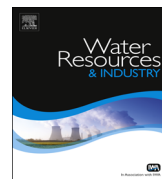




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Assessment of activated carbon prepared from corncob by chemical activation with phosphoric acid

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ABSTRACT

Corncob, which is the main waste from corn agricultures in Egypt, has been used as a raw material for the preparation of different activated carbons. Activated carbons (ACs) were prepared by chemical activation with concentrated H_3PO_4 acid; followed by pyrolysis at 400, 500 and 600 °C. Different ACs have been used for the removal of methylene blue (MB) dye from aqueous solutions. Batch adsorption experiments were performed as a function of initial dye concentration, contact time, adsorbent dose and pH. Adsorption data were modeled using the Langmuir and Freundlich adsorption isotherms. Adsorption of MB on AC1 ($R^2=0.9868$) and AC2 ($R^2=0.9810$) followed Langmuir model with maximum monolayer sorption capacity of 28.65 and 17.57 mg/g, respectively. Adsorption onto AC3 was better fitted to Freundlich isotherm model ($R^2=0.9823$).

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1. Introduction

The world-wide high level of production and use of dyes generates colored wastewaters, which give cause of environmental concern. Textile companies, dye manufacturing industries, paper and pulp mills, tanneries, electroplating factories, distilleries, food companies and a host of other industries discharge colored wastewater [1]. As a matter of fact, the discharge of such effluents in the

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environment is worrying for both toxicological and esthetical reasons. The methods of color removal from industrial effluents include biological treatment, coagulation, floatation, adsorption, oxidation and membrane filtration. Among the treatment options, adsorption appears to have considerable potential for the removal of color from industrial effluents. It has been found that the performance of the adsorbents depends on their textural properties as porosity and surface area [2]. Adsorption onto activated carbons is a well known process for micro-pollutants removal [3]. It has many advantages over several other conventional treatment methods for wastewater treatment. These include (i) less land area (half to quarter of what is required in a biological system); (ii) lower sensitivity to diurnal variation; (iii) not getting affected by toxic chemicals; (iv) greater flexibility in the design and operation and (v) superior removal of organic contaminants [4]. Activated carbon is perhaps the most widely used adsorbent for the removal of many organic contaminants which are biologically resistant. Adsorption with activated carbon is usually an expensive treatment process and this promoted a growing search to prepare economical activated carbon. Many kinds of plant biomasses are natural renewable resource that can be converted into activated carbon, either by physical or chemical activation. The advantage of using agricultural by-products as raw materials for manufacturing activated carbon is that these raw materials are potentially less expensive to manufacture [5]. The technology to manufacture activated carbon of good quality is not fully developed in developing countries; therefore, there is a need to produce activated carbon from cheaper and readily available materials, which can be used economically on a large scale. According to what was mentioned, providing an adsorbent, which is cost-effective and environmentally comparative, for the removal of dyes from aqueous solutions seems to be so vital. The activated carbon has been produced from variety of biomass i.e., agricultural wastes [6], date stone [7], peanut shells [8], marigold straw [9], oil palm shell [10], cotton wove waste [11], and almond shell [12].

In this work, three different ACs were prepared from corncob by chemical activation using phosphoric acid followed by pyrolysis at different temperatures. They were used as adsorbents to remove methylene blue dye from aqueous solution. The kinetic data and equilibrium data on batch adsorption studies were carried out to understand the adsorption process. The effect of adsorption parameters such as pH, adsorbent concentration, contact time and initial dye concentration is also reported.

2. Materials and methods

2.1. Adsorbate

Methylene blue, a basic dye is a product of LOBA Company, Egypt. It was used as received without further purification. A stock solution of methylene blue (50 mg/l) was prepared and suitably diluted to the required initial concentration. All chemicals were of analytical reagent grade.

2.2. Adsorbent preparation

The corncob collected from agriculture lands near Benha city were washed several times with distilled water and left to dry, and then it was cut into small pieces, grained and sieved to an average particle size (0.05 mm). The raw material was subjected to chemical treatment followed by pyrolysis. The activation was carried out by impregnation of the corncob samples with phosphoric acid in a ratio of 1:2 (w/w) for 24 h, and then washed with distilled water several times until pH reached 4. After that, the samples were dried at 100 °C then pyrolysed in a muffle furnace in absence of air at 400, 500 and 600 °C for 2 h. After pyrolysis, the resulting samples were washed with distilled water until the pH of the washing solution reached 6–7. The activated carbon samples were dried at 100 °C and kept dry till use. The resulting AC samples produced are abbreviated as AC1, AC2 and AC3 according to the activation temperatures (400, 500 and 600 °C, respectively).

2.3. Adsorption studies

Batch adsorption experiments were performed at room temperature (25 ± 2 °C) in a set of Erlenmeyer flasks (250 ml) where solutions of dye (50 ml) with different initial concentrations (5–50 mg/l) were placed. Equal doses of 2.0 g/l of particle size (150 μ m) ACs were added to dye solutions. The pH of the solutions was adjusted to the required value by adding either 0.1 M HCl or 0.1 M NaOH solution. The concentration of methylene blue in the supernatant solution before and after adsorption was determined using a double beam UV spectrophotometer (Shimadzu Model: UV 1601, Japan) at maximum wavelength of 665 nm. Blanks containing no dye were used for each series of experiments as controls. A calibration curve of absorbance versus concentration was constructed using spectrophotometric data. The percentage removal of dye was calculated using the following relationship:

$$\% \text{Dye Removal} = \frac{C_0 - C_e}{C_0} \quad (1)$$

where, C_0 and C_e are the initial and final (equilibrium) concentrations of dye (mg/l), respectively. The amount of adsorption at any time, q_e (mg/g), was calculated by:

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (2)$$

where, C_0 and C_e (mg/l) are the liquid-phase concentrations of dye at initial and any time, respectively. V is the volume of the test solution (l), and W is the mass of adsorbent used (g).

2.4. Pore size and surface area of ACs

Temperature has been reported to play an important role in producing optimum surface area of activated carbon. For the characterization of different activated carbon (AC) samples was carried out by N_2 adsorption at 77 K using Autosorb I, supplied by Quantachrome Corporation, USA. The BET (N_2 , 77 K) is the most usual standard procedure used when characterizing an activated carbon.

2.5. Iodine number

According to U.S. Environmental Protection Agency Technology Transfer Manual (1972), about 2.0 g of activated carbon sample was grind to pass a 0.25 μ mesh sieve to produce approximately 1.0 g carbon. The product added was to 10 ml of 5.0 mass% HCl solutions in flask and put on hot plate for 30 s, then cooled at room temperature and a 100 ml of standardized 0.10 N iodine solution was added to the flask. The flask stopperd and shaken vigorously for 30 s. After filtration, the remaining solution was titrated with 0.10 N sodium thiosulphate solution. The results are shown in (Table 1).

2.6. The point of zero charge (pH_{pzc})

The pH_{pzc} is an important characteristic for any activated carbon as it indicates the acidity/basicity of the adsorbent and the net surface charge of the carbon in solution. To measure the pH_{pzc} , the pH of the solution is maintained by using 0.1 N HCl and 0.1 N NaOH solutions. The pH_{ZPC} values of different

Table 1
Characterization of the activated carbon.

Activated carbon type	AC1	AC2	AC3
Activation temperature (°C)	400	500	600
Surface area (m ² /g)	700	633	600
Micropore volume (cm ³ /g)	0.011	0.009	0.003
Iodine number (mg/g)	632	945	485

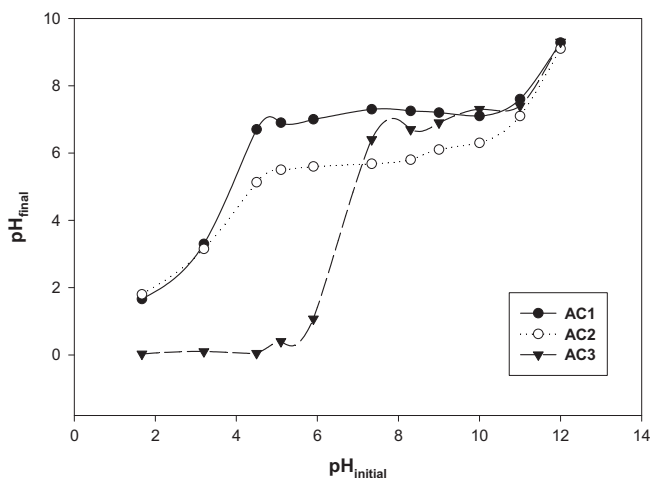


Fig. 1. Determination of point zero charge of different ACs.

activated carbon used for the adsorption experiment are determined by using solid to liquid ratio of 1:1000. For this, 0.1 mg of AC is added to 100 ml of water with varying pH from 2 to 12 and stirred for 24 h. Final pH of the solution are plotted against initial pH of the solution and shown in Fig. 1. The results of the determination of the pH_{pzc} for AC1, AC2 and AC3 are 7.4, 5.7 and 7.0, respectively.

3. Results and discussion

3.1. Effect of contact time

The contact time between adsorbent and adsorbate is one of the most important parameters that significantly affect the performance of dye removal. The effect of contact time on the percentage removal of MB dye was investigated at fixed initial dye concentration (25 mg/l) and adsorbing dose (2 g/l). As shown in Fig. 2, the dye removal increased rapidly in case of AC1 in the first 10 min of contact and then more slowly until equilibrium was obtained. This can be explained by the strong attraction forces between the positive sites of cationic dyes and the anionic sites of the activated carbon [13]. On the other hand, the rate of removal is slower at the beginning for AC2 and AC3 but it gradually increased with time until it reached equilibrium. The time taken to reach equilibrium was about 45 min for AC1 and 120 min for AC2 and AC3 for the dye concentration used.

3.2. Effect of initial concentration of dye

The effect of initial concentration of dye on the removal percent and removal efficiency (in terms of mg/g) on various adsorbents is studied as shown in Fig. 3a and b, respectively. The percentage removal of the dye was found to decrease with the increase in initial dye concentration from 5 mg/l to 50 mg/l. It can be attributed that the active sites on adsorbent for dye removal decreases when dye concentration increases [14]. In the same time, the adsorption capacity of the three types of activated carbon increased with increasing of the initial dye concentration. This indicates that there exist reductions in immediate solute adsorption, owing to the lack of available active sites required for the high initial concentration of MB. Similar results have been reported in the literature [15,16]. The results show that the adsorption capacity of dye increases from 4.9 to 46.0, 4.8 to 39.3 and 4.6 to 37.5 mg/g as the initial dye concentration increases from 5 up to 50 mg/l for AC1, AC2 and AC3, respectively.

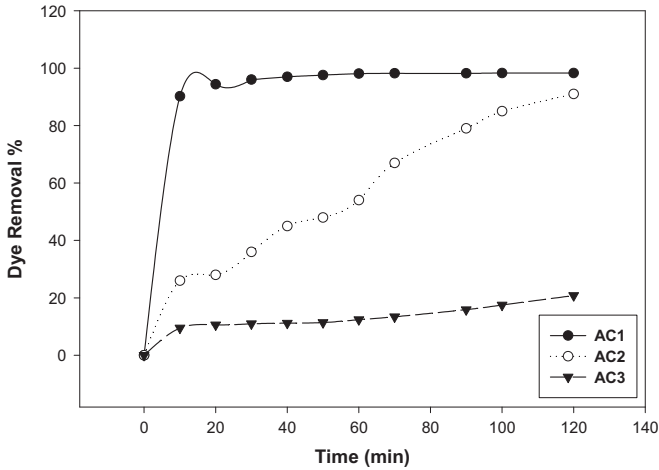


Fig. 2. Effect of contact time on the removal of MB dye.

3.3. Effect of dose of adsorbent

The effect of dose of adsorbent on the percentage removal of MB dye at pH 8.2 is shown in Fig. 4. The percentage removal of the dye increased with the increase in dose of adsorbent. This may be due to the increase in availability of surface active sites resulting from the increased dose of the adsorbent [17]. While adsorbent dose increases from 1 to 5 g/l, the dye removal percent increases from 96.2% to 99.6%, 98.6% to 99.4% and 41.6% to 91.2% at equilibrium time for AC1, AC2 and AC3, respectively.

3.4. Effect of pH

The effect of initial pH of dye solution on the percentage removal of MB was studied by varying the initial pH under constant process parameters. The results are shown in Fig. 5. The dye adsorbed by different types of carbons was higher at higher pH. The optimum pH was attained at pH higher than 8. As the pH of the solution increased, the dye adsorbed increased considerably. The same behavior was observed by many authors [18,19]. The combined influence of all the functional groups of activated carbon determines pH_{pzc} , i.e., the pH at which the net surface charge on carbon was zero. At $pH < pH_{pzc}$, the carbon surface has a net positive charge, while at $pH > pH_{pzc}$ the surface has a net negative charge [20]. Fig. 3 shows the “pH drift” data, from which the pH_{pzc} of the adsorbent can be determined (pH 9.0). The pH_{pzc} is the point where the curve pH_{final} vs $pH_{initial}$ intersects the line $pH_{initial} - pH_{final}$ [21].

3.5. Adsorption isotherms

In order to optimize the design of an adsorption system to remove the dye, it is important to establish the most appropriate correlations for the equilibrium data for each system. Two isotherm models have been tested in the present study; Langmuir and Freundlich models. Langmuir theory was based on the assumption that adsorption was a type of chemical combination or process and the adsorbed layer was unimolecular. The theory can be represented by the following linear form:

$$\frac{C_e}{q_e} = \frac{1}{q_0 b} + \frac{C_e}{q_0} \quad (3)$$

where, C_e is the equilibrium concentration (mg/l), q_e is the amount adsorbed at equilibrium (mg/g), q_0 signifies the adsorption capacity (mg/g) and b is related to the energy of adsorption (l/mg). The linear plots of C_e/q_e vs C_e (Fig. 6) show that adsorption follows a Langmuir isotherm for AC1 and AC2

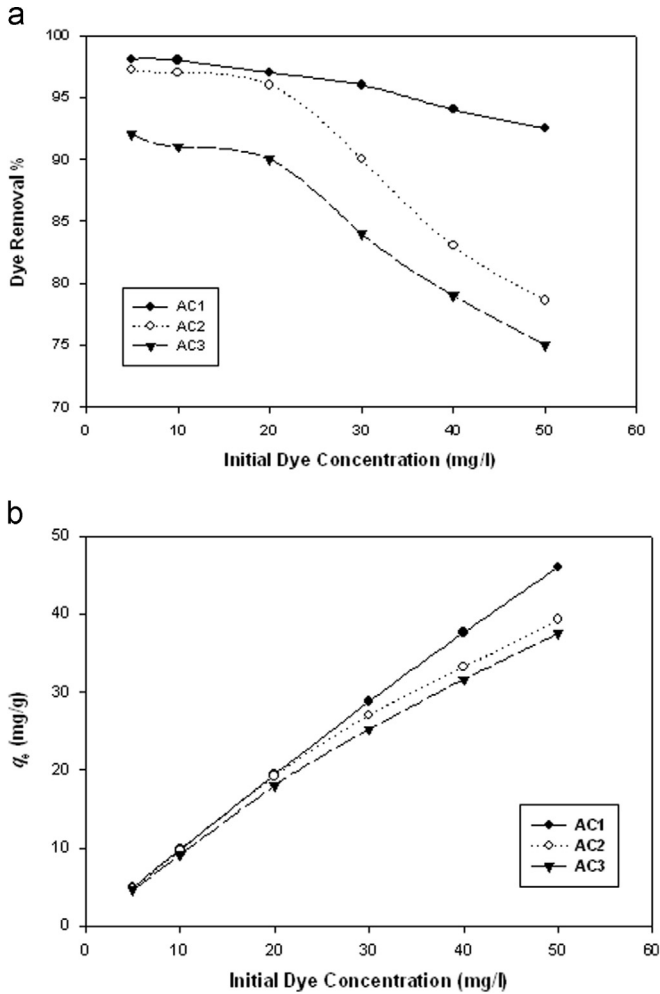


Fig. 3. Effect of initial dye concentration on (a) dye removal percent and (b) removal efficiency (mg/g).

(Fig. 4). Values of q_0 and b were calculated from the slope and intercept of the linear plots and are presented in Table 2. The correlation coefficient values for AC1, AC2 and AC3 were calculated as 0.9868, 0.9810 and 0.9121, respectively. These values show that the adsorption of MB dye on AC1 and AC2 follows Langmuir isotherm model. The essential characteristics of the Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter, R_L [22] defined by

$$R_L = 1/(1 + bC_0) \quad (4)$$

R_L values indicate the type of isotherm. The R_L value implies the adsorption to be unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$) [23]. The R_L values were found to be between 0 and 1 for dye concentrations of 5–50 mg/l for AC1 and AC2 (Table 2).

The Freundlich adsorption model stipulates that the ratio of solute adsorbed to the solute concentration is a function of the solution. The empirical model was shown to be consistent with an exponential distribution of active centers, characteristic of heterogeneous surfaces. The amount of solute adsorbed, q_e , is related to the equilibrium concentration of solute in solution, C_e . The Freundlich

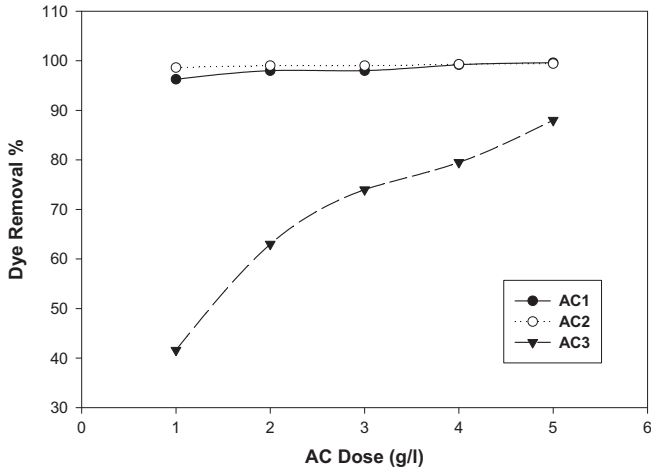


Fig. 4. Effect of different ACs doses on MB dye removal.

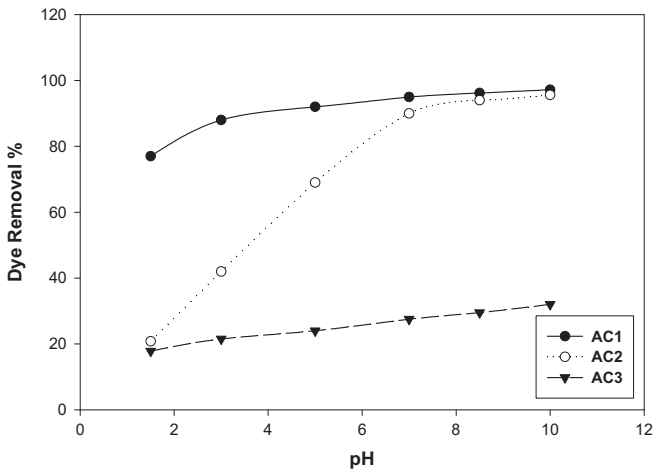


Fig. 5. Effect of initial pH on the removal of MB dye by different ACs.

heterogeneous sorption model is expressed by the following equation [24]

$$q_e = K_F C_e^{1/n} \quad (5)$$

The equation may be linearized by taking the logarithm of both sides

$$\ln q_e = \ln K_F + 1/n \ln C_e \quad (6)$$

The variations of $\ln q_e$ with $\ln C_e$ for MB on different ACs are shown in Fig. 7. It can be seen from the linear relationship that the adsorption of the dye on AC3 only follows Freundlich model ($R^2 = 0.9823$). The values of K_F and n obtained from the intercept and the slope, respectively are presented in Table 2. The $1/n$ value for AC3 is 0.810 (n is greater than 1), indicating that the adsorption of MB on AC3 is favorable [25].

From the data obtained from adsorption isotherms, it can be seen that the adsorption of MB on different ACs follows Langmuir model for AC1 and AC2 and Freundlich model for AC3.

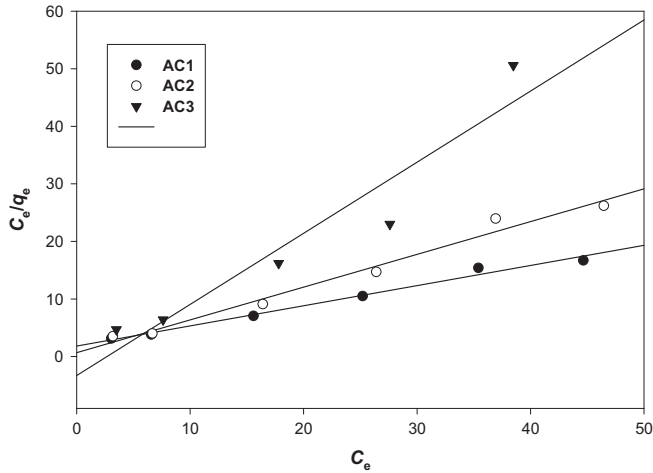


Fig. 6. Langmuir isotherm for adsorption of MB dye different adsorbents.

Table 2

Langmuir and Freundlich adsorption constants for adsorption of MB on ACs.

AC type	Langmuir constants				Freundlich constants		
	q_0 (mg/g)	b (l/mg)	R^2	R_L	$1/n$	K_F (mg/g (l/mg) $^{1/n}$)	R^2
AC1	28.65	0.0191	0.9868	0.511–0.913	0.349	0.7838	0.8775
AC2	17.57	0.0839	0.9810	0.192–0.704	0.567	1.0957	0.5084
AC3	0.809	–2.636	0.9121	–	0.810	7.6329	0.9823

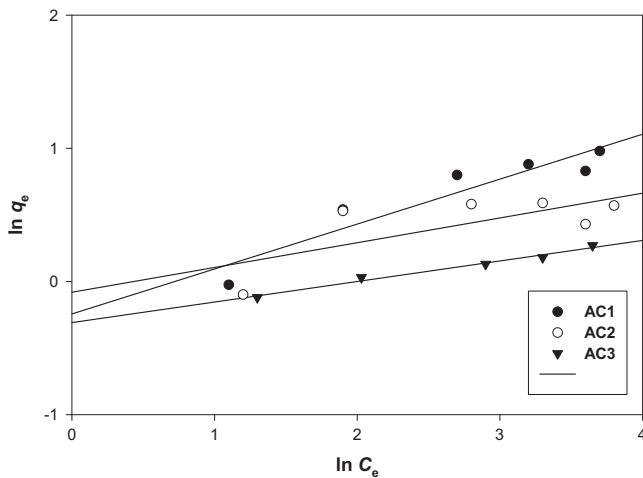


Fig. 7. Freundlich isotherm for adsorption of MB dyes on different adsorbents.

The maximum adsorption capacities (Q_0) of AC1, AC2 and AC3 for MB were compared with those reported in the literature for different activated carbons prepared from waste agricultural wastes and shown in Table 3.

Table 3
Comparison of adsorption capacities of different activated carbons for adsorption of MB.

Origin of AC	Q ₀ (mg/g)	Reference
Almond shell	1.3	[26]
Apricot stones	4.1	[26]
Coir pith	5.8	[27]
Hazelnut shell	8.8	[26]
<i>Posidonia oceanica</i> (L.)	285.7	[28]
agricultural wastes	398.2	[29]
Oil palm wood	90.9	[30]
biomass	259.2	[31]
buriti shells	274.6	[32]
Rattan sawdust	294.14	[33]
Date stones	316.1	[34]
AC1	28.65	Present work
AC2	17.57	Present work
AC3	0.809	Present work

4. Conclusion

In this work, three different activated carbons were prepared from corncob used successfully as adsorbing agents for the removal of MB dye from aqueous solutions. Adsorption was influenced by various parameters such as initial pH, initial dye concentration and dose of adsorbent. The maximum adsorption of MB dye by corncob occurred at alkaline solutions. Removal efficiency increased with decreasing dye concentration and increasing adsorbent dose. The Langmuir and Freundlich adsorption isotherm models were used for the description of the adsorption equilibrium of MB dye onto activated carbons prepared from corncob. The data were in good agreement with both Langmuir for AC1 and AC2 and Freundlich isotherm for AC3. Since corncob, an agriculture solid waste, used in this study is locally available; the adsorption process is expected to be economically for wastewater treatment.

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