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Treatment of reactive dye solutions by physicochemical combined process

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ABSTRACT

Color and turbidity removal from solutions of two reactive dyes: brilliant blue Levafix EBRA and gold yellow Levafix EG were studied. Coagulation–flocculation by polyaluminium hydroxide (PAH) and bentonite, followed by adsorption on powdered activated carbon (PAC) techniques were used. Optimal parameters of coagulation-flocculation (jar tests) and adsorption (temperature, pH, contact time and PAC concentration) were determined under optimal conditions for maximal dye elimination. 99.89% and 99.02% of color removal were obtained from 100 mg/L of dye solutions, respectively. Bentonite and aluminum used concentrations were 0.50 g/L and 27.6 mg/L (blue EBRA); also 0.37 g/L and 15.0 mg/L (yellow EG), coupled with 1.0 g/L of PAC for each dye. At suitable conditions this combined process was able to efficiently remove color from solutions containing reactive dyes and produce easily settle-able sludge, using moderate amounts of reagents, and thereby the low-cost of treatment.

Keywords: Reactive dye solutions; Coagulation; Flocculation; Bentonite; PAC; Adsorption

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1. Introduction

Large amounts of dyes are used for various industrial applications, especially in the textile industries [1]. A significant proportion appears in the waste waters that must be treated before their discharge into the environment. Reactive dyes belong to the group of anionic dyes soluble in water, such as acid and direct dyes. They are the most used in the textile industry for dyeing cotton. They have complicated aromatic structures that resist to degradation in conventional waste water treatment process because of their stability to oxidizing agents and micro organisms [2]. However, removal of these dyes from textile effluent is a major environmental challenge and there is a permanent requirement to develop an effective processing technology which can efficiently remove these dyes [3].

Elimination of these dyes by the conventional biologic treatments is usually insufficient. Generally they are non-biodegradable and require intense physicochemical processes to be eliminated [4–6]. However, in several cases the used methods are not effective, so the pollution is transferred from a phase to another, and the problem remains unsolved. Moreover, the derivatives of their degradation have harmful effects on the aquatic life ecosystems and causes serious problems [7–10].

Several methods have been proposed for treatment of colored waste water, such as: chemical oxidation [11–13], electrolysis [14–16], biodegradation [17,18], adsorption [19–21], chemical coagulation [22–25] and membrane processes [26–28].

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The experiments showed that conventional biological processes are less efficient in degradation of dye due to their complex structure and molecular size [3]. The number of sulphonate groups in the dye molecule is inversely proportional to the effectiveness of the biological treatment [29]. Shaul et al. [9] showed that on eighteen dyes belonging to various groups, only three were biodegradable. The advanced processes has a good potential to eliminate whole organic carbon, but its effectiveness only for very low concentration of dyes [3,7], the complexity and the cost of the process pose serious problems [30]. Moreover, certain products of oxidation are toxic such as the halogenous products from aldehydes, acids or others. These products increase the carcinogenic properties of dyes [29]. Due to their adaptation at different charges of pollutant and different flow rate, the electrochemical processes are the best, but formation of iron hydroxide sludge is a big disadvantage [22].

Both coagulation and adsorption processes have individually proven to be highly effective in the decolorisation, but the cost of sludge treatment and active carbon powder are relatively high [3,22]. Moreover, the regeneration of the activated carbon is a very delicate and expensive operation [31,32]. Membrane processes are known for their efficiency in the removal of reactive dyes, the disadvantages of these methods are: a high pressure process, a fouling with high concentrations and an insufficient quality of the treated waste water [22]. Kacha et al. [33] showed that the coagulation-flocculation of the acid dye mixtures by the polyaluminium hydroxide associated with bentonite eliminates color efficiently (more than 95%). The presence of bentonite improves quality of the treatment and generates a good decantation of formed sludge [34]. Nevertheless, treated water containing only 1 mg/L of dye is considered as tinted [7]. Other authors are aiming the maximum elimination of the dyes by coupled methods [8,35,36]. The purpose of this work is the maximum of color removal from two reactive dye solutions, by coagulation/adsorption. The used combined process is summarized in a coagulation–flocculation using polyaluminum hydroxide (PAH) associated with bentonite, followed by adsorption of the residual color on activated carbon (TANAC 4500).

2. Materials and methods

2.1. Materials

Bentonite produced and commercialized by national company (ENOF, Maghnia, Algeria) was used. It is a fine powder, easy to use, and of high specific area with montmorillonitic properties [34]. Bentonite was freshly put in suspension in stirred distilled water at a concentration of 1 g/L before being added to the reactor content. It is maintained under continuous agitation in a flask of glass at 20°C.

Two synthetic reactive dyes including azoic bridges in their molecules, with one or many sulphonic groups, enabling them to be anionic in aqueous solution [7] were used: brilliant blue Levafix EBRA and gold yellow Levafix EG commercially available, purchased from DyeStar Co. (France). These two dyes called also: reactive blue 114 and reactive yellow 27 contains respectively chromophoric groups resulting primarily from the families from: difluorochloropyrimidine and dichloroquinoxaline (Figs. 1 and 2). Their maximum wavelengths of absorption (λ_{max}) are: 590 and 432 nm, respectively.

The polymer of aluminum hydroxide solution (PAH), also called Al_{13} was prepared starting from a solution of hexahydrated aluminum chloride ($AlCl_3.6H_2O$, Aldrich), pre-neutralized by sodium hydroxide solution (NaOH, Prolabo) until a hydroxide/aluminum hydrolysis ratio OH/Al = 2.25. The final aluminum concentration in the solution is 0.1 M [33]. The polymerization was carried out in dark glass reactors at 20°C. Bentonite was always added to the stirred colored solution before polyaluminum hydroxide.

2.2. Coagulation studies

Coagulation studies were carried out in 500-cm^3 batch reactors mechanically stirred, using jar-test apparatus. The temperature of solutions was kept constant at 20°C. The pH was adjusted with a 0.1 N hydrochloric acid, HCl (Cheminova Internacional, S.A.) or sodium hydroxide

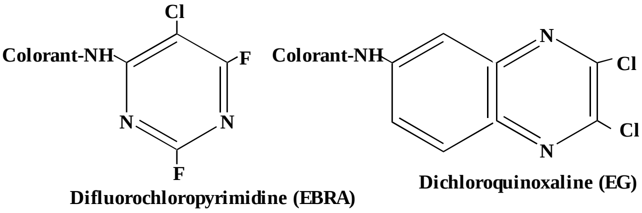


Fig. 1. Molecule structures of the reactive dyes.

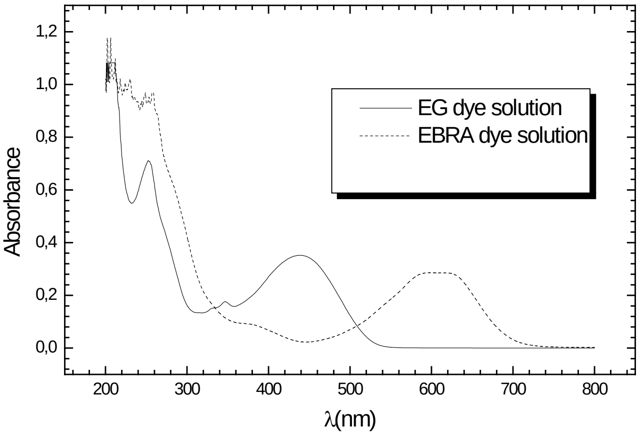


Fig. 2. UV spectra of 100-mg/L EBRA and EG dye solutions.

NaOH solution. In order to standardize the tests, coagulant (PAH) was added initially, under energetic agitation (250 rpm during 2 min), then moderate agitation (60 rpm during 20 min). After, the solutions were decanted for 24 h, a fraction of the supernatant was taken and then color and turbidity were measured. Dye concentrations in water were determined using UV-visible spectrophotometer (SHIMADZU UV-2401PC, Japan). The calibration curves in the range of concentration from 0 to 100 mg/L obey the Beer–Lambert law. Residual dye concentrations were deduced from absorbance values, according to the relation:

$$C = \frac{A}{A_0} C_0 \tag{1}$$

where C_0 and C are the initial and equilibrium (or residual) liquid-phase concentrations (mg/L), A_0 and A are the initial and equilibrium absorbances, respectively. Turbidity was measured using a turbidity meter (HANNA Instruments, LP-2000, Hungary).

2.3. Adsorption studies

Adsorption studies were carried out using commercially available powdered activated carbon (PAC) called TANACARBO 4500, purchased from TANAC S.A. (Montenegro, RS, Brazil). Its physical properties determined by Garnier et al. [37] are listed in Table 1. Increasing amounts of PAC were added to 100 mL of decanted water in a stirred (300 rpm) flask, placed in thermostated baths for 24 h at 25°C. Solid-liquid separation was carried out by filtration under vacuum conditions. The dye concentration was then measured the filtrate.

The adsorption capacity (Q) of the TANACARBO 4500 was determined, for both dyes, according to the expression:

$$Q = \frac{V\,(C_{R_0} - C_R)}{m} \tag{2}$$

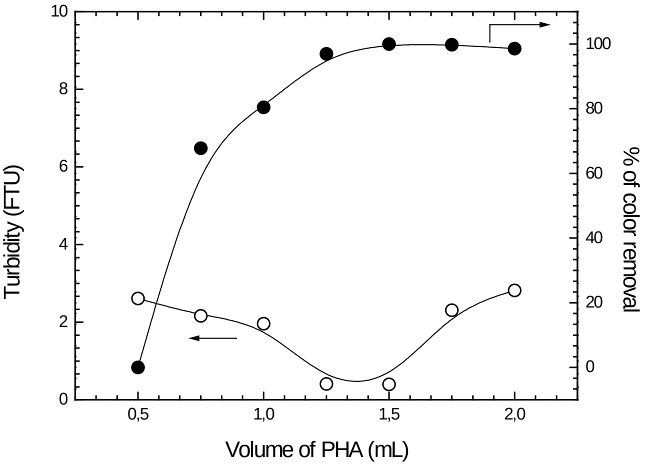
where C_{Ro} and C_R are the liquid-phase residual initial and equilibrium concentrations (mg/L), respectively, V is the volume of solution L, and m is the weight of dry activated carbon (g).

Table 1
Physical properties of powdered activated carbon TANACARBO 4500

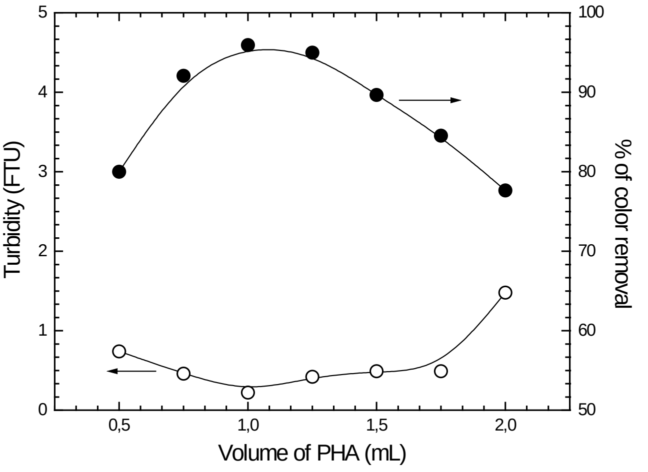
S_{BET} (m^2/g)	740
V_m (m^2/g)	169
C_{BET}	1267
$S_{\mathrm{micropore}}$ (m^2/g)	525
S_{mesopore} (m^2/g)	270
S_{total} (m^2/g)	795

3. Results and discussion

To determine the approximately the volume of PAH able to coagulate the mixture of bentonite and dye in solution, 20 mg/L was initially used as an initial concentration of blue EBRA and yellow EG, mixed with 75 mg of bentonite. The maximum of decolorization was obtained when added. PAH volumes were 1.5 and 1.0 mL (4.05 and 2.7 mg of Al), giving an aluminum/bentonite ratio of 0.054 and 0.036 mg/mg, respectively (Figs. 3a and 3b). To determine the exact concentration of dye in solution to be removed by these amounts of PAH/bentonite, further solutions with increasing dye concentrations were tested. A higher decolorization was observed up to 30 mg/L (from 95.06% to 99.82%) and 40 mg/L (from 96.37% to 97.14%) dye concentration, respectively. After



(a) Blue EBRA

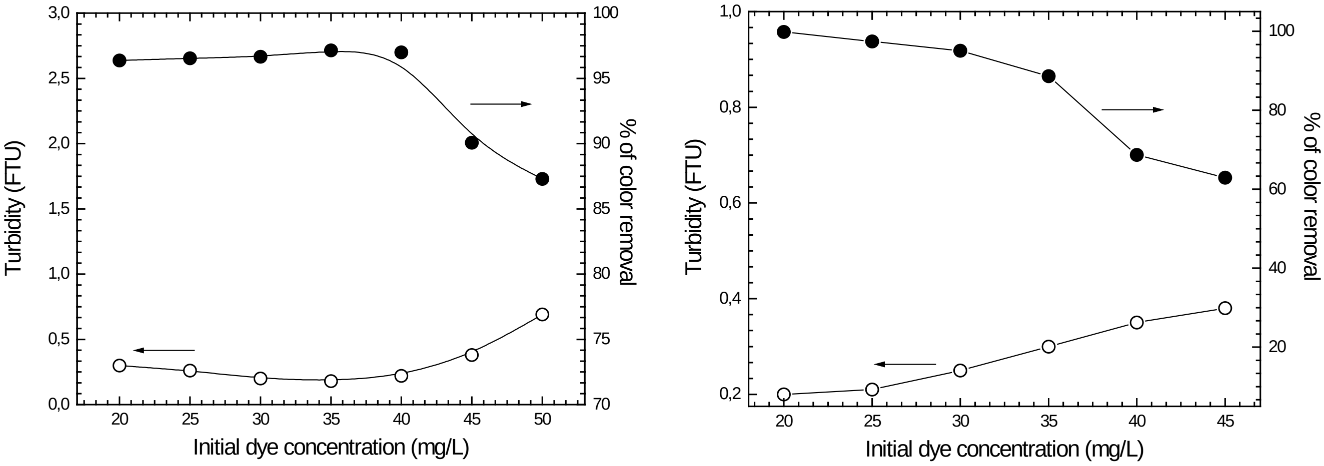


(b) Yellow EG

Fig. 3. Coagulation–flocculation of dyes by PAH/bentonite. Turbidity and color removal as functions of volume of PHA. V_{sample} = 500 mL; C_0 = 20 mg/L; $m_{\mathrm{bentonite}}$ = 75 mg; pH$_i$ = 7 ± 0.05; T = 20°C.

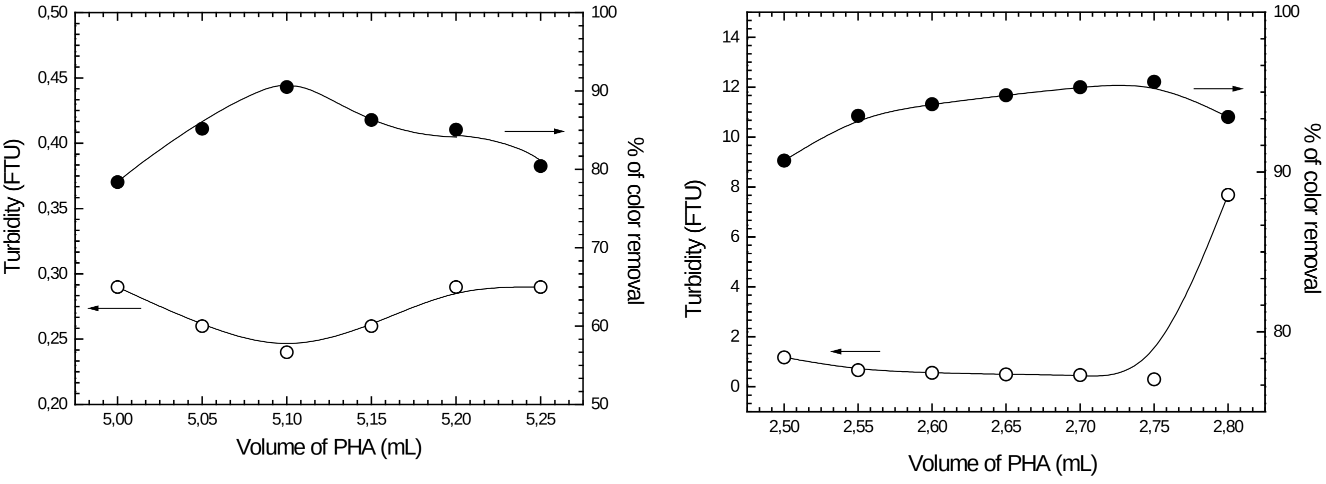
these two concentrations the decolorization efficiency decreased with increasing of dye concentration in the solution (Figs. 4a and 4b). According to these results, for maximum dye abatement of 100-mg/L dye solution, the proportions of PAH/bentonite in the reactor will be as follow: 5.0 mL/250 mg (blue EBRA) and 2.5 mL/187.5 mg (yellow EG). Figs. 5a and 5b show that this assumption is not so exact; the volume of PHA required, in both cases, increased: 5.10 mL and 2.70 mL, respectively, indicating that there is no a linear relationship between the color removal and the amount of PHA/bentonite. This behavior of dye is attributed to the various interactions between the three species in solution which makes a complex precipitation reaction. Coagulation-flocculation of dyes by PHA/bentonite remains, in spite of that, an effective process. Color removal from 100-mg/L dye solutions was 90.51% and 95.65%, respectively. In addition, turbidity removal was 99.37% and 99.56%, respectively. The following coagulation runs were carried out under these conditions.

In several cases color removal from dye solutions by coagulation–flocculation can be influenced by the initial pH of solutions. Aluminum in aqueous solutions exists in several forms, such as monomeric species Al^{3+}, $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_4^-$, polycations species such as $Al_{13}O_4(OH)_{24}^{7+}$, or gibbsite solid form of $Al(OH)_3$. The existence of one or more of these aluminum species is



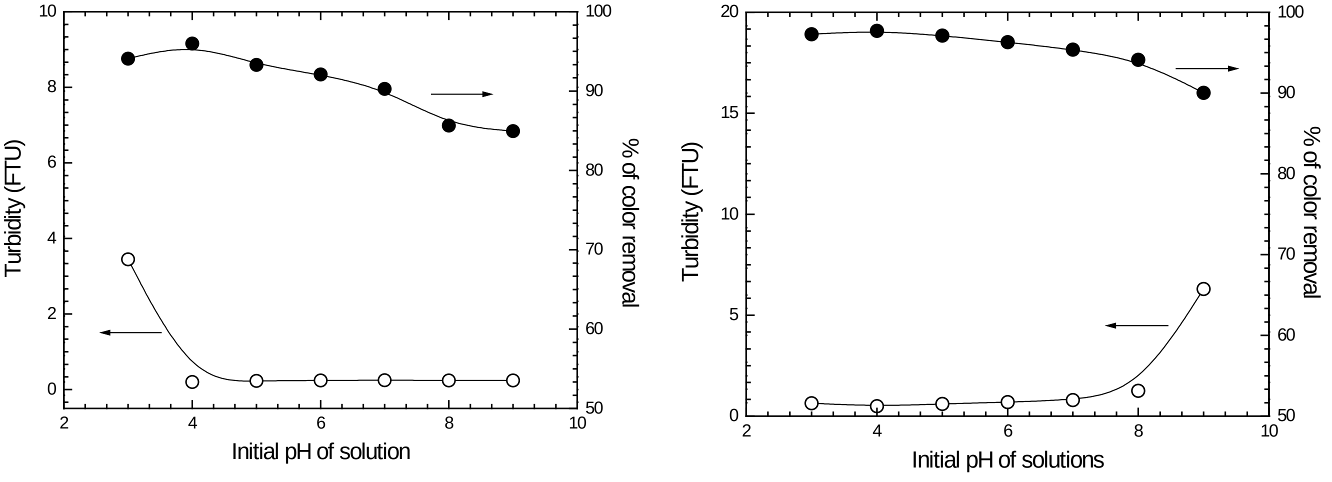
(a) Blue EBRA (b) Yellow EG

Fig. 4. Coagulation–flocculation of dyes by PAH/bentonite at optimal doses of PAH. Turbidity and color removal as functions of initial dye concentration. V_{sample} = 500 mL; $m_{bentonite}$ = 75 mg; V_{PAH} = 1.5 mL (a) and 1.0 mL (b); $pH_i = 7 \pm 0.05$; T = 20°C.



(a) Blue EBRA (b) Yellow EG

Fig. 5. Coagulation–flocculation of dyes by PAH/bentonite. Turbidity and color removal as functions of volume of PHA. V_{sample} = 500 mL; C_0 = 100 mg/L; $m_{bentonite}$ = 250 mg (a) and 187.5 mg (b); $pH_i = 7 \pm 0.05$; T = 20°C.



(a) Blue EBRA

(b) Yellow EG

Fig. 6. Coagulation–flocculation of dyes by PAH/bentonite at optimal doses of PAH. Turbidity and color removal as functions of initial pH of solutions. V_{sample} = 500 mL; C_0 = 100 mg/L; V_{PAH} = 5.1 mL (a) and 2.7 mL (b); T = 20°C.

strongly depending on Al^{3+}/OH^- ratio in the solution and thus, on the pH of the medium. Generally, pH ranged from 3 to 7 generates higher coagulant species, especially poly-nuclear forms of aluminum because the Al^{3+}/OH^- ratio to be situated between 2 and 2.5 (mol/mol), which induced the highest removal of dyes [33]. The effect of pH is investigated between 3 and 9. Figs. 6a and 6b show that the decolorization slightly increases with the decrease of pH. Best results were obtained for pH = 4 for both dyes, with a preference for the yellow EG compared to the blue EBRA (97.18% and 95.15% of color removal, respectively). It was shown that the increase in the initial pH of the solutions between pH 3 and 9 caused a loss of efficiency of approximately 10% for EBRA solutions (from 94.08% to 84.96%), and only 7% for EG solutions (from 97.29 to 90.04). These values are comparable with those previously obtained with acid dyes [33]. Turbidity elimination is not influenced by the pH ranging between 4 and 8 for both dyes. Nevertheless, it was observed that at pH lower than 4 (blue EBRA) or higher than 8 (yellow EG) the elimination of turbidity becomes underprivileged. At pH = 4, residual turbidity was lower than 0.5 FTU for the two dyes, giving 99.47% and 99.29% of elimination, respectively.

Reactive dyes are usually recalcitrant and difficult to remove by one step of decolorization treatment. The adsorption of residual color from treated dye solutions, under optimal conditions, by coagulation on activated carbon was studied as a complementary treatment. 100 mL of supernatant of each decanted solution were investigated. The adsorption efficiency depends on several physicochemical and operational conditions, such as the mass of activated carbon added to the solution, pH of medium and contact time. The effect of these parameters on color and turbidity removal were checked at 25°C.

The number of adsorption sites presented by the carbon amount must be equivalent to the dye molecules or ions to remove from solution. To determine the effect of PAC mass on the adsorption efficiency, several masses of carbon ranged between 25 and 150 mg were added to 100 mL of decanted solution, maintained under agitation during 24 h at 300 rpm. Fig. 7 shows that the adsorption equilibrium was reached when the concentration of PAC was 1g/L. This relatively moderate mass fixes 87.48% and 57.67% of residual color of blue EBRA and yellow EG, at pH = 4.30, respectively. Although the concentration of dyes in solution is low, the PAC remains effective and

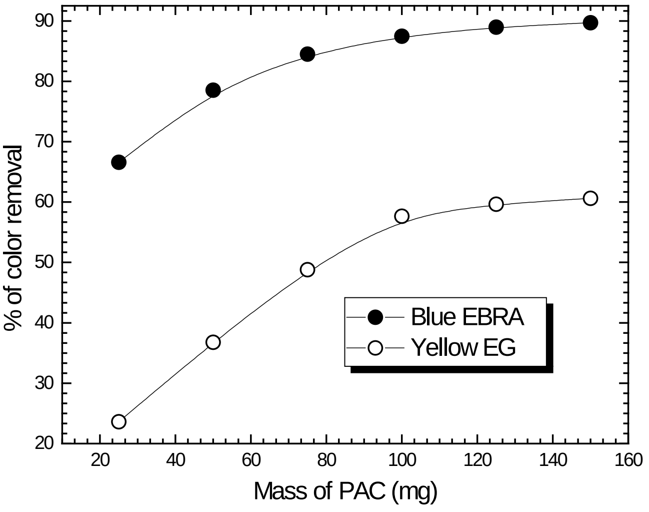


Fig. 7. Adsorption of residual dye concentration on PAC. Color removal as a function of the mass of PAC. V_{sample} = 100 mL; $C_{\text{Ro(EBRA)}}$ = 4.85 mg/L; $C_{\text{Ro(EG)}}$ = 2.82 mg/L; pH_i = 4.30 ± 0.05; T = 25°C; Agit. = 300 rpm; Contact time = 24 h.

offers a strong affinity between the dye molecules and the carbon surface.

The effect of the initial pH of solutions on adsorption was investigated between 3 and 10. The efficiencies of adsorption were slightly affected by the pH value, but pH 6 seems to be optimal for a maximum elimination of the two dyes. Nevertheless, better efficiencies were observed for the blue EBRA compared to the yellow EG: from 86% to 99%, and from 55% to 61% of residual concentration, respectively; that is due probably to the chemical structure. Adsorbability is enhanced when the molecular size and aromaticity of the dyes increase and the solubility decrease [7]. EG is a larger molecule than EBRA but it has a more important ionicity, which decreases absorbance on PAC. In addition, the residual concentration of the blue EBRA in solution is twice more important than that of the yellow EG (4.85 and 2.82 mg/L, respectively), which enabled it to have more contact with the carbon surface (Fig. 8).

Contact time is a very influent parameter in fixing of residual dye concentrations. Short contact time appears insufficient, because it does not permit the formation of the physicochemical bonds between the aqueous dye and the adsorption sites of the solid phase. In addition, more long contact time is some times useless. Fig. 9 shows that the adsorption equilibrium was reached after 6 h of stirring for both the dyes. The adsorption isotherms are adjustable on the Freundlich equation which is an empirically derived model. The mechanism of sorption is mixed between fixed sites and a pseudo-partition process; many layers of fixed sites exist all with different enthalpies (Figs. 10a and 10b):

$$Q = K_F\ C^{1/n} \tag{3}$$

where Q is the adsorption capacity of PAC (mg/g), C is the liquid-phase residual equilibrium dye concentration (mg/L), K_F and n are the Freundlich constants which depend on the nature of adsorbed species and the fixing conditions. The Freundlich relation can be written in its linear form as follows:

$$\log\ Q = \log\ K_F + \frac{1}{n}\log\ C \tag{4}$$

The adsorption capacity of blue EBRA is higher than that of yellow EG (4.75 mg/g and 1.10 mg/g, respectively). The Freundlich constants are listed in Table 2. Table 3 recapitulates the main results obtained by the combined coagulation and adsorption process.

4. Conclusions

The coagulation–flocculation with PAH and bentonite is an efficient process to remove dyes and turbidity from solutions. However, adsorption on PAC can be used as a complementary treatment to remove recalcitrant reactive dyes. The influence of pH on the equilibrium removal is negligible at usual initial concentrations; however, for the coagulation process, the removal rate is a decreasing function of the pH. The adsorption isotherms are adjustable to the Freundlich model. The combination of adsorption with coagulation is able to eliminate almost the whole of reactive dyes from synthetic solutions.

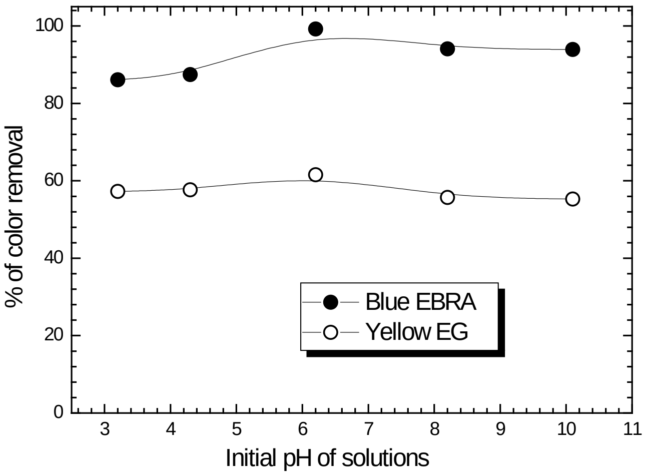


Fig. 8. Adsorption of residual dye concentration on PAC. Color removal as a function of initial pH of solutions. V_{sample} = 100 mL; $C_{\text{Ro(EBRA)}}$ = 4.85 mg/L; $C_{\text{Ro(EG)}}$ = 2.82 mg/L; m_{PAC} = 100 mg; T = 25°C; Agit. = 300 rpm; Contact time = 24 h.

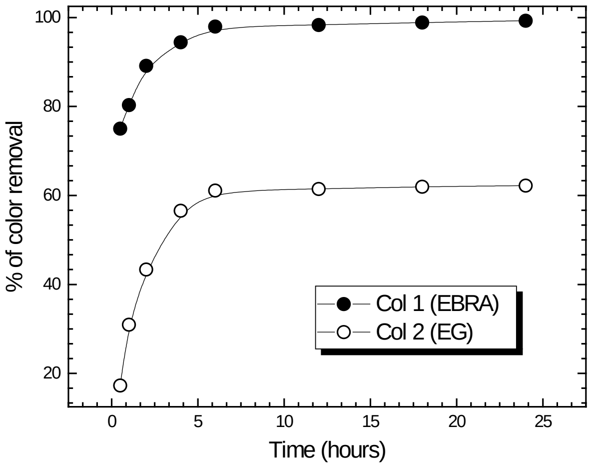


Fig. 9. Adsorption of residual dye concentration on PAC. Color removal as a function of contact time. V_{sample} = 100 mL; $C_{\text{Ro(EBRA)}}$ = 4.85 mg/L; $C_{\text{Ro(EG)}}$ = 2.82 mg/L; pH_i = 6 ± 0,05; m_{PAC} = 100 mg; T = 25°C; Agit. = 300 rpm.

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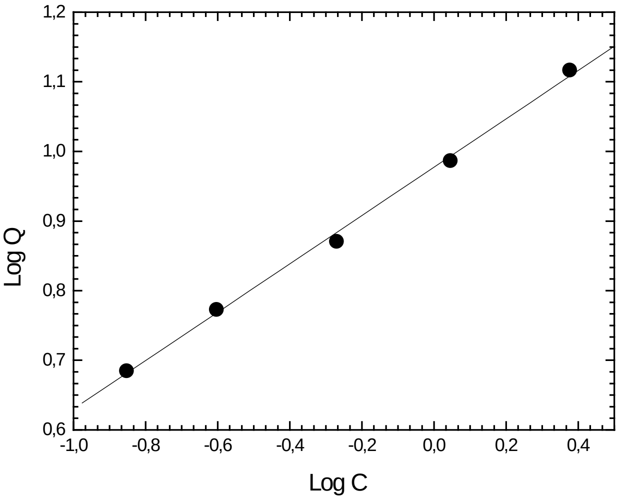
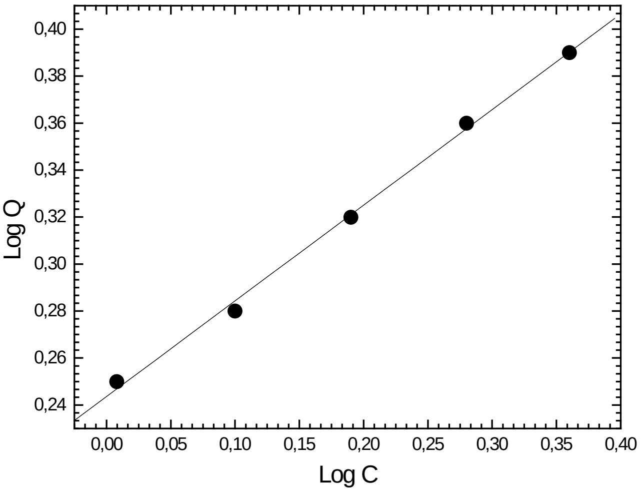


(a) Blue EBRA



(b) Yellow EG

Fig. 10. Adsorption of residual dye concentration on PAC. Modeling by a Freundlich equation.

Table 2
Freundlich parameters

Dye	K_F	n	Q (mg/g)
Brilliant blue EBRA	9.49	2.88	4.75
Gold yellow EG	1.75	2.45	1.10

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Table 3
Summary of the main results obtained by the two methods: coagulation and adsorption

Treatment parameters	Coagulation		Adsorption	
	Blue EBRA	Yellow EG	Blue EBRA	Yellow EG
V_{sample} (mL)	500	500	100	100
C_0 (mg/L)	100	100		
$m_{\text{bentonite}}$ (mg)	250	187.5		
V_{PAH} (mL)	5.10	2.70		
pH	4±0.05	4±0.05	6±0.05	6±0.05
C_{Ro} (mg/L)			4.85	2.82
m_{PAC} (mg)			100	100
Contact time (min)	20	20	360	360
% of colour removal	95.07	97.5	97.97	61.10
% of cumulated elimination	99.89 (Blue EBRA) 99.02 (Yellow EG)			

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