



## Removal of Methyl Orange Dye from Aqueous Solutions using Amberlite LA-2 as an Extractant

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### Keywords:

Methyl orange  
Liquid-liquid extraction  
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Distribution coefficients  
Oading capacities  
Extraction efficiency.

### ABSTRACT

The removal of methyl orange dye, MO, has been accomplished through the use of liquid-liquid extraction from aqueous solutions using Amberlite LA-2 as an extractant. UV-Vis spectroscopy was used to determine the concentration of dye in an aqueous solution. The effect of pH was studied at a specific concentration of dye and Amberlite at contact time. The effect of the initial dye concentration (20-80 ppm), and Amberlite concentration (0.011-0.044 M) on the removal rates of the dye at different contact times (15- 60 min), and temperatures of 20-40 OC have been investigated. The experimental results of extraction were used to calculate the distribution coefficient (KD), loading capacity (Z), and extraction efficiency (E%). The removal of the dye decreases with the increase in the amount of Amberlite. Moreover, there was an increase in the E % with increasing temperature. The best pH was determined to be 5.5. The maximum extraction value of about (97.65%) was obtained.

### إزالة صبغة الميثيل البرتقالية من المحاليل المائية باستخدام Amberlite LA-2 كمستخلص

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### الكلمات المفتاحية:

ميثيل برتقالي  
استخلاص السائل-السائل  
المبادلات الأيونية  
أمبرلايت LA-2  
معاملات التوزيع  
كفاءة الاستخلاص

### المخلص

تمت إزالة صبغة الميثيل البرتقالي، MO، من المحاليل المائية من خلال استخدام طريقة الاستخلاص سائل-سائل واستخدام الأمبرلايت (Amberlite LA-2) كمستخلص. تم استخدام التحليل الطيفي للأشعة فوق البنفسجية (UV-Vis) لتقدير تركيز الصبغة في محلول مائي. تم دراسة تأثير تركيز الصبغة الابتدائي (20-80 جزء في المليون) وتركيز الأمبرلايت (0.011 - 0.044 مول / لتر) على معدلات إزالة الصبغة في ازمنا التلامس المختلفة (15- 60 دقيقة) ودرجات حرارة 20-40 درجة مئوية. أيضا، تمت دراسة تأثير الأس الهيدروجيني عند تركيز محدد للصبغة والأمبرلايت في زمن التلامس ساعة واحدة. تم استخدام النتائج التجريبية للاستخلاص لحساب معامل التوزيع (KD) وسعة التحميل (Z) وكفاءة الاستخلاص (E%). اشارت النتائج الى تناقص إزالة الصبغة مع زيادة كمية الأمبرلايت. علاوة على ذلك، كانت هناك زيادة في E% مع زيادة درجة الحرارة. تم تحديد أفضل أس هيدروجيني ليكون  $pH = 5.5$ . تم الحصول على أقصى قيمة استخلاص (97.65%).

### 1.Introduction

Water pollution is the entry of harmful substances, directly or indirectly, into rivers, seas, oceans, lakes, and underground, biological, or physical water stores [19]. Although industries make many products that are beneficial to mankind, they also produce different wastes in different forms that are responsible for increased risks and pollution. Most of the waste products are discharged into soil and water bodies, which ultimately constitutes a serious warning to humanity and the ecosystem [5, 6].

Dyes can be defined as substances that provide colour when applied or added to another substance through a process of permanent or at least

temporary change [8, 26]. These materials with great colouring ability are widely used in the textile, pharmaceutical, food, cosmetic, plastic, photographic, and paper industries [6, 14]. There are more than 10,000 dyes used in the textile industry alone, approximately 70 % of which are synthetic azo dyes [8, 10]. The urgent need to reduce the number of residual pigments in wastewater has become a major concern in recent years. It is, therefore, essential to remove the dye from wastewater or treat it in such a way to minimize the damage to the environment and also to decolourize the water [1]. Wastewater containing coloured substances can be treated in various ways such as physical and chemical

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methods. Physical treatment methods use the principle of physical forces to separate pigments from polluted water. Physical approaches include various methods such as coagulation, flocculation, sedimentation, adsorption, filtration, reverse osmosis, ultrafiltration, nanofiltration, and ion exchange [2, 21]. Chemical methods by adding chemicals or by chemical reactions (such as reduction, oxidation, and Complex metric methods [1, 2, 20, 21].

In recent years, much attention has been focused on separation techniques such as solvent extraction or liquid-liquid extraction (LLE) and liquid membrane [4, 17, 18]. LLE is based on the principle that a solute can distribute itself in a certain ratio between immiscible solvents, and the extraction process depends on its mass transfer rate [4, 17, 18]. Liquid-liquid extraction of methylene blue [16], methyl violet [15], astacryl blue and astacryl golden yellow were also reported [13], but no previous study was found for methyl orange dye.

Methyl orange is an azo dye readily soluble in water to give intense orange colour. The presence of azo dyes in ecosystems leads to poisoning and carcinogenicity [11]. These negative effects are compounded by its adverse impact on human health, including respiratory problems, diarrhea, vomiting and nausea [11, 12]. The molecular structure of this compound is given in Figure 1. Methyl orange shows red colour in acidic medium and yellow colour in basic medium. Thus, it is usually used in titration for acids. This dye is generally non-biodegradable and is not removed efficiently by the traditional treatment methods such as adsorption on activated carbon, coagulation, sedimentation, chemical oxidation, and biological digestion. Studies indicate that the reductive cleavage of the azo bond by azo reductase enzyme in the liver produces aromatic amines and can even lead to intestinal cancer [3, 7].

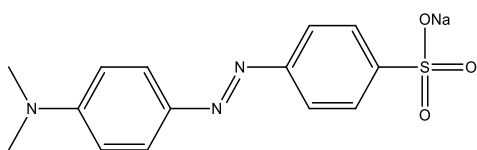


Fig.1: Chemical Structure of Methyl Orange (MO)

This research attempts to find a simple and rapid technique of liquid-liquid extraction (LLE) to remove MO dye from its aqueous solution by using Amberlite LA-2 as an extractant, since it give good result as ionic exchanger [22-24]. In addition, identifying the appropriate extraction parameters, such as the initial dye concentration, the initial Amberlite LA-2 concentration, pH, contact time, and temperature

## 2. Materials and Instrumentation

### 2.1 Chemicals and Instrumentations

The chemicals used are all of a high degree of purity and are produced by well-known companies. Amberlite LA-2 (BDH Laboratory Reagents, 99%), DCM (Pure Chemistry, 98.5 %), hydrochloric acid (CHEMSOLUTE, 37%), methyl orange (Riedel-De Haegen T, 99.9%), sodium hydroxide (Analytical Reagent, 98%), sodium carbonate (Riedel-De Haegen T., 99.8%), these chemicals were used without further purification. The instruments used in this study: UV-Vis spectrophotometer (Shimadzu UV2450 spectrometer), pH device (Thermo),

### 2.2 Preparation of Standard Solutions

Standard MO dye solutions were prepared by dissolving the required amount of dye in a known volume of distilled deionized water. Amberlite LA-2 solution with different concentrations of 0.011, 0.022, and 0.044 M was prepared by taking volumes of 0.25, 0.5, and 1mL, respectively in a 50 mL stander flask, and the volume was completed with dichloromethane solvent and placed in an airtight bottle before use.

### 2.3 Standard Calibration Curve of Methyl Orange

The best wavelength at which the absorption of the MO dye solution occurred was determined by measuring the absorbance of the MO dye solution with a concentration of 20 ppm in a range of wavelengths (400-800nm), where the highest absorption of the dye solution was at 461 nm. To construct the standard calibration curve of MO, the 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 ( $\lambda_{max} = 461 \text{ nm}$ ) as shown in Figure 2. 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 will be used to measure the concentration of the dye in the aqueous phase at the end of each extraction experiment as nshown in Figure 3.

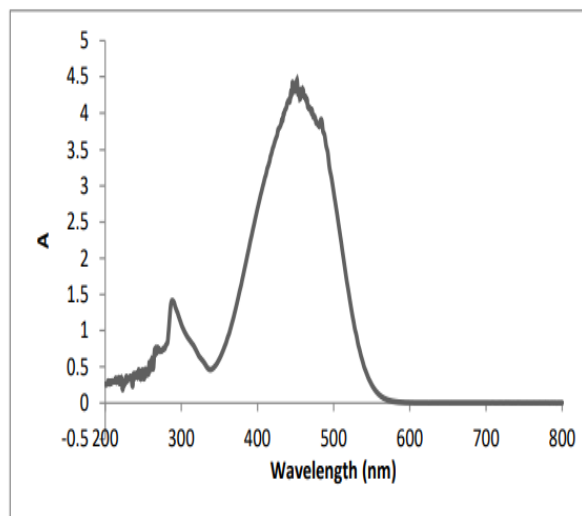


Fig. 2: UV-Vis spectra of methyl orange (MO) with  $\lambda_{max}$  (461 nm)

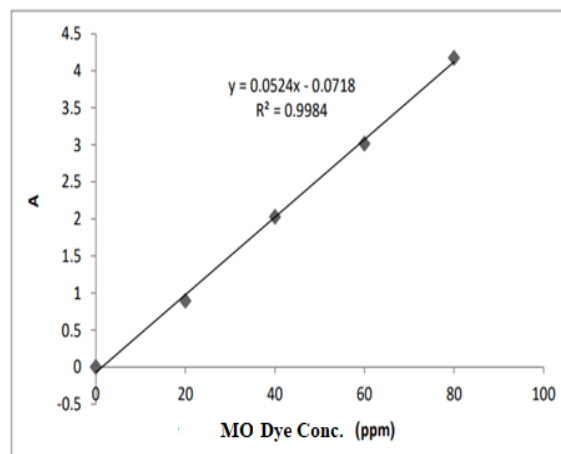


Fig. 3: Standard calibration curve of methyl orange

obtain the distribution ratio ( $K_D$ ), percentage of extracted dye ( $E\%$ ), and loading capacity ( $Z$ ) [13, 18].

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

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

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

where  $[dye]_{org}$  is the dye concentration in the organic phase (ppm),  $[dye]_{aq}$  is the dye concentration in the aqueous phase (ppm),  $[Dye]_{0,aq}$  is the initial dye concentration in the aqueous phase and  $[R_2NH]_0$  is the primary concentration of Amberlite LA-2 in the organic phase.

## 3. Result and Discussion

MO was extracted from an aqueous solution using Amberlite LA-2 in DCM as an extractant. In this section, the effects of initial dye concentration, equilibration time, pH, temperature, and Amberlite LA-2 concentration on removal process were studied and the results were presented in Tables 1- 3.

### 3.1 The Effect of Temperature

The effect of temperature on the extraction of MO dye was studied at two concentrations (40 and 60 ppm) at 20, 25, 30, and 40 °f and at

min 30 and a concentration of (0.022 M) and the obtained results were included in Table (2).

The E% increase of the extracted MO dye was observed with increasing temperature from 20 to 25 and then started to decrease when the temperature was raised to 30 and 40 °C as shown in Fig. (4). The increase in the percentage of dye removal is due to the fact that the extraction process is initially an activating process, so the percentage increases with increasing temperature [9, 25]. When the temperature rises from 25-40 degrees Celsius, the physical bonds that bind the dye molecules with Amberlite LA-2 are weakened, and their transfer to the organic layer is reduced. Moreover, probably decreased due to the evaporation of the solvent at high temperatures.

### 3.2 pH Effect

The effect of pH on the liquid/liquid extraction process was studied by adjusting the pH of the dye solution with a concentration of 60 ppm at different pH values ranging from (0.4 to 11), the data were listed in Table 3, and the best pH for the extraction process was determined. The results indicated that Amberlite has a good performance towards MO in a wide range of pH values. The E% increased from 77 to 97% when the pH value of the MO solutions increased from 0.5 to 5.5. Then E% reverted and decreased at pH values above 7. Since the dye showed strong adsorption at pH=5.5 (pH of pure MO), this value was selected as the optimal pH for the subsequent experiments.

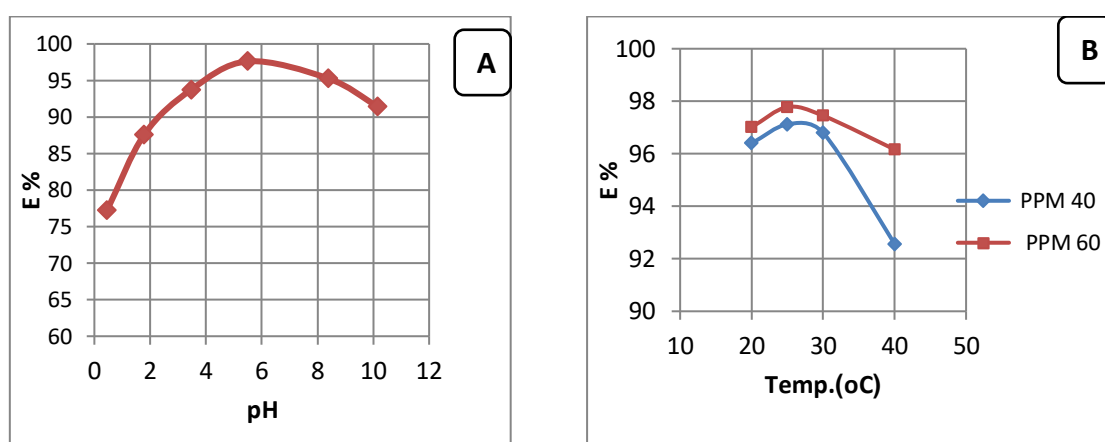
**Table 1: presents a summary of the extraction of MO dye using by Amberlite LA-2**

C <sub>LA-2</sub> (M)	C <sub>MO</sub> (ppm)	t (min)	C <sub>aq</sub>	C <sub>org</sub>	K <sub>D</sub>	Z	E%		
0	20	30	4.798	15.202	3.168	24	4.798		
	40		16.504	23.496	0.769	58.74	16.5		
	60		31.84	28.16	0.884	46.93	31.84		
	80		65.775	14.255	0.216	17.781	65.775		
	0.044	20	15	2.0576	17.9428	8.7217	89.7137	0.405	
			30	2.3435	17.6565	7.5342	88.2824	0.3986	
			45	2.0763	17.924	8.6324	89.6183	0.4074	
			60	2.5153	17.485	6.9514	87.423	0.3947	
		0.022	40	15	2.3626	37.637	15.931	94.0935	0.8496
				30	2.458	37.542	15.273	93.855	0.8475
				45	1.9046	38.095	20.002	95.239	0.8599
				60	2.0763	37.924	18.265	94.809	0.8561
0.011			60	15	2.8588	57.141	19.988	95.2354	1.2899
				30	1.9237	58.076	30.191	96.794	1.3199
				45	1.7901	58.21	32.518	97.017	1.3140
				60	1.8855	58.115	30.822	96.858	1.3118
	0.011		80	15	2.9351	77.065	26.256	96.331	1.7396
				30	1.771	78.229	44.172	97.786	1.7659
				45	1.5229	78.477	51.531	98.096	1.7715
				60	2.3435	77.657	33.137	97.071	1.7530
		0.011	20	15	2.6958	17.304	6.4190	86.521	0.7812
				30	1.3464	18.654	13.854	93.268	0.8422
				45	1.0795	18.921	17.527	94.603	0.8542
				60	1.0943	18.906	17.276	94.528	0.8535
0.011			40	15	1.5016	38.498	25.638	96.3979	1.7381
				30	1.4408	38.559	26.7615	96.4359	1.7408
				45	1.4256	38.574	27.0575	96.0559	1.7416
				60	1.5776	38.422	24.354	97.6665	1.7346
	0.011		60	15	1.400	58.5999	41.8535	97.0118	2.6456
				30	1.7929	58.207	32.4647	97.6197	2.6279
				45	1.4282	58.572	41.0116	97.5729	2.6443
				60	1.4562	58.544	40.202	96.2576	2.6431
		0.011	80	15	2.9934	77.006	25.7205	97.5956	3.4766
				30	1.9235	78.077	40.5907	97.6574	3.5249
				45	1.8741	78.126	41.687	97.4186	3.5272
				60	2.0651	77.935	37.738	96.246	3.5185
0.011			20	15	1.4290	18.571	12.996	92.855	1.6768
				30	1.3861	18.614	13.4295	93.067	1.6807
				45	1.2429	18.757	15.092	93.786	1.6937
				60	1.1856	18.814	15.869	94.072	1.6988
	0.011		40	15	1.1063	38.8938	35.158	97.234	3.5068
				30	1.1500	38.845	33.782	97.125	3.5079
				45	1.1500	38.85	33.782	97.125	3.5079
				60	2.2446	37.7554	16.820	94.3885	3.4091
		0.011	60	15	1.3365	58.6635	43.8935	97.7725	5.2969
				30	1.5341	58.4659	38.1105	97.443	5.2791
				45	1.2853	58.7147	45.683	97.8579	5.3016
				60	1.7976	58.2024	32.378	97.004	5.2553
0.011			80	15	1.3504	78.6496	58.242	98.312	7.1016
				30	4.1813	75.8187	18.1328	94.774	6.8486
				45	3.2143	76.7858	23.889	95.982	6.9333

	60	4.4626	75.537	16.9267	94.4217	6.8205	
<b>Table 2: Effect of temperature on the extraction of MO dye by Amberlite</b>							
$C_{LA-2}$ (M)	$C_{MO}$ (ppm)	T (°C)	$C_{aq}$	$C_{org}$	$K_D$	Z	E%
0.022	40	20	20	1.4408	38.5592	26.7615	96.3979
		25	25	1.1521	38.8479	33.7194	97.1198
		30	30	1.2804	38.71961	30.24048	96.79902
	60	40	40	2.9739	57.02611	12.45039	92.56527
		20	20	1.7929	58.20706	32.46466	97.01177
		25	25	1.3334	58.66661	43.99811	97.77768
		30	30	1.5299	58.47014	38.21925	97.45023
		40	40	2.3021	57.69735	25.05693	96.16225

**Table 3: The results obtained when extracting MO dye by Amberlite 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	0.45	13.679	46.321	3.3863	2.1055	77.2
			1.79	7.439	52.561	7.066	2.3891	87.602
			3.47	3.756	56.244	14.976	2.5565	93.74
			5.5	1.7929	58.207	32.4647	2.6458	97.6197
			8.39	2.84	57.16	20.129	2.5982	95.267
			10.16	5.149	54.851	10.653	2.4932	91.419

**Fig. 4:** The effect of A) pH and B) temperature on extraction of MO dye

### 3.3. The Effect of Initial Dye Concentration

The dye solutions with different initial concentrations of MO in the range of 20–80 ppm was tested under constant conditions of Amberlite concentration, temperature, and pH, and the results were combined in Table (1).

The relationship between dye concentration and separation efficiency (E%) was drawn as shown in Figure (4A), which shows the results of the study at room temperature of 25 °C, pH = 5.5, and concentrations of 0.044, 0.022, and 0.011 molarity of Amberlite

Removal percentage began to increase but decreased at the highest concentration of the dye (see Table 1), and this can be explained that the organic layer reaching saturation with dye molecules before it was completely extracted [23, 24].

### 3.4 Effect of equilibration time

According to the results, the amount of dye that is removed from the aqueous phase to the organic phase increases over time, and after 60 minutes, there is a quantitative transfer of the methyl orange dye. Figure (4B) generally demonstrates that as time passes, the dye's concentration in the organic phase steadily rises. The maximum amount of dye was removed in 30 to 60 minutes at concentrations of 80 and 60 ppm.

### 3.5 Effect of Extractant (Amberlite La-2) Concentration

To verify the role of the organic solvent in the extraction process, an extraction experiment was conducted in the absence of Amberlite. From Figure (4C), It was found that 58.74% and 46.93% of the methyl orange dye was extracted from the aqueous phase in the absence of Amberlite, due to the partial solubility of methyl orange in the DCM solvent, which is due to the carbon chain in the acid. The solubility property increases by increasing the concentration of the dye in the organic layer, which indicates that there is an equilibrium process for the distribution of the dye between the two layers [24, 25]. This means that the solvent is a good carrier for a dye.

The effect of different concentrations of Amberlite AL-2 on the extraction of methyl orange was studied, where the varying concentrations of the extract were 0.044, 0.022, 0.11 M, at room temperature (25 °C) and pH = 5.5. In general, the results show that the extraction efficiency decreases with an increase in the concentration of the extractive material (Amberlite LA-2). For example, in the percentage of the extracted dye in Figure 4D, the extraction percentage increases with the increase in the concentration of the extractive material (Amberlite LA-2).

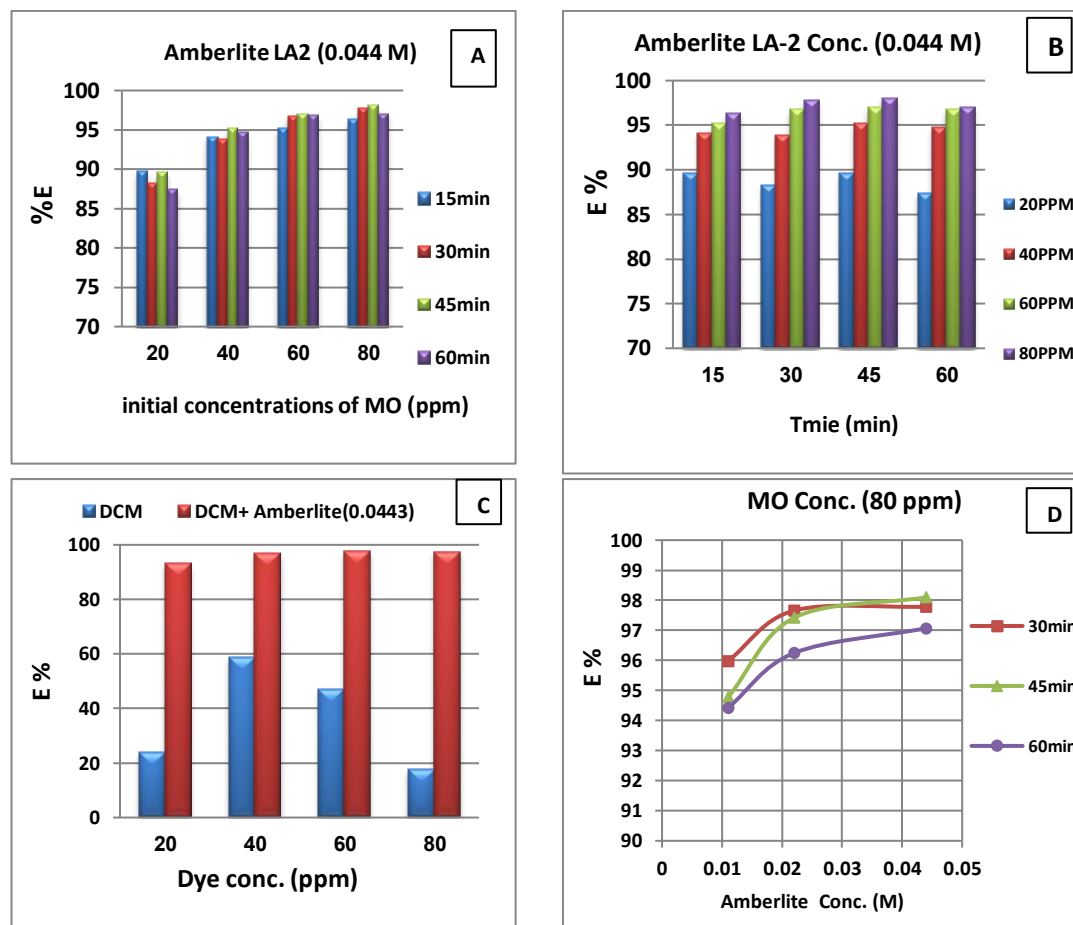
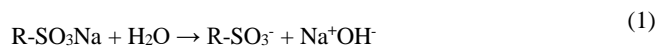


Fig. 4: The effects of: A) initial concentrations of MO (ppm); B) time; C) solvent; D) Amberlite LA-2 concentrations on extraction of MO dye

### 3.6 Extraction Process Mechanics

In this study, significant extraction of MO by secondary amine Amberlite-LA2 was observed. It is suggested that the extraction mechanism be as follows and as shown in Figure (5). First; MO is dissolved in the aqueous phase, and then the sodium sulfonate groups are ionized in MO dye ( $R-SO_3Na$ ) and converted into ions as in the following equation:



Secondly, the process of interaction of the dye with the amine substance is curtailed by the electrostatic attraction between the positive ions of the secondary amine Amberlite LA-2 ( $R_2NH_2^+$ ) and the negative anions of the dye as in the Acid base reaction:

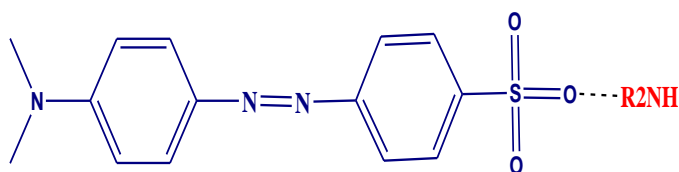


Fig. 5: The complex formed as a result of the electrostatic attraction between the Amberlite cation and the MO dye anion

### 4. Conclusion

After conducting the current study to remove methyl orange dye from water, we reach the following conclusions:

The methyl orange dye was successfully extracted from the aqueous solution by liquid/liquid extraction using amber lite dissolved in DCM as the extract. The values of the methyl orange distribution coefficients in the extraction process were greater than 1 indicating that the dye distribution in the organic layer is higher than in the aqueous layer. The value of the distribution modulus and the loading capacity increase with the increasing concentration of the initial dye and

decrease with the increase of amberlite LA-2. By increasing the pH function, the value of the distribution coefficient and the loading capacity increased, and thus the extraction efficiency up to pH 5.5 started decreasing with the increase in the basicity. With increasing temperature, the value of the distribution coefficient and the extraction efficiency decrease.

### 5. Recommendations

Based on the results obtained in this research, the following recommendations are suggested:

- Using amberlite in the chemistry lab to remove methyl orange dye after titration experiments in order to avoid contamination of sewage water with this dye.
- Removing various chemical pollutants such as dyes and heavy metals from the water before they are released into rivers, seas, or oceans, may help in obtaining a clean environment.
- Studying the possibility of using amberlite to remove other types of pollutants such as heavy metals, oil pollution, and pesticides. Use another solvent instead of dichloromethane.
- Searching for materials that can recover the dye from the organic medium.

### 5. Acknowledgments

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### 6. Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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