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HPLC-DAD DETERMINATION OF METHYL ORANGE DYE FROM SYNTHETIC SOLUTIONS AFTER ADSORPTION TREATMENT WITH ACTIVATED CARBON

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Introduction

Dyes are the first known contaminants in industrial wastewater streams. Various industries such as food processing, paper, cosmetics, leather, textiles, printing, and pharmaceuticals discharge large amounts of wastewater containing dyes polluted with toxic compounds into the environment. Annually, it is estimated that 50,000 tons of organic dyes are disposed of worldwide.

Adsorption is one of the methods that has received a lot of attention due to its advantages such as: moderate cost, process flexibility without excessive sludge production, process simplicity, high efficiency and speed.

Materials and methods

Methyl Orange dye (MO, purity > 95%) was purchased from Sigma-Aldrich (Germany), activated carbon (powder, 10 - 50 µm particle size, 256 m^2/g specific surface area, 14.7 Å pore size and 870 m^2/g total pore area) were bought from Trace Elemental Instruments (Delft, Netherland). Acetonitrile (HPLC grade) and ammonium acetate (≥ 98%), used for HPLC mobile phase, were purchased from Merck (Germany). Sodium hydroxide (≥ 99%) and hydrochloric acid (37%) were used for pH adjustment, were acquired also from Merck (Germany).

The analytical method used in this study was High Performance Liquid Chromatography (HPLC) with UV detection, the wavelength at which the maximum absorption was reached, λ = 425 nm. Chromatographic column used was Acclaim Surfactant Plus (15x3 mm, 3 µm). For this study, 100 mM ammonium acetate at pH 5 (A) and acetonitrile (B) in the isocratic regime (50/50) were used as the mobile phase. The flow rate of the mobile phase was 0.5 mL/min, the injection volume was 10 µL, and the MO elution was carried out in 10 minutes. The range of linearity was between 0.1 and 100 mg/L, with a correlation coefficient R^2 = 0.99, and the quantification limit LOQ = 0.1 mg/L.

To establish the optimum conditions and obtain the highest removal efficiency, the effects of some the operational parameters were tested, namely: activated carbon dose (0.001–0.010 g AC), interaction time (1-120 min), beginning MO dye concentration (5-100 mg/L MO) and pH (3-11, using HCl and KOH solutions of different concentrations). In this study the working volume was set as 50 mL. The MO removal percentage (% R), at different times (t), was determined using the following equation:

$$\% R = (C_0 - C_t) / C_0 \times 100$$

where, C_0 - initial concentration of MO in the solution (mg/L); C_t - MO concentration at time t (mg/L).

Results and conclusions

In this study, the removal of methyl orange dye from aqueous solutions was followed, using active carbon as adsorbent material. Samples were collected at well-established time intervals. The supernatant was centrifuged at 5,000 rpm for 5 min and the MO concentration was analysed using HPLC technique (Figure 1).

Fig. 1. HPLC chromatogram obtained for 10 mg/L MO (a); The UV-VIS spectrum registered for the MO dye (b).

The optimal conditions of the studied remediation process were determined through experiments in which four essential parameters, the activated carbon dosage, the initial concentration of MO, the pH and the contact time in the solutions subjected to the remediation process were varied.

As it can be seen in Figure 2a, the removal percentage of methyl orange increased from 35% to 94% when the amount of the activated carbon was increased from 0.001 g to 0.01 g. Between 0.007–0.01 g of activated carbon, the graph become flat, indicating that the amount of AC is more than sufficient to adsorb all the MO molecules. Data presented in Figure 2b, revealed a fast adsorption process of MO, on the adsorbent material, the time necessary to reach the equilibrium was only 30 min. The highest removal efficiency was observed at 5 mg/L and 10 mg/L MO (Figure 2c).

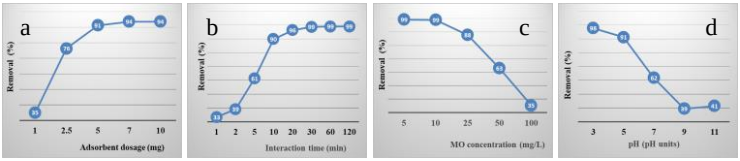

Fig. 2. The effect of adsorbent amount (a), time contact (b), initial dye concentration (c) and pH (d), on the MO adsorption by the activated carbon

Further mathematical modelling based on the results showed a limited number of active sites on the specific surface of AC. pH prove to be a very important parameter for MO adsorption, influencing the interactions between the activated carbon and the MO molecules. The maximum removal efficiency of MO was obtained at pH 3, namely 98%, and drastically decreased to 42% with the increasing of pH at 11.

The results obtained in this study revealed that activated carbon is an efficient adsorbent material for the removal of the methyl orange dye from aqueous solutions.