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# REMOVAL OF FUSCHIN DYE FROM AQUEOUS SOLUTION USING CHITOSAN/MAGNETIC CELLULOSE/SUGARCANE BAGASSE BEADS

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## ABSTRACT

A recent ideal alternative to the current expensive methods of removing the dye from aqueous solution is the use of low cost eco friendly materials as adsorbent. In the present research work, the chitosan/magnetic cellulose/sugarcane bagasse beads were synthesized and utilized as adsorbent for sorptive removal of fuschin dye from aqueous solution in batch mode on laboratory scale. Investigation was done to study the effect of contact time on the adsorption removal efficiencies. Results reveal that around 95% fuschin dye were removed from the aqueous solution and kinetic studies reveals that the pseudo second order kinetic model is followed. Hence from the obtained results it was suggested that the prepared chitosan/magnetic cellulose/sugarcane bagasse beads can be used for the dye effluent treatment at the industrial level.

**KEYWORDS:** Chitosan, magnetic cellulose, sugarcane bagasse, removal, Fuschin dye.

## INTRODUCTION

Dyes are usually considered as synthetic aromatics containing various functional groups. In order to treat finished products, certain chemical dyes were used in many industries. The light penetration in water interfering with water can be reduced by the chemical dyes and also most of them contain suspected carcinogens.[1] Eventhough the dyes has many applications it it is exceeded above a specified limit it will introduce toxicity to human living organisms. These dyes were reported to be harmful to human being causing various health disorders such as allergy, skin irritation, dermatitis, mutations and cancer.[2] As a result of these numerous health disorders, it is necessary to reduce or eliminate these life-threatening compounds from wastewater before it is discharged.

The fuchsin dye belongs to the triarylmethane class dyes which are widely used as a colouring agent for textile, leather materials and also in the staining of collagen, muscle, mitochondria and tubercle bacillus. The removal of fuchsin from wastewater systems is of great concern and this is because of its properties of poor biodegradation, toxicity, carcinogenicity and unsightliness.[3] Numerous conventional treatment methods such as membrane separation, chemical coagulation, activated sludge, biodegradation, oxidation, adsorption and photo degradation for dye removal have been extensively explored.[4][5] Lot of researches were carried out by many scientists to remove the fuschin dye from aqueous solution.[6][7]

When compared to the other physical and chemical processes, the adsorption method has gained importance as a cost effective and highly efficient purification process on an industrial application since it is quite effective in producing high-quality effluent without the formation of harmful substances.[8][9][10] In recent years, the development of economical adsorbents by many researchers to treat dyes in wastewater has attracted great interest. Sugarcane bagasse is the major by-product of the sugar cane industry which contains about 50% cellulose, 25% hemicellulose and 25% lignin.[11] Many researchers reported that the modified form of agricultural product sugar cane bagasse acts as an effective adsorbent.

Handojo Djati Utomo and his coworkers studied about the removal of methylene blue dye using chemically modified sugarcane bagasse. Results indicate that the cellulose rich NaOH treated sugarcane bagasse (BSGB) could better remove organic dyes in neutral alkaline water environment.[12] In the year 2015, the adsorption equilibrium study was investigated to estimate adsorption parameters using Freundlich and Langmuir's isotherm models by Mahalakshmi Mathivanan and Saranathan. Results showed that the maximum specific uptake capacity on sugarcane bagasse was found to be 108.67 mg/g.[13] From these studies, it was inferred that sugarcane bagasse has a good potential to be used for small scale industries.

The biopolymer chitosan possessing certain interesting set of characteristics including non-toxicity, biodegradability, biocompatibility and bioactivity has been extensively investigated as adsorbents by many scientists. The chitosan–montmorillonite composites (CTS–MMT) were synthesized and utilized as an adsorbent for acid fuschin dye removal by Chun Yan Cao and his coworkers. Results reveal that the adsorption processes were better fitted by pseudo-second-order model and the Freundlich model.[14] Zuhair jabbar and his coworkers investigated about the adsorption of congo Red (CR) from aqueous solution onto chitosan in a batch system.[15]

Magnetic cellulose is a composite of nanocellulose and magnetic nanoparticles which are found to be evenly distributed onto the cellulose nanofibrils and produced in an inexpensive single-step process. Magnetic cellulose/graphene oxide composite (MCGO) was prepared as a novel adsorbent to dispose of dye wastewater by Haochun Shi and his coworkers. Results showed that the adsorption efficiency of MCGO was still over 89% after recycling for five times.[16] Ghanbarian and his coworkers revealed about the Potentiality of amino-riched nano-structured $MnFe_2O_4$ cellulose for biosorption of toxic Cr (VI) from aqueous solution. The adsorption isotherm parameters showed that the adsorption of Cr (VI) onto this sorbent occurs in multilayer possessing heterogeneous sorption sites.[17]

Based on literature survey three materials namely chitosan, modified sugarcane bagasse and magnetic cellulose were selected to prepare the ternary blended mixture in the form of bead. In our ongoing research, we have focused on preparing, characterizing and describing the new alternative adsorbent namely chitosan/magnetic cellulose/sugarcane bagasse ternary beads for the best removal of fuschin dye from aqueous solution. The effects of contact time on the adsorptive removal of fuschin dye from aqueous solution was investigated and based on this the removal rate kinetics for fuchsin adsorption onto chitosan/magnetic cellulose/sugarcane baggasse ternary beads was also studied and all the results were discussed.

## MATERIALS AND METHODS

### Materials

Chitosan was kind gift from Indian Sea foods Cochin, Kerala. The raw material sugarcane baggasse was bought from the local sugarcane juice shops of Vellore. The cellulose polysaccharide was purchased from Sisco Research laboratories, Pvt. Limited. Certain chemicals namely orthophosphoric acid, sodium hydroxide, urea, ferric chloride and glacial acetic acid were procured from Nice Chemicals Pvt Limited and Thomas Bakers Chemicals, Pvt Ltd, Mumbai.

**Preparation of chitosan solution**

A vicous homogeneous chitosan solution was prepared by dissolving about 2 gram of chitosan in minimum amount of 3% acetic acid solution.

**Preparation of phosphoric acid treated sugarcane bagasse activated carbon**

Initially the sugarcane baggasse collected from the juice shop was washed thoroughly under running water to remove any leftover sugar and dirt. This washed sugarcane baggasse was then dried in sun for two days and this dried sugarcane bagasse was cut into fine pieces (0.2 inches in size) and then soaked in 25% orthophosphoric acid for a period of 4 hrs. After 4 hours is over, it is thoroughly washed with distilled water until it reaches the neutral pH. It is then kept for drying process in an electric oven at 120°C for three hours and followed by this treatment, these dried samples were pyrolysed at 300°C for 3 hours in the muffle furnace. The product carbon which is obtained named as phosphoric acid treated sugarcane bagasse activated carbon was cooled, powdered using mortar and pestle and stored in a tight container until further use.

**Preparation of magnetic cellulose**

About 2gm of cellulose were dispersed into a pre-cooled solution of 8 g of NaOH, 80 mL of distilled water and 12 g of urea. In order to get the clear transparent solution, the above prepared mixture was then stirred for 5 min and stored in a freezer for one hour.[18] By utilizing the coprecipitation method, the magnetic cellulose was prepared as follows: About 4.5 g of Ferric chloride and 2 g of manganese nitrate with 2:1 mole ratio were dissolved in 100 ml double distilled water. It was then stirred for a period of 5 minutes at room temperature. The initially prepared dissolved cellulose mixture was then added to the 2:1 ratio mixture (ferric chloride: manganese nitrate) dropwise under constant stirring and followed by this process, this complete solution mixture was then placed in ultrasound bath and sonicated for 60 min at 80°C. The final obtained dark precipitate was then separated by filtration, washed several times with distilled water and dried at 70°C for 24h.

**Preparation of chitosan/magnetic cellulose/ sugarcane bagasse beads (4:1:1 ratio)**

In order to prepare the ternary chitosan/magnetic cellulose/ sugarcane bagasse beads, about 0.5 g of the above prepared magnetic cellulose and 0.5 g of the above prepared phosphoric acid treated sugarcane bagasse activated carbon is added to the above prepared homogenous viscous chitosan solution. This ternary solution mixture was then mixed well using magnetic stirrer by complete stirring process for half an hour. Followed by this complete stirring process, this emulsion like appearance of solution mixture was then poured as small droplets into 100 ml of 5% sodium hydroxide. The ternary beads so obtained was then allowed to stay for some time (15 mins) and the filtered, washed with water, dried and then stored for further analysis. A photograph of the chitosan/magnetic cellulose/ sugarcane bagasse ternary bead was represented below (Fig-A).

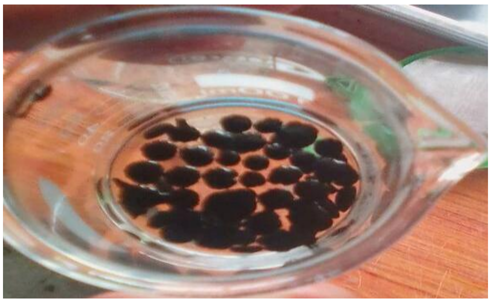
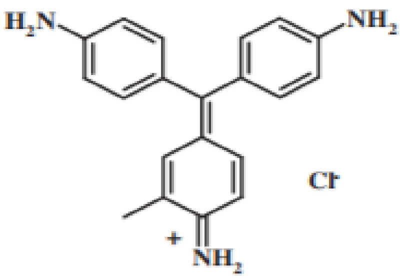


**Fig. A: Photograph of the chitosan/magnetic cellulose/ sugarcane bagasse ternary bead.**

**Batch Adsorption studies**

**Experimentation**

The adsorption experiments were carried in a batch process at room temperature by using an aqueous solution of Fuschin dye. The chemical structure of fuchsin treated as cationic dye is presented below.




**Chemical structure of Fuchsin**

In order to prepare stock solution, dissolve 1 g of Fuschin dye in 100 ml of distilled water (1000mg/L). The above prepared 1000 mg/L stock solution was suitably diluted to the required initial concentration (200ppm). An accurately weighed amount of chitosan/magnetic cellulose/sugarcane baggasse (adsorbent) (0.1g) was added to 100ml of the fuschin dye solution (adsorbate) taken in stoppered conical flask. This solution mixture was then shaken well in orbitrary shaker for a definite time (60minutes) at a room temperature. After the completion of shaking process, the above mixture was filtered and the filterate collected was then analysed for the fuschin dye adsorption using the photoelectric colorimeter technique ie the concentration of the dye remaining after adsorption process was determined at 480 nm, using photoelectric colorimeter (Perkin-Elmer Lambda 25). The biosorption studies were carried out with the same procedure in batch mode to identify the effect of contact time (60 min-300 min) on the biosorption of fuschin dye. The percentage of dye removal was calculated by using following equation respectively

$$\text{Removal } (\%) = \frac{C_o - C_f}{C_o} \times 100$$

Where $C_o$- Initial concentration of fuschin dye solution (mg/L) and $C_f$ - Final concentration of fuschin dye solution (mg/L).

**Characterization**

**FT-IR Spectral analysis**

Perkin Elmer 200 FT-IR spectrophotometer were utilized in the present research work to record the FT-IR spectra of prepared samples in the wave number range of 500-4000cm$^{-1}$ during 64 scans with resolution of 2 cm$^{-1}$.

## RESULTS AND DISCUSSION

**Fourier transform IR spectroscopy**

Fourier transform infrared (FTIR) spectroscopy is a powerful analytical tool for identifying chemical constituents and according to the vibrational modes of their molecular functional groups this technique helps to elucidate compound structures in various forms in real-world samples. The identity of pure compounds can be confirmed from the unique collection of absorption bands in FT-IR spectrum and also in addition the presence of specific impurities can be detected from this technique. In order to determine the existence of interactions among the components in chitosan/magnetic cellulose/ sugarcane bagasse ternary bead, the FTIR spectra of pure chitosan and chitosan/magnetic cellulose/ sugarcane bagasse ternary bead were recorded and analyzed respectively. The FT-IR spectral details of pure chitosan was shown in Fig.1.

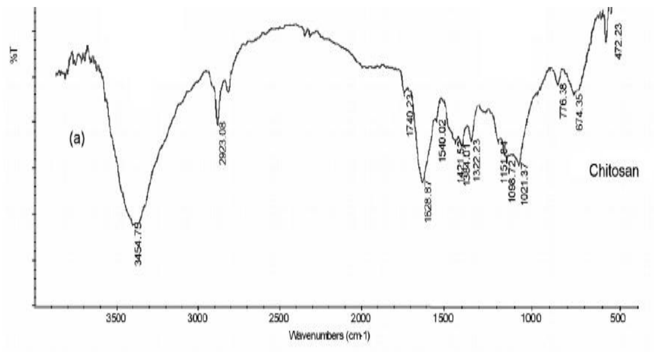



**Fig. 1: FT-IR spectrum of pure chitosan.**

The FT-IR spectrum of pure chitosan shows a broad band at 3454.75 $cm^{-1}$ which is due to the OH stretching (Esam A El-Hefian et al., 2010). The band at 1628.87$cm^{-1}$ is assigned for C=O stretching (amide-I band) O=C-NHR while the small peak obtained at 1540.02 $cm^{-1}$ corresponds to N-H bending.[19] The bands at 2923.08 $cm^{-1}$, 2884.23 $cm^{-1}$ and 1421.52 $cm^{-1}$ were attributed to the $CH_2$ bending due to pyranose ring.[20] The OH in plane bending band of alcoholic group present in the chitosan can be observed at 1384.01 $cm^{-1}$. The skeletal vibrations involving the C-O-C stretching bands was observed at 1151.84 $cm^{-1}$ and 1098.72 $cm^{-1}$ (C-O-C bridge as well as glycoside linkage).[21] Certain absorption bands obtained at 1021.37 $cm^{-1}$, 674.35 $cm^{-1}$ and 472.23 $cm^{-1}$ was assigned to the C-C stretching, NH wagging and C-C bending vibrations.

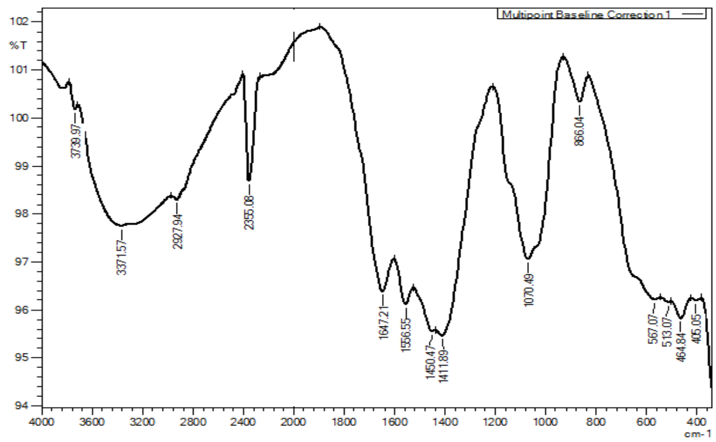



**Fig. 2: FT-IR spectrum of chitosan/magnetic cellulose/ sugarcane bagasse beads.**

The FT-IR spectrum of the chitosan/magnetic cellulose/ sugarcane bagasse ternary beads showed nine major peaks at the ranges of 3371.57 $cm^{-1}$, 2927.94 $cm^{-1}$, 2355.08 $cm^{-1}$, 1647.21 $cm^{-1}$, 1556.55 $cm^{-1}$, 1450.47 $cm^{-1}$, 1070.49 $cm^{-1}$, 567.07 $cm^{-1}$ and 513.07 $cm^{-1}$. Broad absorption band appeared in the range of around 3371.57 $cm^{-1}$indicate the presence of intermolecular hydrogen bonded OH stretching in alcohols. Furthermore, there was one characteristic absorbance band located at 2927.94 $cm^{-1}$, which corresponds to the stretching vibrations of the −C−H, which are methyl, methylene, and methane of the cellulose. Certain strong absorption bands observed at 2355.08 $cm^{-1}$, 1647.21 $cm^{-1}$, 1556.55 $cm^{-1}$, 1450.47 $cm^{-1}$ was attributed to $NH_3^+$ stretching, carbonyl stretching and –CH bending. The wide band at about 1070.49 $cm^{-1}$ appears to conclude the presence of amorphous silica (asymmetric stretching vibration of Si (Al)–O–Si bonds).[22] Additionally, peaks belonging to carbonate anion (from calcite) were also identified: 1450.47 $cm^{-1}$ (asymmetric stretching vibration of $CO_3^{2-}$ anion) and 866.04 $cm^{-1}$(out-of-plane bending mode of $CO_3^{2-}$).[23] The sharp peak around 567.07 $cm^{-1}$ might be attributed to the symmetric stretching of the band of Si-O-Si and 464.84 $cm^{-1}$ band of the O-Si-O (quartz).

From the observed results, it was evident that when compared to pure chitosan, in case of chitosan/magnetic cellulose/sugarcane baggase ternary beads certain additional bands were appeared due to the stretching vibrations of the −C−H, which are methyl, methylene and methane of the cellulose, asymmetric stretching vibration of Si (Al)–O–Si bonds and asymmetric stretching vibration of $CO_3^{2-}$ anion. The appearance of these new peaks concluded that the three components namely chitosan, magnetic cellulose and sugarcane baggase were blended effectively in ternary bead formation.

## Batch Adsorption Studies

### Factors influencing the adsorption of fuschine dye

In the present study, the potentiality of chitosan/magnetic cellulose/sugarcane bagasse ternary bead as adsorbent for the removal of fuschin dye from aqueous solution as a function of contact time has been studied and the results were investigated below in detail.

### Effect of contact time on the adsorption of chromium

The effect of contact time for the removal of fuschin dye by chitosan/magnetic cellulose/ sugarcane bagasse ternary bead is as shown in fig. 3. It is observed that the dye is rapidly adsorbed in the first 60min up to 80% and then adsorption rate decreased gradually and reached equilibrium in about 90 min upto 98%.[24] Further agitation beyond this time (90 minutes) did not lead to any increase in the adsorption of the dye and on the basis of this result it was evident that the optimum contact time was found to be 90 minutes for treating 100ml of 200 ppm fuschin dye solution with removal efficiency of around 98%. From Fig. 3, it was observed that initially due to a larger surface area of the ternary bead, the percent fuschin dye removal is higher for the adsorption of fuschin dye but after a certain period of time (>90 minutes) as the surface adsorption sites become exhausted, the increasing contact time didn't provide any changes in further percent removal of fuschin dye and this might be due to the attainment of equilibrium.

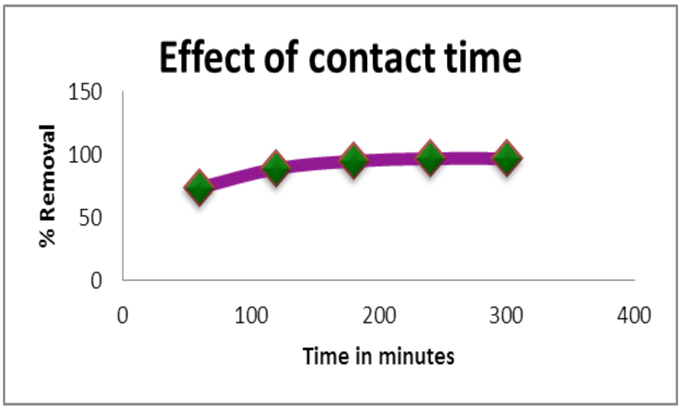



**Fig. 3: Effect of contact time on the adsorption of Fuschin dye removal from aqueous solution using chitosan/magnetic cellulose/sugarcane baggasse bead.**

### Kinetic studies

In the present research work, two kinetic models namely Lagergren pseudo first-order kinetic model and pseudo second order kinetic model has been studied to investigate the chemical kinetics of solid liquid adsorption process.[25] The kinetic measurements was carried out by agitation of 100 mL solutions of Fuschin dye with initial concentrations 200 mg/L and 0.1 g of each adsorbent at different time intervals from 30 to 150 min. In order to determine the adsorption process of fuschin dye onto prepared chitosan/magnetic cellulose/sugarcane baggasse bead, the Larergren model has been effectively used in this work**.**

### Pseudo first order kinetic model

The expression for pseudo-first-order rate equation is given as follows.[26]

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} \text{ ------(1)}$$

where $q_e$ – adsorption capacity at equilibrium (mg/g)

$q_t$ – adsorption capacity after certain time 't'(mg/g)

$k_1$ is the rate constant of the pseudo-first-order adsorption process ($min^{-1}$).

The plot log ($q_e$ - $q_t$) versus time (t), give the respective values of $k_1$ and $q_e$ from slope and intercept values. The pseudo first order kinetic plot for fuschin dye removal from aqueous solution using chitosan/magnetic cellulose/sugarcane baggasse bead was shown in Fig.4.

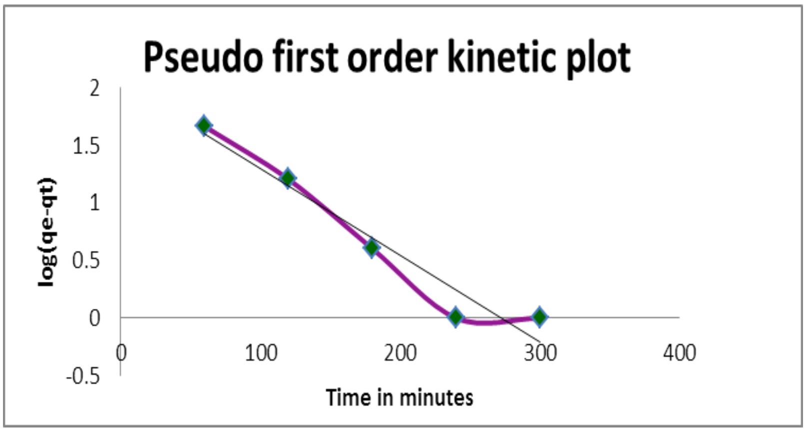



**Fig. 4: Pseudo-first-order sorption kinetic plot for adsorption of Fuschin dye removal from aqueous solution using chitosan/magnetic cellulose/sugarcane baggasse bead**

**Pseudo second order kinetic model**

For the full-scale batch process, the kinetics of adsorption of an adsorbate by any adsorbent is required for selecting optimum operating conditions.[27] In order to model the adsorption of adsorbate by adsorbent surface, the pseudo-second order kinetics equation has been often successfully used in many works.[28] Pseudo second order kinetic model rate equation is expressed as follows:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e} \quad \text{----(2)}$$

Where h = $k_2 q_e^2$ (mg $g^{-1}min^{-1}$) can be regarded as the initial adsorption rate as *t* tends to 0;
$k_2$ is the rate constant of second order rate constant of adsorption process (g $mg^{-1}min^{-1}$)
$q_e$ is the amount of fuschin dye adsorbed at equilibrium.

Pseudo second order kinetic plot has been drawn by taking t/qt along y axis and time in minutes along x axis. From the slope and intercept of straight line plot of t /$q_t$ versus time (t), the values of second order rate constant ($k_2$) and the equilibrium adsorption capacity ($q_e$) were calculated respectively. The pseudo second order kinetic plot obtained for the removal of fuschin dye from aqueous solution was represented in Fig.5.

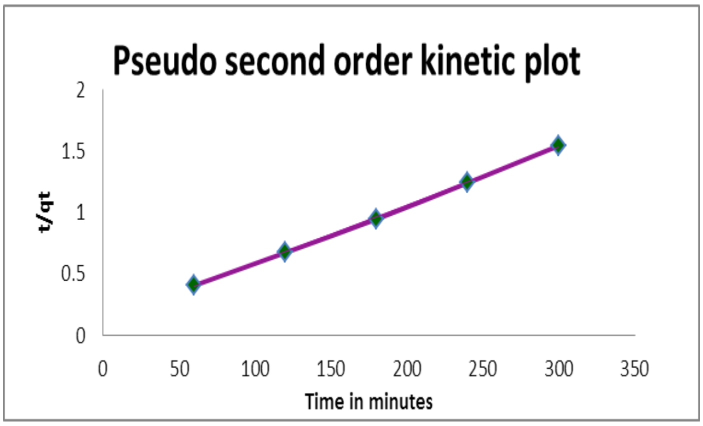



**Fig. 5: Pseudo-second-order sorption kinetic plot for adsorption of Fuschin dye removal from aqueous solution using chitosan/magnetic cellulose/sugarcane baggasse bead.**

From the plots represented in above Fig.4 and Fig.5, values of the pseudo-first-order rate constant, pseudo-second-order rate constant and the equilibrium adsorption capacity has been determined. The calculated values of pseudo-first-order rate constant, pseudo-second-order rate constant and the equilibrium adsorption capacity along with the corresponding correlation coefficients was presented in Table-1.

**Table-1: Comparison between Lagergren pseudo-first-order and pseudo-second-order kinetic models for Fuschin dye removal from aqueous solution using chitosan/magnetic cellulose/sugarcane baggasse bead.**

| Sample | Pseudo-first-order kinetic model | | | Pseudo-second-order kinetic model | | |
|---|---|---|---|---|---|---|
| | $q_e$ (mg/g) | $k_1(min^{-1})$ | $R^2$ | $q_e$ (mg/g) | $k_2$ (g $mg^{-1}$ $min^{-1}$) | $R^2$ |
| Chitosan/ magnetic cellulose /sugarcane baggasse beads | 112.90 | 0.1727 | 0.9453 | 208.33 | 0.00021 | 0.9992 |

From the above obtained results it was evident that, pseudo second order kinetic model yield a very good straight line when compared to the pseudo first order kinetic model and this was concluded from the obtained corresponding higher correlation coefficient ($R^2$) value for the pseudo second order model (correlation coefficient, $R^2$ = 0.9761) when compared to the pseudo first-order model (correlation coefficient, $R^2$ = 0.7825). Hence from the above obtained results, it has been shown that in case of the adsorption of fuschin dye onto chitosan/magnetic cellulose/sugarcane baggasse bead, the pseudo-second-order model provided a better fit indicating that the process is controlled by chemisorption when compared to pseudo first order kinetic model.

## CONCLUSION

Batch adsorption experiment was carried out to determine the feasibility of using chitosan/magnetic cellulose/sugarcane baggasse bead to remove fuchsin dye from aqueous solution. Results indicate that the prepared modified chitosan/magnetic cellulose/sugarcane baggasse ternary bead has shown higher adsorption efficiency for the Fuschin dye which resulted in 97% removal. The evaluated results of effect of contact time on the adsorption efficiency indicate that the optimum contact time of around 90 minutes itself is enough to treat around 97% of the wastewater and kinetic studies reveals that the pseudo second order kinetic model shows a better fit. The overall results conclude that the in the near future, the dye contaminated wastewater can be treated with suitably prepared modified chitosan/magnetic cellulose/sugarcane baggasse bead.

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