Libyan Journal of Ecological & Environmental Sciences and Technology
(LJEEST)

LJEEST
ISSN: 2710-5237
www.srcest.org.ly/jou

https://doi.org/10.63359/drfc9462

Liquid-Liquid Extraction of Tartaric Acid from Aqueous Solutions by Amberlite LA-2 as Liquid Ion Exchanger

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ARTICLE INFO

Vol.4 No. 2 Dec., 2022
Pages (5-12)

Article history:
Revised form 01 Sebtamber 2022
Accepted 15 Sebtamber 2022

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Keywords:
Tartaric acid, liquid-liquid extraction, Amberlite LA-2, distribution coefficient, loading coefficient, Extraction percentage

ABSTRACT

A liquid/liquid extraction method was used to extract tartaric acid using the Amberlite LA-2 anion exchanger. The extraction efficiency of tartaric acid was determined under the influence of both the initial concentration of the acid and the initial concentration of the extractant Amberlite LA-2 at a contact time ranging from 15 to 60 minutes and at 25ºC. The effect of pH and temperature effect on extraction efficiency was also studied under conditions of contact time of 30 minutes using an initial concentration of tartaric acid and Amberlite LA-2 to be 0.05 N and 0.022 N, respectively. The titration technique with sodium hydroxide was used to determine the concentration of tartaric acid in the aqueous layer before and after the extraction procedure. The experimental data were used to calculate distribution coefficients (K_D), loading coefficients (Z), and extraction efficiency E%. The obtained results indicated that the extraction percentage E, %, increased with the increase of Amberlite LA-2 concentration, pH and temperature. An extraction efficiency of 99.5% was obtained, and a maximum loading of 1.318. The extraction process mechanism was also proposed.

استخلاص السائل السائل لحمض الطرطريك من المحاليل المائية بواسطة Amberlite LA-2 كمبادل أيونى سائل

عائشة أحمد العباسى * هنادي عبد السلام هديل على سليمان

تم استخدام طريقة الاستخلاص السائل/السائل لاستخلاص حمض الطرطريك باستخدام المبادل الأنيوني . AMBERLITE LA-2 تم تعيين كفاءة الاستخلاص لحمض الطرطريك تحت تأثيركلا من التركيز الابتدائي للحمض وتركيز الابتدائي للمادة المستخلصة AMBERLITE LA-2 وذلك عند زمن تلامس يتراوح بين 15 إلى 60 دقيقة وعند 25 م^{O} . وايضا تمت دراسة تأثير الأس الهيدروجيني وتأتير درجة الحرارة على كفاءة الاستخلاص وذلك عند ظروف من زمن تلامس وقدره 30 دقيقة باستخدام تركيزابتدائي لحمض الطرطريك و مادة AMBERLITE LA-2ليكون N 0.05 و N 0.022 على التوالي. استخدمت تقنية المعايرة بهيدروكسيد الصوديوم لتقدير تركيز حمض الطرطريك في الطبقة المائية قبل وبعد عملية الاستخلاص. استخدمت البيانات التجريبية المتحصل عليها لحساب معاملات التوزيع (K_D) ومعاملات التحميل (Z) وكفاءة الاستخلاص (E%) .أشارت النتائج التي تم الحصول عليها إلى أن نسبة المئوية للاستخلاص ,% E, تزداد مع زيادة تركيز AMBERLITE LA-2و pH ودرجة الحرارة. تم الحصول على كفاءة استخلاص بنسبة 99.5 % ، وأقصى تحميل 1.318. ايضا تم اقتراح آلية عملية الاستخلاص.

INTRODUCTION

Carboxylic acids are frequently used in the pharmaceutical sector as a nutrient, additive, or raw material (Babu., 2008; Uslu, Baykal, Gök, Kırbaşlar, & Santos, 2020). Modern manufacturing of carboxylic acids uses either chemical synthesis or a fermentation process, which are two of the most source currently in use(Dziezak, 2016). There are several studies indicating that carboxylic acid purification processes account for more than 30% of the total processing costs in such processes (Murali, Srinivas, & Ahring, 2017). Thus, the extraction of the Dicarboxylic acids from their source using environmentally friendly methods has gained interest (Uslu *et. al*., 2020).

Tartaric acid is an important dicarboxylic acid that is widely used in chemical, pharmaceutical, food, and other industries (Dziezak, 2016; Uslu *et. al*., 2020). The largest single application of tartaric acid is as a raw material for the manufacture of emulsifiers used to improve baking, in the food industry (such as jams, fruit juices, pickles, and soft drinks), as an acidifier in grape and lemon flavored beverages, gelatin desserts, jams, jellies, and hard desserts. The acid monopotassium salt, commonly known as "cream of tartar," is used by industries such as ceramics, textiles, tanning, and photography as well as pharmaceuticals)Dziezak, 2016; Goldberg & Rokem, 2009; Joye, 2019(. Since tartaric acid has so many uses in a wide range of different industries, it was necessary to find ways to purify it and obtain it in its purest form(Anyasi, 2017; Murali *et. al*., 2017; Uslu *et. al*., 2020). There are many published publications concerned with the purification of tartaric acid using solvent extraction techniques (Eda *et. al*., 2018; Inci, Asçi, & Tuyun, 2011; Marinova, Kyuchoukov, Albet, Molinier, & Malmary, 2004; Poposka, Prochazka, Tomovska, Kostadin, & Grizo, 2000; Sharma, Singh, Wasewar, & Athankar, 2017; Uslu, 2007).

Separation using solvent extraction is one of the most important methods used in purification, where the acid is extracted in the organic phase and reacted with a carrier (additive), which leads to a higher extraction efficiency. Such as the use of amines with primary alcohol or kerosene for the efficient extraction of different carboxylic acids from the fermentation mixture)urali, 2017(. One of the primary disadvantages of using solvent extraction for on-site product recovery during fermentation is the difficulty in finding a common, biocompatible solvent with a high product recovery coefficient. Another problem is the ability of the solvents to operate at fermentation pH (ranging from 5.5 to 7), which is an interesting range for carboxylic acid production) urali, 2017 ; Yang, White, & Hsu, 1991(. The ability and selectivity to extract tartaric acid are achieved by liquid-liquid extraction (LLE) utilizing a particular extractor containing an appropriate functional group (for example, amine) (Uslu *et. al*., 2020). The choice of solvent depends on two main factors: 1) its good ability to dissolve the substance to be extracted. 2) its ease of separation from the mixture.

The use of water-insoluble acid-base and liquid reagents as liquid ion exchangers was first proposed by Smith and Page in a paper entitled "Acid-binding properties of long-chain aliphatic amines")Smith & Page, 1948; Werner, 1974(. Since then, there has been a growing interest in using bases of high molecular weight as extracts)Werner, 1974(. Amberlite-LA-2 is chemically called N-Lauryl (trialkylmethyl) amine and is a liquid secondary amine of high molecular weight, soluble in most common (non-polar) organic solvents. 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 from low-concentration ore as well as the separation of iron, cobalt, and nickel in hydrochloric acid solutions)Kunin & Winger, 1962; Werner, 1974(. Moreover, Amberlite LA-2 was used to purify many organic acids including L(+) Tartaric Acid)Inci *et. al*., 2011,(Formic Acid)Uslu, Bayat, Gökmen, & Yorulmaz, 2009(, Propionic Acid)Aşçı & İnci, 2009(, Picric Acid)Uslu, 2016(, Malic Acid)Uslu & İsmail Kırbaşlar, 2010(, Fumaric Acid)Kloetzer, Ilica, Cascaval, & Galaction, 2019(, Glycolic Acid)Aşçı & İnci, 2009(, and Levulinic Acid & Malic Acid)Uslu & Kırbaşlar, 2013(. The main objective of this project is to purify tartaric acid using the liquid-liquid extraction technique. Studying the ability of the negative ion exchanger represented by the substance Amberlite LA-2 as an extract of tartaric acid. Studying some of the factors affecting the extraction process to obtain the best conditions for the extraction process, such as the effect of the initial concentration of the acid. Effect of concentration of the extracted substance (Amberlite LA-2), the effect of pH, effect of contact time, and effect of temperature.

MATERIALS AND INSTRUMENTATION

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1 Chemicals and instrumentations

The chemicals used are all of a high degree of purity and are produced by well-known companies: Tartaric acid (99.5 %), Amberlite LA-2 (99 %), Dichloromethane (99.8%), sodium hydroxide (98%), hydrochloric acid (37%) and sodium carbonate (99.8%). a pH device (Thermo).

2 Preparation of standard solutions

Standard tartaric acid solutions (0.1 N) were prepared as a stock solution by dissolving the required amount of 7.5045 g in a standard flask (1 L). Amberlite LA-2 solutions with different concentrations of 0.022, 0.044, and 0.133 N, were 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. A solution of (0.1M) sodium hydroxide was prepared by dissolving (4 g) in a standard flask (1L). To adjust the concentration, the solution was titrated against a standard solution of hydrochloric acid in the presence of a phenolphthalein indicator. A standard solution of (0.1N) hydrochloric acid was prepared and the concentration was adjusted by titrating it with (0.1M) sodium carbonate in the presence of an orange methyl indicator. Various concentrations (0.022, 0.044, 0.133 molars) of Amberlite LA-2 were prepared in a 50 mL beaker by taking (1/2, 1, and 3 mL) of liquid amberlite in dichloromethane (DCM), noting that the prepared solutions were prepared in The same day, in order to avoid evaporation of the solvent, and that the room temperature at the time of the experiment was 25 degrees Celsius.

3 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 tartaric acid. The bottles with the mixture are placed in a shaking water bath (200 rpm) at a certain temperature. The bottles are shaken for the desired time, then the aqueous layer is separated from the organic layer using a separating funnel. Immediately 4 mL was withdrawn from the aqueous layer to measure the concentration of tartaric acid by titration against sodium hydroxide. To obtain the best conditions for the extraction process, the effect of the initial concentration of tartaric acid (0.075, 0.05, 0.025, 0.01 N) and the initial concentration of Amberlite LA-2 (0.022, 0.044, 0.133 N), as well as the effect of temperature (25, 30, 40 °C) and time (15, 30, 45, 60 and 90 min), were studied. Moreover, the effect of the pH on the extraction process was studied at different pH values (1,3,5,9,11,13). The percentage distribution (K_D) and percentage of the extracted dye (%E) were calculated.

RESULT AND DISCUSSION

3.1 Theoretical approach

The distribution of the solute between the two immiscible solvents is described by the law of distribution. The molecules of solute HA are distributed between two organic and aqueous phases, then the equilibrium can be stated as follows:

$$HA_{aq} \leftrightarrows HA_{org} \qquad (1)$$

Where aq and org refer to the organic and aqueous phases, respectively. The distribution coefficient of the solute, (K_D), is expressed by eq. (1).

$$K_D = \frac{[HA]_{org}}{[HA]_{aq}} \qquad (1)$$

Where: $[HA]_{org}$ and $[HA]_{aq}$ are the concentration of HA in the organic and aqueous layer. This means that the greater the distribution coefficient, the closer the extraction will be. The loading coefficient (Z) is defined as the maximum concentration of solute that a solvent can load under specific conditions)Rice, Irving, & Leonard, 1993(. The loading factor Z can be expressed by the following equation:

$$Z = \frac{[HA]_{org}}{[R_2NH]_0} \qquad (2)$$

where $[R_2NH]_0$ is the initial concentration of Amberlite LA-2. The extracted percentage, E%, is expressed by the extraction efficiency, E%, by the following equation (3):

$$E\,\% = [1-(\frac{[HA]}{[HA]_0})]\times 100 \qquad (3)$$

where $[HA]_0$ is the initial concentration of the acid in the aqueous phase.

2 Extraction optimization

In order to achieve the objectives of this research, we studied the extraction of tartaric acid from its aqueous phase by studying the chemical equilibrium system between Amberlite LA2 dissolved in organic DCM and the aqueous phase of tartaric acid.

The distribution coefficients (K_D), loading factor (Z), and percentage of extraction (E%) were studied. The parameters affecting the extraction process were studied, namely the initial concentration of tartaric acid, the initial concentration of Amberlite LA-2, temperature, contact time, and pH. The focus was on testing such conditions to obtain the best results for the extraction process so each experiment was conducted three times and results were collected in Tables 1, 2, and 3, which respectively give the experimental results of the extraction in the presence and absence of Amberlite in the organic layer.

The distribution coefficient, K_D, values ranged from 0.489 to 221.2, and these values depend on the conditions of the experiments of the concentration of both tartaric acid and Amberlite and the effect of temperature, pH, and contact time. In the most of experiments, the distribution coefficients were greater than 1 as the tartaric acid has a higher concentration in the organic layer than in the aqueous layer. when the distribution coefficient values are less than 1 means that the acid is present in the aqueous layer. this was only at a lower concentration of amine (0.022M). The loading factors were ranged from 0.054 to 1.318. The loading factor in general increases with an increasing initial concentration of the acid, pH, and temperature. However, the highest value of the acid loading factor was obtained when the acid concentration was (N 0.05) and the amine concentration was low (0.022N) at room temperature.

Table 1. Characteristic performance data obtained by liquid-liquid extraction of tartaric acid by Amberlite LA-2

C_{LA-2} (N)	C_{HA} (N)	t (min)	C_{aq}	C_{org}	K_D	Z	E%
0.022	0.01	15	0.001463	0.008538	5.837607	0.3881	85.375
	0.01	30	0.001	0.009	9	0.4091	90
	0.01	45	0.001	0.009	9	0.4091	90
	0.01	60	0.001	0.009	9	0.4091	90
	0.025	15	0.00475	0.02025	4.263158	0.9205	81
	0.025	30	0.00675	0.01825	2.703704	0.8295	73
	0.025	45	0.008875	0.016125	1.816901	0.733	64.5
	0.025	60	0.009875	0.015125	1.531646	0.688	60.5
	0.05	15	0.021	0.029	1.380952	1.318	58
	0.05	30	0.0215	0.0285	1.325581	1.2955	57
	0.05	45	0.022	0.028	1.272727	1.2727	56
	0.05	60	0.024	0.026	1.083333	1.1818	52
	0.075	15	0.050375	0.024625	0.488834	1.1193	32.83333
	0.075	30	0.0475	0.0275	0.578947	1.25	36.66667
	0.075	45	0.047125	0.027875	0.591512	1.2671	37.16667
	0.075	60	0.04775	0.02725	0.570681	1.2386	36.33333
0.044	0.01	15	0.001375	0.008625	6.272727	0.196	86.25
	0.01	30	0.000875	0.009125	10.42857	0.2074	91.25
	0.01	45	0.00075	0.00925	12.13333	0.2102	92.5
	0.01	60	0.000375	0.009625	25.66667	0.2188	96.25
	0.025	15	0.00375	0.02125	5.666667	0.483	85
	0.025	30	0.0025	0.0225	9	0.5114	90
	0.025	45	0.00175	0.02325	13.28571	0.5284	93
	0.025	60	0.00125	0.02375	19	0.054	95
	0.05	15	0.003875	0.046125	11.90323	1.0483	92.25
	0.05	30	0.00375	0.04625	12.33333	1.05114	92.5
	0.05	45	0.0025	0.0475	19	1.07955	95
	0.05	60	0.0025	0.0475	19	1.07955	95
	0.075	15	0.03375	0.04125	1.222222	0.9375	55
	0.075	30	0.025875	0.049125	1.898551	1.1165	65.5
	0.075	45	0.0255	0.0495	1.941176	1.125	66
	0.075	60	0.0205	0.0545	2.658537	1.2386	72.66667
0.133	0.01	15	0.0009	0.0091	10.11111	0.06842	91
	0.01	30	0.000625	0.009375	15	0.07049	93.75
	0.01	45	0.000375	0.009625	25.66667	0.07237	96.25
	0.01	60	0.00025	0.00975	39	0.07331	97.5
	0.025	15	0.002	0.023	11.5	0.17293	92
	0.025	30	0.001375	0.023625	17.18182	0.17763	94.5
	0.025	45	0.000625	0.024375	39	0.18327	97.5
	0.025	60	0.00025	0.02475	99	0.18609	99
	0.05	30	0.00125	0.04875	39	0.36654	97.5
	0.05	30	0.000625	0.049375	79	0.37124	98.75
	0.05	45	0.0004	0.0496	124	0.3729	99.2
	0.05	60	0.00025	0.04975	199	0.37406	99.5
	0.075	15	0.00175	0.07325	41.85714	0.55075	97.66667
	0.075	30	0.0015	0.0735	49	0.55263	98
	0.075	45	0.001	0.074	74	0.55639	98.66667
	0.075	60	0.000875	0.074125	84.71429	0.55733	98.83333

3.2.1 Effect of the initial concentration of acid

The efficiency of extraction in general increases with the increase in the initial concentration of the acid. For example, the percentage of extracted tartaric acid increases from 91% to 97.6% when the initial concentration is increased from 0.01 to 0.075 N. The increase in extraction with increasing initial concentration can be explained by the fact that the organic medium under these conditions can load more acid molecules and has not reached saturation, as shown in Figure (1).

3.2.2 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 Amberlite. From the results of the experiments, it was found that there is an amount of tartaric acid higher than 50% that was extracted from the aqueous phase to the organic phase in the absence of Amberlite LA-2 C, due to the unlimited solubility of tartaric acid in the DCM solvent, which is due to the acid carbon chain, meaning that the solvent is a good carrier of acid. In the presence of Amberlite, the E% of tartaric acid extraction increased from 90.0 to 97.5% with an increase in the concentration of Amberlite AL-2 from 0.022 to 0.133 N as shown in Figure (2).

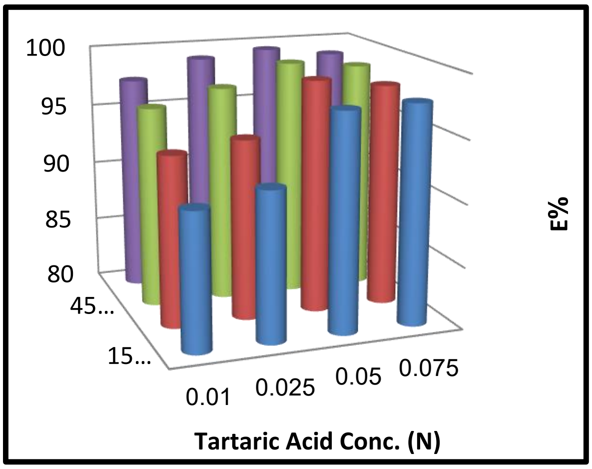

Figure 1: Effect of initial concentration of tartaric acid, on the extraction process at 25 ºC

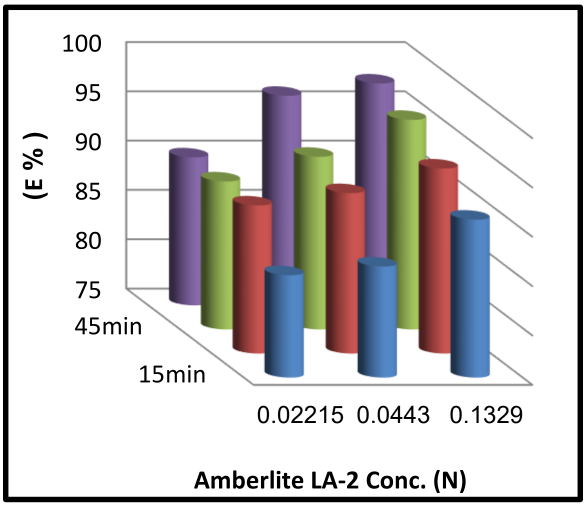

Figure 2: Effect of initial concentration of Amberlite LA-2 on the extraction process at 25 ºC

3.2.3 The effect of contact time

The results showed that the acid extracted from the aqueous phase to the organic phase increases with time, and the quantitative transfer of tartaric acid takes place after 60 minutes. In general, Figure (3) shows that when time increases; The acid concentration in the organic phase increases gradually.

3.2.4 Effect of pH

The percentage of extraction increases with the increase of pH from pH = 2 to pH = 5. The percentage of extraction remains constant after that when the pH value is raised to 13. This is because when the pH values increase, it leads to an increase in the dissolution of the acid and thus ease of its transfer. to the organic phase by electrostatic attraction as shown in Figure (4).

3.2.5 Temperature effect

The percentage of extracted acid with a concentration of 0.025 M was observed to increase from 90% to 99.05% with increasing temperature from 25 to 40 degrees Celsius, respectively. The increase in the percentage of acid is due to the fact that the extraction process is an activating process that increases with increasing temperature.

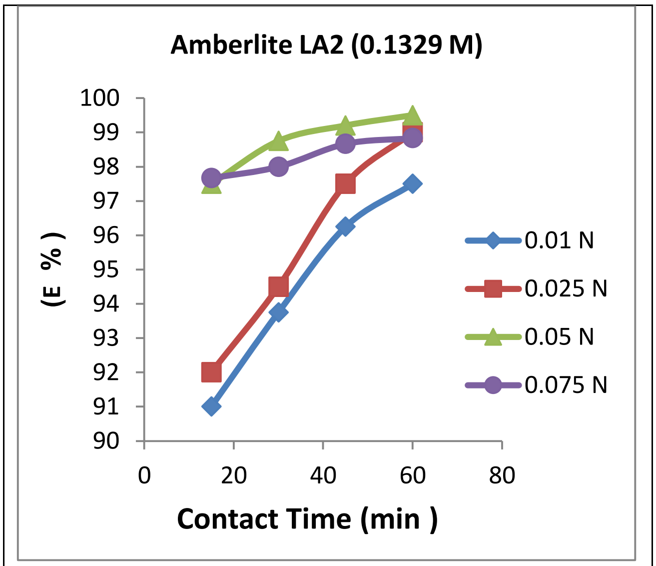

Figure 3: Effect of the contact time on the tartaric acid extraction

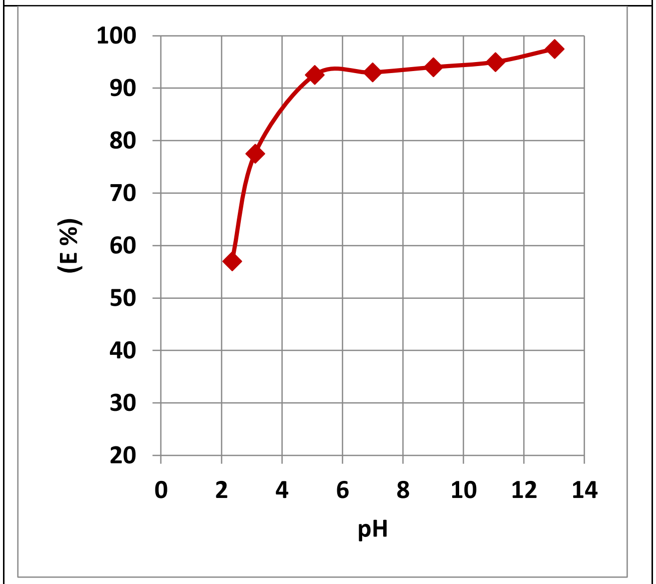

Figure 4: Effect of pH on the tartaric acid extraction

Table 2: The results obtained when extracting tartaric acid by Amberlite under the pH effect.

C_{LA-2} (N)	C_{HA} (N)	t (min)	pH	C_{aq}	C_{org}	K_D	Z	E%
M0.022	0.05N	30	2.35	0.0215	0.0285	1.325581	1.2955	57
			3.11	0.011	0.039	3.444	1.7727	77.5
			5.08	0.004	0.046	12.333	2.0909	92.5
			11.06	0.003	0.048	19	2.1818	95
			13.01	0.001	0.049	39	2.2273	97.5

Table 3: Effect of temperature on the extraction of tartaric acid by Amberlite LA-2

C_{LA-2} (N)	C_{HA} (N)	t (min)	C_{aq}	C_{org}	K_D	Z	E%
0.044 M	0.025 N	15	0.0025	0.0225	9	0.5114	90
		30	0.000113	0.024888	221.2222	0.56564	99.55
		45	0.000238	0.024763	104.2632	0.5628	99.05
		60	0.001875	0.023125	12.33333	0.52557	92.5
	0.05 N	15	0.00075	0.04925	65.66667	1.11932	98.5
		30	0.0091	0.049088	53.79452	1.11564	98.175
		45	0.005	0.045	9	1.02273	90
		60	0.000225	0.049775	221.2222	1.13125	99.55

3.3 Extraction process mechanics

The mechanism of complex formation was proposed to explain the adsorption of acid in the organic phase, where the scientist Weinstein [1] determined the structure of the complex consisting of amine molecules - and acid molecules in the organic layer. He suggested that the acid reacts with the amine directly to form an ionic pair, then the [OH] group in another molecule of the acid forms a hydrogen bond with the [C=O] group in the carboxyl group of the first acid to form the compound shown in Figure (5)

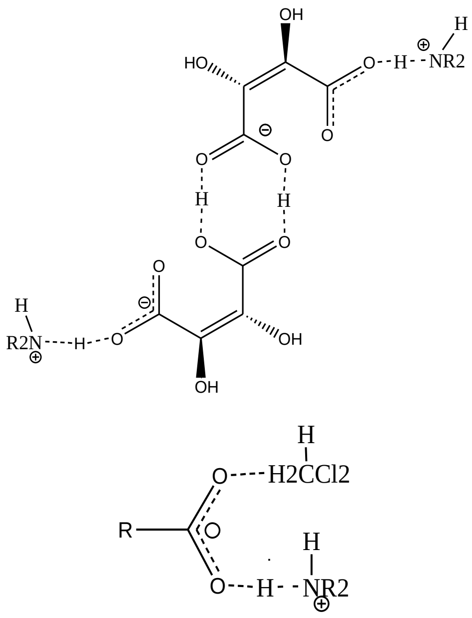

Figure 5: The mechanism of acid adsorption in the organic phase

CONCLUSION

Tartaric acid was successfully extracted from the aqueous solution by an extraction process using Amberlite LA-2 as extract dissolved in DCM as an organic solvent. The distribution coefficients of tartaric acid in the extraction process had values greater than 1 indicating that the distribution of the acid in the organic layer is higher than in the aqueous layer, and the highest value of the distribution coefficient was 221.2. High loading coefficients ranging from 0.054 - 1.318 were obtained. The increase in the concentration of tartaric acid in the medium also led to a decrease in the distribution coefficient. The extraction efficiency of tartaric acid decreases with increasing acid concentration and increases with increasing Amberlite LA-2 and the highest extraction values of 99.5% were obtained. By increasing the pH values, the distribution coefficient value and the extraction efficiency increase. With increasing temperature, the value of the distribution coefficient and the extraction efficiency increase.

ACKNOWLEDGMENTS

We would like to thank the Chemistry Department of Sebha University for supporting this work and the Libyan Petroleum Institute in Tripoli for their efforts in providing some of the chemical analysis techniques.

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