SYNTHESIS OF CHITOSAN BEADS/ACTIVE CHARCOAL AS ADSORBENT OF YELLOW ACID 25 AND ACID RED 73

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SYNTHESIS OF CHITOSAN BEADS/ACTIVE CHARCOAL AS ADSORBENT OF YELLOW ACID 25 AND ACID RED 73

ABSTRACT. Synthesis of chitosan/activated carbon beads has been carried out as an adsorbent for Acid Yellow 25 and Acid Red 73 in the solution. The adsorbent preparation was carried out by dissolving chitosan in 2.5% (v/v) acetic acid and then adding activated carbon. The beads were printed in NaOH solution and then washed until neutral. Chitosan/activated carbon bead adsorbents were characterized using FTIR and SEM before and after the adsorption-desorption process. Optimum conditions were determined by adsorption studies at various contact times, initial concentrations, and pH for each dye. Desorption studies were carried out using NaOH desorption medium at various concentrations and desorption times. The results showed that the optimum adsorption conditions for AY-25 were a contact time of 90 minutes with an initial concentration of 600 mg L^{-1} at pH 4 which resulted in an adsorption capacity of 501.8 mg g^{-1}. Meanwhile, AR-73 was optimum at 120 minutes with an initial concentration of 300 mg L^{-1} at pH 4 which resulted in an adsorption capacity of 297.5 mg g^{-1}. The adsorption of the two dyes followed the pseudo-second-order kinetic model and agreed with the Langmuir isotherm model. The effective desorption process was carried out with 1 M NaOH for 3 hours. The regenerated adsorbent can be used for the adsorption process three times.

KEYWORDS: adsorption, chitosan beads/activated carbon, Acid Yellow 25, Acid Red 73

SINTEZA PERLELOR DE CHITOSAN/CĂRBUNE ACTIV CA ADSORBANT PENTRU COLORANȚII ACID YELLOW 25 ȘI ACID RED 73

REZUMAT. S-a realizat sinteza perlelor de chitosan/cărbune activ ca adsorbant pentru coloranții Acid Yellow 25 și Acid Red 73 în soluție. Adsorbantul a fost preparat prin dizolvarea chitosanului în acid acetic 2,5% (v/v) și apoi adăugarea de cărbune activ. Granulele au fost impregnate în soluție de NaOH și apoi spălate până la pH neutru. S-au caracterizat adsorbanții sub formă de granule de chitosan/cărbune activat folosind FTIR și SEM înainte și după procesul de adsorbție-desorbție. Condițiile optime au fost determinate prin studii de adsorbție la diferiți timpi de contact, concentrații inițiale și pH pentru fiecare colorant. Studiile de desorbție au fost efectuate folosind mediu de desorbție NaOH la diferite concentrații și timpi de desorbție. Condițiile optime de adsorbție pentru AY-25 au fost, conform rezultatelor, un timp de contact de 90 de minute cu o concentrație inițială de 600 mg L^{-1} la pH 4, ceea ce a dus la o capacitate de adsorbție de 501,8 mg g^{-1}. Pe de altă parte, AR-73 a fost optim la un timp de contact de 120 de minute, cu o concentrație inițială de 300 mg L^{-1} la pH 4, ceea ce a dus la o capacitate de adsorbție de 297,5 mg g^{-1}. Adsorbția celor doi coloranți a urmat modelul cinetic de ordinul doi și a fost în acord cu modelul izoterm Langmuir. S-a realizat un proces de desorbție eficient folosind NaOH 1 M timp de 3 ore. Adsorbantul regenerat poate fi utilizat de trei ori pentru procesul de adsorbție.

CUVINTE CHEIE: adsorbție, granule de chitosan/cărbune activ, colorant Acid Yellow 25, colorant Acid Red 73

LA SYNTHÈSE DE BILLES DE CHITOSANE/CHARBON ACTIF COMME ADSORBANT POUR LES COLORANTS ACID YELLOW 25 ET ACID RED 73

RÉSUMÉ. La synthèse de billes de chitosane/charbon actif a été réalisée comme adsorbant pour les colorants Acid Yellow 25 et Acid Red 73 dans la solution. La préparation de l'adsorbant a été réalisée en dissolvant du chitosane dans de l'acide acétique à 2,5 % (v/v), puis en ajoutant du charbon actif. Les billes ont été imprimées dans une solution de NaOH puis lavées jusqu'à neutralité. Les adsorbants chitosane/billes de charbon actif ont été caractérisés par FTIR et SEM avant et après le processus d'adsorption-désorption. Les conditions optimales ont été déterminées par des études d'adsorption à différents temps de contact, concentrations initiales et pH pour chaque colorant. Des études de désorption ont été réalisées en utilisant un milieu de désorption NaOH à différentes concentrations et temps de désorption. Les résultats ont montré que les conditions optimales d'adsorption pour le colorant AY-25 étaient un temps de contact de 90 minutes avec une concentration initiale de 600 mg L^{-1} à pH 4, ce qui aboutissait à une capacité d'adsorption de 501,8 mg g^{-1}. D'autre part, le colorant AR-73 était optimal à 120 minutes avec une concentration initiale de 300 mg L^{-1} à pH 4, ce qui entraînait une capacité d'adsorption de 297,5 mg g^{-1}. L'adsorption des deux colorants suivait le modèle cinétique de pseudo-second ordre et était en accord avec le modèle isotherme de Langmuir. Le processus de désorption efficace a été réalisé avec du NaOH 1 M pendant 3 heures. L'adsorbant régénéré peut être utilisé pour le processus d'adsorption trois fois.

MOTS CLÉS : adsorption, billes de chitosane/charbon actif, colorant Acid Yellow 25, colorant Acid Red 73

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INTRODUCTION

Colored waste is one of the environmental problems that arise as a result of industrial activities. The dyes contained in the waste make it difficult to degrade in the environment because they tend to be stable against light and oxidizing agents and can survive under anaerobic conditions [1]. If this colored waste is disposed of into the environment, it can be harmful because of the potential toxicity carried by dyes [2]. In addition, dyes that enter the waters can block the penetration of sunlight so they must be prevented from entering water bodies and causing disruption of the ecological balance and the process of photosynthesis [3–5]. Examples of dyes commonly used in the textile and leather tanning industries are Acid Yellow 25 (AY-25) and Acid Red 73 (AR-73). Both of these dyes are included in the anionic dyes which have toxic potential in the environment.

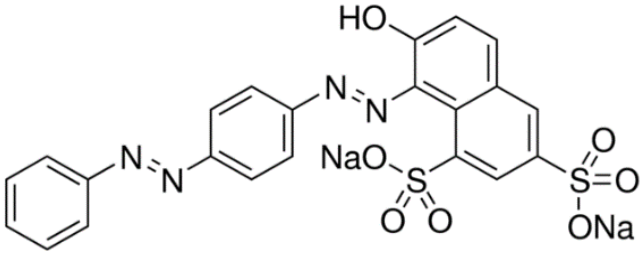

Figure 1. Structure of AY-25

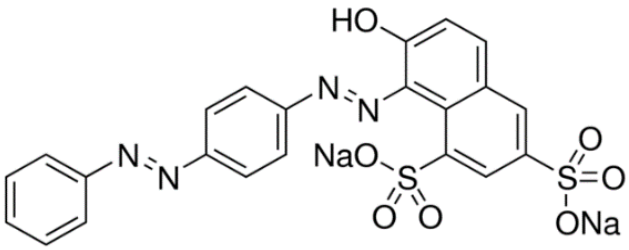

Figure 2. AR-73 structure

Waste-containing dyes can be treated by various methods, including ion exchange [6], ozonation [7-8], photocatalyst [9, 10], electrochemistry [2], oxidation [5], and adsorption [11-13]. In general, these methods can be used for the processing of dyes, but have some drawbacks, including requiring a large amount of energy, using chemical additives, and producing intermediates during the reaction.

Chitosan is the result of the deacetylation of chitin which has many applications. The use of chitosan, which is quite flexible, in various fields is due to its biodegradable nature, high compatibility, specific functional groups, low toxicity, and large enough molecules [4]. Characteristics of chitosan that can be utilized in the adsorption process are the presence of hydroxy and amine groups. However, chitosan has weaknesses such as forming colloids when in contact with water, being soluble in acids and very easily biodegradable. Therefore, to overcome these weaknesses, chitosan is usually modified by adding cross-linking agents or by impregnation, one of which is with carbon material [14, 15]. Several studies have been carried out using chitosan as an adsorbent, including the use of chitosan-silica for the adsorption of azo dyes with the adsorption parameters studied including the effect of contact time, initial adsorbate concentration, composition, and studies related to desorption [16]. Chitosan modified with activated carbon to form beads can be used for adsorption under the influence of pH. This is due to the electrostatic interaction between the charged adsorbate and the adsorbent at a certain pH [17].

EXPERIMENTAL

Materials and Methods

Materials

Chitosan and activated carbon with technical grades as well as chemicals produced by MERCK with analytical degrees of purity which include glacial acetic acid (CH_3COOH), sodium hydroxide (NaOH), and hydrochloric acid (HCl). In addition, Acid Yellow 25 (AY-25), Acid Red 73 (AR-73), and distilled water were used as solvents and washers.

Methods

Synthesis and Characterization of Chitosan/Activated Carbon Beads

1 g of chitosan was dissolved in 60 mL CH_3COOH 2.5% (v/v) and then stirred until all dissolved. Then, 1 g of activated carbon was added to the chitosan solution while stirring for 60 minutes. The resulting suspension was then dripped into 200 mL of 2.5% (w/v) NaOH solution and allowed to stand for 24 hours. The resulting beads are then washed with distilled water until the pH of the distilled water before and after washing is the same. The resulting chitosan/activated carbon beads were then dried at 100 °C. Chitosan/activated carbon beads were characterized by FTIR and SEM before and after the adsorption-desorption process.

Determination of Contact Time and Optimum Initial Concentration

A total of 0.025 g of adsorbent was added to 25 mL of AY-25 and AR-73 solutions each with a solution concentration of 100 mg L^{-1}. After that, stirring was carried out with a magnetic stirrer for 10 minutes, then the concentrations of AY-25 and AR-73 were measured after the adsorption process using a Uv-Vis Spectrophotometer with λ=393 nm (AY-25) and 512 nm (AR-73). Action steps were repeated for time variations of 20, 30, 45, 60, 90, 120, 180, 240, and 480 minutes with various concentrations of 50, 100, 200, 300, 450, 600, 800, and 1000 mg L^{-1}.

Optimum pH Determination

A total of 0.025 g of adsorbent was added to 25 mL of AY-25 and AR-73 solutions with pH 2, 4, 6, 8, and 10, respectively with various concentrations of 50, 100, 200, 300, 450, 600, 800, and 1000 mg L^{-1}. After that, adsorption was carried out during the optimum time. The next step is measuring the concentration of the dye after the adsorption process.

Adsorbent Desorption and Regeneration Studies

AY-25 and AR-73 solutions were prepared at optimum concentration conditions. The solution was used for adsorption with the adsorbent at the optimum time and pH conditions, respectively. Furthermore, the used chitosan/activated carbon adsorbent beads were dried and then used for desorption studies.

Desorption studies were carried out in 0.5 M NaOH and 1 M NaOH solutions for 1 and 3 hours. The desorption solution was then analyzed using a UV-Vis spectrophotometer. The adsorbent that has been used for desorption studies is then dried. These adsorbents were reused for the adsorption process of AY-25 and AR-73 under their respective optimum conditions. This procedure is repeated until the adsorbent is no longer effective for the adsorption process.

RESULTS AND DISCUSSIONS

Characterization of Chitosan/Activated Carbon Beads

FTIR spectra of chitosan/activated carbon beads show an absorption peak at wave number 3314 cm^{-1} which describes the stretching vibration of the –OH group which overlaps with the NH group, 2876 cm^{-1}, indicating the stretching vibration of -CH, 1573 cm^{-1} which is the bending vibration –NH2, 1369 cm^{-1} is the bending vibration absorption of –CH groups in –CHOH, and the absorption peak at 1019 cm^{-1} is the stretching vibration of –CO in CONH. After adsorption, new absorption peaks appeared at wave numbers 1186 and 808 cm^{-1} for AY-25 and

1171 and 844 cm^{-1} for AR-73. The absorption peaks at 1186 and 1171 cm^{-1} indicated the presence of –C–N=N–C– bonds of the dye molecule and 808 and 844 cm^{-1} for –C–S stretching vibrations of SO_3. The FTIR spectra of the adsorbent after desorption shown in Figure 3 shows the disappearance of the absorption peak –C–N=N–C–. This indicates that the desorption process with NaOH is effective enough to release dye molecules from the chitosan/activated carbon bead adsorbent although there are still dye molecules remaining on the surface of the adsorbent as indicated by the presence of –CS stretching vibration absorption peaks. This is in line with previous research which showed a shift in wave number and a new absorption peak after the adsorption of Yellow 42 dye [18].

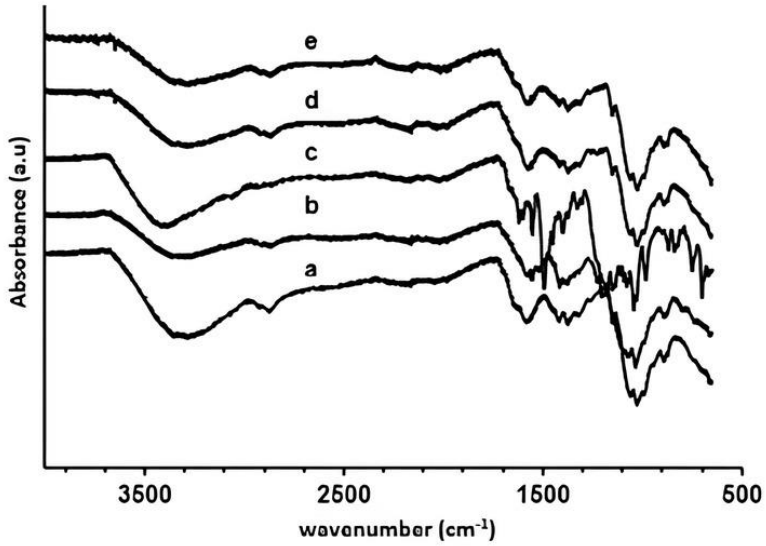

Figure 3. FTIR spectra of: a) chitosan bead/activated carbon adsorbent, b) adsorption of AY-25, c) adsorption of AR-73, d) desorption of AY-25, e) desorption of AR-73

Figure 4 shows that the chitosan/activated carbon beads have pores of various sizes with irregular shapes. This pore can be used to adsorb dye molecules. Whereas the adsorbent that has been used for adsorption shows that the surface of the adsorbent becomes rougher and some lumps cover the pores. This indicates the presence of AY-25 and AR-73 molecules attached to the surface of the adsorbent. SEM image after desorption shows a smoother surface but there are still lumps attached to the surface of the adsorbent. This indicates that the desorption process still leaves dye molecules. This indication is supported by the EDX data in Table 1 which shows a decrease in the percentage of element S originating from AY-25 and AR-73 molecules in the adsorbent after the desorption process.

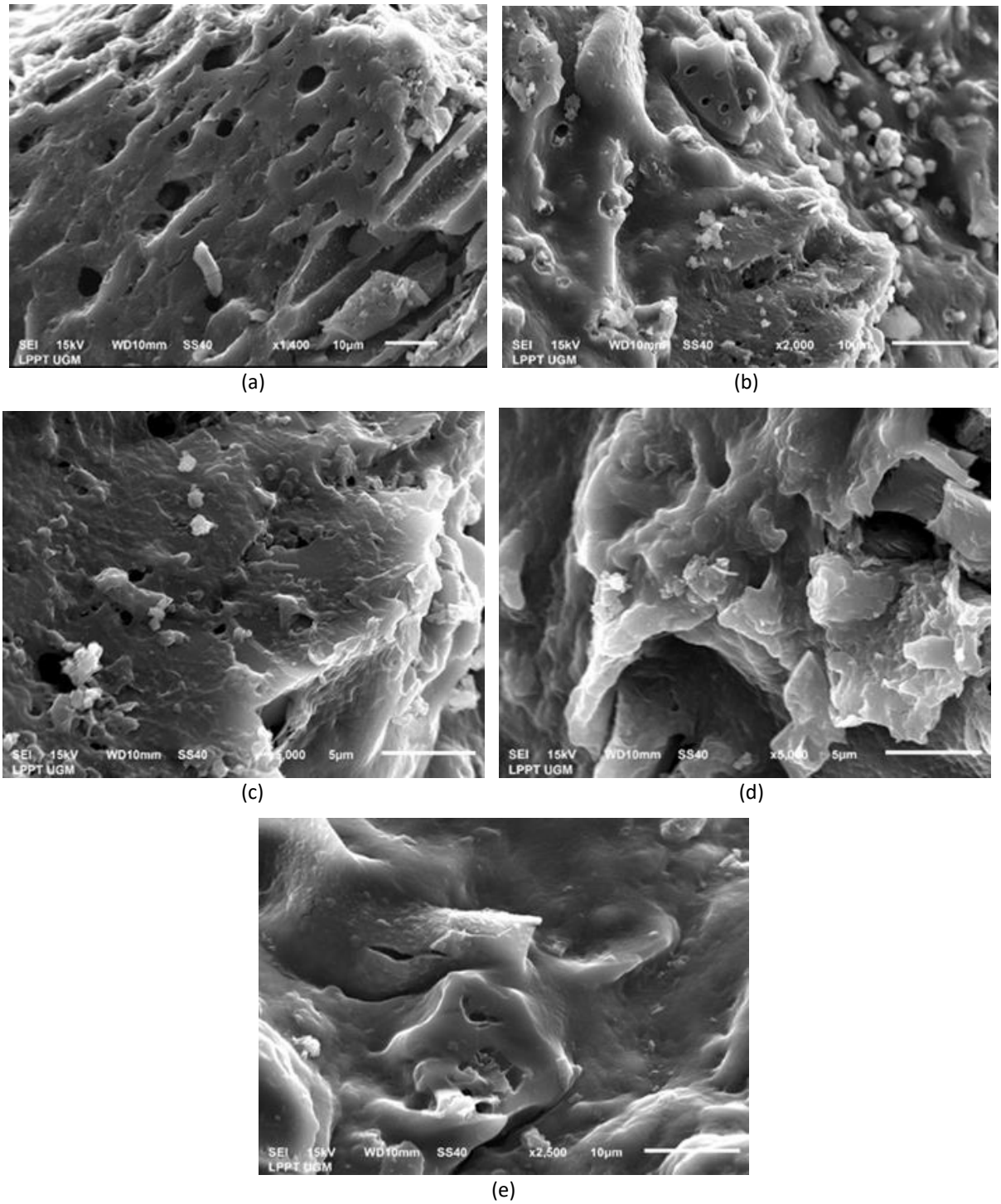

Figure 4. SEM images of: a) chitosan bead/activated carbon adsorbent, b) after adsorption of AY-25, c) after adsorption of AR-73, d) after desorption of AY-25, and e) after desorption of AR-73

Table 1: EDX data before and after the adsorption-desorption process of AY-25 and AR-73

Element	Pristine Adsorbent (%)	Ads AY-25 (%)	Δ% Ads AY-25	Ads AR-73 (%)	Δ% Ads AR-73	Des AY-25 (%)	Δ% Des AY-25	Des AR-73 (%)	Δ% Des AR-73
C	46.32	48.20	+4.06%	53.51	+15.52%	46.13	−0.41%	50.26	+8.50%
N	19.85	20.97	+5.64%	19.78	−0.35%	20.18	+1.66%	19.66	−0.96%
O	33.43	29.33	−12.27%	25.66	−23.24%	32.85	−1.74%	28.85	−13.70%
S	–	1.14	Detected	0.47	Detected	0.34	Residual	0.37	Residual

Determination of Contact Time and Optimum Concentration

Based on Figure 5a and Figure 5b it can be seen that the adsorption capacity increased significantly at the beginning of the process, then tended to be constant. Optimum conditions for AY-25 adsorption occurred at a contact time of 90 minutes and for AR-73 occurred at a contact time of 120 minutes. The increase in adsorption capacity at the beginning of the process occurs because the adsorbate attaches to the active site of the adsorbent which is still empty so that the dye molecules diffuse more easily on the surface of the adsorbent. The adsorption capacity of AY-25 and AR-73 increased with increasing contact time, but after the optimum contact time, the adsorption capacity tended to be constant. This happens because the number of active sites that are empty on the adsorbent has decreased [19].

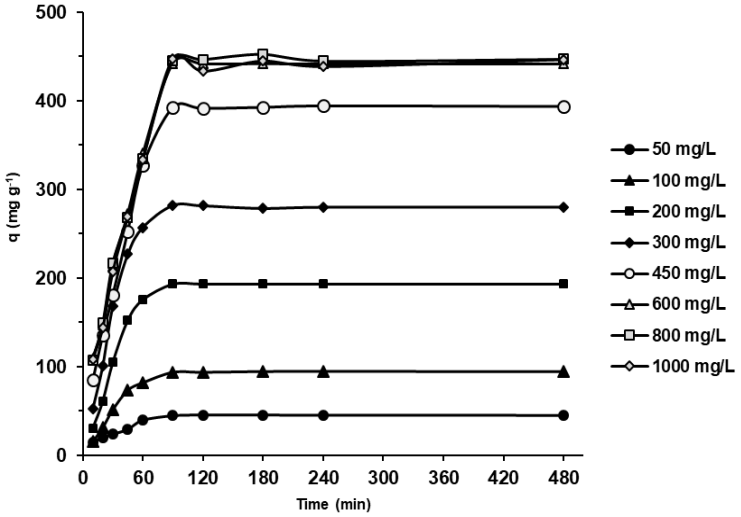

Figure 5a. Graph of determination of contact time and initial concentration of AY-25

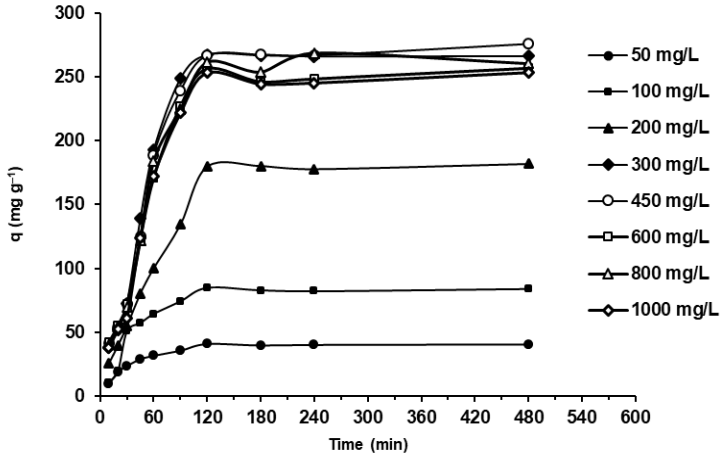

Figure 5b. Graph of determination of contact time and initial concentration of AR-73

The difference in the optimum time between AY-25 and AR-73 is due to the difference in the adsorption rate of the two dyes. Adsorption speed or rate can be determined using kinetic equations. This study uses first-order, second-order, pseudo-first-order, and pseudo-second-order kinetic equations. A summary of the parameters of various kinetic models is presented in Table 2.

Table 2: Adsorption kinetics parameters of AY-25 and AR-73 in solution on chitosan/activated carbon beads

Kinetic Models	Parameter	AY-25	AR-73
q_e experiment (mg g^{-1})		442.53	267.38
Order one	k'_1 (min^{-1})	2.6×10^{-3}	4.6×10^{-3}
	R^2	0.5354	0.5240
Order two	k'_2 (min^{-1})	1.0×10^{-5}	6.0×10^{-5}
	R^2	0.5963	0.5607
Pseudo first order	k_1 (min^{-1})	0.0143	0.0137
	R^2	0.4887	0.5568
	q_e calculation (mg g^{-1})	79.51	88.23
Pseudo second order	k_2 (g mg^{-1} min^{-1})	7.9×10^{-5}	5.5×10^{-5}
	R^2	0.9917	0.9564
	q_e calculation (mg g^{-1})	476.19	312.50
	H	17.96	5.43

Table 3: Parameters of Langmuir and Freundlich isotherms for adsorption of AY-25 and AR-73

Model	Parameter	AY-25	AR-73
	q_{max} experiment (mg g^{-1})	442.53	267.38
Freundlich	K_f	52.79	44.27
	1/n	2.56	3.29
	R^2	0.7011	0.5556
Langmuir	K_L (L mg^{-1})	0.04	0.08
	R_L	0.02	0.01
	q_{max} (mg g^{-1})	476.19	263.16
	R^2	0.9950	0.9949

The kinetic model suitable for the adsorption of AY-25 and AR-73 is a pseudo-second-order kinetic model. One of the things that affect the rate of adsorption is the size of the adsorbate molecule [20]. AY-25 with a molecular weight of 549.55 g mol^{-1} which is smaller than AR-73 with a molecular weight of 556.48 g mol^{-1} causes AY-25 to diffuse faster than AR-73. This is supported by data on the optimum contact time for AY-25 which is faster than AR-73.

Other parameters determined in this study were the initial concentrations of AY-25 and AR-73. Figures 5a and 5b show that the adsorption capacity increases as the adsorbate concentration increases, then at a certain concentration it tends to be constant. A significant increase in adsorption capacity at the beginning of adsorption was caused by an increase in the concentration of the adsorbate, causing a concentration gradient that accelerated diffusion [21]. This research resulted that the optimum initial concentration of AY-25 was 600 mg L^{-1} while AR-73 had an optimum initial concentration of 300 mg L^{-1}.

One way to determine the type of interaction between adsorbate and adsorbent is through the determination of isotherms that describe adsorption capacity as a function of the equilibrium concentration of adsorbate at a constant temperature [22]. The isotherms studied in this study are Langmuir and Freundlich isotherms. Parameter data for Langmuir and Freundlich isotherms are presented in Table 3 which shows that the appropriate coefficient of determination is the Langmuir isotherm for the adsorption of AY-25 and AR-73. The agreement with the Langmuir isotherm indicates that the maximum adsorption of AY-25 and AR-73 occurs when a monolayer layer is formed on the surface of the adsorbent with constant adsorption energy and there is no interaction between the adsorbate molecules and neighboring active sites [22, 23]. This study indicated that the adsorption was influenced by the interaction between the sulfonate groups of the dye molecules and the amino groups of chitosan.

Optimum pH Determination

Figures 6a and 6b show that pH 4 is the optimum pH for the adsorption of AY-25 and AR-73. The figure also shows a tendency to decrease the adsorption capacity of the two

dyes as the pH increases. This occurs because at low pH conditions the increased concentration of H^+ ions promotes the protonation of amino groups on the adsorbent surface ($–NH_2$ to $–NH_3^+$), leading to a positively charged surface that enhances electrostatic interactions with negatively charged adsorbate molecules, although ζ-potential measurements were not performed in this study [24, 25]. Chitosan in a low pH environment will cause the protonated $–NH_2$ group to become $–NH^{3+}$ [24]. Therefore, the electrostatic interaction between the adsorbate in the form of anionic dyes and the adsorbent will take place optimally at low pH. The decrease in adsorption capacity when there is an increase in pH is due to the fact that at a pH that tends to be neutral, chitosan will tend to form $–NH_2$ compared to $–NH^{3+}$ so that it will be more difficult to bind to anionic dyes [25]. An increase in pH also causes the number of OH- ions to increase, causing competition with anionic dye molecules [26].

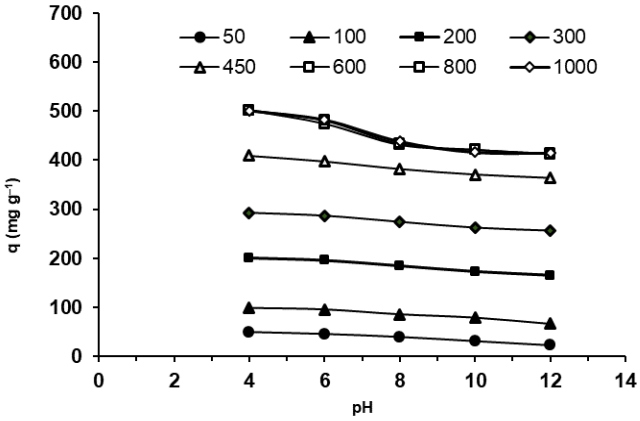

Figure 6a. Graph of determining the optimum pH of AY-25

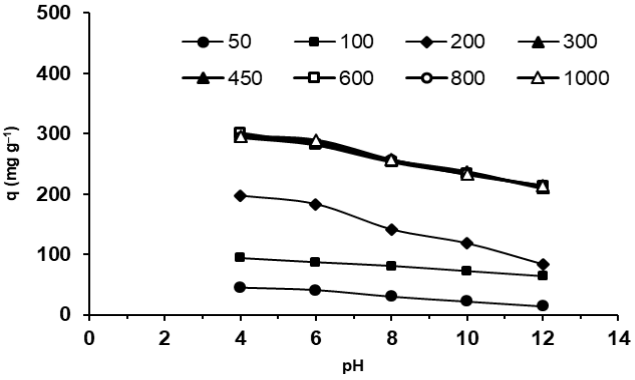

Figure 6b. Graph of determining the optimum pH of AR-73

Desorption Study

Figure 7 shows that as the NaOH concentration and desorption time increased, the amount of AY-25 and AR-73 released increased. This is caused by the weakening of the electrostatic interaction of the sulfonate groups of dyes with chitosan due to an increase in pH which causes chitosan to tend to form $–NH_2$ resulting in the release of adsorbate molecules [26]. From these results, it can be predicted that the most dominant interaction in the adsorption of AY-25 and AR-73 using chitosan/activated carbon beads is the electrostatic interaction of the dye sulfonate groups with $–NH^{3+}$ from chitosan in an acidic condition. The highest desorption percentages of AY-25 and AR-73 were produced by 1 M NaOH desorption solution with a desorption time of 3 hours, namely 50.57% for AY-25 and 57.60% for AR-73.

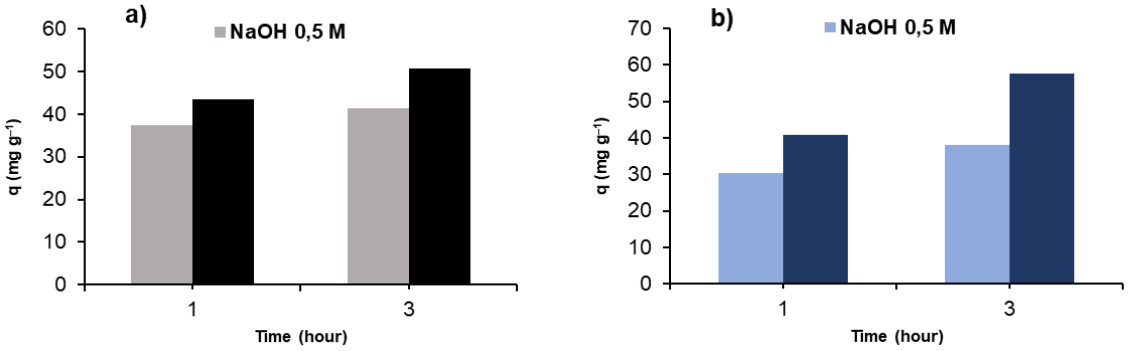

Figure 7. Desorption percentage of (a) AY-25 and (b) AR-73 at different desorption times using NaOH solutions of two concentrations; the darker bars represent 1.0 M NaOH, while the lighter bars represent 0.5 M NaOH

CONCLUSIONS

This research produced chitosan/ activated carbon beads which could be used as dye adsorbents for Acid Yellow 25 and Acid Red 73 with the optimum contact time for AY-25 adsorption at 90 minutes while for AR-73 at 120 minutes. Adsorption of both dyes followed a pseudo second-order kinetic model. The adsorption capacity of both dyes increased with an increasing initial concentration of the solution and optimally took place at pH 4. Chitosan/activated carbon bead adsorbents using NaOH would result in an increase in percent desorption with increasing concentration and time. The regenerated chitosan/carbon bead adsorbent can be reused for adsorption three times.

Conflict of Interests

No potential conflict of interest was stated by the authors.

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