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The effects of bed column and compaction granulation processes of new granules on malachite green adsorption

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Abstract. A new generation of grains based on an iron organo-inorgano montmorillonite complex and gluten (G) as an inert bonding agent called Fe-COIMG and were prepared by the dry compression granulation method. Their efficiencies were evaluated in tests for the removal of Malachite green cationic dye (MG) by adsorption in fixed bed columns. The effects of dynamic adsorption parameters such as bed depth, initial concentration and inlet flow rate were investigated. The breakthrough curves were exploited by the Thomas and Yoon and Nelson models using nonlinear regression. The corresponding results showed a good agreement with the experimental results obtained indicating that the model predictions were appropriate for the design of the column.

Keywords: Granulation, Pillared clay, Gluten, Malachite Green, Adsorption.

1 Introduction

Adsorption is a physical technique which involves the adherence of particular molecules onto the surfaces of adsorbent. Adsorption techniques employing solid adsorbent have been widely used to remove certain classes of chemical pollutants from waste-water, such as cationic dyes. Over the past decade, the Pillared Inorganic–Organic Clays (PIOCs) have been used increasingly as an alternative to activated carbon and other adsorbents due to their environmental compatibility, low cost, high adsorption capacity and simplicity of use. They are prepared in various ways, generally by incorporation of metallic polycations replacing the exchangeable cations in the interlayer space of clay minerals. Organic–inorganic modification of clay minerals offers the potential to remove both organic and inorganic pollutants from wastes, due to their intriguing structural diversity, but present many difficulties for separation of effluents especially in continious adsorption [1-7]. In this way, Due to continuous wastewater flow, batch reactor, the most reported method in POICs and GOICs adsorption studies, is not appropriate for industrial applications. In order to obtain basic engineering data, continuous flow system is an effective process suitable for large wastewater volumes and cyclic sorption/desorption; fixed-bed reactor is a simple model for the determination of bed operation life span and regeneration time.

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The performance of such adsorption process is described through the concept of the breakthrough curve. The time for breakthrough appearance and the shape of the breakthrough curve are very important characteristics for process design because they directly affect the feasibility and economics of the sorption phenomena.

Following previous works [8–10], the present study was focused on evaluating the performance of the Iron Pillared Inorganic–Organic Clays with Gluten granules (Fe-PIOCG) prepared by compaction granulation in the removal of Malachite Green color from textile wastewater in a fixed-bed reactor. The color uptake capacity of granules was investigated as a function of flow rate, initial dye concentration.

2 Materials and methods

2.1 Preparation of sorbent

The adsorbent used in these studies was the Iron Pillared Inorganic–Organic Clays with Gluten granules (Fe-PIOCG) prepared by compaction granulation according to a previous method [3]. The $800-1200 \ \mu m$ size range of Fe-PIOCG was used in these studies.

2.2 Column adsorption experiments

Column experiments were performed in a fixed bed mini Perspex column reactor with an internal diameter of 1 cm and a length of 15 cm packed with Fe-PIOCG, size of granules is 800 to 1200 μ m. Wall effects were neglected since the column diameter was at least more than 10 times larger than the particles size. The experiments were conducted by pumping a MG solution at different concentrations 5, 10 and 20 mg/L in up flow mode through the fixed-bed with a peristaltic pump (ISMATEL 829B) at a specified flow rate.

And then in order to determine the effect of the presence of another competing dye, Rhodamine effluent (RB) is mixed in the solution with a ratio r = 1, and a concentration for each dye at 10 mg/L. Samples were collected at regular intervals for analysis from the top of the column and the residual concentrations of MG were determined by UV spectrophotometry using the Spectrophotometer model Shimadzu 170 UV/visible at $\lambda_{max} = 617$ nm for MG and 553 nm for RB.

3 Results and Discussion

After studying the discontinuous reactor adsorption of MG and RB dyes on Fe-PIOCG bridged clay grains, in single-component single systems and binary mixtures (multicomponent system), we were interested in examining the effectiveness of these new Fe-PIOCG grains in dynamic continuous reactor adsorption.

As previously notified, we worked with Malachite Green (MG) as a single solution. The operation is spread over a period of 24 to 100 hours (maximum) in order to be able to correctly draw the different bore curve profiles.

Parameters C_o (mg.L ⁻¹)	Volume (mL)	Rupture Time (Hours)	Saturation Time (Hours)	q (mg.g ⁻¹)
5	42000	25	140	33.76
10	39000	20	130	62.70
20	7500	0,5	25	24.11

Table 1 Parameters for MG adsorption as a function of initial concentration

The breakthrough curves (C/C0 = f(t)) obtained when studying the effect of the different operating parameters all have the same shapes.

3.1 Effect of initial dye concentration

The study of the influence of the initial concentration is carried out by varying the initial concentrations of the MG between 5, 10 and 20 mg. L^{-1} while maintaining the pH of the mixture at 6, the bed height (H) at 15 cm and the feed rate (Q) at 5 mL.min⁻¹.



Fig.1 Breakthrough curves showing the influence of the initial concentration on the adsorption of the MG.

Figure 1 shows the breakthrough curves obtained for the different concentrations studied. Examination of these curves shows that the overall appearance is a function of the initial MG concentration. Indeed, for an initial high concentration 20 mg.L⁻¹, the solute breakthrough time and adsorption equilibrium appear more quickly. In this case and due to the significantly higher mass transfer, the active sites of the adsorbent grains become saturated more quickly.

On the other hand, and for an initial low concentration of solute 5 mg. L^{-1} , diffusion becomes slower, which results in a slower mass transfer and a late appearance of the breakthrough curves.

3.2 Effect of flow rate

In this experiment, the breakthrough curves obtained at different flow rates (2 and 5 ml.min⁻¹) with an initial concentration of 10 mg L^{-1} and a bed depth of 15 cm with an initial pH of 6 are given in Figure 2.



Fig. 2 Influence of feed rate on MG breakthrough curves.

These findings are consistent in that the higher the feed rate, the higher the quantities of dye brought into contact with the grains and the faster the saturation of the bed. The results we obtained are in agreement with those observed by Cheknane et al and Ararem et al [1, 2, 3].

3.3. Effect of bed height

The influence of bed height is studied by comparing the breakthrough curves obtained (Figure 3) for MG with the heights of 5 and 15 cm while keeping constant the pH u medium, the initial dye concentration of 10 mg.L^{-1} and the feed rate at regime II equal 5 ml.min⁻¹.



Fig. 3 Breakthrough curves showing the influence of bed height on MG adsorption.

Examination of the breakthrough curves reveals a delay of these curves at higher heights of the adsorbent bed corresponding to high masses of GSMPM-Fe grains that provide more adsorption sites.

Thus, at relatively shallow bed depths, the breakthrough curves become tenser and reveal a rapid exhaustion of the adsorbent beds.

4 Conclusion

Synthesized granules were characterized with various techniques and dynamic adsorption of Malachite green MG over it was carried out successfully. Adsorption of MG was found to be high initially and it then decreases with an increase in the injected volume. It was found that adsorption increases with increase in the contact time between MG dye and granules adsorbent.

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