

## Evaluation of the performance of adsorption and coagulation processes for the maximum removal of reactive dyes

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### Abstract

Physicochemical processes of adsorption and coagulation were systematically evaluated for the removal of reactive dyes (Orange 16 and Black 5) in a laboratory scale experimental setup. The effectiveness of combined processes of adsorption and coagulation for complete removal of dyes was also investigated. The right sequence of operation was identified for the combined treatment system. A coconut-based powdered activated carbon (PAC) was used as an adsorbent and alum chloride was chosen as a coagulant. The results indicated that adsorption capacity of Orange 16 was much higher than that of Black 5. Also, adsorption capacity on PAC was highly dependent on the pH of solution. The dye removal efficiencies for 100 mg l<sup>-1</sup> of Black 5 and Orange 16 by coagulation were almost 99% and 80% under the determined optimal conditions for Black 5 (250 mg l<sup>-1</sup> coagulant dose and pH 6) and for Orange 16 (350 mg l<sup>-1</sup> coagulant dose and pH 6). Coagulation followed by adsorption was found to be more efficient than having adsorption prior to coagulation. There was a significant increase in adsorption capacity of PAC for the combined process where coagulation was carried out prior to adsorption. The combined coagulation–adsorption process has the capability of complete dye removal and thus total decolourization, reduction in coagulant and adsorption amounts and thereby produce less amount of sludge. © 2005 Elsevier Ltd. All rights reserved.

**Keywords:** Coagulation; Adsorption; Reactive dyes; Combined treatment system; Aluminium chloride; Activated carbon

### 1. Introduction

Synthetic dyes have been extensively used in many industries such as textile, leather tanning, paper production, food technology, photoelectrochemical cells, hair colorings etc. [1–5]. Although the exact number and amount of dyes produced in the world is not known,

it is estimated to be more than 100,000 commercially available dyes with over  $7 \times 10^5$  tons of dye-stuff produced annually [1–3]. However, it has to be emphasized that the overwhelming majority of synthetic dyes currently used are the highly water soluble azo-reactive dyes. Azo dyes are characterized by the existence of nitrogen nitrogen double bonds and the presence of bright colour is due to these azo bonds and associated chromospheres. Even the presence of very low concentrations of dyes (less than 1 mg l<sup>-1</sup>) in the effluent is highly visible and is considered undesirable. These reactive dyes are the most problematic compared

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to other forms of dyes [6] and must be removed from wastewater completely. Apart from being aesthetically displeasing, these synthetic dyes cause considerable environmental pollution, are toxic to some aquatic organisms and are of serious health risk to human beings. They lead to greater public concern and present legislation problems. However, removal of these dyes from wastewater is a major environmental challenge and there is a constant need to have an effective process that can efficiently remove these dyes. Moreover, tightening government legislation is enforcing to treat textile wastewater to an increasingly high standard.

There are many processes available for the removal of dye by conventional treatment technologies including biological and chemical oxidation, chemical coagulation, foam flotation, electrolysis, biodegradation, advanced oxidation, photocatalysis and adsorption [1–3,6]. Conventional biological processes are less efficient in degrading synthetic dyes due to the complex structures, molecular size and nature [7–9]. The cell uptake was shown to be inversely proportional to the number of sulfonate groups [10]. Shaul et al. [9] studied the removal of 18 kinds of azo dyes by biological process and found that only three were biodegraded. Advanced oxidation processes ( $H_2O_2/UV$ ,  $O_3$ ) have the potential to eliminate organic carbons in wastewater; however, they are too expensive and complex apart from being effective only for very low concentrations of organic dye wastes [1,6,11,12]. Moreover, depending on the initial materials, additional by-products, such as, halides, metals, inorganic acids, organic aldehydes and organic acids may be produced [6,11,12], which could have increased carcinogenic or toxic properties. Both coagulation and adsorption processes have individually proven to be highly effective in the decolourization [2–4,6,13–15]. However, the reported performances of the processes have varied depending on the nature of dye, adsorbent or coagulant used and the operation conditions. Recently, it is becoming more and more evident

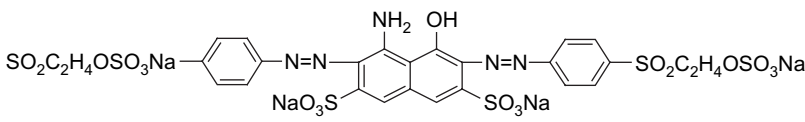
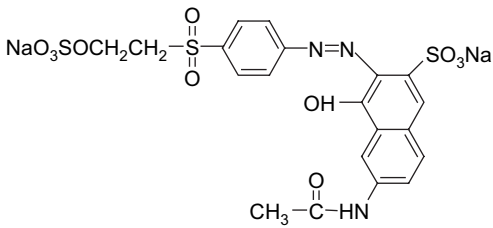
that the use of combined processes is able to overcome the disadvantages of individual unit operations and enhance the overall treatment performance [3,16]. However, there is only limited research on the coagulation of reactive dyes and the combined treatment process of adsorption and coagulation.

The objectives of the present work were to optimize the coagulation and adsorption processes individually and determine the effectiveness of combined process of aluminium chloride coagulation and powdered activated carbon (PAC) adsorption for the complete removal of reactive dyes. Further, the sequence of treatment operation was altered and the overall efficiencies of dye removal were compared. The performance of the combined system was compared with the results when only coagulation and adsorption were present.

## 2. Experimental

The experiments were carried out using two commercially available reactive dyes namely, Black 5 and Orange 16, purchased from Aldrich Co. (USA). Their chemical structures and absorbance values are listed in Table 1. Coconut-shell based PAC was obtained from James Cumming & Sons PTY Ltd. (Australia). The powdered activated carbon was rinsed with distilled water, boiled for 3 h in distilled water, then dried at 393 K for 5 h and stocked inside desiccator. In order to obtain important physical properties of PAC, nitrogen adsorption–desorption isotherms were carried out using an ASAP 2010 volumetric adsorption apparatus (Micromeritics) at 77.4 K. The surface area was calculated by the BET method [17]. The pore size distribution was measured by BJH (Barrett, Joyner and Halenda) method using the desorption data (Fig. 1). The determined physical properties are listed in Table 2. The surface area was found to be  $1200\text{ m}^2\text{ g}^{-1}$  and the average pore diameter was 30 Å, which belong to the

Table 1  
Dye structures and their  $\lambda_{\text{max}}$  values

Dye	Structure	$\lambda_{\text{max}}$ (nm)
Reactive Black 5		598
Reactive Orange 16		491

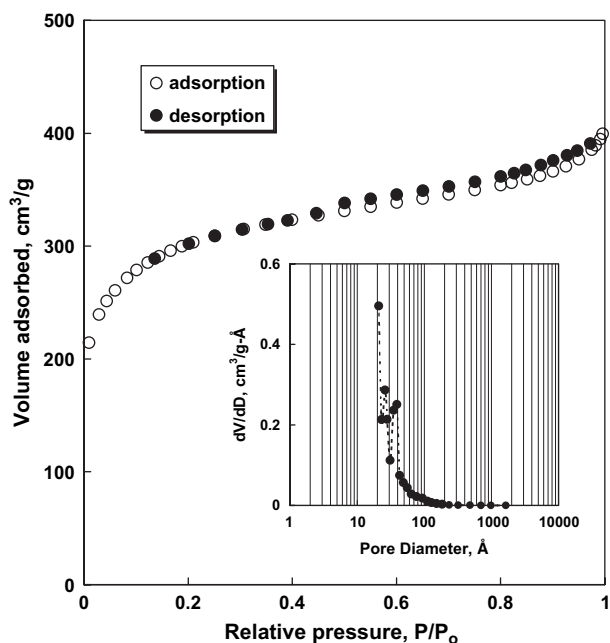


Fig. 1. Nitrogen adsorption and desorption isotherms with pore size distribution for coconut-shell based PAC.

mesopore range. Fig. 2 shows the scanning electron micrograph (SEM) of PAC to examine the particle shape. Aluminium chloride ( $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ ) (Junsei Co., Japan), having a gram molecular weight of 241.45, was used as a coagulant. Dye concentration was evaluated using UV spectrophotometer (Shimadzu UV-160A, Japan) at 598 and 491 nm for Black 5 and Orange 16, respectively. Optimum pH and coagulant concentration were determined based on the dye removal efficiency. Zeta potential (Photal Otsuka ELS-8000, Japan) was measured to examine the underlying surface charge to obtain further insight in the mechanism of removal. All samples were analyzed as per the standard procedures [18].

## 2.1. Individual process optimization

### 2.1.1. Adsorption studies

Adsorption experiments were conducted by adding different amounts of PAC into the flask containing 100 ml of synthetic wastewater. After shaking in a constant temperature incubator at constant temperature (298.15 K) for 3 days to give sufficient contacting time for equilibrium, samples were taken from the flask and filtered through a Whatman glass microfilter. The filtrate was then measured for the dye concentration. The results of adsorption experiments were used to determine equilibrium isotherms for both the dyes. The adsorption capacity ( $q$ ) of PAC was determined from the expression

$$q = \frac{V(C_0 - C)}{m} \quad (1)$$

Table 2

Characteristics of powdered activated carbon (PAC)

Specification	PAC-HA
Raw material	Coconut-based
BET surface area, $\text{m}^2 \text{g}^{-1}$	1200
Moisture content, %	10
Bulk density, $\text{kg m}^{-3}$	425
Mean pore diameter, Å	30
Mean particle diameter, $\mu\text{m}$	34

where,  $C_0$  and  $C$  are the initial and equilibrium (or residual) liquid-phase concentrations ( $\text{mol m}^{-3}$ ), respectively,  $V$  is the volume of solution ( $\text{m}^3$ ), and  $m$  is the weight of dry activated carbon (kg). On the other hand, adsorption kinetic experiments were conducted in a Carberry-type batch adsorber ( $1.0\text{--}2.0 \times 10^{-3} \text{ m}^3$ ) at 300 rpm to obtain concentration decay curves as a function of time.

### 2.1.2. Coagulation studies

Coagulation studies were conducted using Jar-Test apparatus. Coagulant was added to the dye solution and was rapidly mixed (100 rpm) for 2 min followed by slow mixing (40 rpm) for 30 min and then allowed to settle for 1 h. The pH was adjusted by adding HCl or NaOH. The supernatant after the treatment was examined for residual dye concentration.

## 2.2. Combined treatment system

The schematic sequence of the combined treatment process is shown in Fig. 3. The pH of each treatment stage was adjusted based on the optimum pH values obtained from the individual processes. The overall system efficiency was evaluated from the difference in dye concentration between the influent and the end of the treatment train.

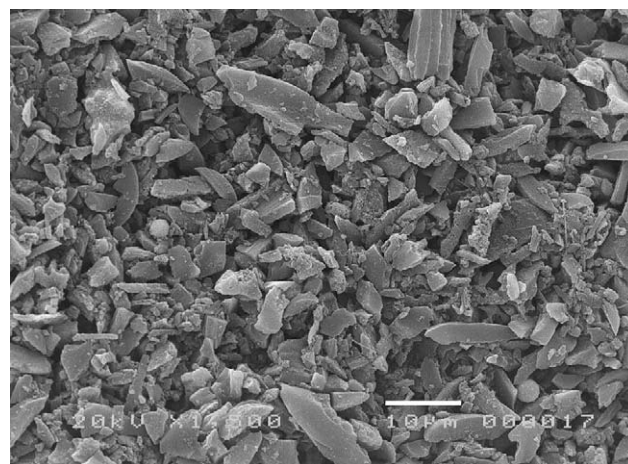


Fig. 2. SEM image of PAC.

### 3. Results and discussion

#### 3.1. Adsorption study

Adsorption isotherms are the most important information for analyzing and designing an adsorption process [19,20]. The adsorption capacity depends on the properties of adsorbate and adsorbent. Fig. 4 shows the adsorption isotherms of Black 5 and Orange 16 on PAC at 298.15 K. The adsorption capacity of Orange 16 was much higher than that of Black 5. The greater affinity of Orange 16 onto PAC than that of Black 5 can be attributed to the chemical structure as well as solubility of the dyes. The molecular structures of the dyes have a significant effect on the extent to which they will adsorb to activated carbon. In general, adsorption capacity on activated carbon is enhanced by increasing molecular size and aromaticity, and by decreasing solubility, polarity and carbon chain branching [3]. Black 5 is of larger molecular size than Orange 16 (Table 2) but it contains more sulphonic acid groups, which decrease the adsorption capacity on PAC. Similarly, the presence of several surface groups (carboxylic, lactonic, phenolic, carbonyl and etheric types) was noted on the activated carbons [17,21]. Adsorption occurs by the donor–acceptor complexation mechanism where atoms of the surface functional group donate electrons to the sorbate molecules [17]. The solid lines (Fig. 4) are the predicted results with Sips isotherm. Among the well-known isotherms such as Langmuir, Freundlich and Sips isotherm models (Table 3), Sips model was found to satisfactorily describe the adsorption of the two reactive dyes studied. The isotherm parameters were determined by minimizing the mean percentage deviations between experimental and predicted amounts adsorbed, based on a modified Levenberg–Marquardt method (IMSL

routine DUNSLF). The object function,  $E(\%)$ , represents the average percent deviation between experimental and predicted results as follows:

$$E(\%) = \frac{100}{n} \sum_{k=1}^n \left[ \frac{|q_{\text{exp},k} - q_{\text{cal},k}|}{q_{\text{exp},k}} \right] \quad (2)$$

Here,  $n$  is the number of experimental data,  $q_{\text{exp},k}$  is the experimental adsorption capacity, and  $q_{\text{cal},k}$  is the calculated adsorption capacity. The determined isotherm parameters of sorbates on PAC are listed in Table 4. The determination of external and internal mass transfer coefficients within porous activated carbon is an important task. Especially, internal mass transfer is usually the rate-controlling step in most adsorption processes [19]. Internal mass transfer within porous sorbents depends on the mechanism of adsorbate transport; namely, pore diffusion, surface diffusion, and others. However, the mechanism of adsorbate transport cannot be completely understood because of its complicated characteristics. In this work, the surface diffusion model was chosen. The detailed description on the diffusion model equations and numerical technique to solve model equations are given elsewhere [19,20,23]. Among various methods for determining the internal diffusion coefficient, the most general method is to compare the experimental concentration decay curves and the predicted values using the specified model. Fig. 5 illustrates the concentration decay curves of Black 5 and Orange 16 on PAC in a batch adsorber. One of the possible reasons for faster adsorption kinetics for Orange 16 than Black 5 could be attributed to their different molecular sizes (Table 2). The solid lines in Fig. 5 are the predicted results by employing surface diffusion model. The external and internal diffusivities

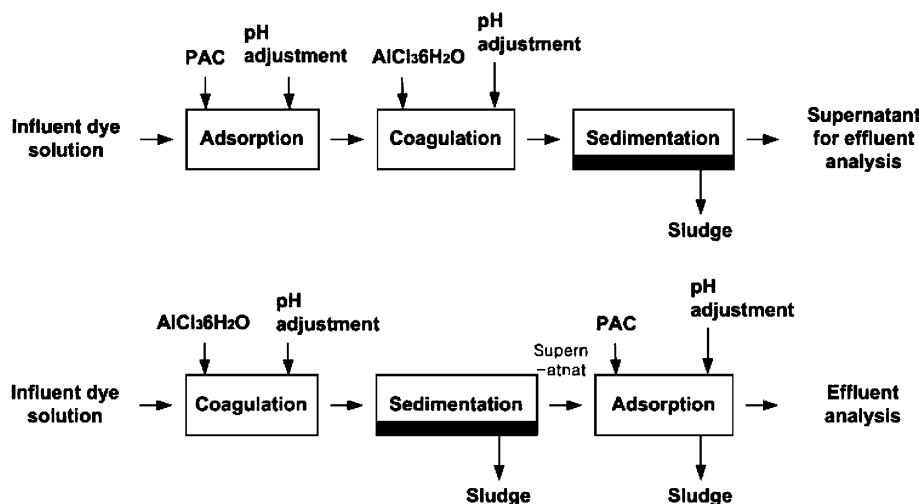


Fig. 3. Schematic sequence of combined treatment processes.

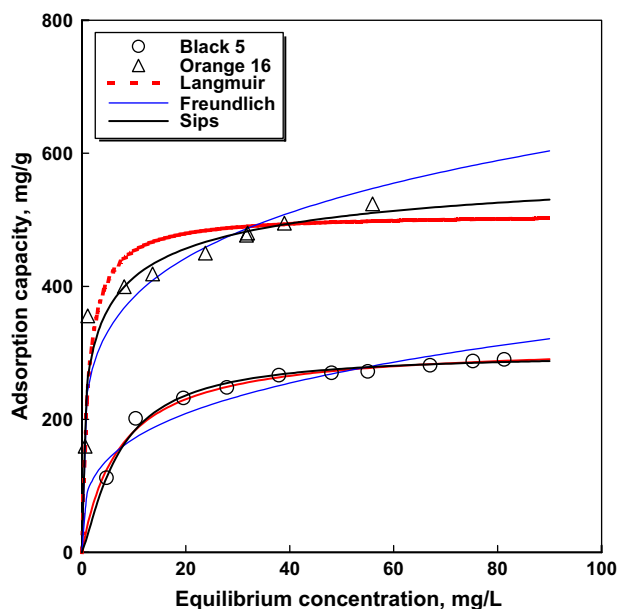


Fig. 4. Adsorption isotherms of Black 5 and Orange 16 on PAC at 298.15 K (equilibrium time – 3 days).

of Orange 16 ( $1.84 \times 10^{-5} \text{ m s}^{-1}$  and  $2.40 \times 10^{-14} \text{ m}^2 \text{ s}^{-1}$ ) were one-order-of-magnitude greater than that of Black 5 ( $2.65 \times 10^{-6} \text{ m s}^{-1}$  and  $5.37 \times 10^{-15} \text{ m}^2 \text{ s}^{-1}$ ) as listed in Table 5. Among various operating parameters affecting adsorption capacity and kinetics for liquid-phase adsorption, pH is the most important factor. Fig. 6 shows the effect of pH on the removal efficiency of reactive dyes on PAC at 298.15 K. A strong influence of electrostatic (columbic) effect was evident in the physical adsorption between the adsorbent and the adsorbate [15,21,22]. Results showed that the adsorption capacity of two reactive dyes was maximum at pH 4 under the study conditions.

### 3.2. Coagulation study

Fig. 7 shows the removal efficiencies of Orange 16 and Black 5 reactive dyes at different coagulant doses. It was found that maximum removal efficiency was about 99.9% and 80% for Black 5 and Orange 16, respectively. The coagulant dose of 350 mg/L for Orange 16 and 250 mg/L for Black 5 was required in order to achieve this removal for 100 mg/L dye concentration. The difference in removal efficiency may come from the different chemical structures of two reactive dyes and

Table 3  
Pure component isotherms used

Name	Model equation	Parameters
Langmuir	$q = \frac{q_m b C}{1 + b C}$	$q_m, b$
Freundlich	$q = k C^{1/n}$	$k, n$
Sips	$q = \frac{q_m b C^{1/n}}{1 + b C^{1/n}}$	$q_m, b, n$

Table 4  
Isotherm parameters for reactive dyes at 298.15 K

Isotherms	Parameters	Black 5	Orange 16
Langmuir	$q_m$	$3.138 \times 10^2$	$5.090 \times 10^2$
	$b$	0.138	0.809
	$E(\%)$	2.63	9.32
Freundlich	$k$	$0.883 \times 10^2$	$2.387 \times 10^2$
	$n$	3.483	4.851
	$E(\%)$	8.31	10.81
Sips	$q_m$	$2.991 \times 10^2$	$6.286 \times 10^2$
	$b$	0.083	0.650
	$n$	0.787	4.851
	$E(\%)$	1.90	9.15

from the difference in their solubilities. Solubility differences are in turn attributed to different chemical structures. The nature of the curve also indicates the possible existence of a stoichiometric relation between the dye and the coagulant. The amount of aluminium ions in the coagulant determines the amount of destabilization of dyes. As the coagulant dose alters the amount of flocs formed and their settling tendency, the removal efficiency of dye decreased with the increase in coagulant dose, above a certain optimum dose. The coagulation effect was strongly dependent on the pH of the solution and had a narrow optimum pH range as shown in Fig. 8. pH 6 was found to be the optimum pH for coagulation (Fig. 8). The decrease in the concentration of dissolved aluminium hydrolysis products when pH was increased resulted in the decrease in coagulation efficiency with the increase in pH beyond pH 6. The results of the coagulation efficiency were in good agreement with zeta potential values (Fig. 9).

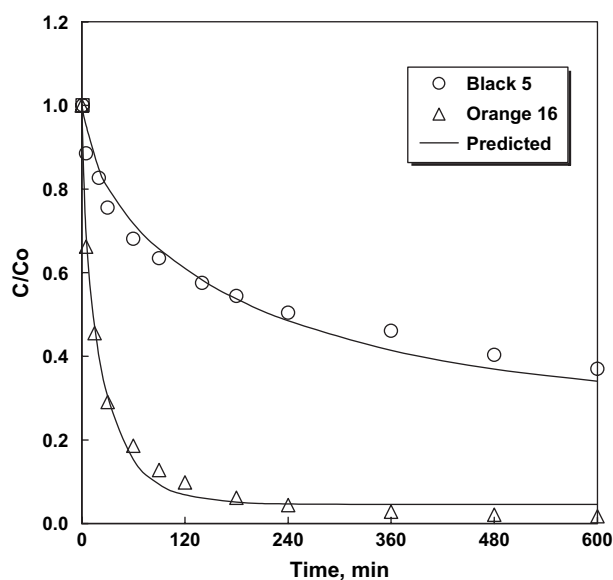


Fig. 5. Concentration decay curves of Black 5 and Orange 16 on PAC (mixing speed 300 rpm; initial dye concentration: Black 5 – 100 mg/L, Orange 16 – 100 mg/L; PAC dose – 0.5 g/L).



Table 5

The external and internal mass transfer coefficients determined in a batch adsorber

Dyes	External mass transfer coefficient, $k_f$ ( $\text{m s}^{-1}$ )	Internal mass transfer coefficient, $D_s$ ( $\text{m}^2 \text{s}^{-1}$ )
Black 5	$2.65 \times 10^{-6}$	$5.37 \times 10^{-15}$
Orange 16	$2.84 \times 10^{-5}$	$2.40 \times 10^{-14}$

Adsorption of hydrogen ions or positive-charged ions, such as aluminium ions, on negatively charged dye surfaces results in charge neutralization causing the net surface charge to be almost zero at pH 6, which results in the maximum dye removal (Fig. 9). When the zeta potential of the particles is approaching towards zero, removal efficiency is improved (Fig. 8). There is limited resource on the coagulation mechanism of reactive dyes. However, from the accumulated evidence, the proposed mechanism is thought to be complex and could involve combination of several mechanisms including, charge neutralization (electrostatic interaction) and precipitation of dye molecules with hydrolyzed monomeric and polymeric alumino complexes, complex formation between dye and alumino precipitates, adsorption, bridge formation and surface precipitation on alumino hydroxide solid precipitate [4,24,25].

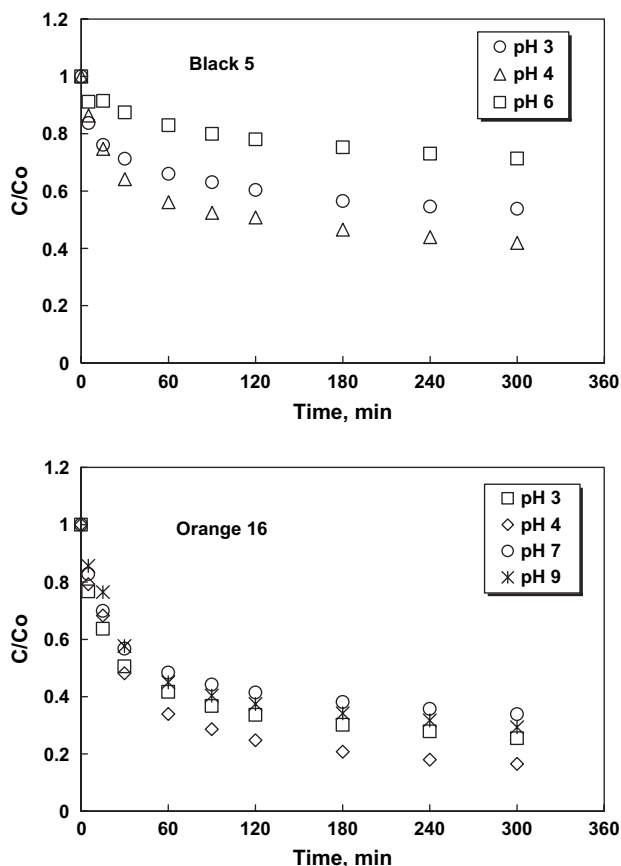


Fig. 6. Effect of pH on the removal efficiency of reactive dyes.

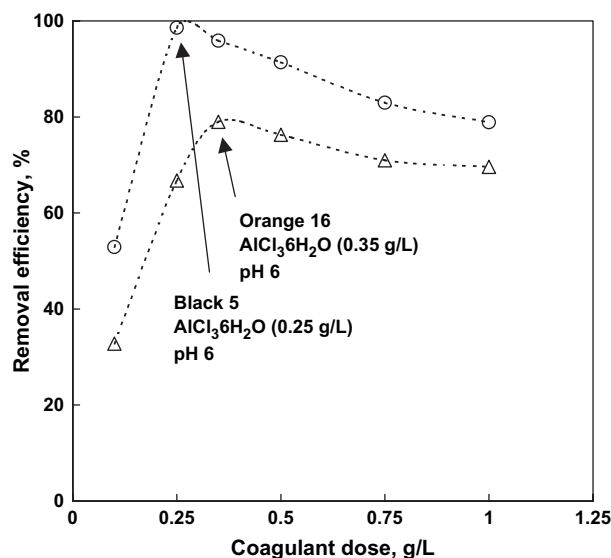


Fig. 7. Removal efficiency of reactive dyes in terms of coagulant dose (dye concentration: Black 5 – 100 mg l<sup>-1</sup>, Orange 16 – 100 mg l<sup>-1</sup>; pH 6).

### 3.3. Combined process study

By combining adsorption and coagulation operations the overall dye removal efficiency was significantly increased. Table 6 shows the comparison of removal efficiencies for Orange 16 dye with two different treatment sequences of coagulation followed by adsorption and adsorption followed by coagulation. It is evident that the overall performance of the system having coagulation prior to adsorption (95.7%) was more superior to having adsorption followed by coagulation (79.1%). Moreover, it was found that the amount of increase in adsorption capacity of PAC (25%) with pre-coagulation was much greater than the amount of

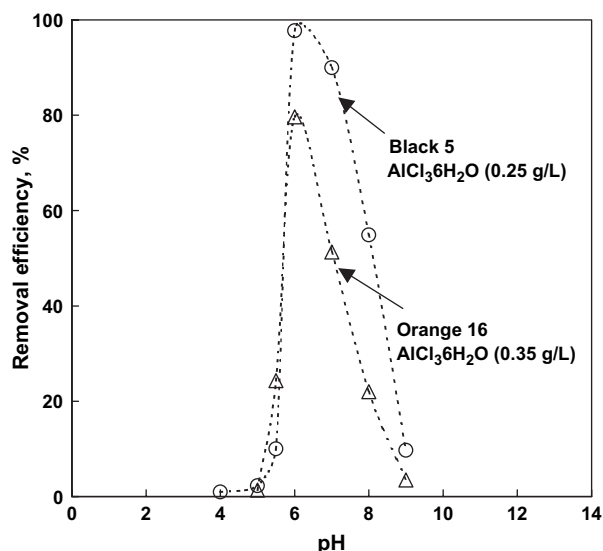


Fig. 8. Removal efficiency of reactive dyes at various solution pH.

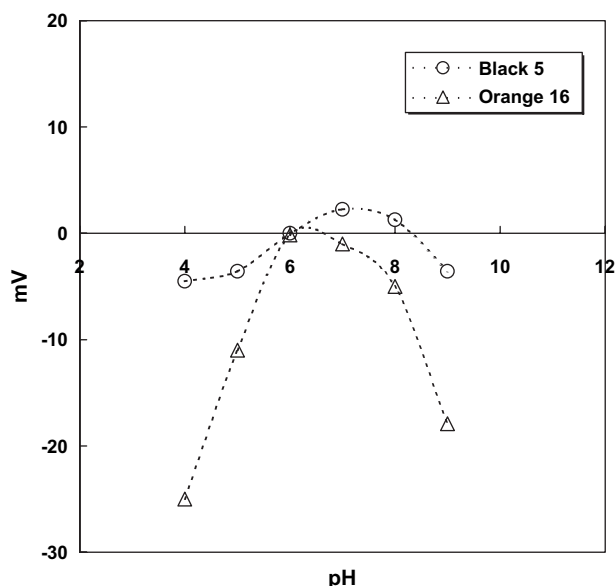


Fig. 9. Zeta potential values at different pH (dye concentration: Black 5 – 100 mg l<sup>-1</sup>, coagulant dose – 0.25 g l<sup>-1</sup>; Orange 16 – 100 mg l<sup>-1</sup>, coagulant dose – 0.35 g l<sup>-1</sup>).

increase in coagulation efficiency (5%) with pre-adsorption. A previous study having a combined system of coagulation followed by adsorption for reactive dyes (Reactive Red 45 and Reactive Green 8) noted significant high dye removal [3]; however, the performance could not be compared with our present study as there was no indication of the initial dye concentration used. Further, the adsorption process was not individually optimized prior to combining with coagulation. However, the present study has systematically addressed all these issues and extends further in analyzing the right sequence of treatment operations, which are very important in order to maximize the overall efficiency and reduce the amount of coagulant and adsorbent usage.

Fig. 10 shows the concentration decay curves of Black 5 and Orange 16, at different PAC doses for the combined system of coagulation followed by adsorption. Complete dye removal was achieved for both the dyes. The overall removal efficiency of Orange 16 was

Table 6

Removal efficiencies of Orange 16 dye for the combination of adsorption and coagulation systems [coagulation pH 6, adsorption pH 4; original dye concentration 100 mg l<sup>-1</sup>; AlCl<sub>3</sub> dose 0.35 g l<sup>-1</sup>; PAC dose 0.05 g l<sup>-1</sup>]

Process	Individual process removal efficiency (%)	Overall removal efficiency (%)
Coagulation prior to adsorption	Coagulation (80) Adsorption (80)	95.7
Adsorption prior to coagulation	Adsorption (30) Coagulation (73)	79.1

greater than Black 5. The results indicated that, under the given study condition, 250 mg l<sup>-1</sup> of initial dye concentration required 350 mg l<sup>-1</sup> of coagulant dose and 200 mg l<sup>-1</sup> of PAC to completely remove Orange 16 and 250 mg l<sup>-1</sup> of coagulant dose and 252 mg l<sup>-1</sup> of PAC for complete removal of Black 5. Several combination processes like coagulation/ozonation [26,27], coagulation/oxidation [24] have been tried earlier. Sarasa et al. [26] found that upon combination of coagulation and ozonation, the performance of ozonation was not very effective in dye removal. Similarly, Kim et al. [4,24] noted that when coagulation and chemical oxidation processes were combined, the later was found to be less effective. However, it is evident from our study that the combined coagulation and adsorption process is very effective and had the capability of complete dye removal and thus total decolourization, reduction in coagulant and adsorption amounts and thereby produce less amount of sludge.

#### 4. Conclusion

For the complete removal of reactive dyes, coagulation and adsorption processes were investigated. The optimum conditions of coagulant dose and solution

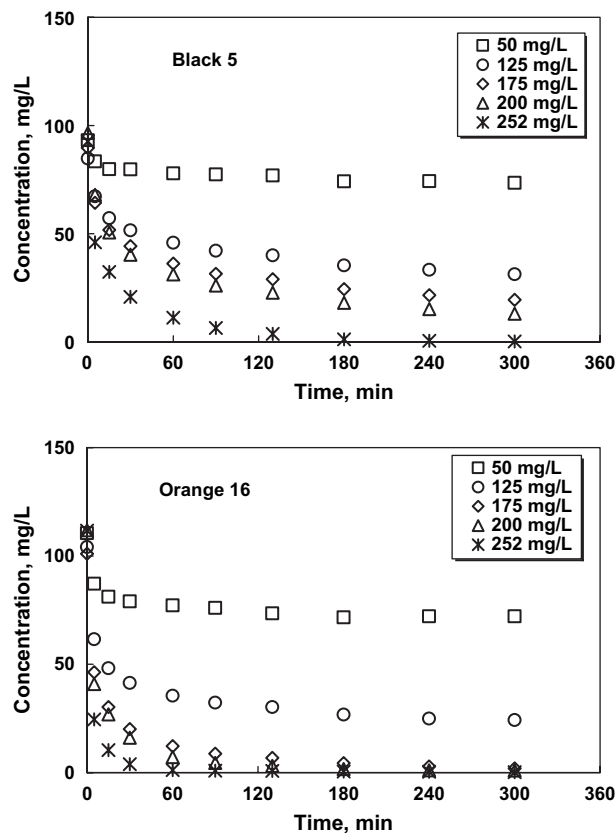


Fig. 10. Concentration decay curves of reactive dyes after coagulation in terms of PAC dose for the combined process.

pH were  $250 \text{ mg l}^{-1}$  at pH 6 for Black 5 and  $350 \text{ mg l}^{-1}$  at pH 6 for Orange 16. However, the removal efficiency of Black 5 by coagulation was somewhat greater than that of Orange 16. Adsorption capacity as well as adsorption kinetics of Orange 16 was much higher and faster than that of Black 5 because of the different molecular dimension, solubility and surface functional groups. While combining the treatment processes, the performance of the system having coagulation followed by adsorption was much more greater than having adsorption followed by coagulation, in terms of overall dye removal and chemical requirement. The combined process of coagulation/adsorption achieved complete decolorization (99.9%) of both reactive dyes from wastewater. From this study, it is evident that the combined coagulation–adsorption process has a great potential application for reactive dye removal. It has the capacity for the production of high quality treated water and allows the reduction in the use of coagulant and adsorbent, which helps in the reduction of amount of sludge produced.

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