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Optimisation of the Coagulation-Flocculation of Reactive Dye Wastewater Using Novel Inorganic-Organic Hybrid Polymer

C. Xin-Hui Su, T. Tow Teng*, N. Morad, M. Rafatullah

School of Industrial Technology, Universiti Sains Malaysia, 11800, Penang, Malaysia

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A novel inorganic-organic hybrid polymer of magnesium chloride-polyethylene oxide ($MgCl_2$-PEO) was prepared, characterised and applied in the coagulation-flocculation of a reactive dye, Cibacron Blue F3GA (RCB). The hybrid polymers were prepared in various ratios and their conductivity and viscosities were measured. For the application in the coagulation-flocculation of RCB, the hybrid polymer of 90% $MgCl_2$:10% PEO ratio was selected as it showed the highest conductivity and lowest viscosity. The factors that affect coagulation-flocculation of RCB, namely initial dye concentration, initial pH, agitation speed, agitation time and hybrid polymer dosage, were studied using fractional factorial design and response surface methodology (RSM). The process was also optimised, with respect to colour removal and chemical oxygen demand (COD) reduction efficiency. The five factors studied showed significant effects toward the colour removal and COD reduction of RCB. The process was optimum at initial dye concentration of 173 mg/L, pH 11.13, agitation speed of 150 rpm, agitation time of 6 minutes and hybrid polymer dosage of 1020 mg/L. Under these optimum conditions, maximum colour removal of 99.76% and COD reduction of 92.09%, were achieved.

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INTRODUCTION

Coloured wastewater is produced by various industries, such as the textile, printing, food and pharmaceutical industries. It is a well-known environmental problem as dyes have complex structures and are recalcitrant. Dyes in wastewater cause high COD and biochemical oxygen demand (BOD) in the water bodies and are toxic to aquatic organisms as they tend to chelate metal ions[1]. Reactive dyes are highly soluble in water. They produce a bright and complete colour range, with excellent wet and light fastness, and are widely used for cellulosic fibres in textile industries[2]. Reactive dyes in wastewater cause high alkalinity, high concentration of organic materials and strong colour [3].

Coagulation-flocculation is a widely applied treatment among the industries for the treatment of dye wastewaters. Recently, the use of hybrid polymer flocculants in coagulation-flocculation has gained increasing attention due to the greater performance and advantages compared to the conventional coagulants [4]. The superior performance of these hybrid polymer flocculants are a result of the combination of coagulating and flocculating components which give a product of higher molecular weight and increased aggregation power [5]. By producing a single product with combined functionality, only one process is needed in the process compared to the conventional coagulation-flocculation process which involves the dosing of the coagulant and flocculant separately.

Hybrid polymer flocculants could be classified into inorganic-organic, organic-organic, organic-inorganic and inorganic-inorganic flocculants, where the inorganic-organic parings have been most widely developed [4]. However, many of the inorganic-organic hybrid polymers that have been studied are aluminium or iron based. For example, aluminium hydroxide-polyacrylamide (Al(OH)3-PAM) [6], polyaluminium

* Corresponding author: T. Tow Teng
E-mail: ttteng@usm.my
Tel.: +604 6532215; Fax: +604 6573678

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chloride-epichlorohydrin dimethylamine (PACl-EPI-DMA) [7] and ferric chloride-polyarylamide (FeCl3-PAM) [8]. Magnesium is known to be the second most plentiful cation in seawater in addition to its convincing coagulation property[9].

In the present study, an inorganic-organic hybrid polymer flocculant consisting of magnesium chloride (MgCl2) and polyethylene oxide (PEO) were prepared through physical blending and was used to remove the reactive dye, Cibacron Blue F3GA (RCB) from wastewater. The use of MgCl2 as a coagulant is less compared to aluminium and iron-based coagulants. While the use of polyacrylamide (PAM) as organic flocculant has been widely studied by researchers, the use of PEO has not been studied. PEO is a non-ionic, water soluble polymer with high molecular weight [10]. The MgCl2-PEO hybrid polymer flocculants were prepared in various ratios and their conductivity and viscosities measured to identify the most suitable ratio of inorganic coagulant to organic flocculant to be used in coagulation-flocculation. Then, the initial dye concentration, initial pH, agitation speed, agitation time and hybrid polymer dosage were studied to optimize the process.

MATERIALS AND METHODS

Magnesium chloride (MgCl2.6H2O; assay ≥ 99.0%) was obtained from R & M Chemicals, Polyethylene oxide (PEO; assay ≥ 99.0%) was obtained from Alfa Aesar, Reactive Cibacron Blue F3GA (RCB; CAS Number: 12236-82-7) was obtained from CIBA. The chemicals were used without further purification.

Preparation of synthetic dye aqueous solution

The aqueous solution of RCB was prepared by dissolving 0.1 g of RCB in distilled water to make 1 L of dye solution. The molecular weight of RCB is 840.1 a.m.u.. The initial pH of the dye solution ranged from 6.0-6.5.

Preparation of hybrid polymer

Aqueous solutions of MgCl2-PEO with desired compositions (90%:10%, 70%:30% and 50%:50%) were prepared and stirred for 24 hours at room temperature to obtain a homogenous solution. Only these three ratios of hybrid polymer flocculants were studied as a high content of polymer would cause an increase in cost and this is not favourable.

Conductivity and viscosity measurements

The conductivities of the hybrid polymers were measured using HACH, Sension5 conductivity meter.The viscosity measurement was carried out using Ubbelohde viscometer of the hybrid polymers of various ratios at 30 ± 0.05˚C. The viscosity was calculated using Eq. 1:

$$\eta = A\rho t \qquad (1)$$

where η is the dynamic viscosity, A is the calibration constant of the capillary viscometer, ρ is the solution density and t is the flow time.

Jar test

Synthetic aqueous dye solution was prepared according to Section 2.2. The pH of the solutions (pH 10-12) were adjusted using 0.1M HCl and 0.1M NaOH. The dye solutions (150 mL) were introduced into six beakers and hybrid polymer added to each beaker at different dosages (500-1500 mg/L). The samples were mixed at the desired speed (100-300 rpm) and time (3-15 min). After mixing, the solutions with flocs were allowed to settle for 30 min and the supernatant collected for colour and COD analysis.

Analytical methods

The COD of the solution was measured using the APHA standard methods for examination of water and wastewater, Method NO. 5220D (closed reflux, colorimetric method) [11]. The COD of the samples before and after treatment were measured using a spectrophotometer (HACH DR/2800). The percentage of COD reduction was calculated by Eq. 2:

$$COD\ reduction\ (\%) = \frac{C_i - C_f}{C_i} \times 100\% \qquad (2)$$

The colour concentrations of the dye solution before and after treatment were measured by UV-Vis through wavelengths 200-800 nm using Shimadzu UV-1601PC UV-Visible Scanning Spectrophotometer. The percentage of colour removal was calculated by Eq. 3:

$$Colour\ removal\ (\%) = \frac{C_i - C_f}{C_i} \times 100\% \qquad (3)$$

where $C_i$is the initial colour concentration (mg/L) and COD (mg/L) of solution and C_f is the the final colour concentration (mg/L) and COD (mg/L) of supernatant.

Design of experiment

For the design of experiment, fractional factorial design was used. The design was generated by a statistical software, Minitab 16. Five factors were studied, namely, initial dye concentration (A), initial pH (B), agitation speed (C), agitation time (D) and hybrid polymer dosage (E). The effect of each factor at different levels were determined through 2^{5-1} fractional factorial design. The response for the design was expressed as percentage of colour removal and percentage of COD reduction of RCB dye. For the optimization of the coagulation-flocculation process, a second-order polynomial response surface model [12] was used:

$y = \beta_o + \Sigma\beta_i X_i + \Sigma\beta_{ii} X_i^2 + \Sigma\beta_{ij} X_i X_j$

where y is the predicted response, β_o is the constant coefficient, β_i is the linear effect, β_{ii} is the square effect, β_{ij} is the interaction effect and X_i and X_j are the coded values of the variables.

RESULTS AND DISCUSSION

Conductivity and viscosity measurements

The conductivity of the hybrid polymers were measured and the results are shown in Fig. 1. The conductivity of the hybrid polymer increased with the concentration of magnesium salt because the addition of inorganic salt ionised the PEO aqueous solution [9].

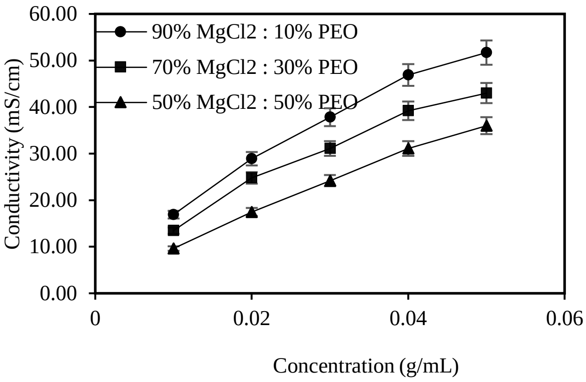


Figure 1. Conductivity of $MgCl_2$-PEO hybrid polymer aqueous solutions

From Fig. 2, it is shown that the viscosities of the hybrid polymer aqueous solutions increased with increasing concentrations of magnesium salt. At higher concentrations of magnesium salt, the network of the PEO could be destroyed [13].

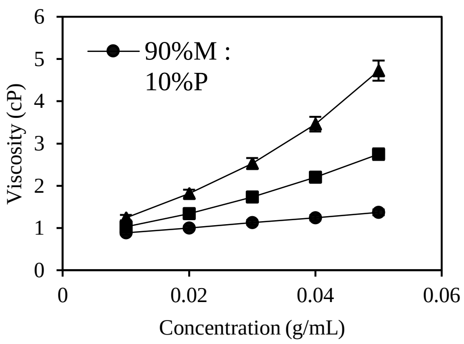


Figure 2. Viscosity of $MgCl_2$-PEO hybrid polymer aqueous solutions

For the application in flocculation of RCB dye aqueous solution, the hybrid polymer of ratio 90% $MgCl_2$:10%PEO was chosen because it has the highest conductivity and lowest viscosity. With lower viscosity, the network of the polymer is not destroyed; hence, the flocculation ability of the polymer is still high. Higher conductivity of hybrid polymer shows that the concentration of magnesium ions in the solution is higher. This will lead to higher adsorptive sites with positive electrostatic surface charge for better coagulation performance [14].

Screening of factors for coagulation-flocculation

The effects of five factors, i.e. initial dye concentration, initial pH, agitation speed, agitation time and hybrid polymer dosage on the performance of $MgCl_2$-PEO hybrid polymer on the colour removal and COD reduction of RCB dye aqueous solution using 2^{5-1} fractional factorial design. The treatment factors occur at two levels, a high range denoted by +1 and low range denoted by -1. The levels of the five factors studied are shown in Table 1. The experimental results for the screening process are shown in Table 2.

TABLE 1. Factors and levels of fractional factorial design

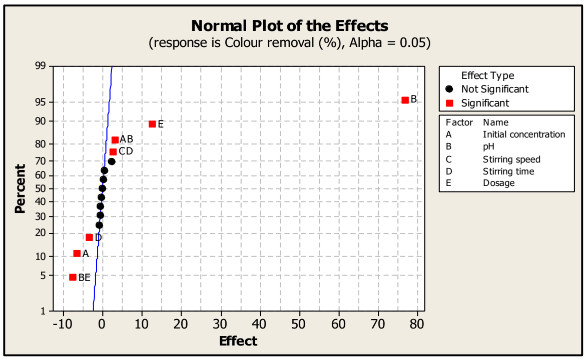
Terms	Factor	Level		
		-1	**0**	**+1**
A	Initial dye concentration (mg/L)	100	200	300
B	pH	10	11	12
C	Stirring speed (rpm)	100	200	300
D	Stirring time (min)	3	7.5	15
E	Flocculant dosage (mg/L)	500	1000	1500

The significance of each factor was evaluated using the normal probability plot of standardized effect with p = 0.05 (Fig. 3). From the plots, it can be seen that the main factors of initial dye concentration (A), pH (B),

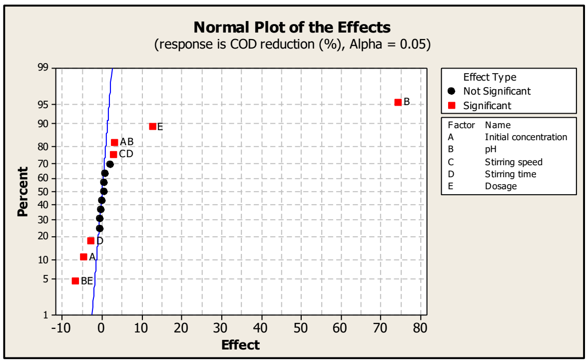
TABLE 2. 2^{5-1} fractional factorial design matrix for RCB dye wastewater treatment using $MgCl_2$-PEO hybrid polymer

A, Initial dye concentration (mg/L)	B, pH	C, Stirring speed (rpm)	D, Stirring time (min)	E, Dosage (mg/L)	Colour removal (%)	COD reduction (%)
300	12	100	15	500	90.04	82.37
300	10	300	3	1500	44.00	37.05
300	12	100	3	1500	99.80	91.73
300	10	100	3	500	12.00	4.32
300	10	100	15	1500	20.00	15.11
300	12	300	15	1500	99.76	92.09
100	10	300	3	500	16.67	9.63
300	12	300	3	500	91.24	85.97
100	12	300	3	1500	99.08	91.11
100	10	100	15	500	8.33	3.70
100	12	100	3	500	97.08	88.15
100	10	100	3	1500	33.33	27.41
100	12	300	15	500	98.67	89.63
100	12	100	15	1500	99.67	91.11
300	10	300	15	500	4.00	0.36
100	10	300	15	1500	16.67	11.11

stirring time (D) and flocculant dosage (E), and the interactions of pH and flocculant dosage (BE), initial dye concentration and pH (AB), and stirring speed and



a



b

Figure 3. Normal probability plot for (a) colour removal (%) and (b) COD reduction (%)

stirring time (CD) are significant towards both colour removal and COD reduction of RCB dye wastewater. The factors A, D and BE, which are situated on the left hand side of the straight line indicate negative effects while factors B, E, AB and CD situated on the right side of the line indicate positive effects on the responses. The effect of pH is the largest on both colour removal and COD reduction. This is proven by the location of point B on the normal plots, which is furthest away from the straight line.

The initial pH of the wastewater is an important factor towards the performance of the coagulation-flocculation process and the hybrid polymer flocculants are pH sensitive, similar to conventional coagulants [9]. During flocculation, the $MgCl_2$ component of the hybrid polymer would dissociate into magnesium ions (Mg^{2+}) and are converted into hydroxides [15]. The initial dye concentration reflects the amount of colloids present in the wastewater. At higher initial dye concentrations, a higher colloid content is present. This affects the flocculant dosage required for high efficiency in the flocculation process. However, an overdose of flocculant could cause the restabilisation of the flocs [16]. The performance of the coagulation-flocculation process is also dependent on the stirring speed and time applied to the wastewater. These two factors affect the size, strength and structure of flocs formed during coagulation-flocculation [17]. In the coagulation-flocculation using hybrid polymer flocculants, the two-step rapid and slow mixing are combined into one process, where a moderate stirring speed and time can be applied. A moderate stirring speed and time is required to ensure there is sufficient contact between flocculants and colloids. When the stirring speed is too fast or stirring time too long, the vigorous agitation provides high shear force and this would cause the flocs to be irreversibly broken down [17].

The empirical models in terms of coded factors for both colour removal and COD reduction are expressed in Eqs. 4 and 5, respectively.

$$\text{Colour removal } (y_1) = 98.33 + 3.52A + 14.51B - 5.81D + 10.00E - 30.55B^2 + 0.03C^2 + 0.09AB - 3.88CD + 0.59BE \quad (4)$$

$$\text{COD reduction } (y_2) = 97.78 + 2.57A + 12.33B - 6.98D + 9.32E - 29.51B^2 - 0.12\,AB - 3.81CD + 1.21BE \quad (5)$$

After the screening of factors, the process was optimised using central composite design (CCD). Table 3 shows the results obtained from the design.

The analysis of variance (ANOVA) for colour removal and COD reduction are shown in Table 4 and 5, respectively. The results of the ANOVA analysis show that the effects of the main factors (linear and square terms) and their interactions are indeed significant toward both the responses, with p-values less than 0.05. The values of coefficient of determination (R^2) measure the overall performance of the experiment. For colour removal and COD reduction, the R^2 values obtained were 99.22% and 99.16%, respectively. This indicates that only 0.78 and 0.84% of the variation of the model is unexplained.

Optimisation of flocculation process

The coagulation-flocculation of RCB using $MgCl_2$-PEO hybrid polymer was optimised to obtain the conditions for maximum colour removal and COD reduction. From the analysis of experimental data obtained (Table 3), the optimum conditions for the process were initial dye concentration of 173.13 mg/L, pH 11.13, stirring speed 149.91 rpm, stirring time 6 min, and hybrid polymer dosage 1019.875 mg/L. The predicted outcomes under these optimum conditions are 100% colour removal and 94.57% COD reduction. However, the actual outcomes obtained through experimental work were 99.76% colour removal and 92.09% COD reduction.

The three dimensional response surface plots for colour removal and COD reduction are shown in Figs. 5

TABLE 3. Results of CCD for colour removal and COD reduction of RCB dye using $MgCl_2$-PEO hybrid polymer

Initial concentration (mg/L)	pH	Stirring speed (rpm)	Stirring time (min)	Dosage (mg/L)	Colour removal (%)	COD Reduction (%)
300	10	300	15	1500	22.13	19.46
100	12	300	3	1500	99.44	92.88
200	11	200	9	1000	99.64	91.48
300	10	100	15	500	18.03	12.22
300	12	300	3	1500	98.85	93.67
200	11	200	9	1000	99.36	91.03
300	12	100	3	1500	99.06	93.67
100	10	100	15	1500	14.08	10.33
300	10	100	3	500	13.93	9.95
200	11	200	9	500	98.89	87.00
200	12	200	9	1000	99.82	91.93
200	11	200	9	1000	99.64	91.03
100	10	300	15	500	18.31	11.21
100	12	300	15	1500	99.3	91.04
100	12	100	15	1500	99.72	92.92
300	12	300	15	1500	98.73	90.95
100	12	100	3	1500	99.86	92.88
200	11	200	9	1000	99.68	91.03
100	10	100	15	500	14.08	9.36
100	10	100	3	1500	11.27	8.44
100	12	100	15	500	96.62	90.81
300	10	100	15	1500	30.33	24.43
200	11	200	9	1000	99.59	90.58
300	12	300	15	500	84.02	79.19
100	12	300	3	500	98.45	89.91
100	12	300	15	500	96.20	90.33
200	11	200	9	1000	99.59	90.13
300	12	100	3	500	86.48	80.09
200	11	200	15	1000	99.77	91.48
200	11	200	9	1500	99.55	90.58
300	12	300	3	500	98.97	90.50
200	11	300	9	1000	99.41	90.58
100	10	300	15	1500	15.49	11.76
200	11	200	9	1000	99.45	90.13
300	10	300	3	1500	9.84	4.98
100	11	200	9	1000	93.66	87.67
200	10	200	9	1000	99.5	90.58
300	10	300	3	500	5.74	1.36
300	10	300	15	500	1.64	1.36
200	11	200	3	1000	98.91	88.79
200	11	100	9	1000	99.59	91.48
200	11	200	9	1000	99.64	91.48
300	12	100	15	1500	98.32	90.05
200	11	200	9	1000	99.32	90.13
100	12	100	3	500	97.75	90.13
100	10	300	3	1500	22.54	17.37
300	11	200	9	1000	93.85	87.33
200	11	200	9	1000	99.5	91.03
100	10	100	3	500	14.08	9.68
100	10	300	3	500	16.9	10.44
300	12	100	15	500	81.56	76.02
300	10	100	3	1500	18.03	11.31

TABLE 4. ANOVA analysis for colour removal

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	20	62723.2	62723.2	3136.2	23.0	0.000
Linear	5	43798.6	43798.6	8759.7	64.2	0.000
Square	5	13867.1	13867.1	2773.4	20.88	0.000
Interaction	10	5057.5	5057.5	505.8	3.7	0.002
Residual error	31	303.5	303.5	9.8		
Total	51	63026.7				

R^2 = 99.22%; R^2 (adj) = 99.03%

and 6, respectively. The interaction between the significant factors displayed clear peaks, while other factors were controlled at centre point (initial concentration 200 mg/L, initial pH 11, stirring speed 200 rpm, stirring time 9 min and flocculant dosage 1000 mg/L). This shows that the optimum conditions for both colour removal and COD reduction are defined in the design boundary and that it is possible to maximise these responses while controlling the other factors at the centre point.

TABLE 5. ANOVA analysis for COD reduction

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	20	58841.1	58841.1	2942.1	22.76	0.000
Linear	5	41374.8	41374.8	8275.0	64.00	0.000
Square	5	17466.3	17466.3	3493.3	25.65	0.000
Interaction	10	4453.3	4453.3	445.3	3.44	0.004
Residual error	31	331.8	331.8	10.7		
Total	51	59172.9				

R2 = 99.16%; R2 (adj) = 98.09%

The optimum hybrid polymer dosage obtained in this study was 1019.875 mg/L and this value is much less compared to that if the conventional MgCl2 coagulant was used. To achieve similar efficiency, the MgCl2 dosage required would be around 4000 mg/L [14]. This is due to the higher molecular weight of the hybrid polymer that has enhanced aggregating power, forming larger flocs. This advantage is beneficial to the industries as a lower coagulant dosage reduces the cost of the treatment. Above the optimum dosage of hybrid polymer, the colour removal and COD reduction decreases. This could be due to the re-suspension of flocculated dye molecules into the supernatant [18].

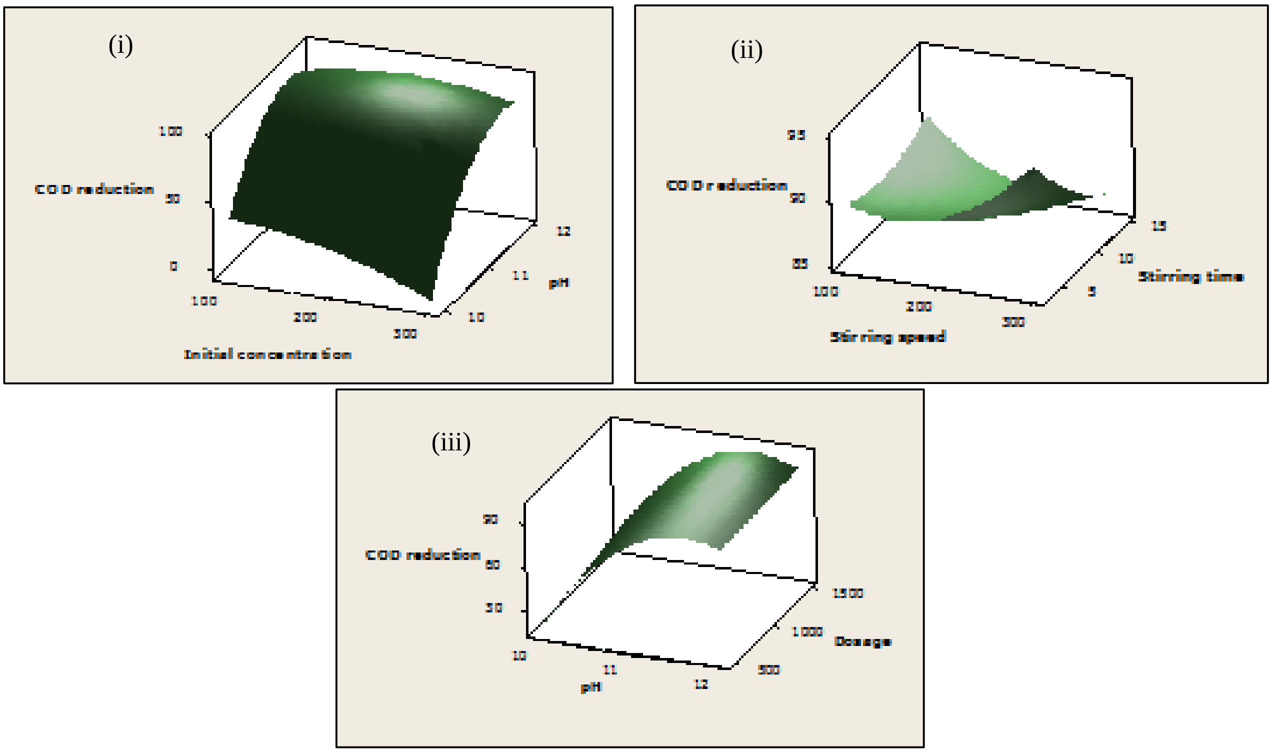


Figure 5. Three dimensional surface plots for RCB dye colour removal as a function of: (i) initial concentration and pH; (ii) stirring speed and stirring time; and (iii) pH and flocculant dosage

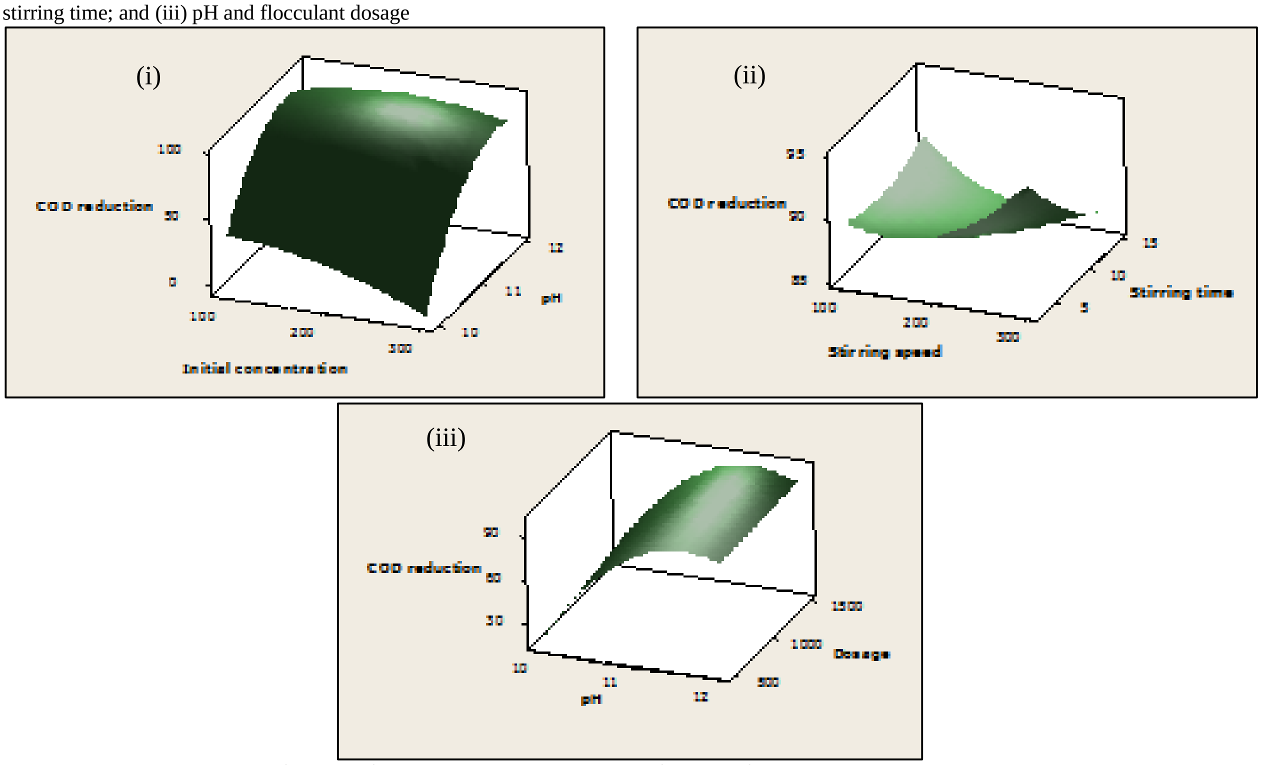


Figure 6. Three dimensional surface plots for RCB dye COD reduction as a function of: (i) initial concentration and pH; (ii) stirring speed and stirring time; and (iii) pH and flocculant dosage

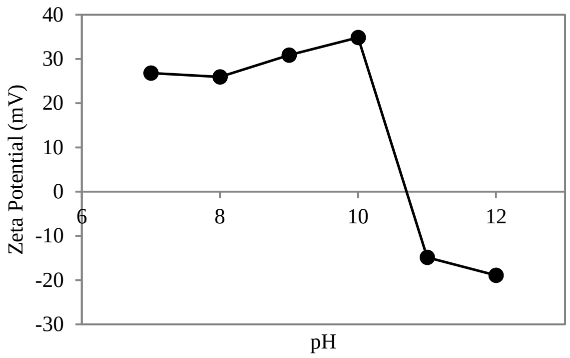


Figure 7. Effect of pH on zeta potential of RCB wastewater

The optimum pH of the hybrid polymer is similar to that of the conventional $MgCl_2$ coagulant because the hybrid polymer consists of 90% $MgCl_2$ and only 10% PEO. At this pH value, the Mg^{2+} ions are converted into hydroxides ($Mg(OH)_2$), which provide large adsorptive sites and positive electrostatic surface charge for it to act as an efficient coagulant [14]. The zeta potential of the solution was also measured at varying pH values to understand the coagulation mechanism of RCB dye when $MgCl_2$-PEO hybrid polymer was used. The results are shown in Fig. 4. Based on the figure, the zeta potential of the solution ranged from 26.79 mV to -18.88 mV. The zeta potential passed through 0 between pH 10 and 11. This point is called the point of zero charge, where the anions and cations are in equilibrium. This shows that the $MgCl_2$-PEO hybrid polymer functions through the charge neutralisation mechanism. At the point of zero charge, the solution pH is considered to be optimum. The point of zero charge obtained from the zeta potential curve is close to the optimum pH value obtained from the optimisation experiments. Charge neutralisation occurs between the anionic groups of RCB dye and the positively charged $MgCl_2$-PEO hybrid polymer, forming large flocs.

The stirring speed of 149.91 rpm for 6 min is another great advantage of using hybrid polymer flocculants in place of conventional coagulants. The coagulation and flocculation are integrated into one step and this saves time in the mixing process compared to the conventional two-step process.

The results of the coagulation-flocculation of RCB dye aqueous solution with $MgCl_2$-PEO hybrid polymer (Table 2 and 3) showed that there is a slight difference between the percentage of colour removal and COD reduction for all the runs. Similar observations were also found in the works of Wong, Teng [19] and Yeap, Teng [20]. This phenomenon could be explained by the partial degradation of RCB into smaller organics that are retained in the solution after the coagulation-flocculation. The presence of these organics in the solution contribute to the lower percentage of COD reduction throughout the experiment compared to the percentage of colour removal. This shows that the hybrid polymer flocculant does not only function to flocculate the dye molecules, it also functions to degrade the complex dye molecule into organic molecules of lower molecular weights. To fully understand this phenomenon, a detailed study could be carried out in the future.

CONCLUSION

The inorganic-organic component ratio in the hybrid polymer that is most suitable to be used as flocculant is 90% $MgCl_2$: 10% PEO due to the high conductivity and low viscosity. The use of $MgCl_2$-PEO hybrid polymer as a novel flocculant has shown great potential for the treatment of RCB. The five factors studied, namely, initial dye concentration, pH, stirring speed and time, and the flocculant dosage show significant effect towards the performance of the hybrid polymer flocculant. Among these factors, solution pH had the largest effect on the colour removal and COD reduction of RCB. The optimum colour removal and COD reduction of 99.76% and 92.09%, respectively, for the process could be obtained at initial dye concentration of 173.13 mg/L, pH 11.13, stirring speed 149.91 rpm, stirring time 6 min and flocculant dosage of 1019.875 mg/L.

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Persian Abstract

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چکیده

در این مطالعه پلیمر ترکیبی آلی-غیرآلی جدید $MgCl_2$_PEO ساخته شد و برای انعقاد رنگ واکنشی Cibacron Blue F3GA(RCB) مورد استفاده قرار گرفت.این پلیمرها در نسبت های مختلف تهیه و ضریب هدایت و ویسکوزیته برای محاسبه گردید. در ادامه پلیمر ترکیبی با نسبت ۹۰٪ $MgCl_2$:10%PEO برای انعقاد RCB، به علت داشتن ضریب هدایت بالا و ویسکوزیته کم انتخاب شد. فاکتورهایی مانند غلظت رنگ اولیه،pH اولیه،سرعت همزدن، زمان همزدن و مقدار مصرفی پلیمر ترکیبی در انعقاد RCB اثر گذار بودند که با استفاده از طراحی فاکتوریل کسری و روش پاسخ سطح(RSM) مورد بررسی قرار گرفتند. همچنین در این فرایند بهینه سازی با توجه به حذف رنگ و راندمان کاهش COD نیز صورت گرفت. پنج عامل مورد مطالعه اثرات قابل توجهی نسبت به حذف رنگ و کاهش COD از RCBنشان داد. مقادیر بهینه فرایند در غلظت رنگ اولیه ۱۷۳ میلی گرم بر لیتر، پی اچ ۱۱/۱۳، سرعت همزدن ۱۵۰ rpm زمان همزدن ۶ دقیقه و مقدار مصرفی پلیمر ترکیبی۱۰۲۰ میلی گرم بر لیتر بدست آمد و تحت این شرایط به ماکزیمم حذف رنگ ۹۹,۷۶٪ و کاهش 92.09% COD دست یافته شد.