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Maleic Acid Separation from Aqueous Solutions Utilizing Amberlite LA-2

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ABSTRACT

This study investigates the potential uses of Amberlite LA-2 for the recovery of maleic acid from an aqueous solution. The effects of the initial concentration of maleic acid and Amberlite LA-2, pH, contact time, and temperature were determined and evaluated. The experimental results of extraction were used to calculate the distribution coefficient (K_D), and extraction efficiency (E%). The results show that the extraction efficiency increased with the increase in the initial concentration of maleic acid, where the percentage increased from 92.65% to 99.01% when the concentration of acid was increased from 0.01 to 0.075 N. The percentage of maleic acid extraction was also increased from 85.3 to 98% with the increase in the concentration of Amberlite LA-2 from 0.044 to 0.22 M. The acid extracted from the aqueous phase to the organic phase increases with time, and the quantitative transfer of maleic acid occurred after 60 minutes. At a concentration of 0.05 M, the percentage of extracted acid was observed to increase from 98.8% to 99.95% when temperature was increased from 25 to 40 ºC. The highest percentage of acid extraction was recorded at pH=3, which is (90.3%). The maximum loading modulus reached a value of 1.6877 at a concentration of 0.044 M of the secondary amine.

1 Introduction

One of the most important methods in the science of separation is the application of liquid-liquid extraction (Blumberg, 1988; Raynie, 2000). This application requires ion exchange materials that differ from conventional materials not only in their functional groups but also in their physical form (Nasef, 2012). The use of water-insoluble acid-base and liquid reagents as liquid ion exchangers was first proposed by Smith and Page (Smith & Page, 1948; Werner, 1974). Since then, there has been a growing interest in using bases and high molecular weight acids as extracts (Kunin & Winger, 1962; Werner, 1974). As far as the importance of 'liquid exchangers' is concerned, all high molecular weight amines have been named liquid anionic exchangers (Khopkar, 2007). The use of ion exchange resins in the adsorption process by ion exchange is more efficient(Lo, Baird, & Hanson, 1991; Nasef, 2012). For the separation of carboxylic acids, the resins used are mostly strong or weak base resins, which contain tertiary or quaternary amines as the ion exchange group (Dethe, Marathe, & Gaikar, 2006). The carboxylic acids are usually recovered from ion exchange resins by rinsing with sodium hydroxide and can be concentrated through evaporation and then hydrolysis to produce a pure acid for further processing (urali, 2017). These amines may be primary, secondary, tertiary, or quaternary, depending on the number of hydrogens being replaced by the R-type groups to form the compound. For example, if R_3NH^+ is a tertiary amine and it undergoes a reaction with the anion as the following:

$$(R_3NH^+\ ) + X^- \rightarrow (R_3NH^+X^-) \qquad (1)$$

Then, an uncharged complex is formed. This amine salt, in the presence of an anionic compound for example (Y), wıll undergo anion exchange as:

$$(R_3NH^+X^-) + Y^- \rightarrow (R_3NH^+Y^-) + X^- \qquad (2)$$

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The entire product is called "liquid anion exchangers" (Khopkar, 2007). The liquid-liquid extraction method is very useful in recovering compounds from mixtures of materials; the principle of extraction depends on the factor of distribution of materials between two immiscible liquids. A part of the substance will be transferred to the second solvent, forming two layers of two unmixed liquids, where each of them contains a certain percentage of the substance (Blumberg, 1988; Ricci, 1980). When the extraction process reaches equilibrium, i.e. after shaking and stabilization, the concentration proportion of the extracted substance in the organic and aqueous phases is a constant, and this is known as the distribution coefficient K_D. Extraction of an organic acid (HA) with a secondary amine (R_2NH) can be described by the following reaction:

$$HA + {}^*R_2NH \rightarrow {}^*(HA)\cdot(R_2NH) \qquad (3)$$

Where; HA represents the undissociated portion of the acid present in the aqueous phase while species in the organic layer are marked with an asterisk (*).

Amberlite LA-2 (N-Lauryl (trialkylmethyl) amine) is a liquid secondary amine of high molecular weight and is soluble in most common (non-polar) organic solvents (A. AL-abbasi, Alammarwy, A., & Abdulljaoad, S., 2022; A. Al-abbasi, Emmhemad, H., Suliman, H., 2022). Amberlite LA-2 is insoluble in water making it of remarkable interest in a wide range of different applications such as aqueous mineral separation processes in wastewater treatment and food processing industriery. Hydrometallurgy is one of the main applications of Amberlite in the field of mineralogy, it has been mainly used in the recovery and purification of uranium mainly from low-concentration ore as well as separating iron, cobalt and nickel in hydrochloric acid solutions (A. Al-abbasi & Kassim, 2011; A. Al-abbasi, Tan, & Kassim, 2010; Belkher, 2019; Lo et al., 1991; Raynie, 2000). Numerous organic acids, such as L(+) tartaric acid, formic acid, propionic acid, picric acid, malic acid, fumaric acid, glycolic acid (A. Al-abbasi, Emmhemad, H., Suliman, H., 2022; Y. S. Aşçı, and Ismail, I., 2009) (H. Uslu, Bayat, Gökmen, & Yorulmaz, 2009), (Kloetzer, 2019), (H. Uslu, 2016), (H. Uslu, and & Kırbaşlar, 2010), (Khopkar, 2007; Kloetzer, 2019) and levulinic acid and malic acid (H. Uslu, Datta, Santos, & Öztürk, 2019) were purified using Amberlite LA-2.

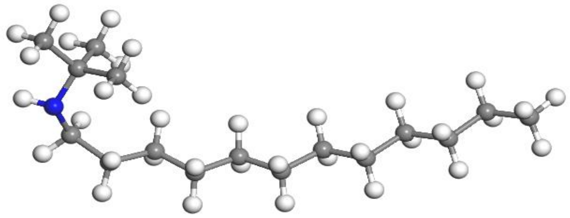


Figure 1: The chemical structure of Amberlite LA-2

Maleic acid, maleic anhydride, and fumaric acid (Figure 1) are multifunctional chemical intermediates that find applications in every field of industrial chemistry (Wiley). Each molecule contains two carbonyl acid groups and a double bond at the position. Maleic anhydride and maleic acid are raw ingredients in the production of resins, coatings, lubricants, copolymers, and ink production (Khalifa, 2018; Wojcieszak et al., 2015). Maleic acid can be formed by hydrolization of maleic anhydride. Isomerization of maleic acid produces fumaric acid which is a trans isomer of maleic acid concerning the double bond in the chemical molecule (Wiley).

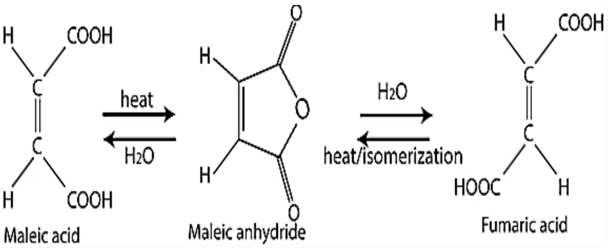


Figure 2: The chemical structure of maleic acid, fumaric acid, and maleic anhydride.

Moreover, in a recently published work by our group, high extraction ratios of about 97.65% and 99.9% respectively were obtained when Methyl Orange and Congo Red Dyes were removed by using Amberlite LA-2 (A. AL-abbasi, Alammarwy, A., & Abdulljaoad, S., 2022; A. AL-abbasi, Ehwedi, A., Ahmida, K., and Saleh,F., , 2023). Therefore, this study aims to investigate the extraction of maleic acid from Amberlite LA-2 by optimizing the extraction process in terms of the; initial acid and Amberlite LA-2 concentration, contact time, pH finally temperature. Furthermore, the mechanics of the extraction process of maleic acid with an anion exchanger in aqueous solutions were studied.

2 Experimental Section

2.1. The Chemicals and Instruments are used

The chemicals used are all of a high degree of purity and are produced by well-known companies. The type of chemicals used are; Maleic acid (Park Scientific Limited U.K, 99.8%), Amberlite LA-2 (BDH Laboratory Reagents, 99 %), Dichloromethane (Pure Chemistry, 99.8%), Sodium hydroxide (Analytical Reagent, 98%), Hydrochloric acid (Chemsolute, 37%), Sodium carbonate (Analytical Reagent, 99.8%), pH device (Thermo Electron/orion 3 star/USA) and water bath.

2.2. Preparation of Standard Solutions

Standard maleic acid solutions (0.1 N) were prepared as a stock solution by dissolving the required amount of 11.607g in a standard volumetric flask (volume 1 L). The volume was filled to the mark with distilled water. A solution of hydrochloric acid (0.1M) was prepared by taking the desired volume of concentrated acid 36 % in a standard volumetric flask 1L volume of distilled water and the acid concentration was adjusted by titrating using 0.1M sodium bicarbonate. The sodium hydroxide solution (0.1M) was prepared by dissolving 4g in volume 1L distilled water then the resulting solution was titrated with a standard solution of 0.1M hydrochloric acid. Different concentrations (0.044, 0.133, and 0.22 M) of Amberlite LA-2 were prepared in a 50 ml beaker by taking (1, 3, 5 mL) of liquid Amberlite and the volumes were diluted with dichloromethane solvent (DCM), the prepared solutions were simultaneously used on the same day to avoid evaporation of the solvent.

2.3 Extraction Experiment

Two equal quantities (10 mL each) of the previously prepared organic (DCM +Amberlite LA-2) and aqueous layers (tartaric acid) are taken in a 1:1 ratio and placed in tightly sealed containers. The mixture was shaken (200 rpm) in a water bath at desired temperature and time. A separating funnel was used to separate the aqueous layer from the organic layer after reaching equilibrium in each experiment. The lower organic layer is withdrawn from the separating funnel, then 4 ml of the aqueous layer is withdrawn for titration with sodium hydroxide (0.1 N), in the presence of phenolphthalein as the indicator to determine the amount of the remaining maleic acid in the aqueous phase. In addition, the extraction process was studied in the absence of Amberlite, so that 10 ml of two different concentrations of an aqueous solution of maleic acid 0.05M and 0.025M were taken separately then placed in an airtight bottle and 10 ml of dichloromethane (DCM) was added so far next step is shaken for 30 minutes at a temperature of 25°C. Experiments were carried out three times and their arithmetic average was taken. The distribution coefficients (K_D), extraction yields (E%) and loading factors (Z) can be determined using the below equations ;

$$K_D = \frac{[\mathrm{HA}]_{\mathrm{org}}}{[\mathrm{HA}]_{\mathrm{aq}}} \tag{4}$$

$$E\% = \left(1 - \frac{[\mathrm{HA}]_{\mathrm{aq}}}{[\mathrm{HA}]_{0,\mathrm{aq}}}\right) X\,100 \tag{5}$$

$$Z = [\mathrm{HA}]_{\mathrm{org}} / [R_2NH]_o \tag{6}$$

Where $[Dye]_{org}$ and $[Dye]_{aq}$ are the dye concentration in the organic layer and the aqueous layer, respectively, and $[R_2NH]_0$ is the initial concentration of Amberlite LA-2. (A. AL-abbasi, Ehwedi, A., Ahmida, K., and Saleh,F., , 2023; A. Al-abbasi, Emmhemad, H., Suliman, H., 2022; H. Uslu, Baykal, Gök, Kırbaşlar, & Santos, 2020).

3 Results and Discussion

The distribution coefficients (K_D) and loading coefficient (Z) were studied and the percentage of extraction (E%) of the acid between the aqueous and organic phases to the organic class. The initial concentration of the acid and the concentration of the extractive substance (Amberlite LA-2), temperature, contact time, and finally pH were explored as the parameters influencing the extraction process. The experimental results of the extraction were compiled in Tables 1, 2, 3, and 4, which are listed in that order.

According to the collected data in Tables (1 and 2), the values of the distribution coefficients are typically higher than 1.0. This is explained by the fact that acid is distributed more widely in the organic phase than in the aqueous phase. The calculated distribution coefficient is displayed in Tables (1, 2, 3, and 4). It can be seen that all of the distribution coefficients increase as the initial acid concentration is raised from 0.1 to 0.075 M, demonstrating that the starting acid concentration has some positive effects on the distribution coefficient.

According to the results presented in Table (3), the greatest value of KD was 100 after an hour of combining 0.075 M and 0.044 M of maleic acid with an Amberlite AL-2, respectively. The high value of KD indicates that there is more maleic acid present in the organic phase than there is in the aqueous phase.

In general, the research results indicated Tables 1, 3, and 4 suggested that the loading factor would rise as the initial concentration of maleic acid did. However, as the concentration of Amberlite-LA2 declines, the loading percentage increase

3.1 The Effect of the Initial Concentration

In order to investigate the effect of the initial concentration of maleic acid on extraction efficiency %, maleic acid solutions with varied beginning concentrations in the range of 0.01–0.075 N were studied under circumstances of constant Amberlite–LA2 concentration, temperature, and pH. At 0.044 M of Amberlite-LA2 concentration, 25 °C, pH= 3.06, and various time intervals, the results are displayed in Table (2) and Figure (3). The overall findings show that as the initial acid maleic content increased, the extraction efficiency increased similarly.

Table 1: The results of the extraction of maleic acid induce the effect of the concentrations of the extracted and extracted substance.

C_{LA-2}	C_{HA}	t (min)	C_{aq}	C_{org}	K_D	Z	E%
0.044	0.01	15	0.00075	0.00926	12.51351	0.2104	92.6
		30	0.000375	0.009625	25.67	0.2187	96.25
		45	0.00025	0.00975	39	0.2215	97.5
		60	0.0002	0.0098	49	0.2227	98
	0.025	15	0.001388	0.023613	17.01802	0.5366	94.45
		30	0.000625	0.024375	39	0.5539	97.5
		45	0.000375	0.024625	65.66667	0.5596	98.5
		60	0.00025	0.02475	99	0.5625	99
	0.05	15	0.001225	0.048775	39.81633	1.1085	97.55
		30	0.000813	0.049188	60.53846	1.1179	98.375
		45	0.0007	0.0493	70.42857	1.1204	98.6
		60	0.0004	0.0496	124	1.1272	99.2
	0.075	15	0.00105	0.07395	70.42857	1.6806	98.6
		30	0.000775	0.074225	95.77419	1.6869	98.9666
		45	0.000825	0.074175	89.90909	1.6857	98.9
		60	0.000738	0.074263	100.6949	1.6877	99.0166
0.133	0.01	15	0.00113	0.00887	7.849558	0.0666	88.7
		30	0.000963	0.009038	9.38961	0.0679	90.375
		45	0.00085	0.00915	10.76471	0.0687	91.5
		60	0.000863	0.009138	10.5942	0.0687	91.375
	0.025	15	0.001888	0.023113	12.24503	0.1737	92.45
		30	0.001175	0.023825	20.2766	0.1791	95.3
		45	0.001083	0.023918	22.09469	0.1798	95.67
		60	0.001313	0.023688	18.04762	0.1781	94.75
	0.05	15	0.001563	0.048438	31	0.3641	96.875
		30	0.001238	0.048763	39.40404	0.3662	97.525
		45	0.001288	0.048713	37.83495	0.3662	97.425
		60	0.0015	0.0485	32.33333	0.3646	97
	0.075	15	0.00225	0.07275	32.33333	0.5469	97
		30	0.00175	0.07325	41.85714	0.5507	97.6666
		45	0.001713	0.073288	42.79562	0.5510	97.7166
		60	0.0017	0.0733	43.11765	0.0551	97.7333
0.22	0.01	15	0.001713	0.008288	4.839416	0.0376	82.875
		30	0.0011	0.0089	8.090909	0.0404	89
		45	0.001375	0.008625	6.272727	0.0392	86.25
		60	0.001463	0.008538	5.837607	0.0388	85.375
	0.025	15	0.0025	0.0225	9	0.1022	90
		30	0.001463	0.023538	16.09402	0.1069	94.15
		45	0.00155	0.02345	15.12903	0.1065	93.8
		60	0.001575	0.023425	14.87302	0.1064	93.7
	0.05	30	0.0031	0.0469	15.12903	0.2131	93.8
		30	0.002325	0.047675	20.50538	0.2167	95.35
		45	0.001716	0.048283	28.12621	0.2194	96.5666
		60	0.001525	0.048475	31.78689	0.2203	96.95
	0.075	15	0.003337	0.071662	21.47191	0.3257	95.55
		30	0.002437	0.072563	29.76923	0.3298	96.75
		45	0.002312	0.072687	31.25806	0.3303	96.9
		60	0.002313	0.072688	31.43243	0.3304	96.9166

Table 2: Maleic acid extracting in the absence of Amberlite LA-2

C_{LA-2}	C_{HA}	t(min)	C_{aq}	C_{org}	K_D	E%
0	0.01	30	0.0025	0.0075	3	75
	0.025	30	0.0053125	0.0196875	3.705882	78.75
	0.05	30	0.01	0.04	4	80
	0.075	30	0.011625	0.063375	5.451613	84.5

Table 3: The effect of pH on Maleic acid extraction

C_{LA-2}	C_{HA}	t (min)	pH	C_{aq}	C_{org}	K_D	Z	E%
0.133	0.01	30	1.92	0.0025	0.0075	3	0.0563	75
			3.06	0.000963	0.009038	9.38961	0.0679	90.375
			5.76	0.001	0.009	7	0.0676	87.5
			7.34	0.002	0.008	4	0.0601	80
			11.25	0.00375	0.00625	1.667	0.0469	62.5

Table 4: The effect of temperature on Maleic acid extraction.

C_{LA-2}	C_{HA}	t (min)	C_{aq}	C_{org}	K_D	Z	E%
0.044	0.025	15	0.0006	0.0244	40.6666	0.5545	97.6
		30	0.000138	0.024863	180.818	0.5650	99.45
		45	0.000025	0.024975	999	0.5676	99.9
		60	0.0003	0.0247	82.3333	0.5613	98.8
0.044	0.05	15	0.000075	0.049925	665.666	1.1346	99.85
		30	0.000025	0.049975	1999	1.1357	99.95
		45	0.0012	0.0488	40.6666	1.1090	97.6
		60	0.000275	0.0497	180.818	1.1295	99.45

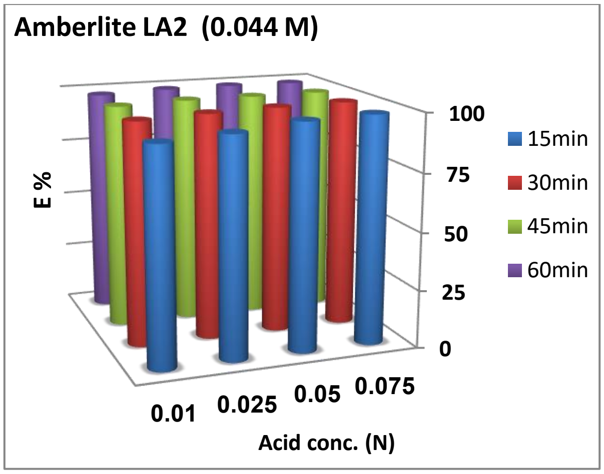


Figure 3: The effect of the initial concentration

3.2 Effect of Extractive Concentration (Amberlite LA-2)

To verify the role of the organic solvent in the extraction process, an extraction experiment was conducted in the absence of the extracting substance (secondary amine). From the results of the experiments shown in Figure (4), it was found that there is an amount of maleic acid above 75% that was extracted from the aqueous phase to the organic phase, due to the unlimited solubility of maleic acid in the DCM solvent, which is depended on to the carbon chain in the acid.

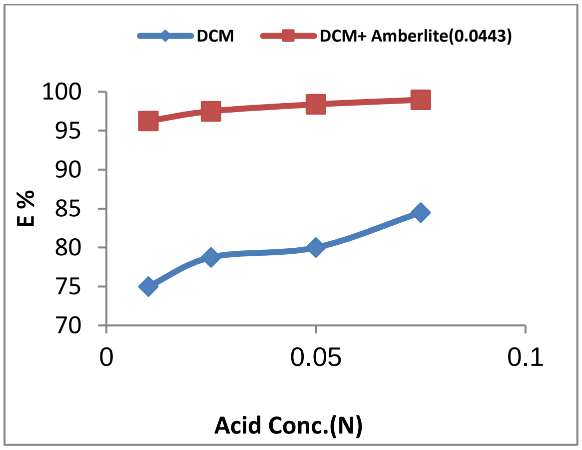
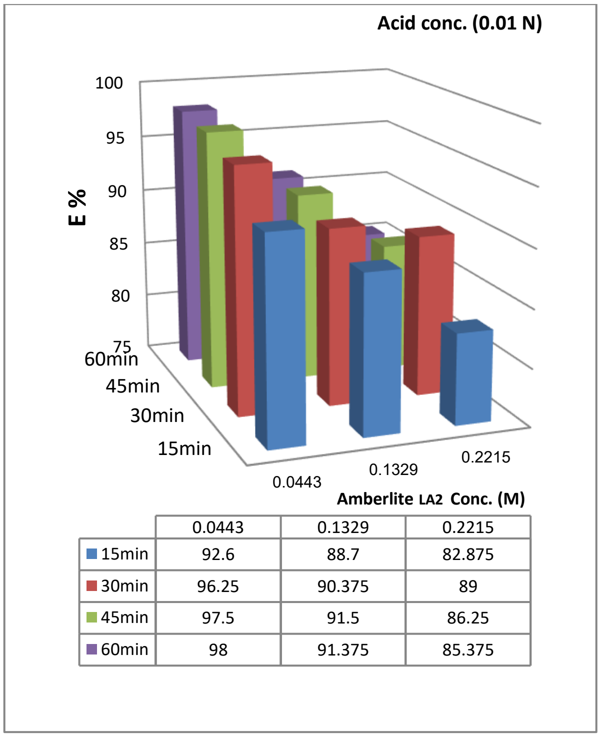


Figure 4: Extraction of maleic acid in the absence of the extract Amberlite LA-2

The effect of different concentrations of secondary amine (Amberlite AL-2) on the extraction of maleic acid was studied, where the varying concentrations of the extract were 0.044, 0.133, 0.22 M at room temperature (25ºC) and pH = 3.06. A relationship was drawn between the secondary amine concentration and the percentage of extraction at different times (15, 30, 45, and 60 minutes) as shown in Figure (5). It was observed that the percentage of maleic acid extraction increased from 85.3 to 98% with an increase in the extract concentration from 0.044 to 0.22 M.



	0.0443	0.1329	0.2215
15min	92.6	88.7	82.875
30min	96.25	90.375	89
45min	97.5	91.5	86.25
60min	98	91.375	85.375

Figure 4: Effect of Amberlite LA-2 concentration on the extraction process

3.3 Effect of pH on the Extraction Process

The pH of the acid solution was adjusted to a range of 2 to 11. The initial maleic acid concentration and Amberlite AL-2 concentration were both held constant, and the solutions' temperature was maintained at 25 °C the maximum maleic acid extraction efficiency, 90.3%, was achieved at pH 3, as illustrated in Figure 5. In order to transfer the solute into the organic phase, the pH of the aqueous solution was therefore appropriate. With an increase in pH, carboxylic acid will dissociate more readily in an alkaline medium, and the organic phase will be able to extract less acid. The competition between the acid anion and the OH- ion for binding sites as a consequence of the presence of the OH- ion in the base solution can be used to explain why the extraction percentage has decreased.

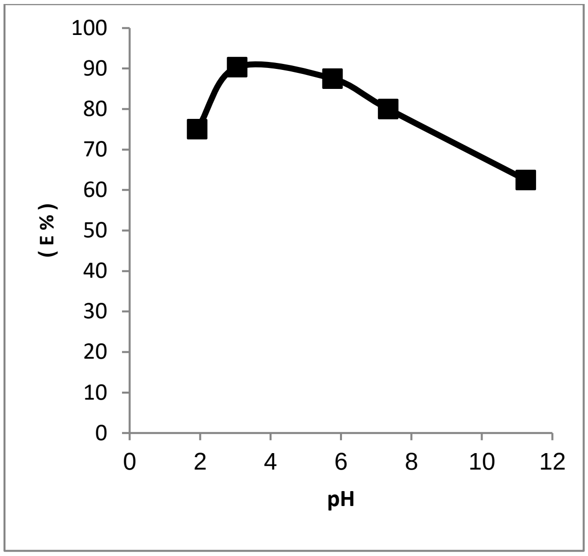


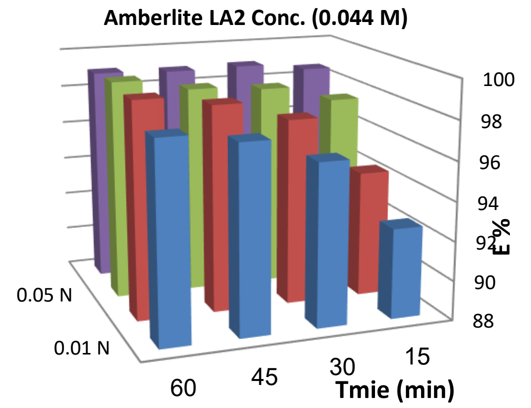
Figure 5: The effect of pH on the percentage of extraction

3.4 Effect of Contact Time

The purpose of this experiment was to determine the time required to reach equilibrium and also to study kinetics. The contact time was varied at 15, 30, 45, and 60 minutes, and the results are shown in Table 1. The results implied that the concentration of acid extracted from the aqueous phase to the organic phase increases with time, and the quantitative transfer of maleic acid took place after 60 minutes (Figure 6). At initial concentration of 0.01 M, the percentage of extraction increased from 92 to 99% when the contact time was prolonged from 15 to 60 minutes.

3.5 Effect of Temperature

The impact of temperature on the extraction of maleic acid from the aqueous phase at two different initial concentrations (0.025 and 0.05 M) was investigated. At a constant extractant concentration of 0.044 M and a fixed contact time of 30 min, the temperature was changed within 25 °C, 30 °C, and 40 °C. According to Figure 7, the proportion of extracted maleic acid with an initial concentration of 0.05 M appeared increase from 98.8% to 99.95% with increasing temperature. The extraction process is an activating process that increases with rising temperature, which is responsible for an increase in the extracted amount of maleic acid.



	60	45	30	15
🟦 0.01 N	98	97.5	96.25	92.6
🟥 0.025 N	99	98.5	97.5	94.45
🟩 0.05 N	99.2	98.6	98.375	97.55
🟪 0.075 N	99.0166667	98.9	98.9666667	98.6

Figure 6: The effect of contact time on the percentage of extraction

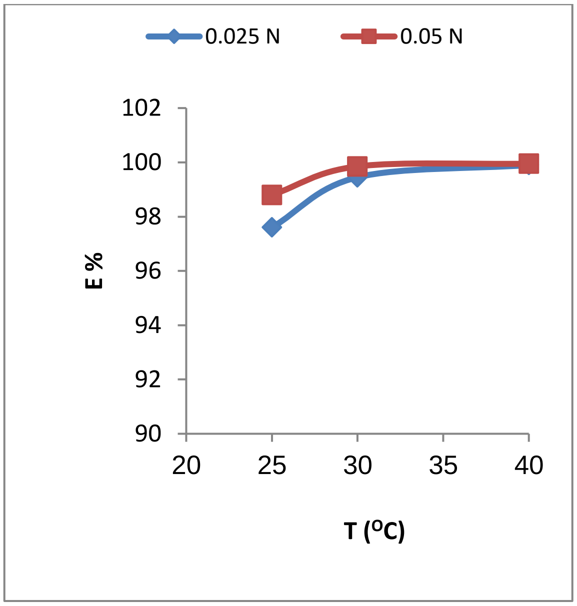


Figure 7: The effect of temperature on the extraction process

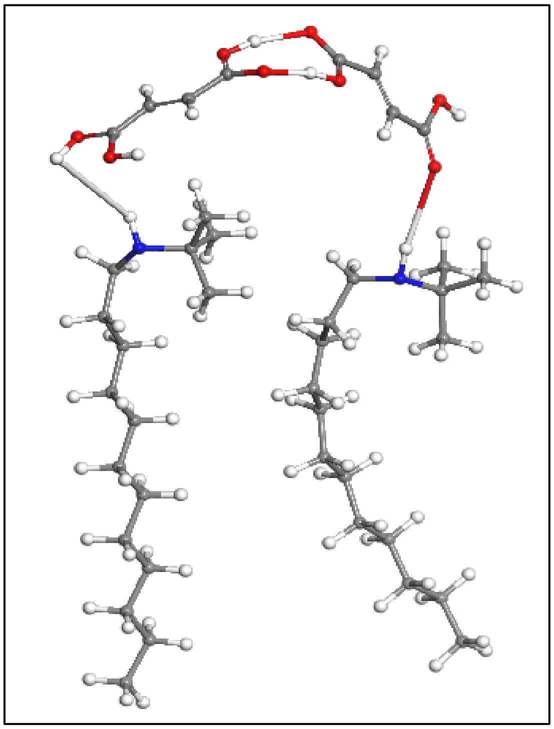


Figure : 8 The complex produced by the interaction of maleic acid and Amberlite LA-2

3.6 Extraction Process Mechanics

Based on research by Winestrin, the mechanism of complex formation is taken out to explain how acid is captured in the organic phase (Wennersten, 1983). To ascertain the molecular structure of the amine and acid mixture in the organic layer, Winestrin proposed that an ion pair forms once the acid and amine mix instantly. As shown in Figure 8, a compound is formed once the [OH] group of one acid molecule hydrogen bonds with the [C=O] group of the carboxyl group of another acid (Y. S. Aşçı & İnci, 2009; Y. S. Aşçı & İnci, 2009).

4 Conclusion

Maleic acid was successfully extracted from the aqueous solution via liquid/liquid extraction process using Amberlite LA-2 dissolved in DCM as an organic solvent. The distribution coefficients of maleic acid for the extraction process had values greater than 1, indicating a higher distribution of acid in the organic layer than in the aqueous layer. The distribution coefficient increased with increasing concentration of maleic acid in the medium. The results show that the extraction efficiency increased with the increase in the initial concentration of maleic acid, where the percentage increased from 95.65% to 99.01% when the concentration was increased from 0.01 to 0.075 M standard. It was also observed that the percentage of maleic acid extraction increases with increasing secondary amine concentration from 0.044 to 0.22 M. The percentage of acid increases with increasing temperature from 25 to 40 ^{o}C. The maleic acid extraction was more efficient in acidic medium, as it was noticed that the separation efficiency decreases as the acidity function increases in the neutral and basic medium.

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Conflict of Interest: The authors declare that there are no conflicts of interest.

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