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## Adsorption characteristics of chitosan hydrogel beads toward removal of Congo red dye from aqueous solutions

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The present study investigated potential application of biopolymer chitosan, in the form of gel beads, for the removal of Congo red (CR) dye from aqueous solution. Chitosan hydrogel beads (CHBs) were prepared by a coupled ionic and covalent co-cross-linking, employing citric acid (CA) and glutaraldehyde (Glu) as an ionic and a chemical cross-linker, respectively. Batch experiments were carried out to optimize the process conditions for efficient removal of CR dye and to determine the adsorption capacity of prepared CHBs. The results showed that the highest removal efficiency of CR was obtained in the pH range from 6 to 10, and that the equilibrium was attained after 180 min. The Langmuir isotherm model provided the best correlation for the adsorption of CR onto CA/Glu co-cross-linked CHBs, with the maximum adsorption capacity ( $q_m$ ) of 544.2 mg g<sup>-1</sup>.

### Introduction

In recent years, many investigations focus on the use of biopolymeric materials for the treatment of wastewaters containing dyes since these materials are non-toxic, biodegradable and can be obtained from renewable sources [1]. Among them, the biopolymer chitosan has caught particular attention because of its high affinity and adsorption capacity toward dyes. According to the literature data [2-4], the use of chitosan in the form of spherical gel beads, instead of flakes or powders, have shown better adsorption characteristics for the removal of dyes from aqueous solutions, primarily due to decreased crystallinity of hydrogels, improved porosity and large pore sizes. Because of that, the diffusion of dye molecules toward a large number of internal -NH<sub>2</sub> and -OH functional groups of chitosan may be facilitated, and consequently, its adsorption capacity could be enhanced.

The aim of the present study was to prepare CHBs via ionic/covalent co-cross-linking and to investigate their adsorption characteristics toward the removal of the anionic CR dye from aqueous solutions in a batch mode. The effects of the initial solution pH, contact time and initial CR concentration were evaluated with respect to the removal efficiency of CR dye from aqueous solution. Pseudo-first-order (PFO) and pseudo-second-order (PSO) adsorption kinetic models were employed to analyze the kinetics of CR adsorption on CHBs. The equilibrium adsorption data were analyzed using Langmuir and Freundlich isotherms.

### Experimental part

Chitosan flakes (medium molecular weight), glacial acetic acid (AA), Glu solution and CA were purchased from Sigma-Aldrich (USA), while the CR dye was purchased from Acros

Organics (Belgium). All the chemicals were analytical reagent grade and used without further purification.

Preparation of chitosan adsorbent in the form of hydrogel beads was done by dissolving 1 g of chitosan flakes in 40 cm<sup>3</sup> of (1) 2 wt% of AA, (2) 1 wt% AA and 1 wt% CA and (3) 2 wt% of CA. Afterward, 5% (v/v) Glu solution was added in each of the 2.5% (w/v) chitosan solutions and the mixtures were magnetically stirred for 2 h at room temperature. Then, the obtained solutions, separately, were dropped into 1 mol dm<sup>-3</sup> NaOH solution to form uniform CHBs. The samples of CHBs (denoted as CHB – 2% AA, CHB – 1% AA, 1% CA and CHB – 2% CA) were kept in the NaOH solution for 24 h. Finally, CHBs were rinsed with deionized water until neutral pH was reached and stored in deionized water until further use.

The study of CR adsorption onto CHBs was carried out in a batch mode. Typically, the experiments were conducted at room temperature by mixing approximately 0.5 g wet CHBs and 10 cm<sup>3</sup> of the CR solution of the desired concentration at pH 6. The mixtures were shaken at a speed of 150 rpm for 24 h. After that, the liquid phases were separated and the residual CR concentrations were determined by UV-Vis spectrophotometer (UV-2600, Shimadzu, Japan) at  $\lambda_{\max}$  of 497 nm. CHBs were dried in an oven at 60°C and then, the mass of the dried CHBs were weighed.

The effect of pH was studied by changing pH of the CR solution (25 mg dm<sup>-3</sup>) from 4 to 12. Kinetic adsorption experiments were performed by mixing 2.5 g wet CHBs and 50 cm<sup>3</sup> of the CR solution (25 mg dm<sup>-3</sup>) at pH 6. The adsorption continued for 24 h and the CR concentration in the solution was measured at predetermined time intervals. The effect of the initial dye concentration was investigated in the range from 1 to 1000 mg dm<sup>-3</sup> at pH 6. The obtained results were discussed in terms of removal efficiency,  $E$  (%), and adsorption capacity,  $q_e$  (mg g<sup>-1</sup>), at equilibrium defined by the equations:

$$E = \left( \frac{C_i - C_e}{C_i} \right) 100$$

$$q_e = \left( \frac{C_i - C_e}{m} \right) V$$

where  $C_i$  and  $C_e$  (mg dm<sup>-3</sup>) are the initial and equilibrium concentrations of CR in the solution, respectively,  $V$  (dm<sup>3</sup>) is the volume of the solution, and  $m$  (g) is the mass of the dried adsorbent.

## Results and discussion

The shape of the CA/Glu co-cross-linked CHBs was nearly spherical with the mean diameter of the gel beads about 3.0 mm. The water content of the three tested CHBs samples was in the range of 97.0-97.5%.

Variations in the initial solution pH affect the surface charge of the adsorbent and the degree of ionization of the adsorbate and, hence, influence the removal efficiency of the adsorbent. The effect of the initial solution pH on the CR dye removal by CHB – 2% AA, CHB – 1% AA, 1% CA and CHB – 2% CA was studied in the pH range of 4-12, while other experimental conditions were constant. The obtained results revealed that the highest removal efficiency (98-99%) of CR for the three tested adsorbents occurred in the pH range of 6-10. According to the literature data [5], it can be assumed that the removal of CR dye is driven by both electrostatic interaction between the positively charged surface of

tested CHBs and the anionic CR dye and some physical forces (like hydrogen bonds or Van der Waals forces). pH 6 was selected as optimal for further experiments.

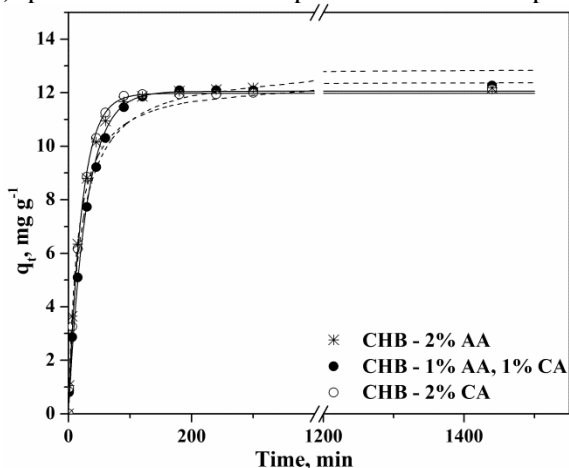


Figure 1. Effect of contact time on the removal of CR dye by CA/Glu co-cross-linked CHBs (solid and dashed lines represent PFO and PSO kinetic models, respectively)

As can be seen from Fig. 1, the adsorption of CR dye on CHB – 2% AA, CHB – 1% AA, 1% CA and CHB – 2% CA took place at relatively fast rate in the first 45 min, then slowed down and finally attained the equilibrium after 180 min of contact. The obtained experimental data were fitted by the non-linear forms of the PFO and PSO kinetic models. The resulting kinetic parameters and the determination coefficients ( $R^2$ ) suggested that PSO model is more suitable for describing the adsorption of CR dye onto the CA/Glu co-cross-linked CHBs.

The equilibrium adsorption data on the effect of the initial CR concentration were fitted using the non-linear Langmuir and Freundlich isotherm models in order to determine the surface properties and the affinity of the adsorbent. The corresponding isotherm parameters and the determination coefficients of these two models are reported in Table 1. Based on the values of the  $R^2$ , it is evident that CR adsorption by CA/Glu co-cross-linked CHBs can be better described by the Langmuir isotherm. CA/Glu co-cross-linked CHBs with the higher content of CA showed better  $q_m$  value compared to other samples, probably due to incorporation of additional carboxylic groups that have positive impact on the features of CHBs.

Table 1 Langmuir and Freundlich parameters for the adsorption of CR onto CA/Glu co-cross-linked CHBs

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	$q_m$ , mg g <sup>-1</sup>	$*K_L$	$R^2$	$n_f$	$**K_f$	$R^2$
CHB - 2% AA	445.8	0.039	0.992	3.52	64.43	0.955
CHB - 1% AA, 1% CA	490.4	0.040	0.999	3.07	65.06	0.963
CHB - 2% CA	544.2	0.046	0.999	2.98	69.49	0.952

13.  $*K_L$ , dm<sup>3</sup>/mg;  $**K_f$ , (mg/g)(dm<sup>3</sup>/mg)<sup>1/n</sup>

In conclusion, the results presented in this study indicate that CA/Glu co-cross-linked chitosan in the form of hydrogel beads can be regarded as an efficient and low-cost adsorbent, which could be potentially applied in the treatment of wastewaters containing



toxic anionic dyes such as CR. Further experiments will be oriented toward its application in dynamic systems.

## **Adsorpcione karakteristike hidrogelova hitozana za uklanjanje boje kongo crveno iz vodenih rastvora**

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Istraživanja u okviru ove studije okrenuta su ka ispitivanju mogućnosti primene hidrogelova hitozana, u obliku sfera, za uklanjanje boje kongo crveno (CR) iz vodenih rastvora. Sfere hidrogela hitozana (CHBs) pripremljene su spregnutim jonskim i kovalentnim umrežavanjem, pri čemu su limunska kiselina (CA) i glutaraldehid (Glu) korišćeni kao jonski i kovalentni umreživač, respektivno. Eksperimenti su rađeni u šaržnom sistemu kako bi se optimizovali uslovi za efikasno uklanjanje CR iz vode i odredili adsorpcioni kapaciteti sintetisanih CHBs. Eksperimentalni rezultati su pokazali da je efikasnost uklanjanja CR najveća u pH intervalu od 6 do 10, a da se ravnoteža uspostavlja nakon 180 minuta. Langmirova izoterma pokazala je najbolju korelaciju za adsorpciju CR boje na CA/Glu umreženim sferama hidrogelova hitozana, pri čemu maksimalni adsorpcioni kapacitet ( $q_m$ ) iznosi 544.2 mg g<sup>-1</sup>.

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