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Modification of corn stalk for high-performance adsorption of Coomassie Brilliant blue dye in simulated polluted water: kinetic study

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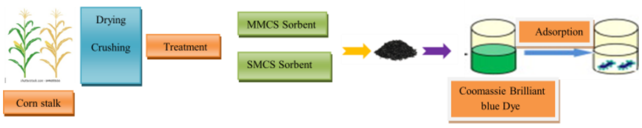
Graphical abstract



Abstract

In the present work, corn stalk was used as a low cost adsorbent for removal of anionic dye (Coomassie Brilliant blue "CBB"). Corn stalk was treated with two methods to inform Modified corn stalk with magnetic particles (MMCS) and Acid hydrolysis of corn stalk using sulfuric acid (SMCS). Comparison between raw and treated corn stalk in dye removal was done. The effect of various parameters as dye concentration (5 to 25mg/L), particle size (4mm-125µm), adsorbent dose (1 to 10g/L), shaking rate (100 to 250rpm), Temperature (15 to 45°C) and pH (3 to 11) were investigated versus time interval. The results summarize that the highest % removal of dye was 85% by using raw corn stalk followed by SMCS about 80% and 50% for MMCS. Different kinetic models were applied; the results suggested that adsorption of CBB dye for raw and treated corn stalk follows the pseudo second order model.

Keywords: Coomassie Brilliant blue, anionic dye, kinetic, adsorption, corn stalk.

1. Introduction

Water is essential for the survival of all living organisms. Today contamination of freshwater systems with a large variety of pollutants could be a subject of great concern. Out of all the contaminants present in industrial effluents, dyes are a very important class of pollutants and might be identified by even human eye. Dyes are used as coloring agents in an exceedingly type of industries like textiles, food, paper, rubber, plastics, cosmetics, leather, etc.

The discharge of wastewater from these industries to resources of water causes unavoidable problems because of the toxic and unsightly nature of dyes. The presence of dyes in water in trace amount is undesirable because it has negative effect on environmental and human health due to most of them are toxic, mutagenic and carcinogenic (Soni *et al.*, 2012), and cause severe damage to the central system, systema digestorium and liver of human bodies (Tanga *et al.*, 2019), even at low concentrations. Several techniques like flocculation, adsorption, oxidation, electrolysis, biodegradation, ion-exchange, photo catalysis are employed for the removal of dyes from wastewater. Amongst the varied techniques, adsorption has received considerable attention because of its several advantages in terms of cost, easy operation, flexibility and ease of design and insensitivity to toxic pollutants (Crini, 2005; Rafatullah *et al.*, 2010).

Adsorption can be defined as an important surface phenomenon usually describes the attachment of particles (ions, atoms, and molecules) either from the gas phase or from the solution on the surface of a solid material.

A good adsorbent should possess large surface area containing many active sites. Figure 1 shows the mechanism of adsorption as it occurs in three steps (Kannan and Sundaram, 2001).

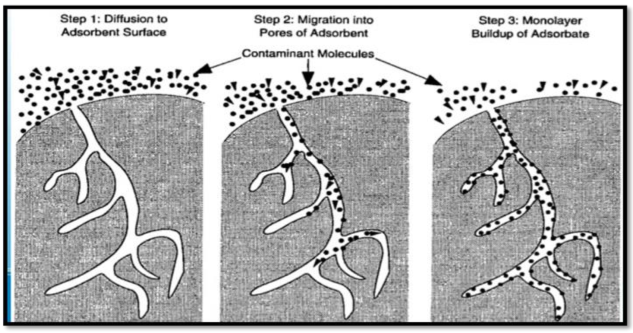


Figure 1. Adsorption mechanism (Kannan and Sundaram2001)

Adsorption process can be classified as physical or chemical adsorption. They are different in: Adsorption of Molecules, Selective process, Formation of layers of adsorbate molecules, Adsorption rate relation with increasing temperature (Lee *et al.*, 2006).

Agricultural and industrial sectors dispose of large amounts of untreated waste, which may pollute the land, water and air, as a result damage the ecosystem. Within the last few years many ideas have been introduced in order to properly dispose of these wastes, such as intensive use as adsorbents. Pollutant removal especially for dye removal where it showed high adsorption capacity.

Agriculture waste is principally composed of three structural components: lignin, cellulose and hemicellulose. The three components have different structures and polarities, in order that they yield different ends up in the method of chemical reactions. The solidity of the three compounds is additionally different when the biomass is treated with acid or alkali solution. As an example, the treatment of biomass with NaOH or KOH can effectively dissolve lignin and hemicellulose; as a result, it may be expected that certain pores are formed when some components are dissolved. Many low cost adsorbents like jack fruit peel (Hameed, 2009a), garlic peel (Hameed, Ahmad, 2009), zeolite (Hor *et al.*, 2016), pine apple stem (Hameed *et al.*, 2009), corn cobs (Reddy *et al.*, 2016), longan shell (Wang *et al.*, 2016), hazelnut shell (Dogan *et al.*, 2009), spent tea leaves (Hameed, 2009b), are reported in literature for the adsorption of dyes. As the plant biomass may be a natural renewable source that may be converted into useful material and energy (Klass, 1998). Corn stalks are agricultural by-products and are currently of no value. Disposal of residue from agriculture is currently a serious economic and ecological issue. However, the abundance and availability of corn stalk as agricultural by-product make them good sources of raw materials for lots of uses, and converting it to adsorbents like carbon represents a possible outlet. Egypt annually produces 3.12 million a lot of corn stalk by-product. Recently, there are several reports on the economic removal of dye.

Bio adsorbents such as rice husk (Ramakrishna and Viraraghavan, 1997), waste coir pith (Klass, 1998) mahogany sawdust, rice husk (Scurlock *et al.*, 2000) orange peel (Shin *et al.*, 1989), bagasse pith (Faust and Aly, 1983), barley husk (Weber Jr, 1972), and banana pith (Ghosh and Bhattacharyya, 2002), banana peels (Munagapati *et al.*, 2018) etc., which have been found to be highly effective, cheap and eco-friendly. New economical, easily available and highly effective adsorbents are still needed. Conversion of corn stalk to a value-added product such as adsorbent will help to solve part of the problem of waste management in Egypt (Husseien *et al.*, 2009).

Aim of this work was Studying the possibility of using corn stalks as adsorbent materials for removal of anionic dyes. Also modification of corn stalks adsorbent by nano magnetic particles and, acid hydrolysis using sulfuric acid was done and study the effect of this on the adsorption process. The influence of different parameters, for example contact time, pH solution, adsorbent dosage, initial dye concentration and temperature, was evaluated in a batch experiment. The kinetic study was fitted by different models, namely pseudo-first-order, pseudo-second order.

2. Materials and methods

2.1. Adsorbate material

Coomassie Brilliant blue dye (CBB) is an anionic dye (Acidic) with λ_{max}=550nm, color index=42660, chemical formula=$C_{45}H_{44}N_3NaO_7S_2$ and M.W=825.97g/mol, as in Figure 2 (Sadia *et al.*, 2012).

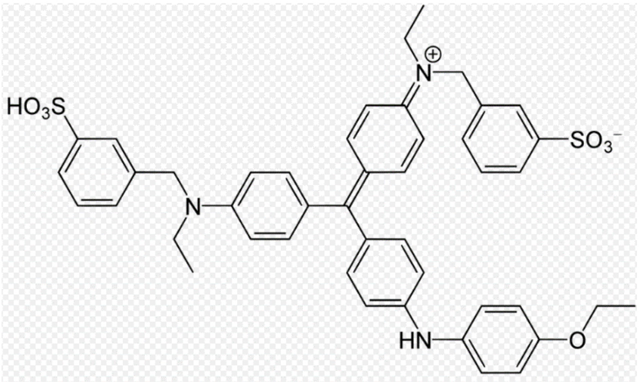


Figure 2. Coomassie Brilliant blue chemical structure (Sadia *et al.*, 2012)

The stock solution was prepared by dissolving 1g of dye in 1000ml of distilled water, the experimental solutions was prepared by diluting the stock solution.

2.2. Adsobent material (corn stalk)

Corn stalk is the selected adsorbent used in this work, it was collected from farms at harvest time and be stripped to remove outer layers then wash to remove any undesirable matters, cut and dried in sunlight then in oven at 80 ^{0}C to remove any humidity. Crush the dried cut corn stalk and sieved to get different particle sizes range from 125µm to 4mm.

2.3. Experimental methods and measurements

2.3.1. Modification of corn stalk with magnetic particles

2.1g of $FeSO_4.7H_2O$ and 3.1g of $FeCl_3.6H_2O$ were dissolved in 80ml of distilled water, the solution was heated to 80˚C with vigorous stirring. Then add 10mL of ammonia with concentration of 25% for complete growth of nano particle crystals. 10g of 250µm corn stalk was added to previous solution with continuous stirring at 80˚C for 30min. let the solution to be cooled and settled then washed with distilled water (Garg *et al.*, 2003).

2.3.2. Modification of corn stalk using acid hydrolysis

Mixing 1:2 (w/w) corn stalk (250µm) to sulfuric acid (98%), stirring manually then left it for 24hr, after that add sufficient amount of distilled water, shake well and then filtrate, this process was repeated until pH reaches 6, then dried the dehydrated corn stalk for 24hr at 80 ^{0}C (Dursun *et al.*, 2007).

2.4. Batch adsorption

0.1g of adsorbent was added to 50ml of different dye concentration solutions; shake the solution at 200rpm for 3 hours at room temperature. The samples were withdrawn at different time intervals, then analyzed using UV/Vis (Ultrospec 2000 - Pharmacia Biotech) spectrophotometer at wave length of 550nm.

The amount of adsorption was calculated using the following equation:

$$q_e=(C_0 - C_e) * V/W \quad (1)$$

C_0 and C_e (mg/L) are the initial and equilibrium concentrations of the CBB dye, respectively, *V*(L), the volume of CBB dye solution, and *W*(g), the weight of dry sorbent used.

The removal efficiency was calculated according to the following equation:

$$\% Re=(C_0-C)/C_0 * 100 \quad (2)$$

Where C_0 and C (mg/L) are the initial and concentration after adsorption at different time intervals (Abu-Saied and Nahla, 2020).

3. Results and discussion

3.1. % Removal of dye by raw corn stalk

3.1.1. Initial concentration

Different CBB dye concentration from 5 to 25mg/L was tested using raw corn stalk (RCS) as shown in Figure 3. By increasing the dye concentration the % removal increase to reach 85% at 10mg/L as the driving force of mass transfer increased which cause % removal increase after that % removal start to decrease to reach 43% at 25mg/L, this behavior may be due to the saturation of adsorption sites on the adsorbent surface. While by increasing time there is a slight increase at the total time interval as the highest value of % removal was at the first 60min.

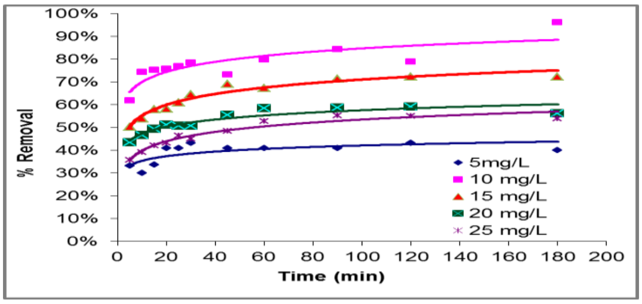


Figure 3. Effect of initial CBB dye concentration on % removal at 25 C, 200rpm.250µm particle size, 2g/L RCS

3.1.2. Particle size

Figure 4, showed the effect of using different RCS particle size ranging from (4mm-125µm). By decreasing the particle size the % removal increased, this may be due to increase of surface area which provided more active sites for adsorption process to take place to reach maximum value at 250µm with value of 72%. With further decrease in particle size, 125µm the % removal decreased. This can be refers to that fine particle convert the adsorbent to emulsion in solution which decrease the active sites available for dye adsorption. Increasing experimental time than 60min has useless effect.

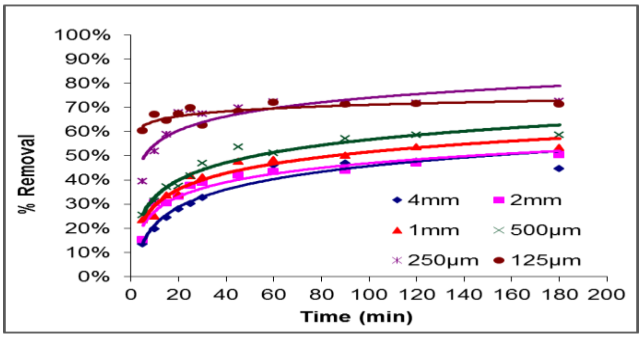


Figure 4. Effect of particle size on % removal of CBB dye at 25°C, 200rpm, CBB concentration of 10mg/L, 2g/L RCS

3.1.3. Adsorbent dose

Different adsorbent dose of RCS are studied as shown in Figure 5. The dose range from 1 to 10g/L, by increasing the adsorbent dose the % removal increased to reach maximum value at 4g/L after that the % removal started to decrease. This behavior can be shown at lower adsorbent dose the adsorbate is more easily accessible while by increasing the dose more than 4g/L the % removal decrease due to accumulation of adsorbent particles which decreasing the active sites for dye removal. The best time interval for highest % removal for different adsorbent doses was 60min.

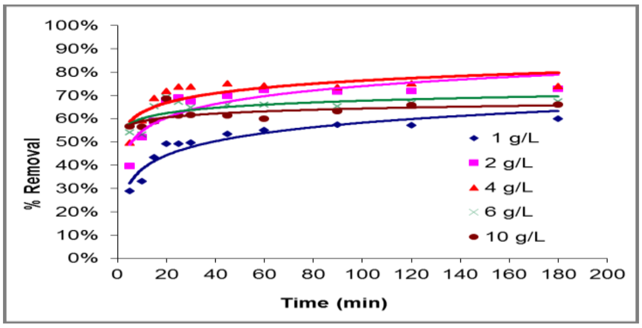


Figure 5. Effect of RCS dose on % removal of CBB dye at 25°C, 200rpm, CBB concentration of 10mg/L, particle size of 250µm

3.1.4. Shaking rate

Figure 6 illustrated the change in stirring speed from 100 to 250rpm comparing with static mode. By increasing the shaking rate the % removal increased to reach maximum value 68% at 200rpm after only 30min then started to decrease to minimum value at 250rpm. The increase of rpm cause decreasing of boundary layer resistance of the transfer adsorbate molecules from the bulk solution to the adsorbent surface, so the diffusion increased with increasing rpm.

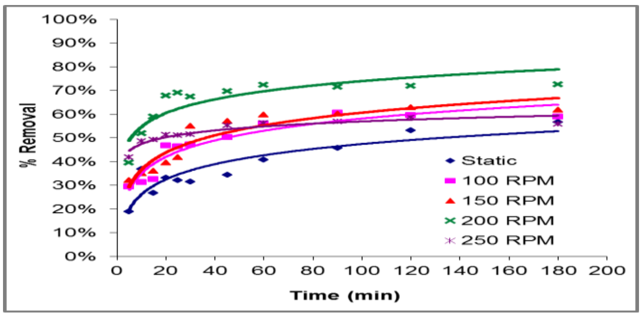


Figure 6. Effect of shaking rate on % removal of CBB dye at 25°C, CBB concentration of 10mg/L, 2g/L RCS, particle size of 250µm

3.1.5. Temperature

Figure 7 showed the effect of changing temperature from 15 to 45°C. It is obvious that the % dye removal increased from 37% to 70% at 25°C after 60min. The decrease of adsorption capacity with increasing temperature than 25 °C indicated that the adsorption was an exothermic process. Increasing temperature caused decrease in the adsorptive forces between the dye species and the active sites on the adsorbent surface resulting of decreasing adsorption capacity.

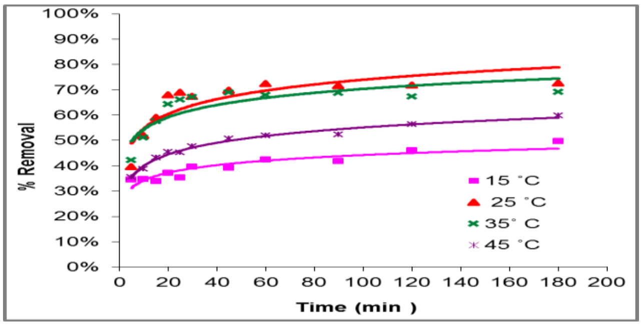


Figure 7. Effect of temperature on % removal of CBB dye at 2g/L RCS, CBB concentration of 10mg/L, particle size of 250µm, 200rpm

3.1.6. pH

The effect of changing pH from 3 to 11 is shown in Figure 8. The figure showed that by increasing the pH value the % removal decreased, as at pH=3 the % removal was 83% after 30min while it was 50% for pH=11. At low pH, the positive charge on the solution interface will increase and the adsorbent surface appears positive charged, which results in an increase in anionic dye adsorption (Ahmad *et al.*, 2020).

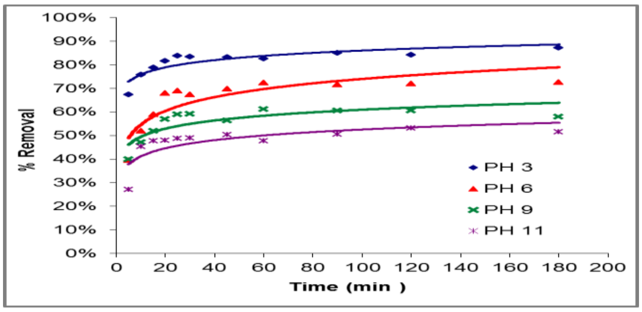


Figure 8. Effect of pH on % removal of CBB dye at 25°C, CBB concentration of 10mg/L, 2g/L RCS, particle size of 250µm, 200rpm

3.2. % Removal of dye by modified corn stalk

3.2.1. Adsorbent dose

Figure 9 illustrated the relation between % removal and time for different adsorbent dose from 1 to 6g/L using corn stalk modified by magnetic particles. The figure showed the same behavior for RCS as by increasing the adsorbent dose the % removal increased and the maximum value was for 4g/L equal 53% after 20min.

While Figure 10 showed the effect of changing dose of acid hydrolyses corn stalk using sulfuric acid" SMCS" on % removal of CBB from the solution. It is cleared that when dose increased from 1g/L to 2g/L as the % removal increased from 37% to 67% after 30min. after that (at 4-10g/L) the increasing of dose has insignificant behavior.

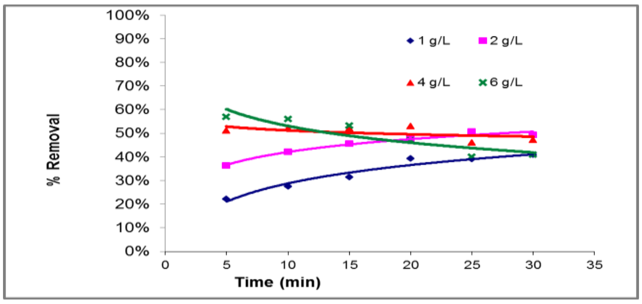


Figure 9. Effect of MMCS dose on % removal of CCB dye at 25°C, CBB concentration of 10mg/L, particle size of 250µm, 200rpm

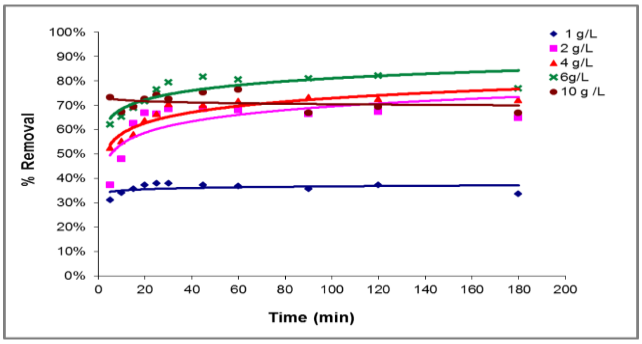


Figure 10. Effect of SMCS dose on %Removal of CBB dye at temperature 25°C, CBB concentration of 10mg/L, particle size of 250µm,200rpm

3.2.2. Agitation rate

Effect of agitation rate was studied in Figure 11. By changing the static to dynamic mode, changing the rpm from 100 to 200rpm the % removal increased to reach the maximum value at 200rpm to reach 50% after 30min.

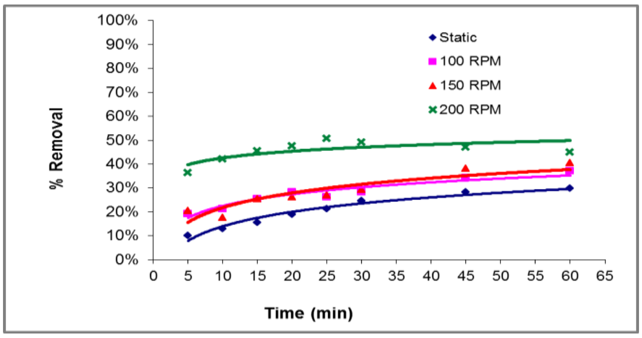


Figure 11. Effect of agitation rate on % removal of CCB dye at 25°C, CBB concentration of 10mg/L, 2g/L MMCS, particle size of 250µm

Figure 12 showed the similar trend for RCS and MMCS in the effect of shaking rate on % removal of CBB to reach the maximum value at 200rpm after 30min which equal 67%.

3.2.3. Temperature

Figure 13 illustrated the relation between changing temperature from 15 to 45°C and % removal. By increasing temperature the % removal reach the maximum value at 25°C=50% after 30min. and started to decrease by increasing temperature to 45°C for the same reason illustrated for RCS.

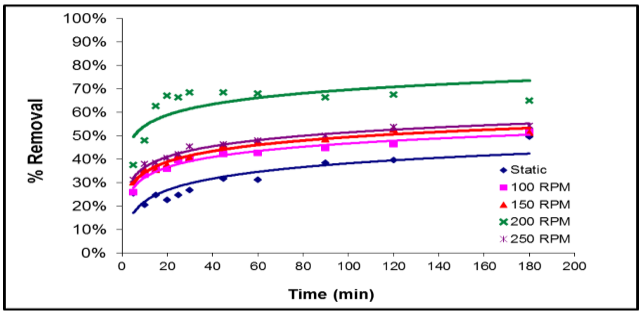


Figure 12. Effect of agitation rate on %Removal of CBB dye at temperature 25°C, CBB concentration of 10mg/L, SMCS dose 2g/L, particle size of 250µm

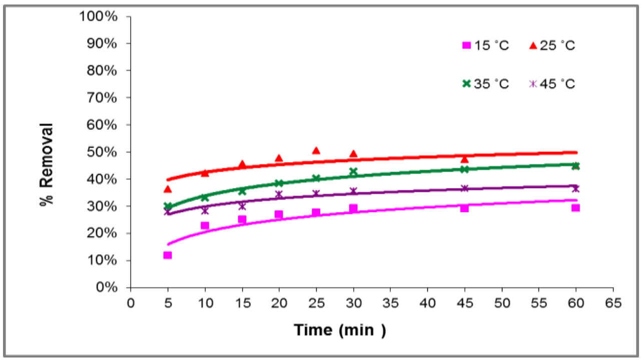


Figure 13. Effect of Temperature on % removal of CCB dye at 25°C, CBB concentration of 10mg/L, 2g/L MMCS, particle size of 250µm, 200rpm

While Figure 14 showed the same trend of relationship between temperature and % removal of CBB in case of RCS and MMCS as the process is exothermic, it is obvious that the % removal increased from 10% to 67% from 15°C to 25°C then decreased again to reach 27% at 45°C.

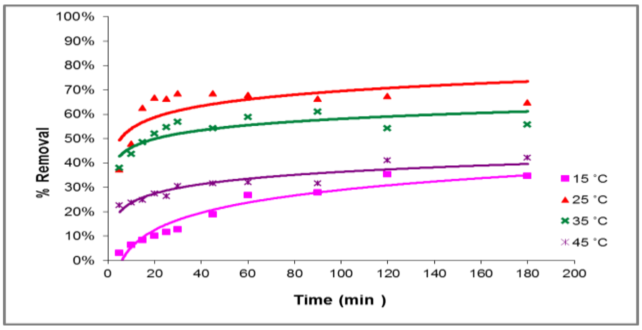


Figure 14. Effect of Temperature on %Removal of CBB dye, CBB concentration of 10mg/L, SMCS dose 2g/L, particle size of 250µm, 200rpm

3.2.4. pH

By increasing pH value from 3 to 11 as shown in Figure 15, the % removal of dye increased to reach 79% at pH= 3 after 30min, the result concludes that the MMCS give high adsorption results in acidic medium than alkaline.

Figure 16 illustrated also the same behavior for RCS and MMCS for the effect of pH on the % removal, as the maximum value was for pH=3 which equal about 80% after 20 min. and decreased to 23% at pH=11.

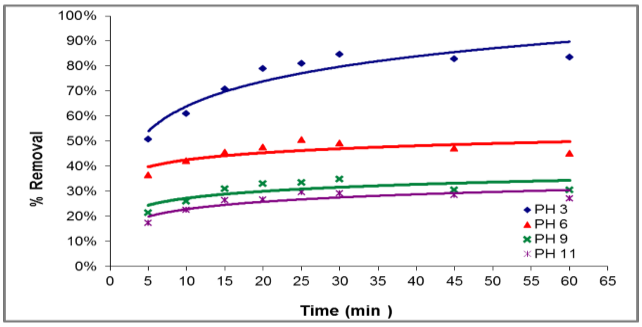


Figure 15. Effect of pH on % removal of CCB dye at 25°C, CBB concentration of 10mg/L, 2g/L MMCS, particle size of 250µm, 200rpm

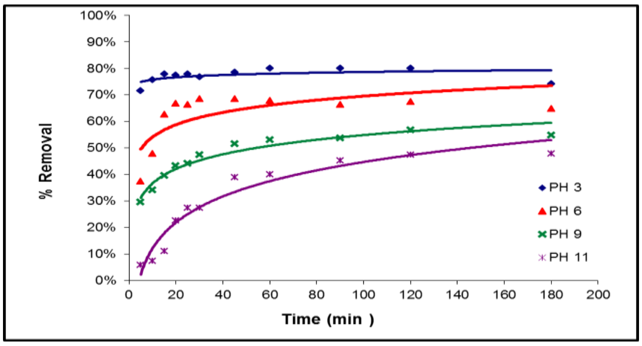


Figure 16. Effect of pH on % Removal of CBB dye at temperature 25°C, CBB concentration of 10mg/L, SMCS dose 2g/L, particle size of 250µm, 200rpm

4. Adsorption kinetics study

In order to investigate the adsorption kinetics of dye adsorption on adsorbent, different models are applied which represented with the following equations: (Sara and Tushar, 2012)

1. The first-order kinetic model:

$$\log C = \log C_0 - \frac{K}{2.303} \times t$$

2. The pseudo-first-order kinetic model:

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

3. The pseudo-second-order kinetic model:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$

Where the equilibrium rate constant is k_1 (1/min), and second order constants is k_2 (g/mg min) q_e is the amount of dye adsorbed on the surface at equilibrium (mg/g), q_t is the amount of dye adsorbed at different times (mg/g).

By applying the first order, pseudo first and second order kinetic models on raw corn stalk at the different concentrations as shown in Figures 17 to 19 and Table 1, it is obvious that the correlation coefficients of pseudo second order were found the highest value and also the theoretical values were very similar to experimental values, this results suggested that adsorption of CBB dye for RCS follows the pseudo second order model.

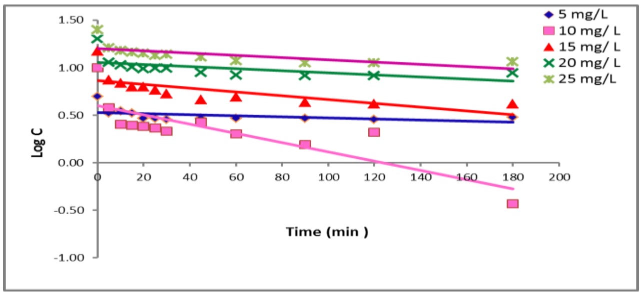


Figure 17. The first order kinetic model for adsorption of CBB dyes using RCS adsorbent at different initial CBB concentrations, 2g/L, 250 µmm pH=, 200rpm at 25°C

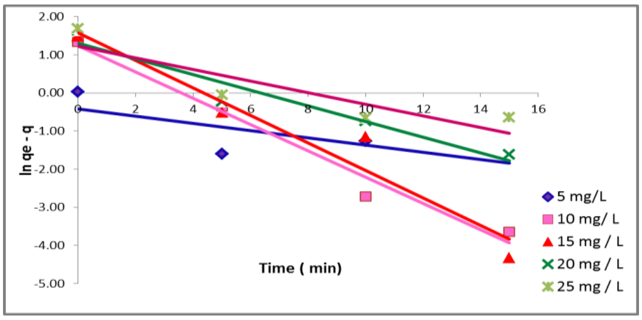


Figure 18. The pseudo first order kinetic model for adsorption of CBB dyes using RCS adsorbent at different initial CBB concentrations, 2g/L, 250 µmm pH=, 200rpm at 25°C

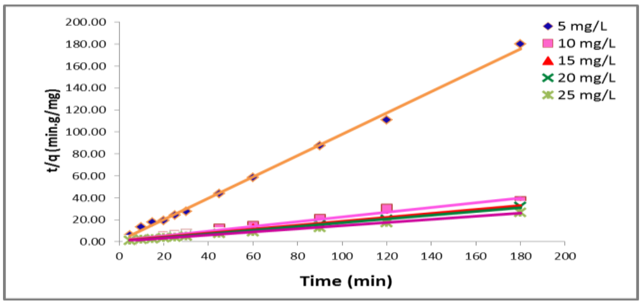


Figure 19. The pseudo second order kinetic model for adsorption of CBB dyes using RCS adsorbent at different initial CBB concentrations, 2g/L, 250µmm pH=200rpm at 25°C

5. Kinetic model comparison for different types of adsorbent used

Figure 20-22 and Table 2 represent the pseudo second order kinetic model for adsorption of CBB using raw and modified corn stalk. The figure showed that the higher value of R_2 was for RCS and MMCS followed by SMCS.

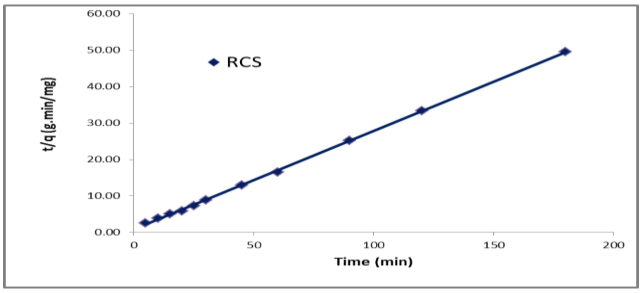


Figure 20. The pseudo second order kinetic model for adsorption of CBB dye using RCS adsorbent, 2g/L, 250µm, 200rpm, pH= 6 at 25°C

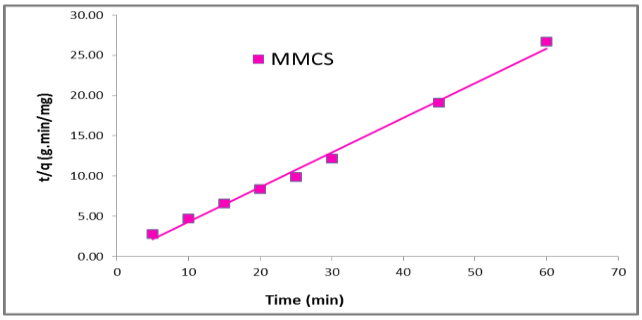


Figure 21. The pseudo second order kinetic model for adsorption of CBB dye using MMCS adsorbent, 2g/L, 250µm, 200rpm, pH= 6 at 25°C

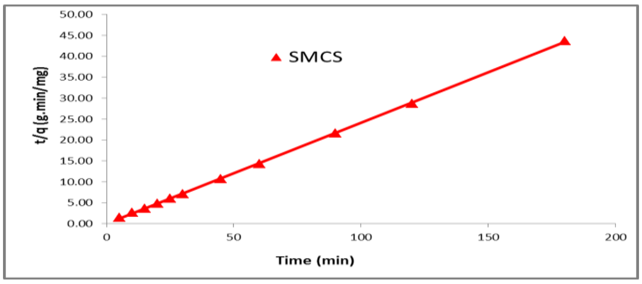


Figure 22. The pseudo second order kinetic model for adsorption of CBB dye using SMCS adsorbent, 2g/L, 250µm, 200rpm, pH= 6 at 25°C

Table 1 Kinetic parameters and the correlation coefficients at different initial CBB concentrations on RCS

C_0 mg/L	$q_{e(exp)}$ mg/g	First order kinetics			Pseudo First order kinetics			Pseudo second order kinetics			
		R^2	C_0 mg/L	K min^{-1}	R^2	q_e mg/g	K_1 min^{-1}	$q_{e(exp)}$ mg/g	R^2	q_e mg/g	K_2 min^{-1}
5	1.026	0.200	3.380	0.0004	0.604	1.511	0.095	1.026	0.997	1.031	1.383
10	3.789	0.679	3.963	0.0092	0.975	3.466	0.345	3.789	0.984	4.651	0.0342
15	4.368	0.482	3.034	0.0046	0.935	4.821	0.360	4.368	0.999	5.587	0.0420
20	5.132	0.317	2.075	0.0023	0.930	3.669	0.205	5.132	0.998	5.814	0.0833
25	5.421	0.453	2.838	0.0023	0.784	3.397	0.152	5.421	0.998	7.042	0.0299

Table 2 Kinetic parameters and the correlation coefficient of pseudo-second -order kinetic model for adsorption of CBB dyes using RCS, MMCS and SMCS at optimum conditions

Adsorbent type	$q_{e(exp)}$ mg/g	Pseudo second order kinetics			
		R^2	q_e mg/g	K_2 min^{-1}	h
RCS	3.395	0.999	3.704	0.0896	1.229
MMCS	2.382	0.994	2.320	-8.444	-45.449
SMCS	4.171	0.999	4.149	3.227	55.550

6. Conclusions

Corn stalk as a one of agriculture waste was tested comparing with rapid and simple modified adsorbent by using two modification methods. The uptake of anionic dye was increased with decrease in initial concentration of dye and particle size of adsorbents and also increased with increase in adsorbent dose used, shaking rate and contact time. The optimum conditions recommended for highest % removal of dye =85% was 10mg/ L initial concentration, 2g/L sorbent with 250µm, 150-200rpm shaking rate at room temperature and acidic solution with pH= 3 at 25°C. By comparing the resulting data across studying different variables of raw and modified corn stalk, the modification did not enhance the adsorbent behavior with valuable amount than raw corn stalk as expected. But modified corn stalk using magnetic particles (MMCS) can be valuable as the adsorbent can be easily collected and reused using a magnet The kinetic adsorption was represented by pseudo-second order model at the optimum conditions for all types of adsorbent used as a best fit model with R^2=0.99. Finally the results indicated that corn stalk could be employed as low cost alternative to commercial activated carbon for anionic dye removal from wastewater.

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