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Introducing a novel chemical method of treatment for dye removal: Removal of Maxillon Blue and Direct Yellow from aqueous solution

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ABSTRACT

Many industrial and chemical dyes are used in many industrial processes, a variety of different uses. Among the most important of these industries, fabric dyeing, many textiles, papermaking, printing and leather processing, and most food products, and other industries, use industrial dyes, and the waste emitted from these dyes in most industries in which most of the polluted waste resulting from these remnants is produced factories. Therefore, the best methods were adopted to treat and dispose of these dyes in order to preserve the aquatic environment .In the current study, the adsorption characteristics of two dyes, Maxillon blue (GRL) and Direct yellow (DY12), from an aqueous solution were evaluated. The effect of several factors, for example, initial concentration, pH solution, temperature, adsorbent mass, and equilibrium time, have been study. The adsorption capacity and percentage of color removed upraised by elevating contact time and surface area, also elevated by the upraised temperature solution for GRL and DY12, and also the adsorption efficiency decreased by the raised adsorbent dosage. The optimum equilibrium for contact time to be completed is found to be (60 min) 1 hour. It is essentially due to the saturation of the active site that does not let further adsorption to take place. The adsorption was proved through utilizing (FT-IR) and (F.E-SEM) analysis. In addition, TGA appears that the NTADCIP/P (AA-co-AM) composite is stable in high temperatures. GRL adsorbent surfaces best adsorption found to be at pH = 10. In fact, adsorption was found to increase through the rise in solution pH. However DY12 maximum dye adsorption was found to be at pH = 3. The applicability of isotherms adsorption to study the adsorption behavior has to been analyzed via isotherm models Freundlech and Langmuir were utilized to illustrate the experimental model and isotherm's constants. It was found the Freundlech model gives the best fits when compare with Langmuer isotherm for two dyes.

Keywords: Treatment, Pollution, Environment, Adsorption, Textile, Dyes.

Article type: Research Article.

INTRODUCTION

Industrial dyes, especially chemical dyes, are considered one of the most dangerous pollutants as a result of the many uses of these dyes in many industries, such as dyeing textiles, fabrics, heirlooms, leather and other industries. Therefore, dyes pose a great danger to human, animal and plant health as a result of their accumulation in very large quantities and in high concentrations in the aquatic environment. The liquid waste that is produced from dye factories contains large quantities of colored waste. It produces annually about 915,000 tons of chemical and synthetic dyes, and 49% of the most dangerous of these dyes are azo dyes. Dyes have a complex aromatic molecular structure and are usually resistant to light, temperature, and oxidants. This distinctive feature makes the color non-degradable and therefore causes the biological accumulation of living organisms, leading to severe

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diseases and disorders (Bozorgpanah Kharat *et al.* 2018; BanimahdKeivani 2018).Pollution of surface and ground waters lead to a danger to human health and the environment because of the potential health risks of their contents, organic and inorganic complex too. The pollution of these waters via dye is a hard job that scientists have been battling to loosen through the last years. Dye has some unwanted aspect effects, like toxicity, mutagenic nature, and carcinogenic (Quesada *et al.* 2019; Danner *et al.* 2019). Many dyes are utilized in several industries like plastics, textiles, printing, dyeing, paper, food, and cosmetics to color the produces that have been factory-made (Al-Hayder & AL-Juboory 2015; Al-Hayder & Al-Hussainawy 2016; Shikha & Jha 2019). Colors are significant features of various wastes liquid where they are easy to detect and access to their sources. Dyes are organic chemicals that on binding by materials, it will give color to the materials. The dye color is provided via the attendance of a group chromophore (Chaturvedi 2019, Alkherraz 2020, Sultana 2021, Sharma 2021, Hatami 2021). A chromophore is a configuration consisting of conjugated double bonds containing delocalized electrons (Alkaim*et al.* 2017; Aljeboree & Alshirifi 2018: Hosseinpour *et al.* 2018).Other common chromophoric configurations contain (-C = O) carbonyl; (-N=N-) azo, (-CH=N – or > C = NH) carbon-nitrogen; (-C=C-) carbon; (=NO-OH-or NO2) nitro; (-NO or N-OH) nitroso; and (C=S) Sulphur. The chromogen that is the aromatic structure normally having naphthalene, benzene, or rings anthracene, is kind of a structure of chromogen-chromophore along within an axo-chrome (Hunger 2007; Aljeboree *et al.* 2019). Thus, the object of the current investigation was to study the adsorption method of GRL on chosen surfaces [NTADCIP/P (AA-co-AM) composite] at the condition changes of temperature and pH.

MATERIALS AND METHODS

Adsorbant

The two textile dyes, basic Maxillon blue (G.RL) and Direct yellow 12 (D.Y 12), were obtained from the textile factory in Hilla/Iraq (a Swedish company). Solutions of the test reagents were made via dissolving these dyes in deionized water. The adjusted pH of GRL and DY 12 were 6.13 and 6.02 respectively. The chemical structures and formula and other properties of the two dyes are set as follows:

Maxilon blue (GRL)

The maxilon blue (GRL) is a basic cationic dye. The solution is blue in color. Chemical formula: $C_{20}H_{26}N_4O_6S_2$, λ_{max} =599 nm, and the chemical structure as appear in Fig. 1.

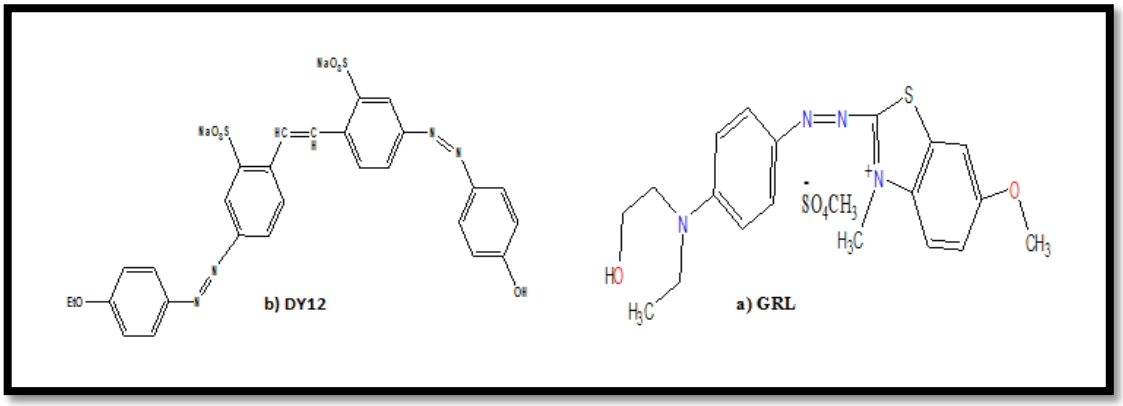

Fig. 1. Chemical structure of dyes: a) maxillon blue GRL; b) direct yellow (DY12; Alrobayi *et al.* 2017).

Direct yellow (DY12)

The Direct yellow (DY12) is a direct dye. The solution is yellow in color. Chemical formula: $C_{30}H_{26}N_4Na_2O_8S_2$, and the chemical structure is shown in Fig. 1. λ_{max} = 403 nm. DY12, a diazo dye usually utilized as a dye cotton and too in the leather and paper industry, due to its stability towards together alkali and acidic solutions being an ethylated product.

Determination of calibration curves of GRL

The calibration curve of several concentrations of GRL and DY12, were prepared in serial dilutions (10-100 mg L^{-1}). Abs. was measured at the λ max for dye and plotted against values t the conc. of GRL and DY12 (Fig. 2).

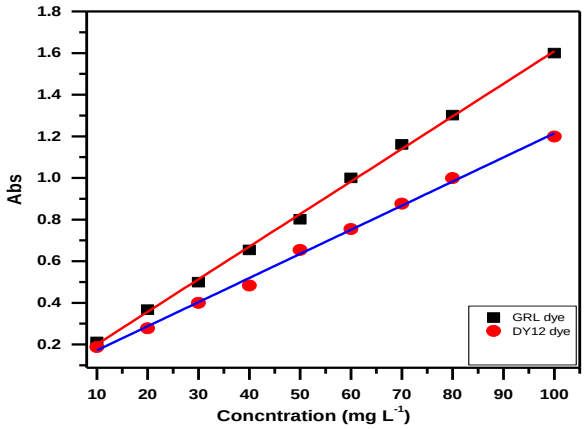

Fig. 2. Calibration curve of maxillon blue (GRL) and direct yellow 12 (DY 12).

Table 1. Statistics result of calibration for several conc. of maxillon blue and direct yellow 12).

Parameters	Proposed Method for GRL	Proposed Method for DY12
λmax (nm)	599	403
Beer's law limit (mgL^{-1})	10-100	10-100
Regression equation	(Y = m X + C) 0.0156X+ 0.04397	(Y = m X + C) 0.0166X+ 0.0555
Slope (m)	0.0156	0.0166
Intercept (C)	0.04397	0.0555
(R^2)	0.9985	0.9972
Color	Blue	Yellow

Prepare (NTADClP/P (AA-co-AM) composites

The compound was prepared via the free radical method. The method included dissolving 0.1 g polymer in 2 mL absolute ethanol, then adding 2 mL acrylic acid in the presence of nitrogen gas for a period and recording from dissolving 0.02 (MBA) for 60 seconds, followed by adding the clamping agent solution, such as acrylic amide KPS (g) in 2 mL distilled water, and also adding the starting solution of potassium persulfate. After taking 0.02 grams and dissolving it in 2 mL distilled water gradually to the reaction mixture and transfer this mixture to a water bath at a temperature of 55 °C for 30 minutes to complete the polymerization process, the polymer is extracted and cut into small pieces followed by washing with distilled water with constant stirring for 6 h, and replacing the water each half an hour until the non-reactive materials are disposed of. Then, it is dry in the oven at 55 °C and ground to become ready to conduct experiments. The following diagram shows the method of preparation.

Adsorption equilibrium experiments

Stock solutions of GRL and DY 12 (1g 1000 mL^{-1}) were prepared, and the series of required concentrations were made via dilutions by DW. The primary tested concentrations of two dyes were 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 mg L^{-1}. The effect of primary pH solution on the two dyes adsorption via NTADCIP/P (AA-co-AM) composite was studied at a concentration of 50 mg L^{-1}. Thereafter, the pH was adjusted utilizing NaOH and HCl at 0.1 N solutions. The influence of weight dosage was studied via agitating in several amounts (0.001, 0.005, 0.008, 0.01, 0.03, 0.05, 0.08 and 0.1 g) of NTADCIP/P (AA-co-AM) composite, at 25 °C of 50 mg L^{-1}. Effect of Ionic Strength of NaCl, KCl, and $CaCO_3$ was studied through the weight of salt (0.05 g).

Shaker water bath adsorption experiment was undertaken as follows: 100 mL of 50 mg L^{-1} for two dyes was mixed thoroughly with 0.03 g NTADCIP/P(AA-co-AM) composite at pH = 6.13 in GRL and 6.02 in DY12 at room temperature for 1 hour by a shaker water bath assuming that the equilibrium has reached. Finally, the sample was centrifuged and then analyzed for residual dye concentration by UV Visible spectrophotometer 1650 spectrophotometer, Japan) at 599 nm and 403 for GRL and DY12 respectively. The amount of dye uptake by NTADCIP/P (AA-co-AM) composite in each flask was calculated utilizing the Eq. 1 (Yang *et al*. 2018):

$$Qe = \frac{\text{Co}-\text{Ce}}{W} * V \quad \textbf{(1)}$$

where (q_e) is quantity of dyes adsorbed via NTADCIP/P(AA-co-AM) composite at equilibrium, C_o and C_e are the primary and equilibrium conc. dyes at the same order, V is the volume dyes (L), and W is the weight dosage (g). The dyes percentage (%) was calculated using the Eq. 2:

$$E\% = \frac{\text{Co}-\text{Ce}}{\text{Co}} * 100 \quad \textbf{(2)}$$

where: C_0 and C_e are primary and equilibrium conc. of GRL and DY12, at the same order.

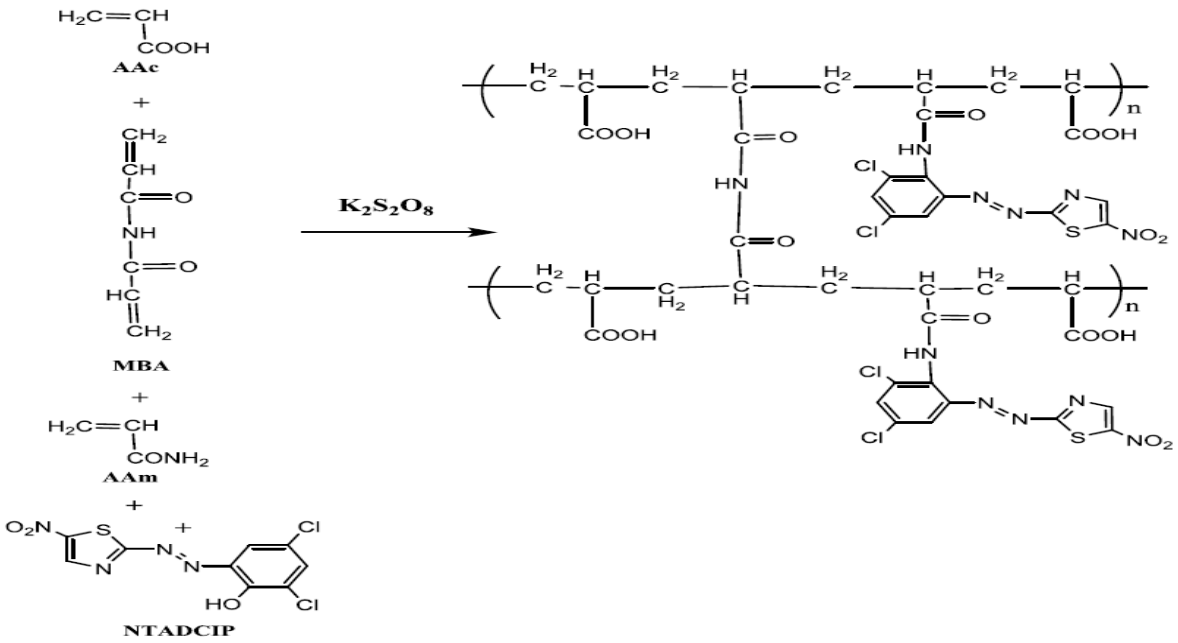

Fig. 3. Preparation of (NTADClP/Poly (AA-co-A.M) composites.

RESULTS AND DISCUSSION

FESEM

The surface (NTADClP/P (AA-co-AM) composites were studied by FE-SEM technology before and after adsorption of GRL and DY12 to know the nature and porosity of the surface, as well as to know the homogeneity between components, shape, size of the particles and the nature of their distribution (Aljeboree2016). The dyes have appeared on the surface, and this is evidence of the adsorption process, as shown in Figs. 4a and 4b.

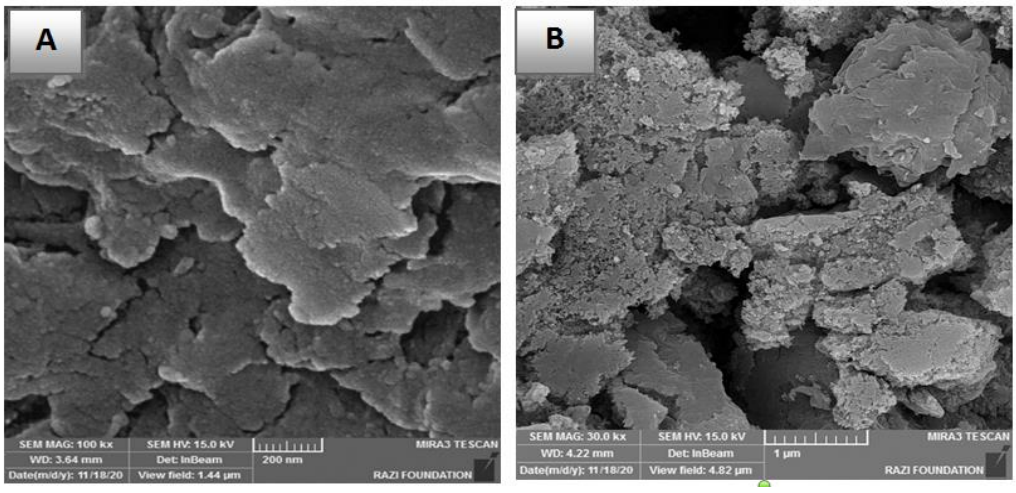

Fig. 4a. Image of FE-SEM after GRL adsorption at different enlarging powers.

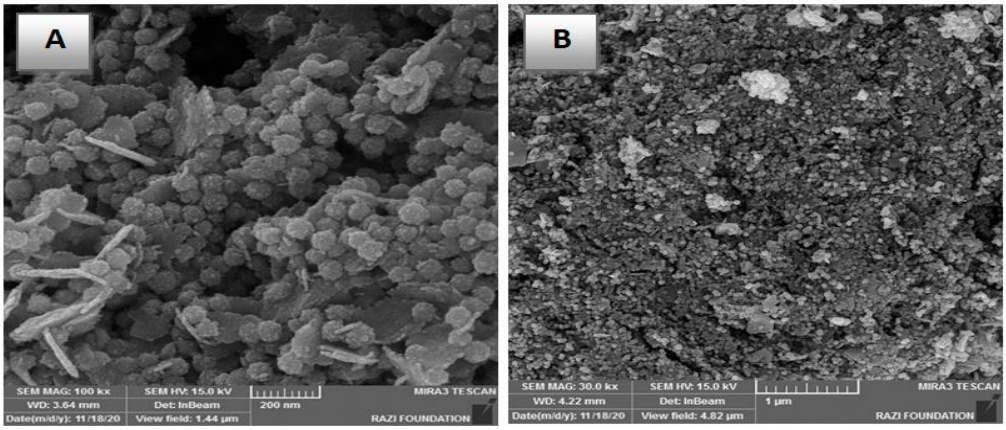

Fig. 4b. Image of FE-SEM after DY12 adsorption at different enlarging powers

Thermogravimetric Analysis (TGA)

A study of the (NTADClP/Poly (AA-co-AM) composites was conducted by employing the technology of TGA. The study revealed that the (NTADClP/P (AA-co-AM) composites are very stable (Fig. 4) at the same temperature body of the human, the (NTADClP/P(AA-co-AM) composites too having to be stable to 319.24 ºC. However, heating the composite in the range of 250-3710 ºC causes 72.35% mass loss, which can be due to the pyrolysis. Thereafter, the (NTADClP/P(AA-co-AM) loses 16.46% of its weight within the range thermal of 374-620 ºC due to the thermal breakage of the interlocking polymeric chains of the (NTADClP/Poly(AA-co-AM).

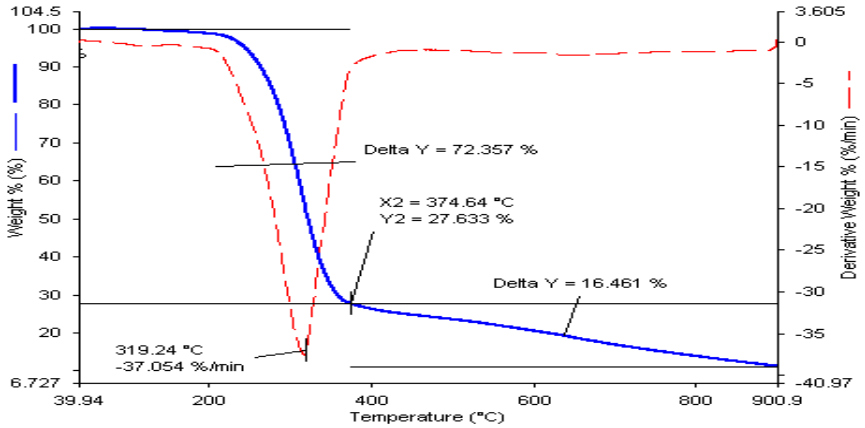

Fig. 5. Analysis TGA of (NTADClP/P (AA-co-AM) composite.

Atomic Force Microscopy (AFM)

A.F.M was utilized to estimation the surface topography of the composite. Fig. 6 appear image three-dimensional of NTADCIP and NTADCIP/P (AA-co-AM). Table 2 also depicts the mean roughness (Ra) and the (Rq) of the surface exhibiting that the composite is greater than the coarse coefficients of the gel NTADCIP confirming that the ketchup may be added. It clearly affected the topography of the surface as the reagents particles spread homogeneous along the surface of the hyaline, which caused great roughness of the surface (Xiong *et al.* 2012).

Table 2. The statistical roughness coefficients of prepared samples

Amplitude Factors	NTADCIP	NTADCIP/P(AA-co-AM)
(Ra)	2.32	7.99
(Rq)	11.10	9.9
surface Skewness (Rsk)	-0.16	-0.0492
surface Kurtosis (Rku)	1.98	2.68

FT-IR characterization for adsorbent/adsorbate

The NTADCIP/P (A.A-co-AM) composite was characterized via spectroscopy IR. Spectra of FT-IR were collected in the mid FTIR-IR from 4000 -400 cm^{-1} with a resolution of 1 cm^{-1}. The spectra of IR of NTADCIP/P(AA-co-AM) composite before and after GRL dye adsorption have appeared in Fig. 6. The FT-IR

pattern appears reduced in the intensity of bands next to the adsorption, such that there is a difference real among NTADCIP/P(AA-co-AM) composite before and after the interaction by GRL dye, suggesting that a PHY-sorption phenomenon happens as a data of attractive forces among the NTADCIP/Poly (A.A-co-AM) surface and GRL dye under investigation (Alkaim & Alqaraguly 2013).

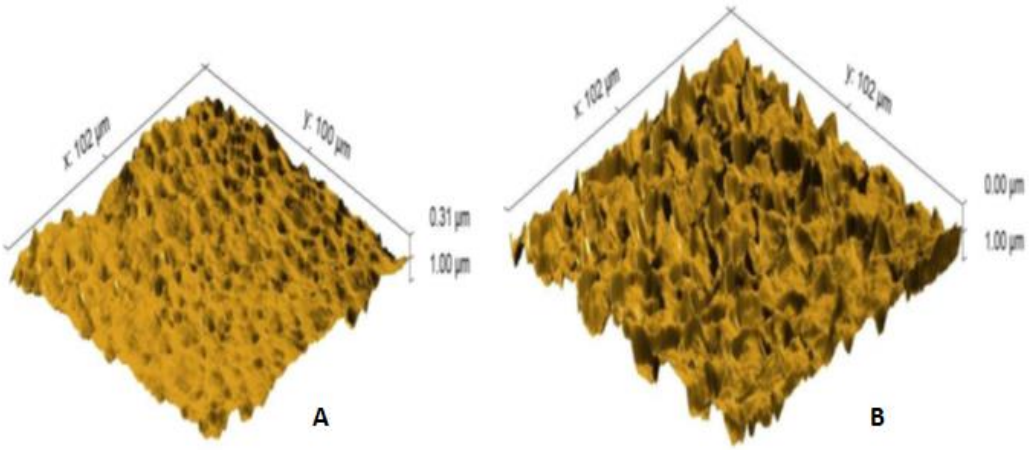

Fig. 6. 3D images of AFM NTADCIP (A), and NTADCIP/P (AA-co-AM) composite (B).

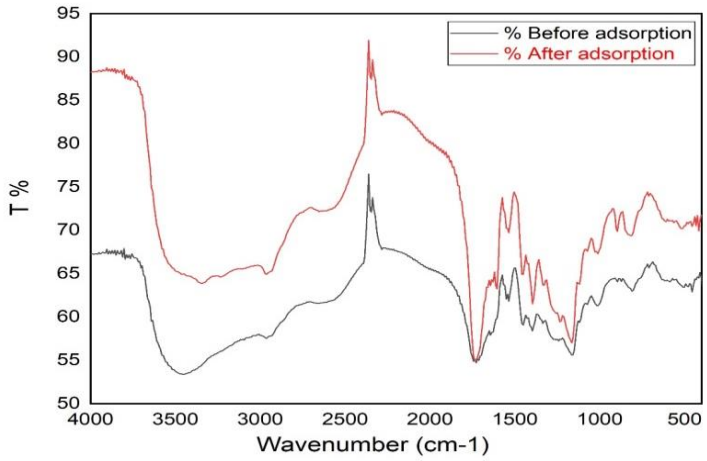

Fig. 7. FTIR spectra of NTADCIP/Poly (AA-co-AM) composite (a) before and (b) after adsorption of GRL.

Effect of contact time

Equilibrium time is one of the significant factors for the assessment of the practical application of the adsorption method. The experimental trial results in adsorption of GRL and DY12 on the surfaces of the NTADCIP/P(AA-co-AM) composite with contact time. The equilibrium data are shown in Figs. 8 revealing that the adsorption efficiency rises by the rise in Equilibrium time to reach the equilibrium because via elevating the time of adsorption, the active sites of surfaces absorbent will saturate, indicating reaching an apparent equilibrium (Del Águila-Carrasco *et al.* 2019) so the adsorption efficiency will decrease. The equilibrium is established within 60 min for all kinds.

Effect of pH

Usually, solution pH of the reaction environment is one of the utmost significant parameters that should be considered in the investigation of adsorption methods by the effect on ionization of contaminants and the charged surface of adsorbent materials. In order to estimation the effects of pH on the percent removal of GRL and DY12 from aqueous solutions via adsorbent, 50 mg L^{-1} for GRL and DY12 solutions were prepared in 100 mL-containers, and the pH was adjusted among 3 (Fig. 7). The E (%) of GRL and DY12 from aqueous solution via NTADCIP/P (AA-co-AM) composite is highly pH-dependent. The optimum solution pH was found to be 6.5 (Abdulsahib *et al.* 2020).

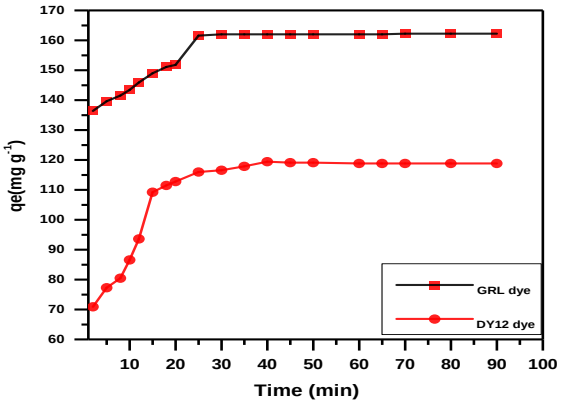

Fig. 8. Effect of change time on adsorption of two dyes (GRL) and (DY12) at 25°C, 1 hour and 0.03 g of dyes as 50 mg L^{-1} in 100 mL water.

Effect of ionic strength

It was noticed that GRL adsorption increase without adding any salt and give higher and best percentage removal, but when added, the amount of NaCl and KCl decreases the removal. It was also noticed that $CaCO_3$ has a negligent effect on adsorption (Karim & Jasim 2019). However, in the case of DY12, the effect of NaCl is larger than KCl, and $CaCO_3$. This gives a conclusion that there is less electrostatic repulsion influence of KCl and CaCO3 than that produced by NaCl on NTADCIP/P (AA-co-AM) composite adsorption (Abdulsahib *et al.* 2020; Fig. 9).

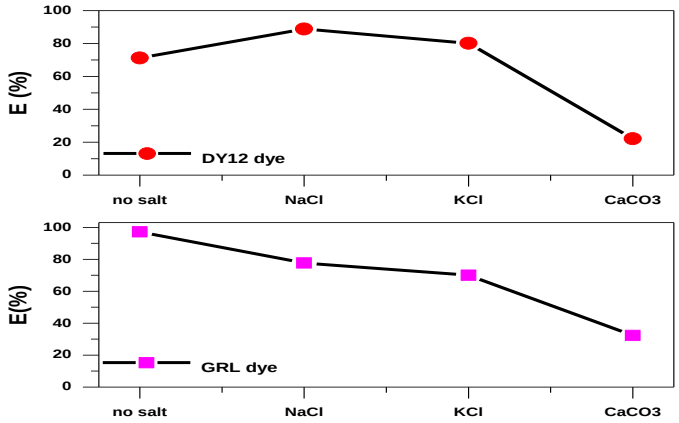

Fig. 9. Effects of the salt adsorption for GRL and DY12 onto surface NTADCIP/P (AA-co-AM) composite at 25°C, 1 hour, 0.03 g of dye as 50 mg L^{-1} in 100 mL water.

The adsorption efficiency of GRL was minimum at pH 3 (48.4053 mg g^{-1}), rise up to pH 10, reached maximum (164.6911 mg g^{-1}). However, DY12 was minimum at pH 10 (51.4051 mg g^{-1}), rise up to pH 3, reached best (151.023 mg g^{-1}) over the primary pH (Fig. 10). At top pH, the surface might get positively charged, which improves the negatively charged GRL anion through electrostatic forces of attraction.

Effect of Adsorbent Dose

Effect of the mass NTADCIP/P (AA-co-AM) composite was needful in arranging to the minutest probable quantity, which appears the maximum adsorption stoichiometric. The different quantities from 0.001 to 0.1 g /100 mL of NTADCIP/P(AA-co-AM) composite (Mahde *et al.* 2018; Figs. 11a and 11b). It is apparent that via raising the mass of adsorbent, the amount of adsorbed GRL and DY12 dyes rises, while adsorption efficiency and the quantity adsorbed per unit weight decrease. It is readily understood that the number of obtainable sites of the adsorption rises via increasing the mass dosage. Thus, data in a rise in the quantity of the adsorbed two dyes. The decrease in efficiency of the adsorption via the rise in the mass of adsorbent is fundamental because active sites stay unsaturated through the reaction of the adsorption whereas the number of the active sites obtainable for adsorption site rises via the increased weight dosage (Aqel *et al.* 2012).

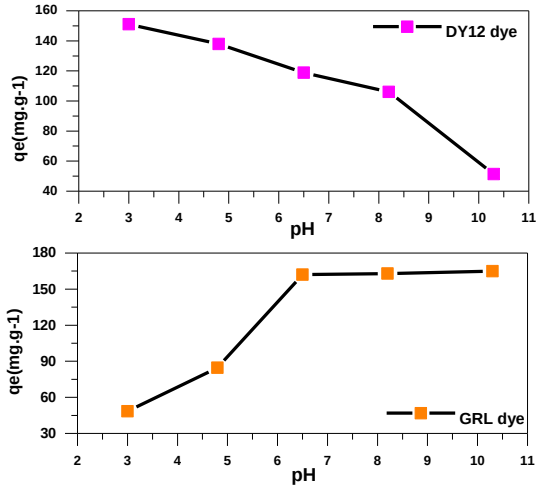

Fig. 10. Effect of solution pH on adsorption of GRL and DY12 on surface NTADCIP/Poly (AA-co-AM) composite at 25°C, 1 hour, and 0.03 g as 50 mg L^{-1} in 100 mL water.

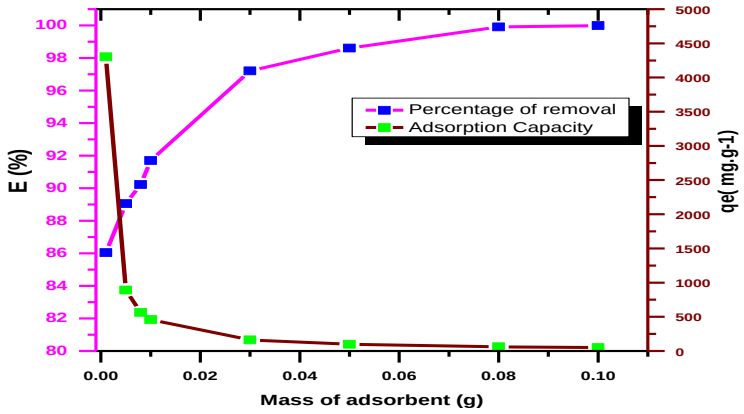

Fig. 11a. Effect of mass adsorbent on adsorption of GRL: (25°C, 1 hour, and pH 6.13).

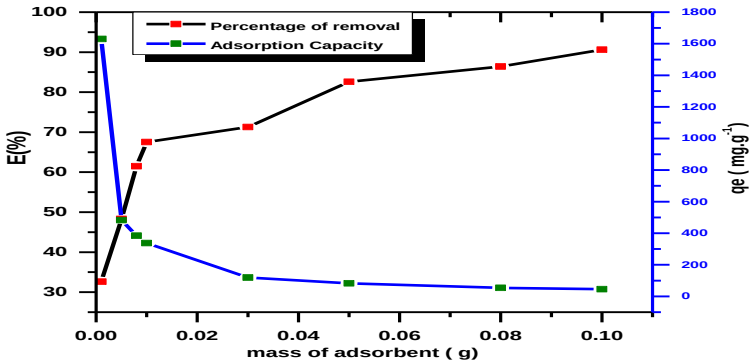

Fig. 11b. Effect of mass adsorbent on adsorption of DY12 (25 °C, 1 hour, and pH 6.13).

Effect of initial dyes concentration

Fig. 10 depicts the plots concerning to the amounts of (E %) GRL and DY12 removal and dyes adsorbed (Qe) against primary concentration C_0 at various experiential conditions. The residual unoccupied active sites were difficult to be taken due to steric barrier among GRL and DY12 adsorbed on the surface of NTADCIP/P (AA-co-AM) composite and the phase solution (Rahdy & Jasim 2019). The production depended on raised leading power of concentration. Gradient with the raise in the primary concern. Fig. 12 depicts that the qe/mg g^{-1} value will enhance by the elevation in primary factor concentration, since the impedance to the agreement of GRL and DY12

from the solution decrease by the high different factor's concentration. In addition, the increased Co amount would mains to a raised rate of adsorption due to the arising in the driving force (Aljeboree *et al.* 2019).

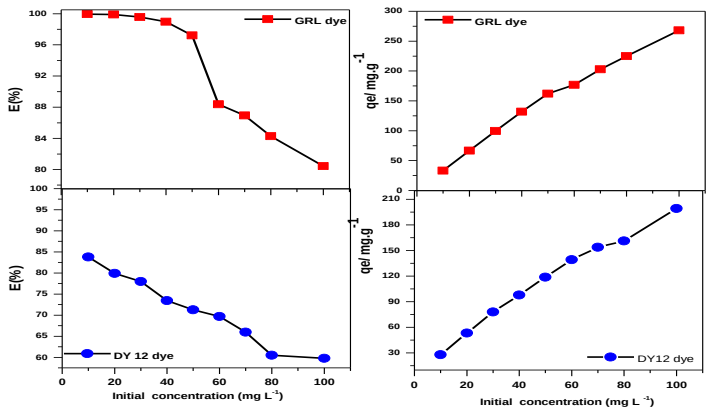

Fig. 12. Effects of primary concentration of GRL and DY12 on the percentage removal and quantity of adsorbed GRL and DY12 (T = 25°C, equilibrium time 1 hour, and pH 6.13 and PH 6.02).

Effect of temperature solution

Impact temperature solution (10 - 35 ºC) on uptake for GRL and DY12 using NTADCIP/P(A.A-co-AM) composite is illustrated in Fig. 13. The result concerning to the adsorption capacity and E (%) of GRL and DY12 on the adsorbent NTADCIP/Poly (A.A-co-AM) composite depend on the temp. It is clear that the removal (E%) and adsorption capacity rised by elevating the solution temp. In the case of GRL, the percentage removal (E%) of 44.045%, 77.896%, 97.207% and 99.987% lead to the adsorption capacity of (74.67, 118.115 [1], 162.012 and 166.646 mg g^{-1}), while in the case of DY 12, E (%) of 13.810%, 31.051%, 71.265% and 78.931% lead to adsorption efficiency of (23.017 [1], 51.752 [1], 118.775 and 131.551 mg g^{-1}) at 10-35 ºC respectively (Ji *et al.* 2010; Arya & Philip 2016).

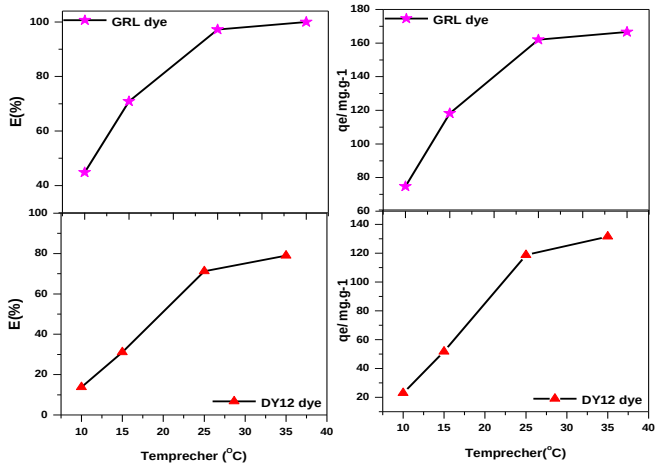

Fig. 13. Effects of temperature on the E (%) and quantity of adsorbed GRL and DY12 on NTADCIP/P (AA-co-AM composite (T = 25°C, 1 hour, and quantity of adsorbent = 0.03g).

Isotherm Freundlich

The isotherm Freundlich is known as Eq. 3 (Ho *et al.* 2002):

$$q_e = K_f C_e^{1/n} (3)$$

where qe is the adsorbent quantity adsorbed / unit weight at equilibrium (mol. g^{-1}), (mg g^{-1}), Ce: the adsorbate equilibrium scam solution next adsorption (mg L^{-1}), (mol. L^{-1}), Kf: capacity factor (L g^{-1}), 1/n heterogeneity factor,

n is a deviation measured of the deviation from adsorption linearity. Its value indicates a nonlinearity unit among adsorption and concentration solution as follows: adsorption route is chemical if the value under the unity adsorption or it is linear if the n value equal to unity, finally the favorable physical route when the value is above the unity (Kumar *et al.* 2010). A plot of q_e vs Ce (Figs. 14 and 15), where the values of K_F and 1/n are obtained from the intercept and slope of the linear regressions (Table 2).

Isotherm Langmuir

The Langmuer model is utmost generally utilized of adsorption of pollutants for liquid solution (Atyaa *et al.* 2019). Another equation was derived by the way of Langmuer on the base of a definite case of the nature adsorption process from solution. The adsorption Langmuir isotherm in Eq. 4 (Langmuir 1918):

$$q_e = \frac{q_0 K_L C_e}{1 + K_L C_e} \quad (\mathbf{4})$$

where q_e is adsorbed amount per unit weight at equilibrium (mg g^{-1}), C_e: conc. of adsorbent equilibrium in solution after adsorption (mg L^{-1}), q_o: the constant of Empirical Langmuir represents maximum qe (mg g^{-1}), K_L: Empirical Langmuer constant (L mg^{-1}). The data of this isotherm have appeared in Figs. 12 and 13, and the Langmuir constants are illustrated in Table 3.

Table 3. Freundlich and Langmuir isotherm models factors GRL and DY12 adsorbed of NTADCIP/P (AA-co-AM) composite at 25 ºC.

Isotherm models	Parameters	NTADCIP/Poly(AA-co-AM) composite Maxilon blue (GRL) dye	NTADCIP/Poly(AA-co-AM) composite Direct yellow DY 12
Langmuir	qm (mg g^{-1})	64.20058 ± 5.210241	269.989 ±18.107
	KL(L mg^{-1})	5.8645 ± 3.14983	0.05683 ±0.00836
	R^2	0.79386	0.98222
Freundlich	K_F	41.822 ± 2.080307	27.8324 ± 2.8429
	1/n	0.19366 ± 0.020802	0.5308 ± 0.03209
	R^2	0.9759	0.98655

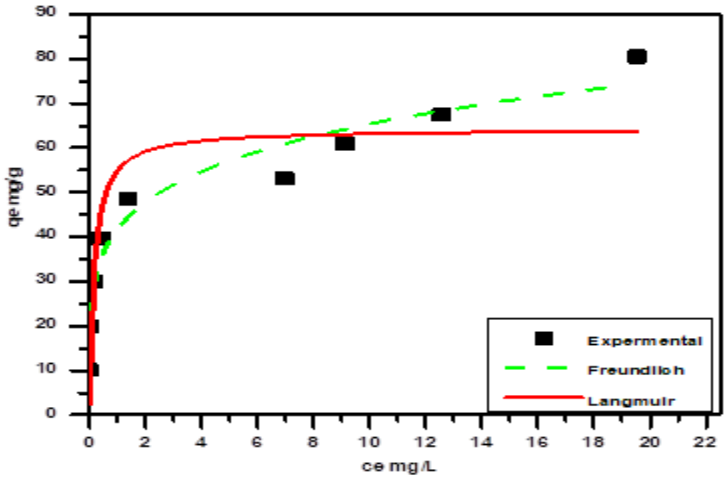

Fig. 14. Several adsorption models nonlinear fit of adsorption of GRL on NTADCIP/P (AA-co-AM) composite primary concentration = 50 mg L^{-1}, Temperature = 25 °C, and weight of surface 0.03 g).

CONCLUSION

Depending on the experimental data of the current study, we can conclude that an NTADCIP/P (AA-co-AM) composite can be utilized for GRL and DY12 adsorption from solution, and the Freundlich model can describe GRL and DY12 adsorption adequately. The two dyes E% depend on the temperature and solution pH. The adsorption optimum equilibrium for equilibrium time to be achieved is reach to be 1 hour. It is essentially due to the saturation of the active site that does not allow further adsorption to take place.

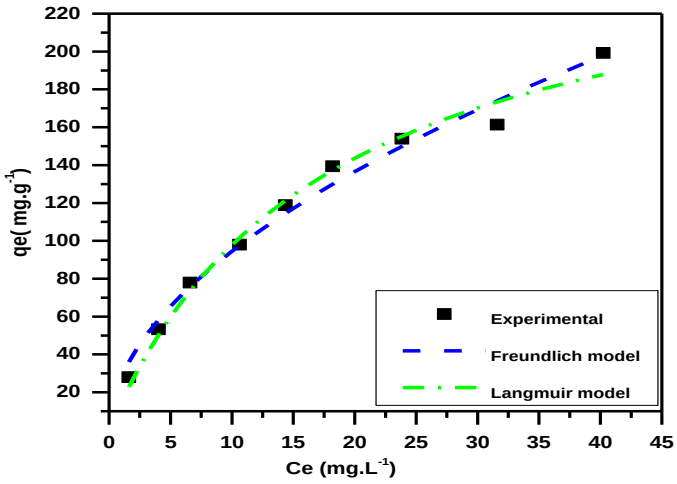

Fig. 15. Several adsorption models nonlinear fit of adsorption of DY12 on NTADCIP/P (AA-co-AM) composite. Primary concentration = 50 mg L^{-1}, Temperature = 25°C, and weight of surface 0.03 g.

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