Removal of Eriochrome Black T from Dye Effluents by Corncob and Treated Corncob

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Abstract. Eriochrome Black T (EBT) is extensively utilized in the industrial sector for dyeing and printing on materials such as nylon, silk, wool, and leather. The wastewater generated from these processes is deemed toxic and poses a significant carcinogenic threat to both humans and aquatic organisms if released untreated into water bodies. In a batch kinetic study, the effectiveness of removing EBT from wastewater using Corn Cob (CC) and Sodium Carbonate-Corn Cob (NCC) was evaluated. Various factors, including RPM, temperature, pH, initial dye concentration, contact time, and absorbent dosage, were examined. The adsorption of EBT dye onto CC and NCC was found to be highly dependent on pH, with an optimal level around 5. The removal efficiency decreased with rising temperatures, with room temperature being identified as optimal. Generally, the removal rate improved with an increase in RPM, but no further improvement was observed beyond 300 RPM. Increasing the absorbent dose from 1 to 3 g/l enhanced the removal of EBT dye, with no additional benefit from further dosage increases. Contact time also played a crucial role; as it increased, so did the removal rate of EBT, although the efficiency plateaued after 90 minutes. It was found that CC and NCC could remove 80% and 85% of the EBT dye, respectively, under optimum parameter conditions. Both CC and NCC demonstrated satisfactory removal rates for EBT dye, offering a cost-effective alternative to expensive activated carbon.

1 Introduction

Use Water is the most vital natural resource necessary for the existence of all living things. Unfortunately, fresh, natural water is currently contaminated with several toxins, making it a significant problem that needs to be addressed by all nations for wastewater treatment before it is released into the water system. In the modern world, wastewater treatment is a challenging goal to achieve, and most developing nations, such as India, are struggling to

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address this issue. An industrial dye discharge are major contributors to freshwater pollution. Most synthetic dyes are classified as aromatic compounds that are soluble in water, have low degradation rates, and are generally stable. Dyes can be roughly categorized into direct, cationic, anionic, solvent, disperse, sulfur, reactive, and vat dyes, depending on their structure, quality, and usage. In addition to these, there are some further classifications. Basic dyes with vibrant colors and cationic dyes are widely used in the industry. The natural water system appears colorful due to the unprocessed dye discharge from enterprises mixing with the waterbody, which is quite evident to the naked eye. Such water poses risks to the ecosystem and is unfit for drinking, farming, and other uses. This color prevents water plants and other aquatic organisms from photosynthesizing.

[1] mentioned that Eriochrome Black-T (EBT) is a direct (azo) dye which is utilized in many industries including textiles, leather, paper, paint, pharmaceuticals and cosmetics. Fig.1 shows the chemical structure of EBT. Discharging waste liquid effluents from these industries into water bodies without appropriate or limited treatment poses a serious threat to the environment and ecosystems. EBT harms ocean habitats; furthermore, it is a cancer-causing agent and can cause severe health issues. Therefore, it is essential to eliminate EBT pollutants from used water prior to discharge or reuse to protect aquatic ecosystems from their harmful effects.

The processing of dyes is highly challenging because they are stable pollutants over extended periods. In addition, dyes are non-biodegradable and resistant to heat and light exposure, making them difficult to remove [1]. It interferes with the ability of aquatic species to grow, affecting photosynthesis. Thus, it is crucial to separate these substances from aquatic wastewater.

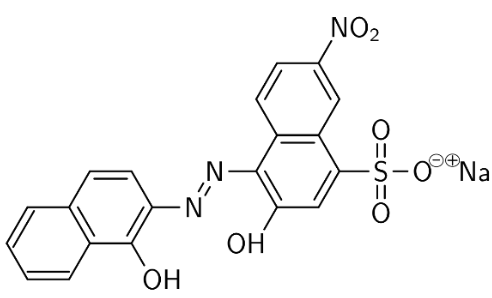

Fig. 1. Chemical structure of EBT

The presence of dyes in ecosystems is hazardous and damaging. Many treatment techniques have been identified to lower the contamination of dye concentration in water bodies, such as physiochemical treatment (ion exchange), enhanced oxidation processes, membrane techniques, adsorption techniques, and biological processes. However, the percentage of dye removal is lower and the process is not cost-effective. Adsorption is a developing approach with the potential for regeneration and sludge-free operation. This makes it superior to the other methods. Owing to its low cost, simple construction, easy maintenance, insensitivity to harmful substances, and efficient dye removal, it is used as a fundamental method for wastewater purification. It involves the physical and chemical interactions of chemicals and ions with a solid surface.

Adsorption mechanisms play an important role in water and wastewater purification, and different adsorbents can be used in these methods. Therefore, it is essential to analyze and investigate deeply to acquire the ideal operating conditions for dye removal prior to using them as adsorbents. While activated carbon is a widely used adsorbent, its production costs are high. This has led researchers to investigate cost-effective, environmentally friendly, and efficient alternative adsorbents for dye removal. Biomaterials have been found to significantly influence the physicochemical properties of an adsorbent. Garlic peel, jackfruit peel, watermelon seeds, tea leaves, etc., are used as inexpensive bio-adsorbents, and it has been claimed for another dye removal. Using biomaterials as adsorbents is not as complex as activated carbon, with minimal processing, affordability, ample availability, good efficiency, effortless regeneration, and lower chemical or biological sludge production. The adsorption efficiency of different types of agricultural byproducts and their operating conditions along with application have been summarized by [2].

[3] explored the removal of Methylene Blue (MB) from effluent using a range of native adsorbents, including banana leaf ash (BLA), charcoal, neem seeds, rice husk ash and coconut coal. Among these options, banana leaf ash with particle sizes starting from 0.053 mm to 0.075 mm was found to be the most effective adsorbent for achieving maximum dye removal. [4] crosslinked bamboo powder and coffee ground waste as filler materials into a composite foam with gelatine and found that these foams could remove different organic dyes. [5] analyzed the physiochemical properties of biochar from agricultural byproducts to study its efficacy in purging dyes from industrial effluents.

[6] has investigated the efficacy of rice husk on the purging rate of four different dyes with different operating parameters and found that the pH of the solution highly influences the purging rate. [7] reported that the equilibrium time of various biochar adsorbents varied between 4 and 5h maximum. [8] reviewed the effectiveness of removing heavy metals and dyes using various agricultural waste products. [9] has examined the different operational parameters of shell-based agrarian waste adsorbents, including pH, dosage, temperature, initial concentration, and duration.

[1] investigated the decolorization of EBT through adsorption on activated carbon (CA) and photocatalytic degradation using TiO2 (PD), employing the Bees Algorithm, Bacterial Foraging Optimization, and Particle Swarm Optimization for modeling. [10] explored the effectiveness of removing EBT from aqueous solutions with activated Typha grass (T. latifolia), reporting an adsorption efficiency of 47.619 mg/g.

[11] has analyzed the adsorption of EBT from a solution by employing waste hemp activated carbon (WHAC), finding a maximum removal efficiency of 44-66% at a dosage of 70 mg. [12] provided a summary of many plant-based bio-adsorbents and their modifications for effectively removing EBT, along with a comparison of different parameters affecting the adsorption process.

Due to its porous nature and surface properties, corncob is an economical adsorbent with promising capabilities for dye removal. [13] explored how effectively corn cob and corn silk can eliminate Cadmium (Cd) and Nickel (Ni) from water. Meanwhile, [14] studied the use of porous biochar derived from corncob for extracting urea from wastewater. [15] has concluded that pretreatment of corncob as an adsorbent, has increase the surface area and it is increasing the ability of removal of heavy metals. Building on the gaps identified in previous studies, our research aims to explore the efficacy of raw and sodium carbonate-treated corn cob in the removal of EBT dye, focusing on optimizing operational parameters for maximum dye removal.

2 Experimental methodologies and design parameter

2.1 Adsorbent Preparation

Corn cob (CC) was purchased from the local market and street vendors, cut into smaller pieces. The Corn Cob was cleaned with distilled water to separate contaminants and sundried for 24 h. The corncob pieces were ground into finer granules and screened through a 600-300 micron sieve and placed in a laboratory drying oven for 24 h at 60°C to extract the moisture. The samples were then stored in a damp-proof container to protect them from atmospheric moisture. The corn cob was treated with NaOH to increase its surface area, which is expected to enhance its dye adsorption capacity. A fraction of tried Corn Cob powder was then kept in a rotary shaker with 0.1 M NaOH (Sodium Hydroxide) solution for up to 6 hours at 300 rpm and rested overnight. It was then removed from the rotary shaker, placed in a laboratory drying oven for 24 h at 60°C, and kept in a damp-proof container. The Corn Cob was designated as CC for distilled washed and NCC for NaOH modified. Fig. 2 shows the preparation process of corncob and treated corncob.

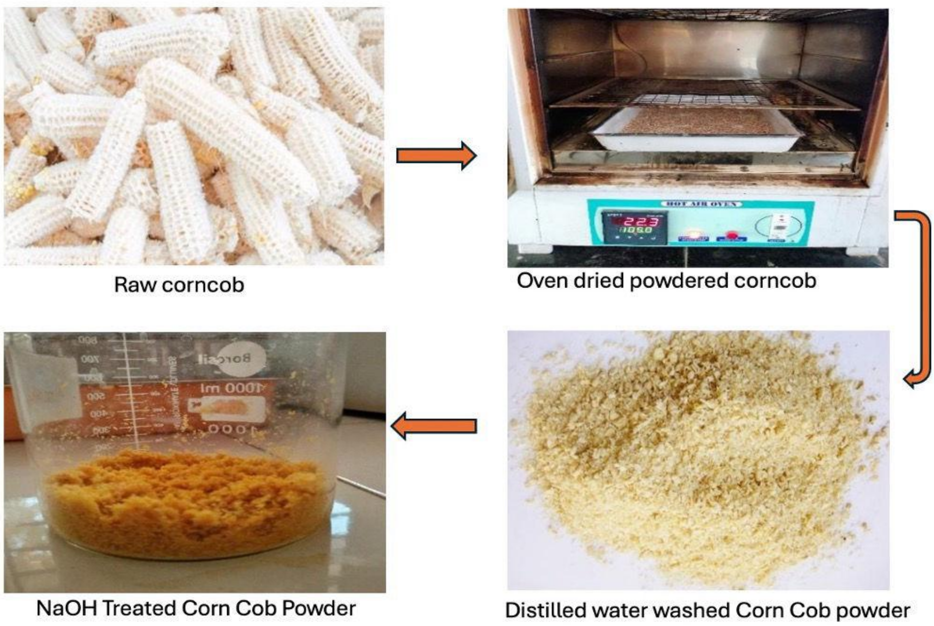

Fig. 2. Preparation corncob and naoh treated corncob

2.2 Standard Solution

A standard solution of 1 g/l of Eriochrome Black T was prepared. All experiments were conducted by mixing the standard solution with deionized water to obtain the required concentrations.

2.3 Adsorbent Characterization

Characterizing adsorbents is crucial for understanding the adsorption mechanism, assessing the rate-limiting steps, and designing parameters for both batch and continuous adsorption processes. Hence, the physicochemical properties and characteristics of the adsorbent were analyzed along with instrumental analysis.

2.4 Batch Adsorption Experiments

The adsorption batch study for CC was performed in 250 mL container using 100 mL of the dye solution. The effects of pH (3 to 10), adsorbent dose (1 to 4 g / 100 mL), temperature (28ºC to 60ºC), contact time (up to 2 hours), initial concentration level (50-100 mg/L) and RPM (100-500) with a mean particle size of 450 microns were studied. Samplings were gathered at regular time intervals and examined using UV-VIS spectrophotometry.

After determining the optimum parameters for CC, the same parameters were used for NCC to perform the adsorption study. The percentage of dye removal was evaluated by the following equation.

$$\text{\% of Dye Removal} = [(C_o - C_e) / C_o] \times 100 \qquad (1)$$

Where, C_0 = Concentration of dye in (mg/l)
C_e = Equilibrium dye concentration in (mg/l)

2.5 Batch Operating parameters

Initially, the effect of rotational speed on adsorption techniques was explored, with experiments conducted at speeds between 100 and 500 RPM. For each sample, the remaining concentration and the percentage of dye removal were analyzed and documented at various RPM levels. The impact of contact period on the adsorption process was also examined to establish the optimal time required for dye removal from wastewater, with tests performed at 30, 60, 90, and 120-minute intervals. Furthermore, the influence of pH on the adsorption of EBT dye by corn cob was assessed, with pH levels adjusted from 3 to 10 using 0.1 M H_2SO_4 and NaOH solutions. The efficiency of the adsorption dosage in the treatment process was evaluated to determine the ideal dose needed for dye removal from wastewater using corn cob adsorbent at dosages ranging from 1 to 4 g per 100 ml of effluent solution. Three different initial dye concentrations (100, 75, and 50 mg/l) were made from the stock concentrate for analysis. The influence of temperature on the adsorption of EBT dye was assessed through three temperature analyses at 28, 45, and 60°C using a thermal incubator shaker, with the process conducted at 300 RPM. The experiment was performed at pH 8, with a dosage of 10 g/L, an initial dye strength of 75 mg/L, and an average particle size of 450 µm. The remaining concentration and percentage of dye removal at different temperatures were studied to comprehend the thermodynamic properties of the adsorption system under varying temperature conditions. Fig. 3 and 4 shows the batch experiment and measurement of pH.

Fig.3. Magnetic stirrer with rpm and temperature

Fig.4. Testing pH of dye solution with pH meter

3 Results and discussions

The present study used CC and chemically modified NCC as bio-adsorbents for purging dyes from bodies of water and in wastewater. CC is a strong, indissoluble, and stable in water, and has a high removal strength. Therefore, it has good physical properties as a bio-adsorbent for dye purging. The main objective of the present study was to identify the optimum operating parameters of CC as a potential bio-adsorbent for the purging of dyes by varying physicochemical operations. All the trial tests were conducted thrice, and the arithmetic average value was utilized for analysis and presented in graphical or tabular form to minimize experimental errors.

3.1 Batch kinetic studies

The laboratory tests and experiments were scrutinized to assess and enhance the different parameter variables for effective dye elimination. The dye absorption potential was investigated through initial screening experiments conducted with demineralized water-washed Corn Cob (CC) and NaOH-treated Corn Cob (NCC). A few trial experiments were performed using dyes to identify the important parameters of adsorption. The various crucial parameters investigated were the initial concentration, adsorbent dose, pH, and equilibrium time. The final experiment was conducted with optimum parameters, that is, at room temperature, pH 5, 300 rpm, 10 g/L dose with a first measured concentration of dye of 75 mg/L, and 100 mL volume, and were analyzed for 90 min. Fig. 5 shows treated water sample with an optimum parameters.

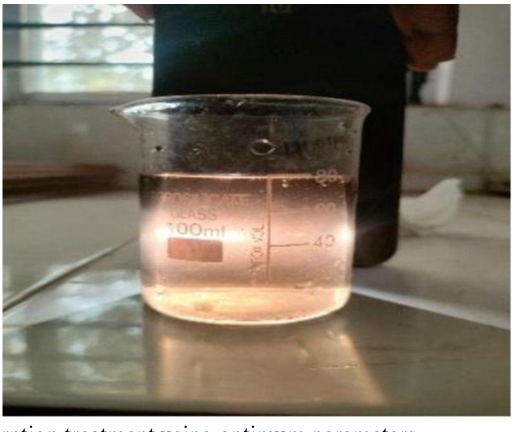

Fig. 5. Dye solution after adsorption treatment using optimum parameters.

The EBT dye removal rate with optimum parameters was 80% for CC and 85% for NCC. The dye adsorption rate increased rapidly in the early stages, and the dye removal rate decreased as it approached the maximum-absorption limit. As observed in the experiments, there was no considerable development in the dye removal after 90 min. After 90 min, the dye concentration in the solution did not decrease; therefore, equilibrium was assumed to have been reached. The dye purging rate of NCC was higher than that of CC because the pretreatment of the adsorbent with NaOH increased its surface area [16].

Equilibrium was considered to be reached at 90 minutes, as the dye concentration remained largely unchanged beyond this point. This is due to the saturation of available sites, which prevents further adsorption from occurring. Some bio-adsorbents exhibit poor operational capacity (i.e., partial solubility) and leaching of colored dyes into the solution because of prolonged contact with water. The use of raw biomaterials as adsorbents may also cause various issues. For instance, the rise in chemical oxygen demand (COD) is due to the discharge of organic solutes available in the plant materials. To overcome these issues, chemical alteration or triggering of raw bio-adsorbents was performed, so that pre-treatment of biomaterials could extract solute organic substances and intensify chelating efficacy.

The adsorption capacity increases with a large surface area; reducing the particle dimensions enhances the surface area in each gram of the adsorbent, contributing to a greater number of binding spots, and the adsorption capacity is augmented. However, an excessive number of fine particles increased the head loss in the column study. A particle size range of–300-600 microns with a mean proportional size of 450 microns was chosen for the studies.

3.2 Effect of RPM

The rate or velocity of agitation significantly affects adsorption. The experiments were performed on EBT dye onto CC by raising the RPM from 100 to 500 with an original concentration of 75 mg/L, 28℃ temperature with pH of 8, and a dose of 10 g/L. It was noted that as the rpm increased, the rate of dye removal also rose, but beyond 300 rpm, the removal rate began to decline. This is because the centripetal force acts on it, resulting in a repulsive force between the CC and dye molecules. Hence, 300 rpm was considered the optimal speed. The influence of the RPM on the purging rate is illustrated in Fig. 6.

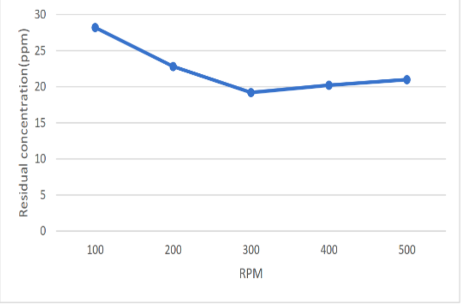

Fig. 6. Residual concentration vs RPM

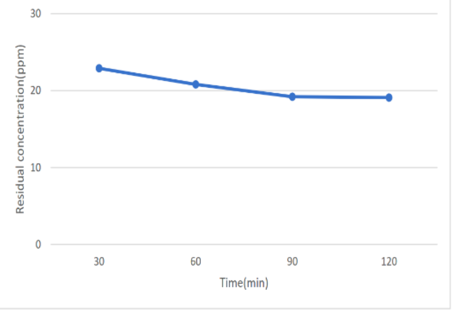

Fig. 7. Residual concentration vs Time

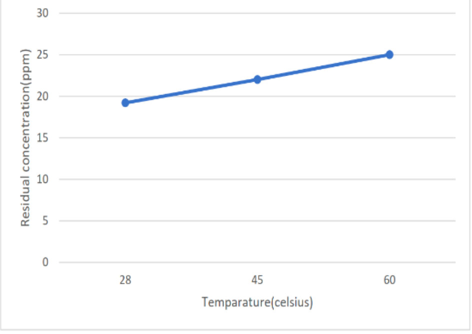

Fig. 8. Residual concentration vs Temperature

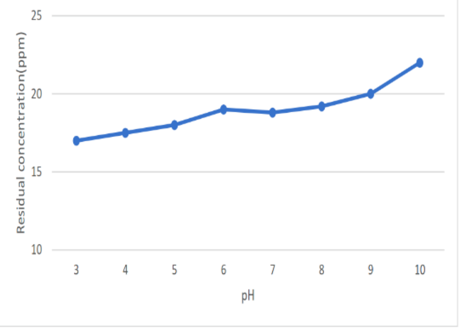

Fig. 9. Residual concentration vs pH

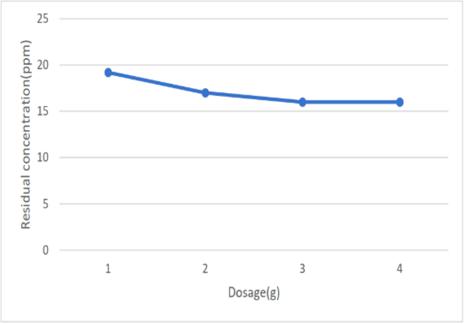

Fig. 10. Residual concentration vs Dosage graph

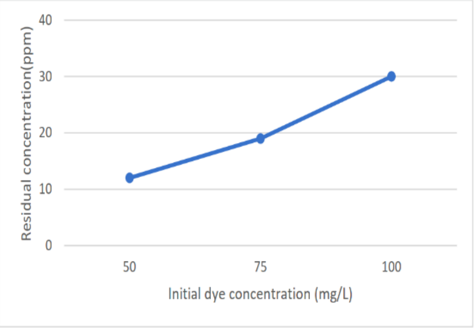

Fig. 11 Residual concentration vs Initial dye concentration

3.3 Impact of Contact Period

To examine the impact of contact duration, the time was extended from 30 to 120 minutes at a speed of 300 rpm, with a pH level of 8, and a dosage of 10 g/L, with a starting dye concentration of 75 mg/L, to facilitate the adsorption of EBT dye onto CC. The adsorption of dye increased as the contact time was extended from 30 to 90 minutes. However, beyond 90 minutes, no notable change was detected because the adsorption sites were already saturated, and further extending the time had no effect. The removal rate peaked at 90 minutes, suggesting that this was the optimal contact time. Figure 7 depicts the effect of contact period on the rate of dye removal.

3.4 Impact of Temperature

The influence of temperature was examined by raising it from 28 to 60°C while maintaining a speed of 300 rpm, a pH level of 8, and a dosage of 10 g/L, with an initial dye strength of 75 mg/L for the removal of EBT dye onto CC. The adsorption of dye diminished as the temperature raised. At 28° C, the removal rate was the highest, so the optimum temperature was taken as 28°C in further experiments. The influence of temperature on dye purging is illustrated in Fig.8.

3.5 Impact of pH

The impact of pH on dye removal is a key factor influencing adsorption. The surface charge of the NCC, the adsorption mechanism, and the ionization degree of the adsorbate's functional group are significantly influenced by pH levels. The pH effects were examined within a range of 3 to 10. At higher pH values, the dye mixture showed a tendency to precipitate. Samples were agitated in a rotational shaker at 300 rpm and 28 °C for 90 minutes, after which the final molar strength of the dye was measured. The purging of EBT dye improved as the pH decreased from 10 to 5, after which it stabilized. Figure 9 illustrates the impact of pH on the removal rate of dye. After analyzing the pH influences, two possible phenomena were observed: the interaction of electrostatic force between the adsorbate and adsorbent including chemical modification of NCC, which involves the chemical effect of alkaline treatment between the adsorbate and adsorbent. More dye purging occurs in acidic than in basic or alkaline environments.

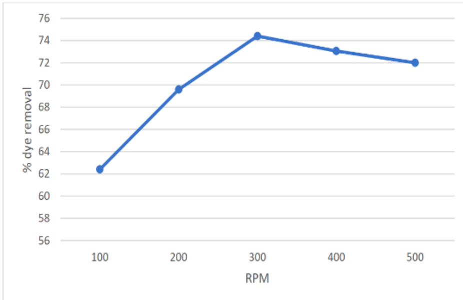

Fig. 12. % of Dye removal vs RPM graph

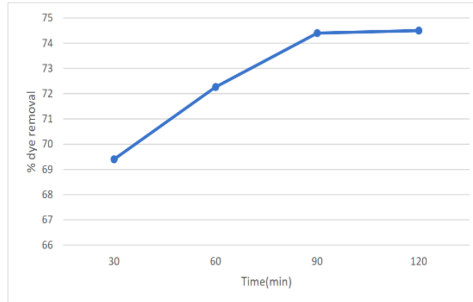

Fig. 13. % of Dye removal vs Time graph

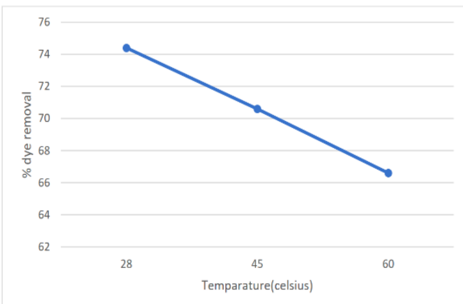

Fig. 14. % of Dye removal vs Temperature graph.

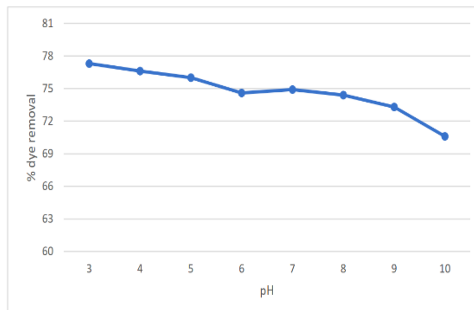

Fig. 15. % of Dye removal vs pH graph

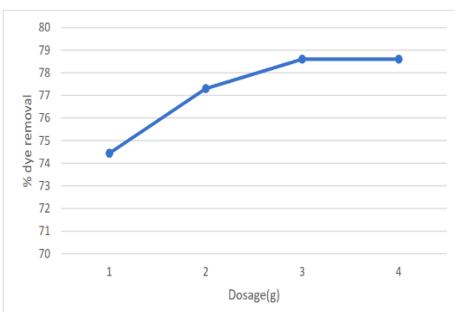

Fig. 16. % of Dye removal vs dosage graph.

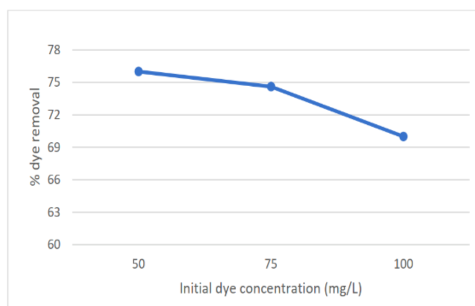

Fig. 17. % of Dye removal vs Initial dye Concentration graph

3.6 Impact of pH

The impact of pH on dye removal is a key factor influencing adsorption. The surface charge of the NCC, the adsorption mechanism, and the ionization degree of the adsorbate's functional group are significantly influenced by pH levels. The pH effects were examined within a range of 3 to 10. At higher pH values, the dye mixture showed a tendency to precipitate. Samples were agitated in a rotational shaker at 300 rpm and 28 °C for 90 minutes, after which the final molar strength of the dye was measured. The purging of EBT dye improved as the pH decreased from 10 to 5, after which it stabilized. Figure 9 illustrates the impact of pH on the removal rate of dye. After analyzing the pH influences, two possible phenomena were observed: the interaction of electrostatic force between the adsorbate and adsorbent including chemical modification of NCC, which involves the chemical effect of alkaline treatment between the adsorbate and adsorbent. More dye purging occurs in acidic than in basic or alkaline environments.

3.7 Impact of Adsorbent Dosage

Adsorbent dosage studies are also paramount for studying the present adsorption methods to obtain the optimum dose for dye purging. By changing the CC dosage from 1 to 4 g/ 100 mL dye solution at a temperature of 28 °C, rotation speed of 300 rpm, and a pH of 8, with a beginning molar strength of dye is 75 mg/L, the best removal percentage rate was 78.5%. With a higher dosage, the percentage of EBT dye purging also increased because of the increase in the number of active pore spots that adsorbed onto CC and NCC. There was a negligible increase after the 3 g dose. The purging of the EBT dye increased with increasing dosage because of the higher number of pore openings on the surface of the adsorbent. Hence, 10 g/L or 1 g/100 mL appears to be an economical and adequate dose of adsorbent for removing EBT dye from wastewater. The impact of the adsorbent dosage on the purging process of the dye effluent is shown in Fig. 10.

3.8 Impact of Initial Dye Concentration

The analysis utilized three initial dye strength: 100, 75, and 50 mg/L. The experiment was conducted at a speed of 300 rpm, a temperature of 28 °C, a pH level of 8, with an adsorbent dosage of 10 g/L, and a particle size of 450 microns. The higher percentage of dye removal was observed at an initial strength of 50 mg/L, followed by 75 mg/L and 100 mg/L, respectively. The difference in dye removal efficiency between 50 mg/L and 75 mg/L was not substantial, leading to the conclusion that 75 mg/L is the optimal initial dye strength. Figure 11 illustrates the impact of initial dye concentration on dye removal, while Figures 12-17 display the percentage of dye removal for various design parameters.

4 Conclusions

The following results and conclusions were drawn based on exhaustive investigations and critical analyses of the experimentally observed data on the adsorption of EBT dye in wastewater by sodium carbonate-treated Corn Cob. In the screening experiment, it was found that CC and NCC could remove 80% and 85% of the EBT dye, respectively, under optimum parameter conditions, starting molar strength of 75 mg/L at 28 °C, a dose of 10 g/L, 300RPM, pH 5, and a exposure time of 90 min. The optimum initial dye strength for purging of EBT dye from a waste-water environment was 75 mg/L. Three dye intensities were used: 50, 75, and 100 mg/L. The 50 mg/L concentration had a slightly higher purging rate than 75 mg/L and 100 mg/L, but 75 mg/L is considered as there is not much difference; as the dye molar concentration rises, the active pore spots of the CC and NCC adsorbent get full, and therefore, no more adsorption occurs. Thus, the modified Corn Cob provides a better purging rate because the augmented active spots on the absorbent result from chemical treatment. The adsorption of EBT dye onto CC and NCC was highly dependent on pH. The optimal pH was 5. Therefore, more significant adsorption occurred, resulting in a higher dye purging rate at pH = 5. The purging of EBT dye increased with an increase in dose from 1 to 3 g, and no change was observed thereafter; however, the % dye removal for 2 and 3 g was not sufficiently high to be considered. Therefore, an economical dosage of 10 g/L was used for further kinetic studies. Regarding the influence of temperature on dye purging, a decrease in the purging rate of the dyes at a higher temperature was observed. Therefore, room temperature is considered optimum. As the RPM increased, the purging rate also increased;

however, after 300 rpm, the purging rate decreased. This is because of the superabundance of the development of centripetal forces, which results in the development of repulsive forces. Consequently, the dye adsorbed on CC and NCC was repulsed. Hence, 300 rpm was considered the optimal level. As the exposure time increased, the dye removal increased, but after 90 min, no change was observed. Hence, 90 min was considered to be the ideal exposure time for the adsorption experiments. CC and NCC provide reasonable removal rates for purging the EBT dye and can be used instead of costly activated carbon.

Conflict of Interest Statement: The authors declare that there are no conflicts of interest.
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