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Modelling and Optimization of Indigo Carmine Adsorption onto P(NIPAM-*co*-AN)/Clay Composite Using Response Surface Methodology

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Abstract

In the present work, a new polymeric adsorbent Poly(NIPAM-*co*-AN)/clay composite was prepared via in-situ polymerization. The copolymerization of N-isopropylacrylamide, acrylonitrile and calculated clay (about 5% w/w) were carried out at 65°C in the presence of 10 mL 1,4-dioxane using AIBN (azobisisobutyronitrile) as an initiator. Characterization of P(NIPAM-*co*-AN)/clay composite was determined by FT-IR techniques. NIPAM-AN/clay composite has been used as a novel adsorbent to remove indigo carmine dye from synthetic effluents. Three important process parameters i.e., initial IC (indigo carmine) concentration (5-30 mg/L), adsorbent dosage (10-80 mg) and contact time (10–180 min) were modeled and optimized to get the best response of indigo carmine removal using a Central Composite Design assembled with Response Surface Methodology. The amount of indigo carmine in the solution was measured using a UV-visible spectrophotometer at a wavelength of 650 nm. Based on the ANOVA (analysis of variance) statistical value, the adsorption of indigo carmine onto P(NIPAM-*co*-AN)/clay composite is highly significant, with very low probability value ($p<0.001$). Twenty test runs were performed and the optimal conditions for indigo carmine adsorption were observed at initial indigo carmine concentration of 17.68 mg/L, the adsorbent dosage of 60 mg and reaction time 115.47 min. The maximum indigo carmine adsorption efficiency under optimal conditions was 76.21%. It was concluded that NIPAM-AN/clay composite has the potential for removal of IC from aqueous solutions.

Keywords: Adsorption, polymeric adsorbent, response surface methodology

Cevap Yüzey Metodolojisi Kullanılarak P(NIPAM-*co*-AN)/Kil Kompozit Üzerine İndigo Karmin Adsorpsiyonunun Modellenmesi ve Optimizasyonu

Öz

Bu çalışmada yeni bir polimerik adsorban olan Poli(NIPAM-*co*-AN)/kil kompoziti yerinde polimerizasyon ile hazırlanmıştır. N-izopropilakrilamid, akrilonitril ve uygun miktarda alınan kilin (yaklaşık %5 w/w) kopolimerizasyonu, 10 mL 1,4-dioksan varlığında 65°C'de, AIBN (azobisizobütironitril) başlatıcısı varlığında gerçekleştirildi. NIPAM-AN/kil kompoziti IC (indigo karmin) boyasını sentetik atık sulardan uzaklaştırmak için yeni bir adsorban olarak kullanılmıştır. Üç önemli proses parametresi olan başlangıç indigo karmin konsantrasyonu (5-30 mg/L), adsorban dozu (10-80 mg) ve temas süresi (10-180 dakika) yanıt yüzeyi metodolojisi (RSM) ile birleştirilmiş. Merkezi Kompozit Tasarım (CCD) kullanılarak modelleme yapıldı ve en iyi indigo karmin giderme yanıtını elde etmek için optimize edildi. Çözeltideki indigo karmin miktarı, 650 nm dalga boyunda bir UV-görünür spektrofotometre kullanılarak ölçülmüştür. ANOVA (varyans analizi) istatistiksel değerine dayalı olarak, indigo karminin P(NIPAM-co-AN)/kil kompoziti üzerine adsorpsiyonu, çok düşük olasılık değeriyle ($p<0.001$) oldukça önemlidir. Yirmi test yapıldı ve indigo karmin İK adsorpsiyonu için optimal koşullar, 17.68 mg/L'lik ilk indigo karmin konsantrasyonunda, 60 mg'lık adsorban dozajında ve 115.47 dakikalık reaksiyon süresinde gözlemlendi. Optimum koşullar altında maksimum indigo karmin adsorpsiyon verimi %76.21' dir. NIPAM-AN/kil kompozitinin indigo karminin sulu çözeltilerden uzaklaştırılması potansiyeline sahip olduğu sonucuna varılmıştır.

Anahtar Kelimeler: Adsorpsiyon, polimerik adsorban, cevap yüzey yöntemi

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INTRODUCTION

In recent years, population growth with increasing needs has led to many industrial developments, which has led to an increase in pollution. Discharge of dye-containing wastewater from different industries into aquatic environments causes serious environmental problems due to the color it contains, reducing sunlight penetration and being toxic to aquatic organisms. The existence of color in wastewater at very fallen concentrations. This is visible and unwanted. Many dyes can cause serious health problems in people, for example allergies, mutations and cancer. Therefore, dye removal from wastewater is essential both environmental and human health (Tanyol, 2017; Zhang et al., 2008). Another name for indigo carmine (IC) is the disodium salt of 5,50-indigo disulfonic acid. This matter is a deep blue polyester dye used to dye fibers, jeans and food industries (Vidya et al., 2017). The removal of dyes from industrial wastewaters is of great importance in recent years due to their toxicity (Vesna et al., 2013). Some dyes are highly toxic in nature, It has been reported that they are insoluble and mutagenic, directly or indirectly affecting human, animal and environmental health. Therefore, the treatment of dye-containing wastewater is an important issue and many methods have been reported in the literature (Boudechiche et al., 2019). Among these methods, adsorption is widely used because of its easy operation, cost-effectiveness and high efficiency. When a solid comes into contact with a liquid (or gas), some liquid is retained by the solid, and if it takes place on the surface of the solid, it is called adsorption. The solid on which the liquid is held is called the adsorbent, and the substance on the surface of the solid is called the adsorbed substance (Yörükoğulları, 1997). Adsorption is a complex process and many parameters affect the adsorption time. These effective parameters are generally the properties of the adsorbent, the adsorbed material, and the adsorption medium (Kayman, 2009). Adsorbents to be used in treatment should have a suitable pore formation and surface chemical combination, a powerful adsorption capacity for adsorbate, big recyclability and reusability for economic, good mechanical events and easy of use and handling (Lozano-Morales et al., 2018). It was aimed to remove IC from aqueous solutions by adsorption using the response surface method (RSM). This is one of the most common statistical and mathematical technical programs used for the optimization of multiple variables. RSM helps to determine the individual and interaction effects of independent variables and process parameters can be optimized with a limited number of experiments (Mirzabe and Keshtkar, 2015). RSM is a method based on statistical technique based on the design of experiments and interpreting the effect of more than one factor at the same time (Ece et al.,2020).

There are many studies on the removal of indigo carmine. In the literature, the use of adsorbents such as activated carbon, fly ash, chitin, chitosan, metal oxides, agricultural waste, polymers, zeolite, alumina, calcium oxide has been previously reported in the removal of IC by the adsorption process. (Ramesh et al., 2017). Mittal et al., (2006); In their study, they developed an inexpensive adsorption method for the removal of indigo carmine, a very toxic indigoid dye class, from wastewater. Bottom ash, which is a power plant waste, and degreased soybean, which is an agricultural waste, were used as waste materials as adsorbent. Cestari et al., (2008); In their study, they used a 2-factorial design to evaluate the quantitative removal of indigo carmine dye from aqueous solutions on glutaraldehyde cross-linked chitosan. Harrache et al., (2019); In their study, they investigated the equilibrium and kinetic parameters of indigo carmine removal from aqueous solutions using activated carbon. In other study, poly(DMAEMA-co-AN) synthesized in the laboratory can be used as an effective adsorbent for the removal of IC from aqueous solutions.

In this present work, a new polymer [P(NIPAM-*co*-AN)] was synthesized from acrylonitrile (AN) and N-isopropylacrylamide by in situ method FRP. Adsorption study of indigo carmine by polymer was made by Response Surface Methodology using a Central Composite Design.

MATERIAL AND METHODS

Materials

N-isopropylacrylamide, acrylonitrile, indigo carmine, 1-4 dioxane and clay were gotten Sigma-Aldrich. As dyestuff, indigo carmine, a blue water-soluble dye with a molecular formula $C_{16}H_8O_8N_2S_2Na_2$ and a molecular weight of 466.36 g/

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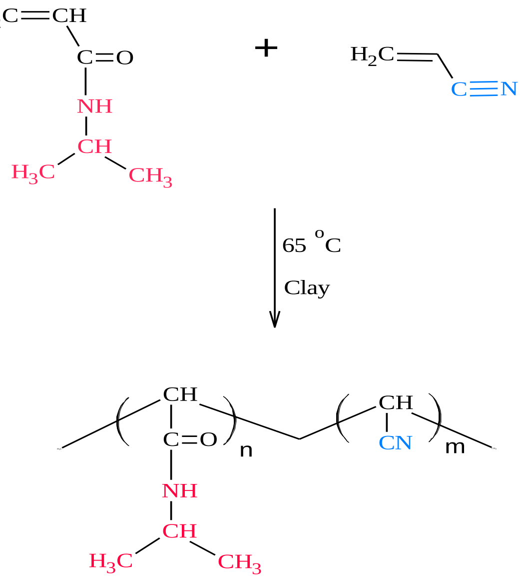
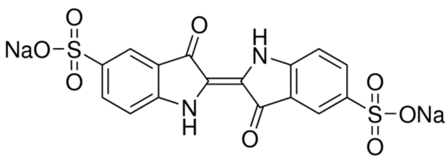
mol was used. 2,2′-azobisisobutyronitrile (AIBN) is initiator for free radical polymerization.

Preparation of bentonite-filled adsorbent via in-situ method

Poly(NIPAM-co-AN)/clay composite polymer was produced by using in-situ FRP (Free radical polymerization). 1 g N-isopropylacrylamide and 1g acrylonitrile monomers were solved within 1,4-dioxane within a polymerization tube. About 0.02 g AIBN and 15% by mass of clay were added to the mixture and degassed inside Ar gas for 10 min. Polymerization was continued for 24 hours in a 65 ^{o}C adjusted oil bath. The copolymer was precipitated within excess diethyl ether. The product was dried under vacuum at 45 ^{o}C for 24h. The prepared of the polymer was illustrated in Scheme 1.



Scheme 1. Synthesis of P(NIPAM-*co*-AN)



Scheme 2. Formation of indigo carmine

Adsorption Experiments

Poly(NIPAM-*co*-AN)/clay composite was used as polymeric adsorbent for experiment. The concentrations of the dye in aqueous solutions were varied from 25 to 100 mg/L by diluting the indigo carmine stock solution (500 mg/L) with distilled water. Batch adsorption studies were carried out in orbital shaker (stirring speed of 400 rpm) using 250 mL erlenmeyer flasks with a volume of 100 mL. In all adsorption works in the batch system, the P(NIPAM-co-AN)/clay composite was taken as $t = 0$ before adding to the solution. 5 mg adsorbent

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(polymer) was added to the flasks. The flasks were then shaken in an orbital incubator at 200 rpm at 30 °C for different contact times. During adsorption, samples were taken into glass tubes at pre-determined time intervals (between 10 to 180 minutes) After adsorption, samples were centrifuged at 4500 rpm for 10 min. The amount of indigo carmine remaining in the solution was determined by UV–Vis spectrophotometer. Measurements were taken at a wavelength of 650 nm. Dye removal yield was calculated using Equation (1):

$$R\% = \frac{C_i - C_e}{C_i} x\ 100 \quad (1)$$

Where C_i and C_e are the indigo carmine concentrations (mg/L) before and after adsorption, respectively.

Experimental design with CCD

Process parameters affecting the removal of indigo carmine were analyzed using the central composite design created using Design Expert Software (7.0 trial version). The color removal percentage was taken as the answer (dependent variable) obtained from the design experiments.

Three independent variables including initial dye concentration (5-30 mg / L), adsorbent dosage (10-80 mg) and contact time (10-180 min.) were used in the Central Composite Design (CCD). The central composite design was carried out with a series of 20 experiments with five levels (-α, -1, 0, 1, + α). These factors and their levels are given in Table 1. The data were analyzed by statistical method (ANOVA).

Table 1. *Factors, levels and date of CCD*

Variables	Code	Level of factors				
		-α	**-1**	**0**	**1**	**+α**
Initial IC concentration (mg/L)	*A*	5	10.07	17.50	24.93	30
Adsorbent dosage (mg)	*B*	10	24	45	66	80
Reaction time (min.)	*C*	10	44.46	95.00	145.54	180

RESULTS AND DISCUSSION

Characterization by FT-IR

Figure 1(a) and (b) show the FT-IR spectrum of P(NIPAM-co-AN)/clay before/after adsorption process. When the two spectrums were compared, there weren't any modification in the bands of the composite later the adsorption event. We can be clarified this result by van der Waals interaction between the dye and the polymer molecules and we can say that adsorption event occurred on the surface of the adsorbent The band at 3452 and 3300 cm^{-1} are assigned to the stretch for the hydrogen- bonded N–H group, the strong peak at 1650 cm^{-1} assigned to (C=O) a stretching vibration and at 1540 cm^{-1} corresponds to N–H vibration of the amide group (–CONH) in NIPA and 2239 cm^{-1} attributed to the nitrile groups (-C≡N) on AN units.

ANOVA Analysis

20 experiments were conducted with the CCD experiment design and the working conditions and response of each experiment are given in Table 2. The regression model equality used to make estimates about the response for given steps of each independent variable. A quadratic model equality is obtained. The equation for indigo carmine (%) in terms of coded factors is given as follows:

$$\text{IC removal, \%} = +68.96 - 0.40A + 13.92B + 6.93C + 9.04AB - 2.95AC + 7.24BC - 8.99A^2 - 10.23B^2 - 15.54C^2 \quad (2)$$

While positive (+) signs define a synergistic effect, negative (-) signs define an antagonistic impact in Equation 2 (Kumar et al., 2009). Here; IC indicates that the initial concentration (A) has a negative effect, the adsorbent dosage (B) and the reaction time (C) have a positive effect. It shows that the batch adsorption process and IC removal decrease with increasing dye initial concentration and

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increases when the amount of adsorbent and contact time is increased (Radnia, et al., 2017).

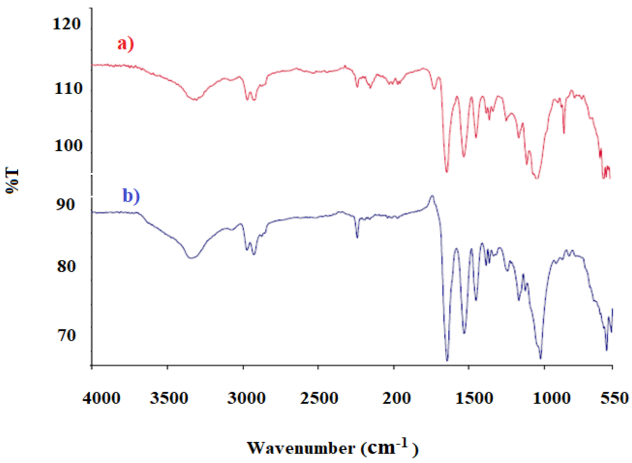

Figure 1. *FT-IR spectrum of P(NIPAM-co-AN)/clay composite (a) before adsorption experiment (b) after adsorption*

Table 2. *Design matrix for three coded factors together with response*

Run	Coded levels			IC Removal, %
	A	***B***	***C***	
1	24.93	0.02	145.54	18.45
2	5.00	0.04	95.00	46.55
3	17.50	0.04	95.00	69.23
4	10.07	0.02	145.54	33.33
5	17.50	0.04	95.00	69.23
6	10.07	0.07	145.54	62.31
7	17.50	0.04	95.00	69.23
8	24.93	0.02	44.46	14.98
9	17.50	0.08	95.00	57.87
10	10.07	0.02	44.46	26.08
11	17.50	0.04	10.00	15.75
12	24.93	0.07	44.46	51.19
13	17.50	0.04	180.00	24.90
14	17.50	0.04	95.00	69.23
15	17.50	0.04	95.00	69.23
16	17.50	0.01	95.00	12.82
17	24.93	0.07	145.54	75.59
18	17.50	0.04	95.00	69.23
19	10.07	0.07	44.46	18.11
20	30.00	0.04	95.00	31.20

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Analysis of variance (ANOVA) was applied to test the fit of the equation for the model and the results is given for indigo carmine removal in Table 3. In order for the results to be meaningful, the observed significance value for the t-test, which is expressed as "Probe>*F*" in the model, should be less than 0.05. If this value (Probe>*F*) is greater than 0.10, the model becomes meaningless for the response variables. Table 3 was indicated, the model fit of the value is more significant. *F*-value (18.15) and *p*-value (<0.0001) (Ece et al., 2021) of the model mean that it is well considerable for IC adsorption onto Poly(NIPAM-*co*-AN)/clay (Torğut et al., 2017; Chawla et al., 2019; Zyaie et al., 2018). It can be said that the larger the *F*-value of the model and the smaller the *p*-value, the more significant the model applied. The probability of such a large "Model *F* Value" occurring due to noise is only 0.01%. The results show that the data of Prob>*F* for *B*, *C*, *AB*, *BC* A^2, B^2 ve C^2 (P<0.05) are all less than 0.05, which shows that these values are statistically significant. In addition, these results show that the amount of P(NIPAM-*co*-AN)/clay is much more effective on the IC removal efficiency than the other two independent variables and is an important parameter, especially due to the lower *p*-values. At the same time, we see that besides the linear effect of reaction time (C), the second order effect is also significant (p< 0.0001). Adeq precision value is used for the model to describe the relationship between quadratic equations and experimental results, and it is required to be greater than four (Arslan-Alaton et al., 2011). Adeq precision value was read as 11.27. It indicates that the model equation for color removal is quite sensitive in estimating experimental results, indicating that there is sufficient signal. Other important data is; the correlation coefficient (R^2) is 0,9423. R^2 value close to 1 predicts better correlation between experimental and predicted values and better model response (Tepe and Dursun, 2014). Besides, it shows that only 6% of the total variation cannot be describe by the model. The program was monitored the Adjusted R^2 value as 0.89. This point out that the indigo carmine adsorption of the model is an important ingredient, provided to well agreement between the experimental and predicted extraction yield. According to the results of all these analyzes, the model is trustworthy and can be used to the design space. These results show that the proposed models are in good agreement with the experimental data (Batur et al., 2020).

Response Surfaces and Contour Plots for Indigo Carmine Adsorption

To research the interaction effects of the two independent factors on the color removal efficiency, 3D (three-dimensional response surface plots were drawn. The response surface plots can be considered as a way to estimate the dye removal efficiency (%) varied values of the data worked. We can see these graphs in Figure 2a-c. Figure 2a shows the response surface plot as a function of beginning dye concentration and adsorbent dosage when time is kept constant at the center step. As shown, the removal efficiency of the dye decreases with rising initial indigo carmine concentration. The decrease in the percentage of dye removal at big IC concentrations may be because of the satiation of the dye adsorption sites of the adsorbent. Rising the adsorbent mass provided more surface field, thus leading to more binding sites for the dye adsorption over P(NIPAM-*co*-AN)/clay (Dil et al., 2017). In Figure 2b, the amount of polymer was kept constant and the interaction effect on the reaction time and percent removal of the IC initial concentration was investigated. The reaction time increased to a certain point (about 95 min). After reaching equilibrium, it has not changed much. This attitude can be clarified by the fact that at low dye concentration, a great number of vacancies are present for adsorption on the P(NIPAM-*co*-AN) surface, but with the increase in dye concentration, the number of these sites decreases (Sharma et al., 2017). In Figure 2c, we can see a 3D graph of the amount of adsorbent and the reaction time when the indigo carmine concentration is kept stationary at the central step. Contact time ensures more diffusion between polymer particles and paint. Higher indigo carmine removal values were achieved with happening at the same time increases in adsorbent dose and shaking time. We see that the adsorption capacity of the IC increases with increasing contact time (Kutluay et al., 2020). It is economically important that the adsorbent dosage and agitation time are low (Pavlovic et al., 2014).

In general, it is important to ensure that the chosen model provides an appropriate approximation to the real system. Figure 3 presents perturbation plots showing the effect of each of the independent variables on the IC. It shows that the initial concentration (A), adsorbent dose (B) and time (C) are highly influenced by the operating parameters of

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IC adsorption (Tepe and Dursun, 2021; Sadhukhan, et al., 2016).

Table 3. *ANOVA and model summary statistic.*

Source	Sum of squares	Degree of freedom	Mean square	*F*-value	*p*-value
Model	9679.47	9	1075.50	18.15	< 0.0001 sig.
A-IC concentration	2.16	1	2.16	0.036	0.8523
B-Adsorbent dose	2646.83	1	2646.83	44.66	< 0.0001
C-Reaction time	656.79	1	656.79	11.08	0.0076
AB	654.13	1	654.13	11.04	0.0077
AC	69.50	1	69.50	1.17	0.3043
BC	418.76	1	418.76	7.07	0.0240
A^2	1163.50	1	1163.50	19.63	0.0013
B^2	1509.17	1	1509.17	25.46	0.0005
C^2	3481.87	1	3481.87	58.74	< 0.0001
Residual	592.71	10	59.27		
Cor Total	10272.18	19			
R^2	Adjusted R^2	Adeq precision	Std. dev.	Mean	
0.9423	0.8904	11.274	7.70	45.23	

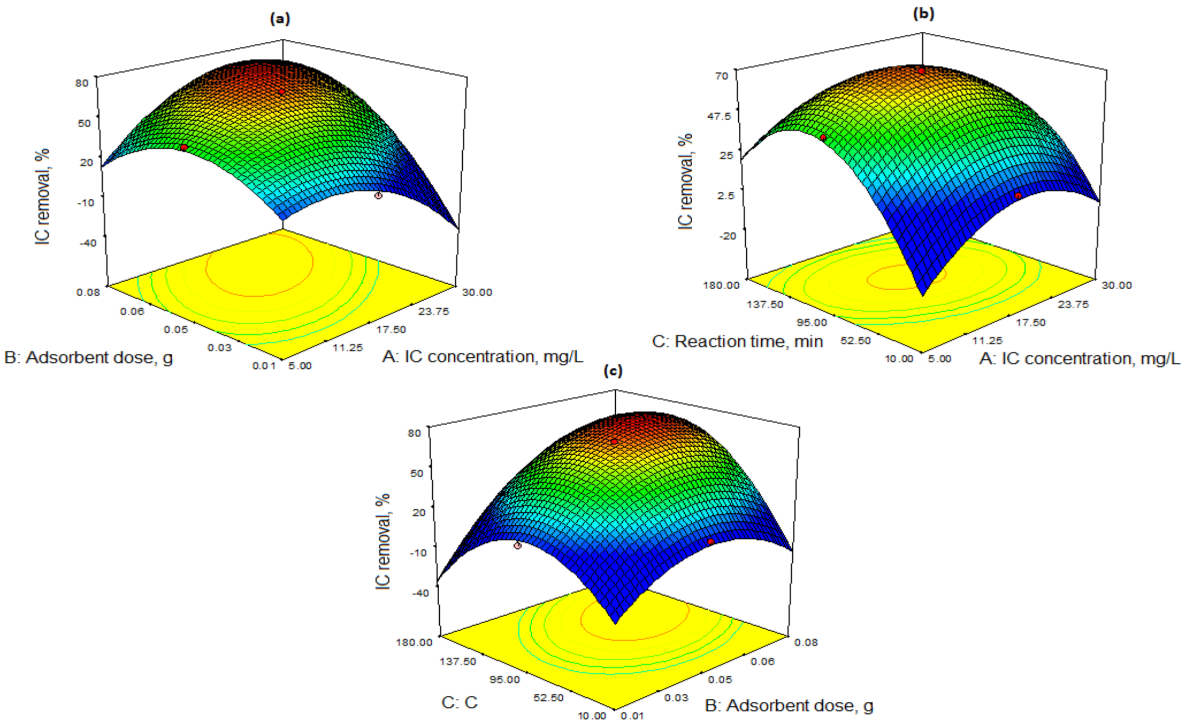

Figure 2. 3D response surface plots

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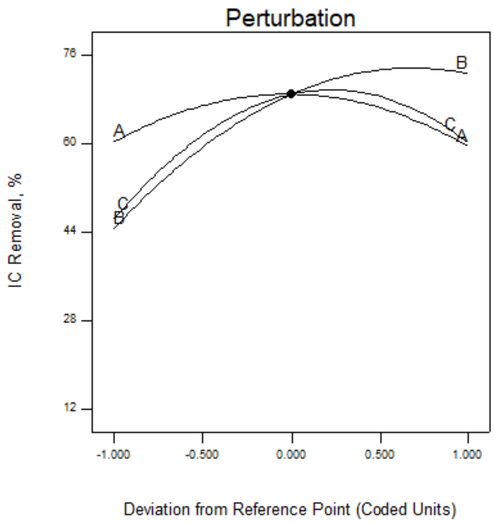

Figure 3. Perturbation plot of IC removal

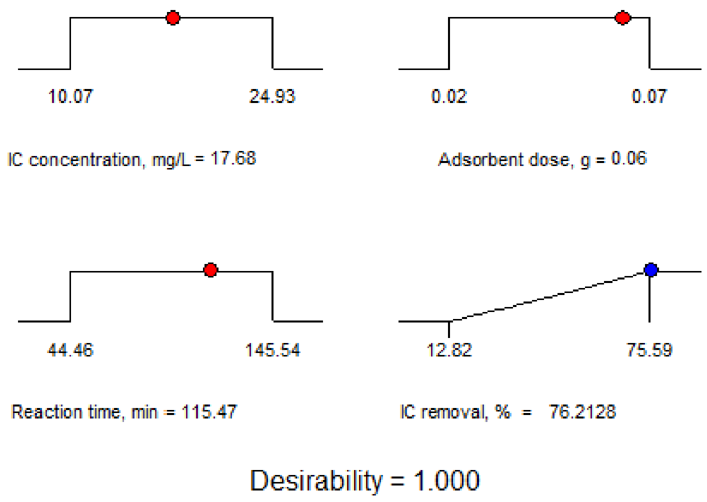

Figure 4. Desirability ramp of IC removal

Optimisation Using Desirability Function

For color removal using batch adsorption, the optimum experimental conditions giving maximum removal efficiency were determined using the desirability function of Design Expert. (Figure 4). The optimum conditions selected are the initial IC concentration of 17.68 mg/L, the amount of polymer 0.06 g, the reaction time 115.47 minutes. Under these conditions, the removal efficiency is 76.21%.

CONCLUSION

In the current study, P(NIPAM-*co*-AC)/clay polymeric adsorbent was synthesized. copolymer was used as an adsorbent for the removal of Indigo Carmine dye from an aqueous solution. The optimum conditions selected are the initial IC concentration of 17.68 mg/L, the amount of polymer
0.06 g, the reaction time 115.47 minutes. The conclusions of the experiments indicated the polymeric adsorbent could remove 76.21% of indigo carmine under optimum conditions. Water pollution is one of the world's most adverse environmental problems. It is important to find a good adsorbent to remove this pollution from the water. We think that the polymer we synthesized will perform this task. In general, P(NIPAM-*co*-AC)/clay because of its top efficiency, simple fabrication and ease of isolation can be used as a reasonable adsorbent for the removal

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of indigo carmine dye from wastewaters. This new adsorbent is very significant due to can be obtained easily and cheaply.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

RESEARCH AND PUBLICATION ETHICS STATEMENT

The author declares that this study complies with research and publication ethics.

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