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Comparison of Adsorption Process by GAC with Novel Formulation of Coagulation – Flocculation for Color Removal of Textile Wastewater

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ABSTRACT: This study evaluates the effectiveness of adsorption process by Granular Activated Carbon (GAC) compared with a novel formulation of coagulation - flocculation process for dye removal from textile wastewater. In this regard, acidic, reactive, disperse and direct red dye are used to prepare the synthetic dye. Dominant wave length for each dye is determined by spectrophotometeric method. Using GAC as adsorbent, equilibrium time and adsorption isotherm of each dye are determined with aid of spectrophotometric method. The results show that GAC can not remove dispersed red dye. Acidic red, direct red and reactive red of 5 mg/L concentration are removed by GAC up to %90, %88 and %43 in 30, 60 and 120 min. (equilibrium time) respectively. Dyes of 50 mg/L concentration are removed up to %93, %30 and %51 in 15, 90 and 150 min. respectively. Adsorption obeys Freundlich isotherm for acidic red, BET isotherm for direct red and Langmuir isotherm for reactive red. This investigation presents a novel formulation of coagulation - flocculation for color removal from textile dye solutions and illustrates its efficiency. Novel formulation of coagulation - flocculation remove direct red, reactive red and disperse red of 5 mg/L concentration respectively up to %93, %91.3 and %57.1. Also the mentioned dyes of 50 mg/L are removed respectively up to %90.8, %91.9 and %70.1.

Key words: Coagulation, Flocculation, Jar test, GAC, Color removal

INTRODUCTION

Wastewater reuse in the textile industry is necessary due to the high water consumption required for its processes. This has to be considered, especially in areas that suffer from water scarcity such as Iran.Although several studies have been conducted, in the developed countries, in to the treatment of water and wastewater through Granular Activated Carbons (Bansode *et al.*, 2004; Ahmedna *et al.*, 2000), adsorbent (Pala and Tokat, 2000; Guohua, 2004), Fenton's reagent (Martinez *et al.*, 2003), wet oxidation (Hung *et al.*, 2003), coagulation - electro - oxidation (Xiong *et al.*, 2000). and advanced oxidation with biological oxidation (Gogate and Pandit, 2004), developing countries still lack basic technology of water and wastewater treatment. In typical dyeing processes, 50-95% of the dye is fixed on to the fiber, and unfixed dyes from subsequent washing operations are discharged in the spent dye-bath or in the wastewaters (Jiraratananon *et al.,* 2000). How ever application of textile treatment methods in an industrial plant becomes difficult due to operational problems and costs. Biological treatment by activated sludge offers high efficiencies in COD removal, but does not completely eliminate the color of the wastewater and frequently operational problems such as bulking appear. Chemical oxidation by ozone, or a combination of UV-radiation and ozone and H_2O_2, has great interest, but the costs are still very high due to the high doses required. Among the above mentioned methods, adsorption is considered to be relatively superior to other techniques because of low cost, simplicity of design, availability and ability to treat dyes in more concentrated form (Meshko *et al.*, 2001); (Kannan and Sundaram, 2001).

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Coagulation - flocculation is also an essential process in water and in industrial wastewater treatment (Ismail, 1978; Abdel-Shafy *et al.*, 1987). Several studies have been reported on the performance and optimization of coagulants, determination of pH and investigation of flocculants addition (Tatsi *et al.*, 2003; Abdel-Shafy *et al.*, 1991). Coagulation - flocculation process has been found to be cost effective, easy to operate and energy saving treatment alternatives (Bromley *et al.*, 2002) Coagulant dosages varies in a wide range aiming at maximum removal efficiency of pollutants using minimum doses at optimum pH (Watanabe *et al.*, 1993; Szpak *et al.*, 1996). Coagulation can be interpreted as the conversion of colloidal and dispersal particles in to small visible floc upon addition of a simple electrolyte. Increasing the concentration of the electrolyte results in a compression of the electrical double layer surrounding each suspended particle, a decrease in the magnitude of the repulsive interactions between particles and destabilization of the particles. The most common coagulant used in wastewater treatment is alum $Al_2(SO_4)_3.4H_2O$ and PAC (Poly Aluminum Chloride), due to its effectiveness in treating a wide range of wastewater type and relatively low cost. The use of performed polymerized forms of Al has become more common as alternative coagulants, such as poly aluminum chloride and poly aluminum sulphate. The higher charge density of poly aluminum chloride species often results in a decrease in the coagulant dose and the associated solids production. These coagulants have the advantage of being more effective at lower temperatures and a boarder pH range than alum (Exall and Vanloon, 2003). Flocculation is used to describe the process whereby the size of particles increases as a result of particle collisions. The purpose of flocculation is to produce particles, by means of aggregation, that can be removed by inexpensive particle - separation procedures such as gravity sedimentation and filtration (Metcalf and Eddy, 2003).Some characteristic properties of different type of poly acryl amid (PAM) as flocculants agent are given in Table 1 (Rahbar *et al.*, 2006).

Meric *et al.*, (2004) studied the effectiveness of fenton's oxidation (FO) process and ozone (O_3) oxidation compared with coagulation - flocculation (CF) process to remove effluent toxicity as well as color and COD from textile industry wastewater. The FO process removed COD at a higher rate (59%) than O_3 (33%) while color removal was similar (89% and 91%, respectively). The CF process removed both COD and color at rates similar to the FO process (Meric *et al.*, 2004). Rahbar *et al.*, (2006) investigated the effectiveness of novel formulation of coagulation - flocculation for color removal from industrial wastewater, with aid of measurement of solid sludge content, suspended solid content percentage of solid recovery, UV absorption in wastewater effluent from two automotive factories. They found that the novel formulation can remove color content from industrial wastewater efficiently up to 96% (Rahbar *et al.*, 2006). Haitan *et al.*, (1999) used Magnesium chloride as a coagulant to investigate the effectiveness in the chemical precipitation method for the removal of coloring matters. The color concentration of dye solutions was measured by visible spectrophotometer. They studied parameters such as the effect of pH, the effect of coagulant and coagulant aid dosages and effect of different coagulants. They found that $MgCl_2$ is capable of removing more than %90 of the coloring material at a pH of 11 and a dose of 4g $MgCl_2$ /L of dye solution and $MgCl_2$ is more effective in removing reactive dye than alum and PAC in terms of settling time and amount of alkalinity required (Haitan *et al.*, 1999).

Table 1. Characteristic properties of different type of poly acryl amide (PAM)

Ionic type	M.W (g/mol)	Charge density (c/g)	Percentage Charge monomer
Anionic PAM	5.5-7×10^6	-260	30.0
Cationic PAM	6.0-7×10^6	+150	19.0
Non - ionic PAM	5.0×10^6	-1.07	____

Al-Degs *et al.*, (1998) investigated the removal efficiency of activated carbon filtrate sorbs 400 (F-400) towards three highly used reactive dyes in the textile industry. In this work the adsorption capacities for the anionic reactive dyes, namely, Remazol Reactive Red were determined. The adsorption capacity data showed high removal ability for the three reactive dyes and a distinguished ability for R.Yellow (Al-Degs *et al.*, 1998). Santhy and Selvapathy (2005) investigated the removal efficiency of activated carbon prepared from coir pith towards three highly used reactive dyes in textile industry. Bach experiments showed that the adsorption of dyes increased with an increase contact time and carbon does. Maximum depolarization of all the dyes was observed at acidic pH also adsorption of dyes was found to follow the Freundlich model (Santhy and Selvapathy, 2005). Mainul Karim *et al.*, (2006) investigated the adsorption of colored compounds from the textile dyeing effluents of Bangladesh, on granulated activated carbons produced from indigenous vegetable sources by chemical activation with Zinc chloride. They established that at optimum temperature (50 ºC) and time of contact (30-40 min) adsorbent loading is (2g/L). Activated carbons developed from Segun saw - dust and water hyacinth showed substantial capability to remove coloring materials from the effluents, and adsorption of reactive dyes by all sorts of activated carbons is higher than disperse dyes (Mainul Karim *et al.*, 2006). In present investigation, GAC as adsorbent and novel formulation of coagulant flocculant are used for color removal from textile wastewater. Novel formulation was originally used by Rahbar *et al.* (2006) for color removal of effluent wastewater in two automotive factories (Iran Khodro and Mehrkam Pars, in Tehran). In this work we used novel formulation as basis of one of our treatment method and try to optimize it for de-colorization of textile dye solutions.

MATERIALS & METHODS

In this investigation, synthetic dye solutions are used for experiments. Four classes of dye with the highest usage rates in Iranian textile industries (direct, acidic, reactive and disperse red dye) were purchased from Bayer-UK. Characteristics of these dyes are presented in Table 2. GAC as adsorbent and novel formulation of coagulant - flocculants have been applied for color removal from textile dye solutions. The ingredients of novel formulation are listed in Table 3. Each component in the formulation has the specific character, which permits to have more efficiency in color removal. Their roles can be gathered in four classifications as below:

Table 3. Ingredients of coagulant - flocculants formulation (Rahbar, *et al.*, 2006)

Role	Component	Concentration (mg/L)
Coagulant	PAC	370
Coagulant	$NaAlO_2$	400
Coagulant	Na_2SiO_3	40
pH adjuster	KOH	80
Coagulant aid	Poly vinyl alcohol	60
Flocculants	PAM	10
pH adjuster	Na_2CO_3	40

- Coagulants: poly aluminum chloride (PAC), sodium aluminates ($NaAlO_2$) and sodium silicate (Na_2SiO_3) are poly electrolyte materials and play the role of coagulant.Dye solutions are synthesized in two concentrations, 5 and 50 mg/L by mixing the powder of every individual dye (direct, acidic, reactive, disperse) with red in distilled water.Two different types of treatments are carried out in the following manner:

a) GAC adsorption

• The equilibrium time of each dye is determined by contacting a known mass of GAC with dye solutions of a predetermined initial concentration (5, 50 mg/L) at a constant agitation speed of 190rpm. Dye solution of 5 mg/L concentration with 0.5gr carbon and 50 mg/L with 2gr of activated carbon are mixed. Each solution was mixed for periods of 15, 30, 45, 60, 90, 120, 150, 180, 240, 300 min.

• To obtain adsorption isotherm, variety dosages of GAC contacting with dye solutions in equilibrium

Table 2. Characteristics of applied dyes

Commercial name	Color index	Characteristics	Type of dye
Direct red 105	Red 12b		Direct red
Carmozin 206	Red 88		Acidic red
		KE3B	Reactive red
Red E3B	Red 60	KE3B	Disperse red

time of each dye as determined above at a constant agitation speed of 190rpm. The dosages of GAC which are used for dyes of 50 mg/L concentration vary between 0.2 to 0.7 g and for 50 mg/L concentration vary between 1 to 7.5 g.

b) Novel coagulation and analysis

Laboratory scale evaluation of novel coagulation and flocculation is performed using a five-place jar test apparatus. The experimental process consisted of three subsequent stages: initial rapid mixing stage at 150 rpm took place for 2 min, followed by a slow mixing stage for 20 min at 30 rpm, and the final settling step lasted for another 30 min. five equal volume beakers are used to examined the five different dosages of coagulants, pH adjuster, flocculant and three different dosages of coagulant aid in each run. Sample bottles are thoroughly shaken for resuspension of possibly settling solids and the appropriate volume of sample is transferred to the corresponding jar test beakers. In this work novel formulation optimized for color removal from dye solutions of 5 and 50 mg/L concentration as follows:

1. Dye solutions of 5 mg/L concentration

1.1. First, the optimum dose of coagulants for every dye solution of 5 mg/L concentration is determined. Known volume of prepared novel solution is added to a jar containing 1L of every dye solution with different coagulant dose. Concentrations of PAC as main coagulant of novel vary between 200, 300, 370, 400 and 450 mg/L, while concentration ratio of novel coagulants is kept constant.

1.2. To investigate the optimum KOH dose as pH adjuster, the coagulants dose of novel are maintained at optimum (as determined above) and then varying dose of KOH mainly 50, 70, 80, 90 and 110 mg/L are added.

1.3. To investigate the optimum coagulant aid (PVA) dose, the coagulants dose of novel are maintained at optimum and varying dose of PVA including 55, 60, 65 mg/L are then added.

1.4. To find the optimum flocculant dose, the coagulants dose of novel are maintained at optimum and varying dose of PAM including 6, 8, 10, 12 and 14 mg/L are added.

1.5. To asses the efficacy of optimum KOH, PVA and PAM dose at the same time on color removal two samples are compared with each other for every dye solution. In both samples, coagulants dose of novel are maintained at optimum. In first sample KOH, PVA and PAM dose of novel aren't changed and in optimum sample, optimum dose are used.

2. Dye solutions of 50 mg/L concentration

2.1. First, the optimum coagulants dose is determined using trial procedure. Although concentration of dyes are increased 10 times, in order to find optimum as well as minimum coagulants dose the optimum coagulants dose of 5 mg/L dye solutions (as determined above) increased by 2, 5, 8, 10 times. Considering economical aspect, best color removal efficiency for each dye is obtained.

2.2. To investigate the optimum KOH dose, considering optimum rate of increase, all KOH doses (which are mentioned for dyes of 5 mg/L concentration) are increased by the same ratio.

2.3. To find the optimum PVA and PAM doses, experiments are repeated accordingly.

The samples are collected before experiments (as raw dye solutions) and after experiments (as treated dye solutions). The color is determined spectrophotometrically at a dominant wave length by spectrophotometric method (no. 2120 standard method), using a Shimadzu UV-Visible spectrophotometer (uv-1201pc).

RESULTS & DISCUSSION

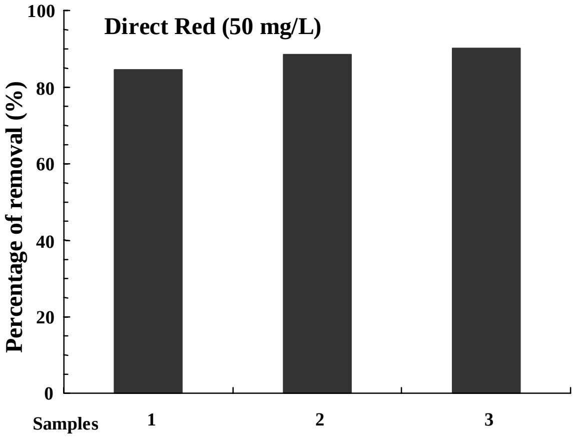
Here, the results are present through various Figures and Tables. The results presented in Table 1 show various dye and related color removal percentile.

Table 4. Equilibrium time and color removal efficiency by GAC

dye	Concentration (mg/L)	Equilibrium time (min)	Color removal (%)
Acidic red	5	30'	90
Direct red	5	90'	88
Reactive red	5	120'	43
Acidic red	50	15'	93
Direct red	50	90'	30
Reactive rec	50	150'	51

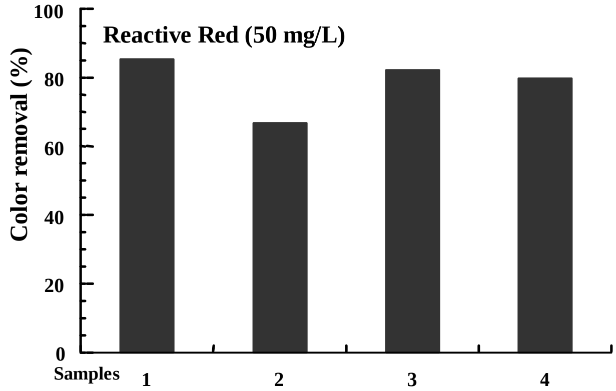
Table 5. Optimum concentration of PAC, $NaAlO_2$, Na_2SiO_3, KOH, PVA and PAM

Dye solutions 5 mg/L	PACmg/L	$NaAlO_2$mg/L	Na_2SiO_3mg/L	KOHmg/L	PAMmg/L	PVAmg/L
direct red	370	400	40	70	6	65
acidic red						
reactive red	400	432.2	43.2	70	10	65
disperse red	450	486.2	48.6	80	6	65



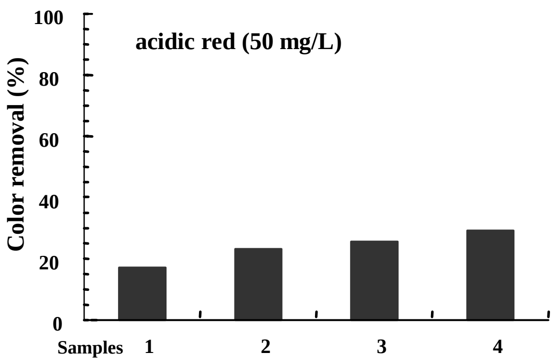
PAC (mg/L)	740	1850	2960	3700
$NaAlO_2$	800	2000	3200	4000
Na_2SiO_3	80	200	320	400
Removal (%)	86.9	87.5	88.9	89.4

Fig. 1. Effect of coagulant dose on removal of direct Red of 5 mg/L concentration



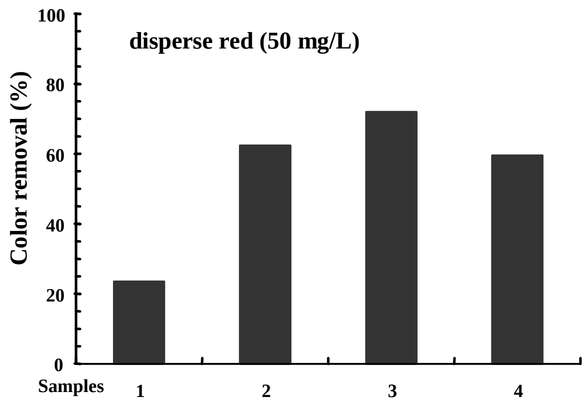
PAC (mg/L)	800	2000	3200	4000
$NaAlO_2$	864.57	2161.42	3458.3	4322.8
Na_2SiO_3	86.45	215.7	345.1	4314.3
Removal (%)	85.2	66.7	82.1	79.7

Fig. 3. Effect of coagulants dose on removal of reactive red of 5 mg/L concentration



PAC (mg/L)	600	1500	2400	3000
$NaAlO_2$	648.57	1621.42	2594.3	3242.85
Na_2SiO_3	64.85	162.14	259.43	324.28
Removal (%)	17.1	23.1	25.5	29.2

Fig. 2. Effect of coagulants dose on removal of acidic red of 5 mg/L concentration



PAC (mg/L)	900	2250	3600	4500
$NaAlO_2$	972.6	2431.43	3890.3	4862.85
Na_2SiO_3	97.26	243.14	389.03	486.28
Removal (%)	23.5	62.3	72	59.6

Fig. 4. Effect of coagulants dose on removal of disperse red of 5 mg/L concentration

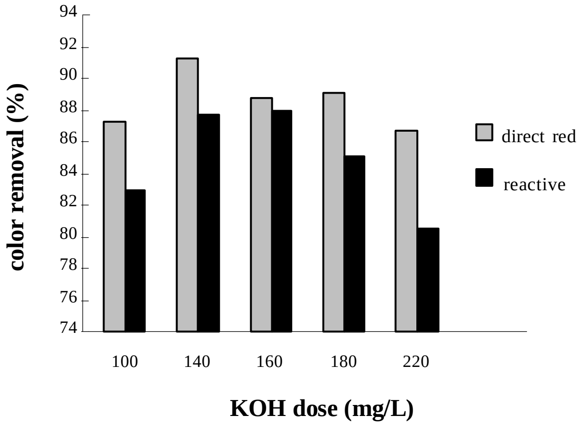


Fig. 5. Effect of KOH dose on removal of direct and reactive red of 50 mg/L concentration

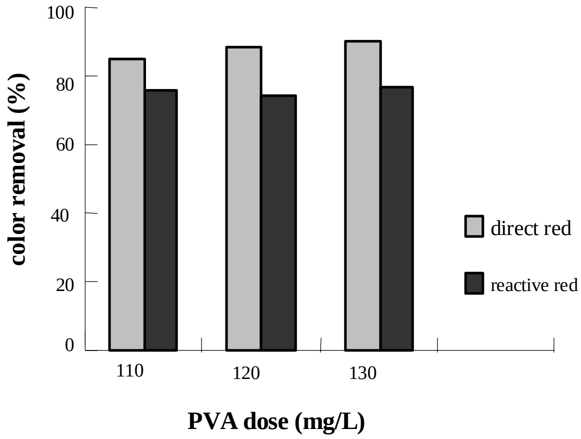


Fig. 7. Effect of PVA dose on removal of direct and reactive red of 50 mg/L concentration

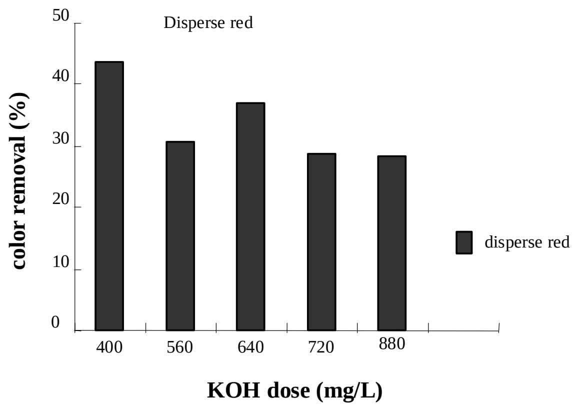


Fig. 6. Effect of KOH dose on removal of disperse red of 50 mg/L concentration

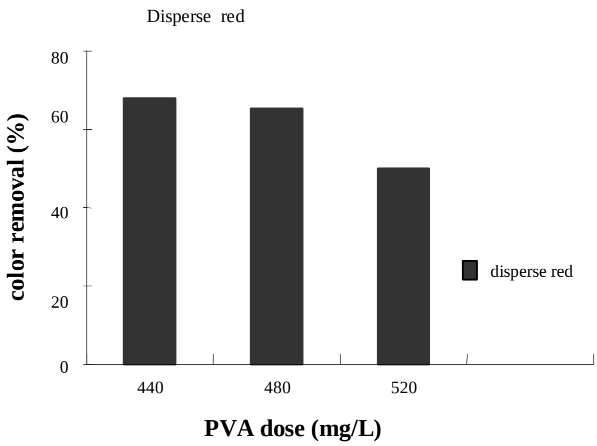


Fig. 8. Effect of PVA dose on removal of disperse red of 50 mg/L concentration

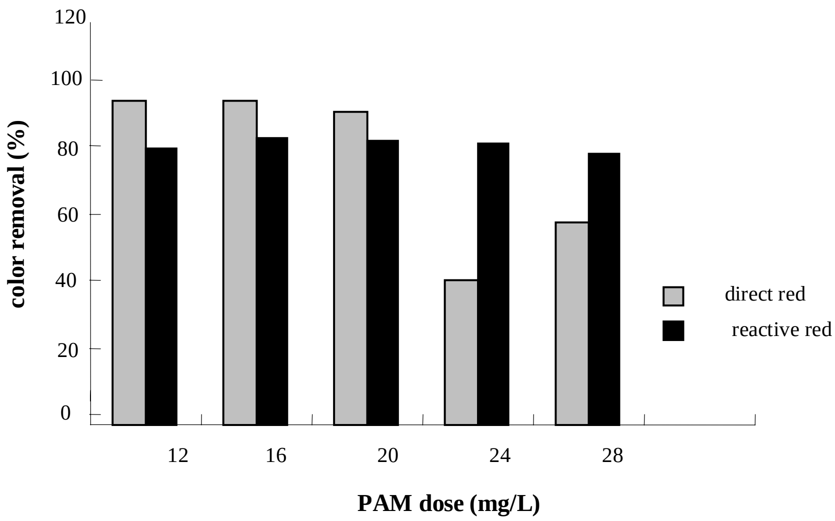


Fig. 9. Effect of PAM dose on removal of direct and reactive red of 50 mg/L concentration

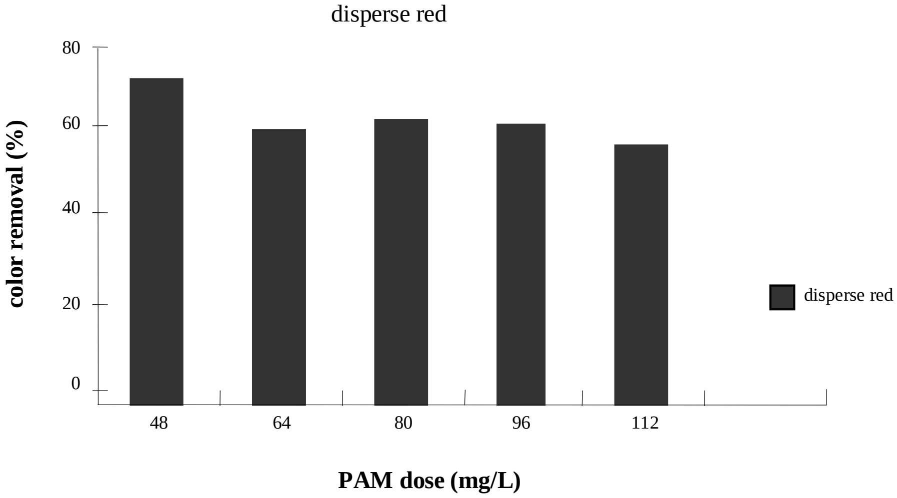


Fig. 10. Effect of PAM dose on removal of disperse red of 50 mg/L concentration

Table 6. Optimum concentration of KOH, PVA and PAM for disperse

Dye solution 50 mg/L	KOH mg/L	PVA mg/L	PAM mg/L
direct red	140	130	12
reactive red	140	110	16
disperse red	400	440	48

Table 7. Removal of direct, reactive and disperse red dye (5 mg/L)

Dye solutions 5 mg/L	PAC mg/L	$NaAlO_2$ mg/L	Na_2SiO_3 mg/L	KOH mg/L	PVA mg/L	PAM mg/L	Na_2Co_3 mg/L	Color removal
direct Red 5 mg/L								
first sample	370	400	40	80	60	10	40	%88.50
optimum sample	370	400	40	70	65	6	40	%93
reactive Red 5 mg/L								
first sample	400	432.2	43.2	80	60	10	40	%89.82
optimum sample	400	432.2	43.2	70	65	10	40	%91.33
disperse Red 5 mg/L								
first sample	450	486.2	48.6	80	60	10	40	%56.08
optimum sample	450	486.2	48.6	80		6	40	%57.14

Table 8. Removal of direct, reactive and disperse red dye (50 mg/L)

Dye solutions 50 mg/L	PAC mg/L	$NaAlO_2$ mg/L	Na_2SiO_3 mg/L	KOH mg/L	PVA mg/L	PAM mg/L	Na_2Co_3 mg/L	Color removal
direct Red 50 mg/L								
first sample	740	800	80	160	120	20	80	%88.91
optimum sample	740	800	80	140	130	12	80	%90.83
reactive Red 50 mg/L								
first sample	800	864.57	86.45	160	120	20	80	%86.91
optimum sample	800	864.57	86.45	140	110	16	80	%91.96
disperse Red 50 mg/L								
first sample	3600	3890.3	389.03	640	480	80	320	%68.86
optimum sample	3600	3890.3	389.03	400	440	48	320	%70.16

According to obtained data, the optimum concentration of PAC, $NaAlO_2$, Na_2SiO_3, KOH, PVA and PAM correspond to best color removal efficiency (Table 5).Effects of different doses of PAC, $NaAlO_2$, and Na_2SiO_3 on the removal of the direct, acidic, reactive and disperse dye of 50 mg/L concentration by novel are shown in Figs. 1, 2, 3 and 4.

As Fig. 1 shows, although first column of the table has fewer percentage of removal but it is economically preferable. As shown, although the concentration of dye has been increased 10 times (50/5), but only twice of coagulants dose are sufficient to remove direct red of 50 mg/L concentration up to %86.9. It is clear that novel formulation is highly recommended for concentrated direct red dye solution for two reasons: (1) high color removal efficiency, and (2) economical aspect. Figure 2 shows low color removal efficiency for acidic red, so further experiments were discontinued. As shown in figure 3, the optimum concentration of PAC, NaAlO2 and Na2SiO3 are 800, 846.57 and 86.45 mg/L, again only twice of coagulants dose are sufficient to remove reactive red of 50 mg/L up to %85.2.

Figure 4 shows the optimum concentration of PAC $NaAlO_2$ and Na_2SiO_3 are 3600, 3890.3 and 389.03 mg/L. so for disperse dye of 50 mg/L concentration coagulants dose should be increased by 8 times which is not reasonable economically and also produce large volume of sludge. Novel formulation is not recommended for disperse red.Effects of KOH dose as pH adjuster on the removal of direct, reactive and disperse red dye of 50 mg/L concentration by novel are shown in Figs. 5 and 6.

Effects of PVA dose as coagulant aid on the removal of direct, reactive and disperse red dye of 50 mg/L concentration by novel are shown in Figs. 7 and 8.Effects of PAM dose as flocculant on the removal of direct, reactive and disperse red dye of 50 mg/L concentration by novel are shown in Figs. 9 and 10.According to information obtained from figure 5 to 10, the optimum concentration of KOH, PVA and PAM for disperse; direct and reactive dye of 50 mg/L concentration is presented in Table 6.Effects of optimum concentration of KOH, PVA and PAM using together at the same time on the removal of direct, reactive and disperse red dye of 5 and 50 mg/L by novel are shown in Table 7 and 8.The results show only a few percentages of improvements in color removal of samples.

CONCLUSION

The results reveal that GAC can not remove disperse red. Acidic red, direct red and reactive red of 5 mg/L concentration are removed by GAC up to %90, %88 and %43 in 30, 60 and 120 minutes (equilibrium time) respectively, while the same dyes of 50 mg/L concentration are removed up to %93, %30 and %51 in 15, 90 and 150 min. respectively. GAC has high adsorption capacity for the removal of acidic red (90%). High adsorption for acidic red is attributed to net positive charge of this dye. Low adsorption capacities for reactive red dye may be due to high molecular weight of this dye. Colloidal molecules of disperse dyes plug the pores of Granular Activated Carbon, so this dye can not be removed by G.A.C. Hence, the experimental results are analyzed by using the Langmuir, Freundlich and BET equations. Adsorption obeys Freundlich isotherm for acidic red, BET isotherm for direct red and Langmuir isotherm for reactive red.

Considering the results, we may conclude that Activated Carbon consists of macro pores. During adsorption initially dye molecule has to first encounter the boundary layer effect and then it has to diffuse from boundary layer film on to adsorbent surface and then finally it diffuse in to the porous structure of the adsorbents. This process will take relatively long contact time. There after adsorption increases very slowly with respect to increasing time of contact. This may be due to the surface saturation with dye molecules and formation of monolayer coverage by adsorbed on the adsorbent surface (Mainul Karim *et al.*, 2006). In all cases, adsorption of color increase with carbon dose. This might be due to the increase in availability of surface active sites resulting from the increase dose and conglomeration of the carbons (Kannan and Karuppasamy, 1998). Optimized novel formulation which is found in this study (considering economical aspect) shows poor results for acidic red dye removal. H^+ which is released from this dye in solution, cause some problems in function of KOH and Na_2CO_3 as pH adjuster.

Direct red, reactive red and disperse red of 5mg/L concentration are removed by this formulation up to %93, %91.3 and %57.1 while dyes of 50 mg/L concentration are removed up to %90.8, %91.9 and %70.1, respectively. Novel is not recommended for removal of disperse red because of high doses of chemicals which are used and large volume of sludge production. This formulation shows more than %90 efficiency for both direct and reactive dyes and is highly recommended for concentrated form of these dyes. Sodium silicate enhances this action. Then, the coagulated particles flocculate by chain S of PAM and float toward free surface of wastewater. Polyvinyl alcohol as coagulant aid agent produces air bubbles among physical bandings (hydrogen bands) and therefore, rain forces the floatation of color as sludge. Since potassium hydroxide is expensive for industrial level of this work, similar but cheaper compound should be studied.

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