indicated that the adsorption kinetics on MR dye is fitted for pseudo second order kinetics. The kinetic data are given in the Table 4. Kinetic models were detected by using the following formulas which is given below.

Pseudo first order equation $\log(q_e-q_t)=\log q_e-\frac{k1t}{2.303}$ 1

Pseudo second order equation $\frac{t}{q_t}=\frac{1}{K_2}-\frac{t}{q_e}$ 2

Table 4 : Kinetic variables for the Degradation of MR Dye onto PRCS

Kinetic models	Parameters	Data
Pseudo- first order	k_1	0.043 min^{-1}
	q_e	11.36 mg/g
	R^2	0.8777
Pseudo-second order	k_2	0.0013 g/mg min
	H	0.429
	q_e	18.93 cal(mg/g)
	R^2	0.9919

Isotherm studies

In this present studies, Langmuir and Freundlich isotherm models were performed. The experimental equilibrium isotherm data were obtained by changing the concentration of MR with 0.25 g/50 mL is shown in Table 5. Equations for these models were illustrated in equations 3 and 4. The results revealed that the Langmuir Isotherm model is best fit for the equilibrium data than Freundlich Isotherm model.

Langmuir $\frac{Ce}{qe}=\frac{1}{qmax}b+\frac{Ce}{qmax}$ 3

Freundlich $\log q_e = 1/n \log(C_e) + \log K_f$ 4

Table 5: Isotherm parameters for the Adsorption of MR

Isotherm models	Parameters	Data
Langmuir Isotherm	Q_m	8.1234 mg/g
	b	2.25 L/mg
	R^2	0.9951
Freundlich Isotherm	N	2.96
	K_F	3.033 mg/g
	R^2	0.9291

Mechanism for dye degradation

The activated *Ricinus Communis* stem is used as a photo catalyst is the primary reason for the degradation of MR dye, when it is exposed to direct solar light irradiation it gives electron hole pairs. The dye degradation is induced by the formed electron hole pairs. The initial dye concentration is the cause for the photo degradation of dyes. Even though it is dye degradation initially adsorption of the dye on the studied adsorbents occurs followed by photo degradation process. At the initial stage more vacant sites are available on the surface on the adsorbent for adsorbing dye molecule when the concentration of the dye is low, but at the later stages the availability of the vacant sites are less on the surface of the adsorbent for adsorbing dye molecule when the concentration of the dye is high. Because, the vacant sites are filled by the dye molecules therefore the rate of dye degradation decreases.

Mechanism of photo degradation occurs in two steps, i.e., Dye adsorption by adsorbent followed by degradation under direct sunlight takes place by the following equation.

Adsorbent+ hν → Adsorbent ($e^- + h^+$)
$O_2 + e^- \rightarrow O_2^{\bullet}$-
$O_2^{\bullet}$- + e^- + $2H^+ \rightarrow 2OH^{\cdot}$

Oxygen plays a vital role in the degradation of dyes. A sunlight, oxygen and hydroxyl ion shows considerable effects on the degradation. If reduction of any one of the species will decrease the degradation capacity of the dye. The formed hydroxyl radical species react with anything to form hydrogen peroxide, which is a considerable active species in photo degradation process.

$2OH^{\cdot} \rightarrow H_2O_2$

The formed hydrogen peroxide cleaved to form $OH^{\cdot}$

$H_2O_2 + h\nu \rightarrow 2OH^{\cdot}$

Degradation of dye by $OH^{\cdot}$ radical

$OH^{\cdot}_{ads}$ + dye → oxidation of the dye

CONCLUSION

In this paper the degradation ability of PRCS is studied. Chemical activation of *Ricinus communis* stem with phosphoric acid gives high yield and high surface area of activated carbon. The physiochemical properties are investigated. The surface analysis of activated carbon was experimented by SEM, EDX, XRD. The effect of pH, Contact time, Adsorbent dose, initial concentration of dye on Degradation of methyl red were analysed. The maximum degradation efficiency of MR dye on PRCS is 82.79%. The degradation of textile effluent, stimulated waste water onto PRCS were analysed by using sunlight irradiation and column studies. The degradation efficiency of textile effluent was found to be 24.65% for PRCS. The degradation efficiency was confirmed by UV spectra. The kinetic studies were also investigated by varying concentration with contact time. The single component equilibrium characteristics of degradation of MR dye by two different isotherm models were investigated. The experimental equilibrium isotherm data were good agreement with calculated values. This paper concluded using activated carbon of *Ricinus communis* stem is an inexpensive adsorbent for the removal of methyl red dye from waste water.

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Conflict of interest

The author declare that we have no conflict of interest.

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