



Congo Red Removal From Aqueous Solutions by Ion Exchanger Amberlite LA-2

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<p>ARTICLE HISTORY</p> <p>Received: 22 August 2022</p> <p>Accepted: 07 February 2023</p> <p>Keywords: Congo red dye; liquid-liquid extraction; Amberlite LA-2; distribution coefficients; loading capacity; extraction efficiency.</p>	<p>Abstract: Amberlite LA-2 was used as an ion exchanger to remove Congo red dye from aqueous solution. The effect of the initial concentration of dye and the initial concentration of Amberlite LA-2 on the efficiency of dye removal from the aqueous phase to the organic phase at different contact times, temperatures and pH values was studied. this research aims to study the potential of using Amberlite LA-2 as an extracting material for Congo red dye removal. UV-visible Spectrophotometer were used to assay the dye concentration in the aqueous solution before and after removal. Distribution coefficients (K_D), loading capacity (Z), and extraction efficiency ($E\%$) were computed using experimental data. The maximum extraction efficiency of 99.69 % was obtained with a loading capacity of 384.366 mol/kg. The obtained results also indicated that the removal of dye increases with the increase in the concentration of Amberlite LA-2. The best removal was obtained in a neutral medium (pH = 7), a temperature of 20 °C. A mechanism of extraction by Amberlite LA-2 was also proposed.</p>
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إزالة صبغة الكونغو الأحمر من المحاليل المائية بواسطة المبادل الأيوني Amberlite LA-2

<p>الكلمات المفتاحية :</p> <p>صبغة الكونغو الحمراء؛ استخلاص سائل-سائل؛ Amberlite LA-2؛ معاملات التوزيع؛ معاملات التحميل؛ كفاءة الاستخلاص.</p>	<p>المستخلص : في هذه الدراسة، تم استخدام Amberlite LA-2 مبدلاً أيونياً لإزالة صبغة الكونغو الأحمر من المحلول المائي، وذلك بدراسة تأثير التركيز الابتدائي للصبغة، والتركيز الابتدائي لمادة Amberlite LA-2 على كفاءة إزالة الصبغة من الطور المائي إلى الطور العضوي في أزمنة، ودرجات حرارة، وقيم أس هيدروجيني مختلفة. استخدمت أطيف امتصاص الأشعة المرئية، وفوق البنفسجية UV-VIS لتقييم تركيز الصبغة في الوسط المائي قبل الإزالة وبعدها. تم استخدام النتائج التجريبية للاستخلاص لحساب معاملات التوزيع (K_D)، وسعة التحميل (Z) وكفاءة الاستخلاص ($E\%$). تم الحصول على أقصى كفاءة استخلاص بنسبة 99.69 %، وقدرة تحميل 384.366 مول/كجم⁻¹. كما أشارت النتائج المتحصل عليها إلى أن إزالة صبغة الكونغو الأحمر تزداد مع زيادة تركيز مادة Amberlite LA-2. حيث أمكن الحصول على أفضل إزالة في وسط متعادل (pH = 7)، ودرجة حرارة 20 °C. كما اقترحت آلية لعمل المبادل الأيوني Amberlite LA-2 مزيلاً لصبغة الكونغو الأحمر من الوسط المائي.</p>
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INTRODUCTION

Environmental pollution is a serious problem facing humans in modern life. These problems have emerged as a result of human technological, industrial and civilized progress. Thus, the problem of aqueous solution pollu-

tion has received great attention from researchers (Hassaan et al., 2017). Organic matter constitutes an important part of the components of industrial wastewater. Organic pollutants have high risk in terms of their long-term impact, as some of them cause cancerous diseases (Padhi, 2012). Dyes are among the

many organic materials polluting water sources. They are used in the textile industries, in printing, in photographic colors, and as additives in the oil industries, as well as in other wide fields (Faust & Aly, 2013). Approximately 10-15 % of these dyes are lost as waste in the water, which is thrown into water sources or soil, causing major problems for plants, animals, and humans (Asthana, 2001; Faust & Aly, 2013). Therefore, finding ways to remove different pollutants is very important to meet the increasing demand for water with increasing population density. Wastewater-containing dyes can be treated by physical, chemical, and biological methods, such as coagulation, fusion, oxidation, membrane filtration, ion exchange, and photocatalytic degradation (Al-Sakkaf et al., 2020). Liquid-liquid extraction is one of the most important of these techniques due to its high efficiency in this field and the simplicity of the technology used for this purpose compared to other methods (Al-Abbasi et al., 2010; Uday et al., 2016).

Congo Red (CR), is an anionic azodye (Hassaan et al., 2017; Padhi, 2012). Congo red dye with the formula $C_{32}H_{22}N_6Na_2O_6S_2$ (Figure 1) is considered a very dangerous dye for various living organisms, and exposure to this dye may lead to human cancer. This dye is sensitive to the change in the pH value, as its red color is stable in the pH range (5-10), and its colour changes to blue in the acidic medium ($pH < 5$) (Kloetzer et al., 2019). Congo red dye has a strong absorption band of max 498 nm in the visible spectrum, giving it a red color. Synthetic dyes such as Congo Red are difficult to decompose due to their chemical, thermal and optical stability. Congo red dye as sodium salt form has a high solubility in water, which makes the process of treating or removing this dye from polluted water very difficult (Saygılı, 2015). The use of water-insoluble acid-base and liquid reagents as liquid ion exchangers was first proposed by (Smith et al., 1937). Since then, there has been growing interest in the use of bases and acids with high molecular weights

as extracts (Werner, 1974). The most important properties of a useful liquid exchanger are low solubility, high selectivity, ability to regenerate in cyclic processes, stability to common reagents under normal conditions, and low cost (Khopkar, 2007; Kunin & Winger, 1962).

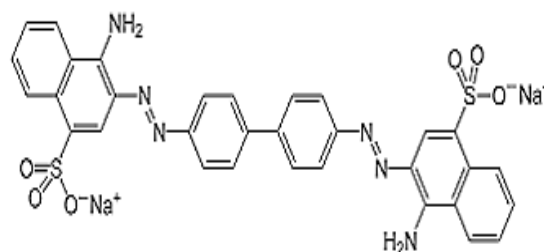


Figure: (1). Chemical structure of Congo red dye

Amberlite LA-2 is chemically named *N-Lauryl (trialkylmethyl)*, and it is a high molecular weight liquid that is soluble in most common nonpolar organic solvents. Hydrometallurgy is one of the main applications of amberlite LA-2 in mineralogy. It has been mainly used in the recovery and purification of uranium from low-concentration samples, as well as the separation of iron, cobalt, and nickel in hydrochloric acid solutions (Lee & Wang, 1995). Moreover, Amberlite LA-2 was used to remove and purify many organic acids, including L(+) tartaric acid (Asçı & İnci, 2009), formic acid (Uslu et al., 2009), propionic acid (Kloetzer et al., 2019), picric acid (Uslu, 2016), malic acid (Uslu & Kırbaşlar, 2010), fumaric acid (Khopkar, 2007; Kloetzer et al., 2019), glycolic acid (Asçı & İnci, 2009), and levulinic acid & malic acid (Uslu & Kırbaşlar, 2013). Removal of Congo Red dye from aqueous solution using Amberlite IRA-400 was also investigated (Sinha et al., 2018). In this study, a maximum loading efficiency (99.99%) of congo red dye was obtained at the conditions pH 4.5, Temp. 303 K, contact time 180 min (Sinha et al., 2018).

In a recently published paper by our group, Amberlite LA-2 was used to remove the Methyl Orange Dye, the maximum extraction

value of about (97.65%) was obtained (Alabbasi & Abdulljoad, 2022; Alabbasi et al., 2022; Alabbasi & Suliman, 2022). Thus, this research aims to study the potential of using Amberlite LA-2 as an extracting material for Congo red dye removal. Optimization of the ideal conditions for the extraction process as the initial dye concentration, Amberlite LA-2 concentration, contact time, pH, and temperature. Moreover, the mechanics of the extraction process of Congo red dye with an anion exchanger in aqueous solutions were studied.

MATERIALS AND METHODS

Chemicals and Instrumentations: The chemicals used are all of a high degree of purity: Congo Red dye (T-Baker Lab chemicals, 100 %), Amberlite LA-2 (BDH, 99 %), dichloromethane (Chemsolute, 98.5 %), sodium hydroxide (Fluka, 98 %), hydrochloric acid (Chemsolute, 37 %) and sodium carbonate (Fluka, 99.8 %). pH device (Thermo) and VU-Visible Spectrophotometer (Shimadzu UV2450 spectrometer.).

Preparation of Standard Solutions: Stock standard Congo red dye solutions (100 ppm) were prepared by dissolving about 10 mg in 100 mL of deionized water. Amberlite LA-2 solution with different concentrations of 0.011, 0.022, and 0.04 M was prepared by taking volumes of 0.25, 0.5, and 1 mL, respectively, in a 50 mL stander flask, and the volume was completed with dichloromethane solvent and placed in an airtight bottle before use.

Standard calibration curve of congo red: The best wavelength at which the absorption of the Congo red dye solution occurred was determined by measuring the absorbance of the Congo red dye solution with a concentration of 20 ppm in a range of wavelengths (400-800 nm), where the highest absorption of the dye solution was at 498 nm. To determine the standard calibration curve of Congo red, standard solutions of 0, 20, 40, 60, and 80 ppm were prepared to form a series of five

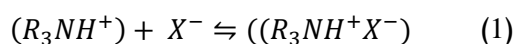
flasks. The absorbance was measured for all flasks at the maximum wavelength of the dye (498 nm) (Vogel, 2000). The relationship between the dye concentration in each flask and the corresponding absorbance was drawn, so a straight line was obtained, and its slope equation was used to measure the concentration of the dye in the aqueous phase at the end of each extraction experiment. The equation for a straight line was $y = 0.0303 x - 0.0007$

Extraction experiments: In tightly capped bottles, two equal volumes (10 mL) of the previously prepared organic and aqueous layers are taken in a 1:1 ratio. The organic layer (Org) consists of an Amberlite LA-2 solution dissolved in dichloromethane (DCM). The aqueous layer (aq.) consists of aqueous solutions of Congo red dye. The bottles with the mixture were placed in a shaking water bath (200 rpm). The bottles were shaken for the define time, and then the aqueous layer was separated from the organic layer using a separating funnel. Immediately, 3 mL was withdrawn from the aqueous layer to measure the absorbance at the λ_{max} of Congo Red (498 nm). To obtain the best conditions for the removal process, the effect of the initial concentration of Congo red dye (20, 40, 60, 80 ppm) and the initial concentration of Amberlite LA-2 (0.044, 0.022, 0.011 M), as well as the effect of temperature (20, 30, 40 °C) and time (15, 30, 45, 60 min), were studied. Moreover, the effect of pH on the extraction process was studied at different pH values (1,3,5,9,11,13). The distribution coefficient (K_D) and percentage of the extracted dye (%E) were calculated.

RESULTS AND DISCUSSION

Congo red was extracted from an aqueous solution using LA-2 in DCM as an extractant. In this section, the effects of the initial dye concentration, equilibration time, temperature effect, and LA-2 concentration were studied, and the results are presented in Table 1.

Theoretical Approach: Liquid–liquid extraction is a physical separation method that aims to remove or separate a certain substance from another substance that has been mixed (Blumberg, 1988) It is defined as the separation of a compound from a mixture utilizing a liquid solvent. This method has proven to be very useful in recovering compounds from mixtures of materials, and it is a method that depends on the solubility of materials in two immiscible solutions, water and organic nonpolar solvents (Blumberg, 1988). The principle of extraction depends on the factor of the distribution of materials between two immiscible liquids (Uslu & Kırbaşlar, 2013). The extraction of Congo red dye with Amberlite LA-2 can be described by the following reaction:



The distribution coefficient (K_D) is the ratio between the concentration of a substance distributed between two immiscible solvents (Seader et al., 2016). The distribution coefficient gives information about the distribution of the dye in the aqueous and organic phases. If the distribution coefficient values K_D are greater than one, the dye concentration in the organic phase is greater than the dye concentration in the aqueous phase, and if the distribution coefficient is less than one, the dye concentration in the organic phase is less than its concentration in the aqueous phase.

$$K_D = \frac{[Dye]_{org}}{[Dye]_{aq}} \quad (1)$$

where $[Dye]_{org}$ is the dye concentration in the organic layer and $[Dye]_{aq}$ is the dye concentration in the aqueous layer. The removal percentage, (E%), is the percentage of solute that moves from one phase to another at equilibrium. It is expressed as the removal efficiency or removal percentage (E%) as in the following equation:

$$E\% = \left(1 - \frac{[Dye]_{aq}}{[Dye]_{0,aq}}\right) \times 100 \quad (2)$$

where $[Dye]_{0,aq}$ is the initial concentration of dye in the aqueous layer. Loading capacity, (Z) indicates the ratio of the concentration of dye in the organic phase at equilibrium to the initial concentration of Amberlite LA-2. The loading capacity (Z) can be expressed by the equation below:

$$Z = \frac{[Dye]_{org}}{[R_2NH]_0} \quad (3)$$

where $[R_2NH]_0$ is the primary concentration of benzoyl thiourea in the organic phase. The larger this number is, the better the material is for the dye extraction process.

Extraction of the congo red: Anionic congo red was extracted from an aqueous solution using Amberlite LA-2 in DCM as an extractant. In this section, the effects of the initial dye concentration, equilibration time, temperature effect, and LA-2 concentration were studied, and the results are presented in Table 1. From Table (1), it is noticed that all K_D values were greater than one, which indicates the preference of the dye for the organic phase over the aqueous phase. It has been noted that the Z value increases as the concentration of Amberlite LA-2 decreases.

To verify the role of the organic solvent on the extraction process, the extraction processes in the absence of Amberlite AL-2 was carried out. Among the results of the experiments shown in the table (1) it was noted that, the 12.528, 31.418, 27.446 and 21.667 ppm of the red Congo dye was extracted from the aqueous phase to the organic phase when the initial dye concentration were 20, 40, 60 and 80 ppm, respectively. This might be explained by the partial solubility of Congo red dyes in the DCM solvent.

From the results of the experiments shown in Table (1) in the absence of Amberlite LA-2, the E % decreases with an increase in the initial dye concentration, as it decreases from 62.64 % to 27.087 % when the initial dye

concentration is increased from 20 to 80 ppm. This is attributed to the saturation of the sol-

vent with the increase in dye concentration.

Table:(1). Characteristic performance data obtained for Congo Red dye by Amberlite LA-2

C _{LA-2} (M)	C _{CR} (ppm)	t (min)	C _{aq}	C _{org}	K _D	Z	E%
0	20	30	7.471947	12.528	1.676678	/	62.64026
	40		28.58218	31.418	1.0992	/	52.363
	60		32.55446	27.446	0.843066	/	45.74257
	80		58.3303	21.667	0.371508	/	27.08746
0.0443	20	15	2.448	17.552	7.1699	0.477359	87.76
		30	1.795	18.205	10.142	0.118	92.356
		45	1.696	18.304	10.79245	0.497811	97.36
		60	3.1155	16.88	5.4181	0.459082	97.14
	40	15	3.0577	36.94	12.081	1.004651	91.025
		30	2.455	37.545	15.2933	1.021105	93.86
		45	1.55	38.449	24.8058	1.045691	98
		60	1.99	59.007	29.6518	1.604803	97.92
	60	15	1.583	58.42	36.9046	1.588838	91.52
		30	1.2	58.799	48.9992	1.599146	96.12
		45	1.069	58.93	55.1263	1.602709	98.22
		60	1.135	38.865	34.2335	1.057005	98.33
	80	15	2.2885	77.71	33.9567	2.113465	84.4
		30	1.66	78.34	47.19277	2.130599	96.68
		45	1.333	78.67	59.017	2.139574	97.162
		60	1.333	78.67	59.017	2.139574	98.33
0.022	20	15	1.246	18.754	15.0514	2.054107	93.77
		30	2.4	17.6	7.33333	1.927711	94.92
		45	1.736	18.264	10.538	2.000438	95.78
		60	0.712	19.288	27.0899	2.112596	97
	40	15	2.032	37.968	18.685	4.158598	88
		30	2.288	37.712	16.482	4.130559	94.28
		45	2.116	37.884	17.9036	4.149398	95.85
		60	1.74	38.26	21.988	4.19058	96.87
	60	15	2.532	57.468	22.6967	6.294414	91.32
		30	2.49	57.51	23.0964	6.299014	94.71
		45	2.532	57.468	22.6967	6.294414	95.78
		60	1.818	58.182	32.0033	6.372618	96.85
	80	15	2.4	77.6	32.333	8.499455	96.44
		30	2.504	77.496	30.9489	8.488059	95.56
		45	2.52	77.48	30.746	8.486309	96.97
		60	1.632	78.368	48.0196	8.583573	97.96
0.011	20	15	2.59	17.4	6.718	7.62322	87.05
		30	2.82	17.18	6.0922	7.526835	93.12
		45	2.62	17.38	6.6336	7.614458	96.073
		60	2.521	17.48	6.9334	7.658269	99.08
	40	15	2.75	37.25	13.5454	16.31983	85.9
		30	2.75	37.25	13.5454	16.31983	93.12
		45	2.59	37.4	14.44	16.38555	95.63
		60	2.455	37.5	15.275	16.42935	96.97
	60	15	2.36	57.6	24.407	25.23549	86.9
		30	2.62	57.38	21.9008	25.1391	93.5
		45	0.363	59.64	164.298	26.12925	99.395
		60	0.277	59.72	215.5957	26.16429	99.69
	80	15	0.7393	79.26	107.2095	34.72508	87.39
		30	2.422	77.58	31.769	33.98905	93.9
		45	0.244	79.756	326.869	34.94239	99.54
		60	0.54	79.46	147.148	34.81271	99.3

Table:(2).Effect of temperature on the extraction of Congo red dye by AmberliteLA-2

C_{LA-2} (M)	C_{CR} (ppm)	T (°C)	C_{aq}	C_{org}	K_D	Z	E%
0.022	40	20	2.288	37.712	16.4825	2.065277	94.28
		25	2.43	37.57	15.4609	2.0575	93.93
		30	0.352	39.648	112.6363	2.1713	99.12
		40	2.884	37.116	12.8696	2.032636	92.79
	60	20	2.49	57.51	23.0963	3.149505	95.85
		25	2.349	57.651	24.5427	3.157227	96.08
		30	1.35	58.65	43.4444	3.211936	97.75
		40	3.908	56.09	14.3526	3.071741	93.48

Table:(3).The results obtained when extracting Congo red dye by AmberliteLA-2 under the pH effect.

C_{LA-2} (M)	C_{CR} (ppm)	t(min)	pH	C_{aq}	C_{org}	K_D	Z	E%
0.022	60ppm	30	1.99	4.238	55.762	13.159	2.534636	92.937
			3.8	5.063	54.937	10.851	2.497136	91.562
			5	5.3036	54.696	10.313	2.486182	91.16
			7	2.49	57.51	23.0964	2.614091	94.71
			9.2	1.003	58.997	58.803	2.681682	98.328
			11.23	2.653	57.347	21.612	2.606682	95.578

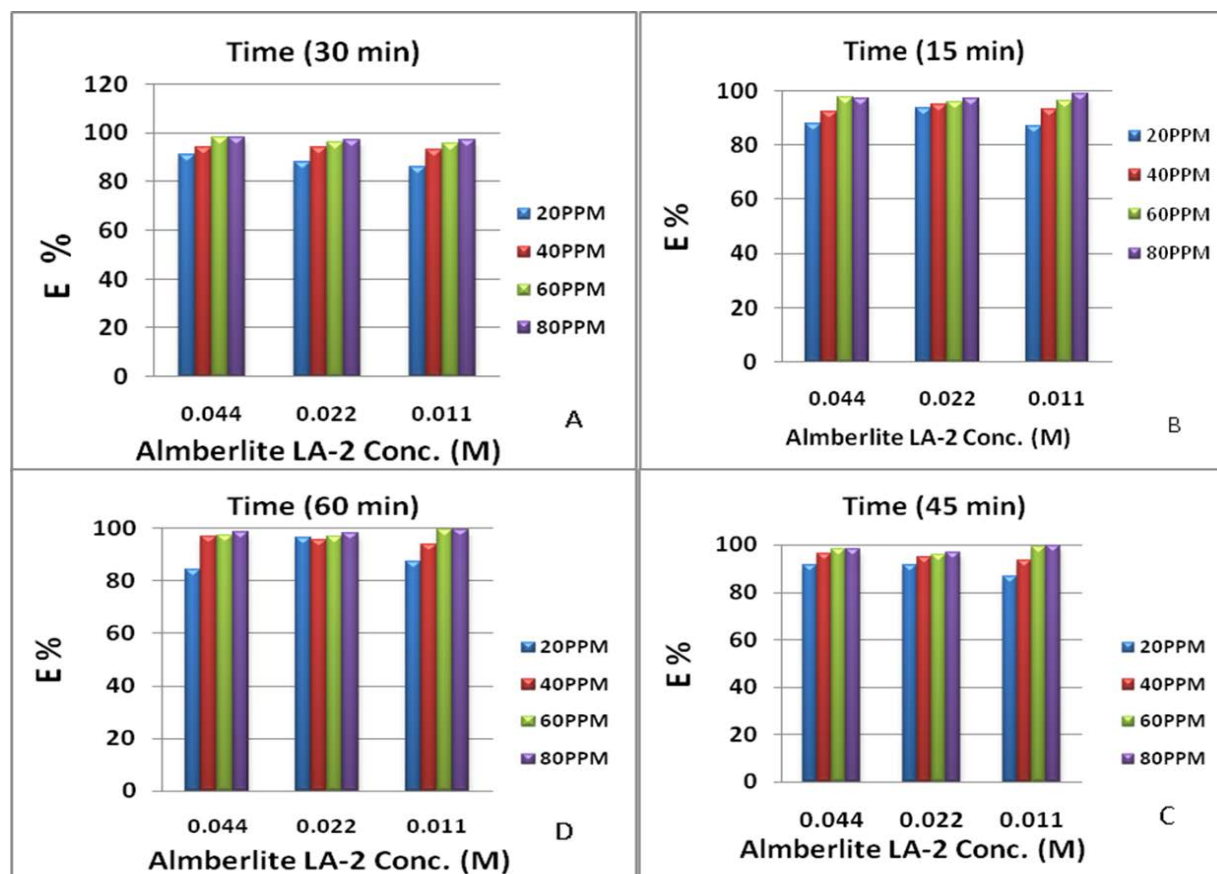


Figure (2). Effect of initial concentration of CR dye, Amberlite LA-2, and contact time on the extraction process

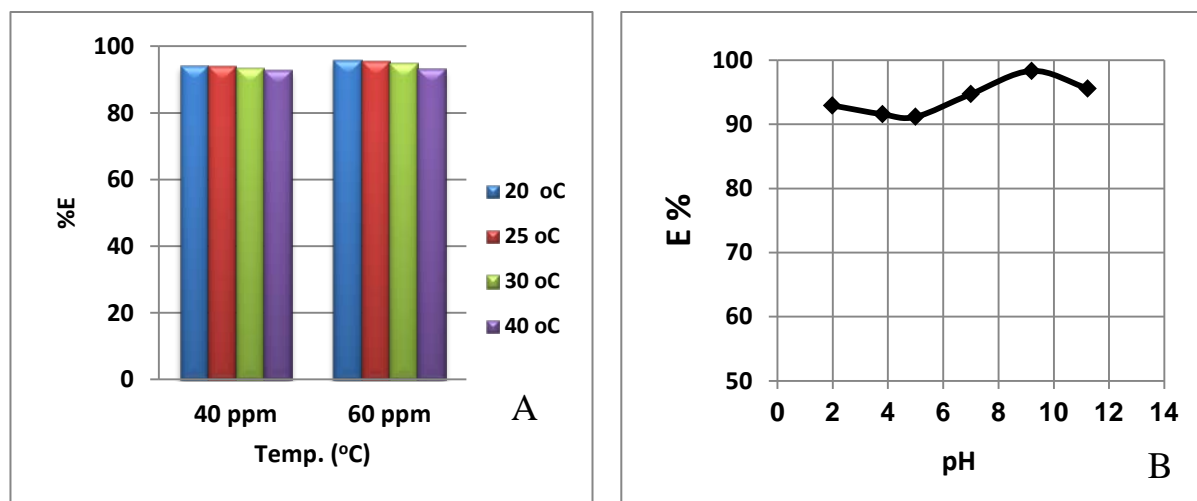


Figure (3). Effect A) temperature B) pH on the extraction congo red dye by Amberlite LA-2

Figures (2 A, B, C, and D) show that E % is an insignificant change when the Amberlite concentration increases from 0.011 to 0.044 M at a constant day concentration and specific time as an example at a day concentration of 80 ppm, the E% is 98.33 %, 97.96 % and 99.3 % for 0.044 M, 0.022 M, and 0.011 M. However, Figure (2) shows that E % increases by increasing the concentration of the initial dye. The results showed that the dye extracted from the aqueous phase to the organic phase increases with time, and the quantitative transfer of the dye takes place after 60 min, this was in good agreements with our previous study on removing of methyl orange by the Amberlite ion-exchanger (Alabbasi et al., 2022).

Figure (3) shows that E% decreases with increasing temperature, which is attributed to the physical bonds between the dye molecules and Amberlite being weakened and its transfer to the organic layer being reduced. Moreover, the possibility of solvent evaporation as it has a low boiling point of 45 °C.

Dye extraction mechanism: In this study, significant extraction of Congo red by Amberlite LA-2 was observed, and the extraction mechanism is suggested as in our previous study (Alabbasi et al., 2022) as follows: First, CR is dissolved in the aqueous phase,

and then the sodium sulfonate groups ($R-SO_3Na$) are ionized as in equation (2):



Second, the interaction of the dye with Amberlite LA-2 is induced by the electrostatic attraction between the Amberlite LA-2 cations ($R_2NH_2^+$) and the negative dye anions, as shown in equation (3):

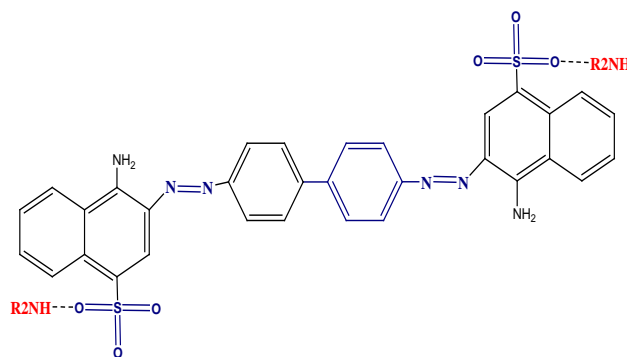


Figure (4). The complex formed by the electrostatic attraction between the positive Amberlite ion and the negative dye ion

CONCLUSION

In this paper, Congo red dye was successfully extracted from an aqueous solution by liquid-liquid extraction using Amberlite LA-2. The values of the distribution coefficients were also determined and these values were greater than 1 which indicates that the pigment distri-

bution in the organic layer is higher than in the aqueous layer. The values of distribution modulus and loading capacity increase with increasing initial dye concentration and decrease with increasing concentration of the substance (Amberlite LA-2). It was noticed that by increasing the value of the acidity function pH, the value of the distribution coefficient and the loading capacity increased, and therefore the extraction efficiency up to 7 pH started decreasing with the increase of the baseline. Increasing the temperature leads to a decrease in the distribution coefficient and extraction efficiency values.

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Author contributions: Dr. A. Al-Abbasi is responsible for the original manuscript's writing, along with its supervision, conceptualization, methodology, writing, review, and editing. Asma Ehmedi is a student who is in charge of the experiments. Khadija Ahmida is responsible for Reviewing and Editing.

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