

Analyses of Ethylenediaminetetraacetic Acid (Edta) in Selected Binary Immiscible Solvents: The Role of Partition Coefficient, k_D

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ABSTRACT

Background: The focus is on the extraction of Ethylenediaminetetraacetic acid from contaminants using binary immiscible solvents. The molecular distributions of EDTA in the binary solvents that are involved are the key factors that determine the magnitude of the partition coefficient.

Methods: Ethylenediaminetetraacetic acid was investigated in carbon tetrachloride-water, diethyl ether-water, and n-hexane-water binary solvents, at 30°C and atmospheric pressure, to determine the partition coefficients and the influence of interfering reactions, such as dimerization, ionization, etc. occurring with the distributions of the acid in the binary solvents and to ascertain the best binary solvent for the analyses of the acid. These analyses were done by titration. EDTA solute was introduced into the solvents and the concentrations of the pure molecules were calculated from the titration measurements.

Results: Ethylenediaminetetraacetic acid has the distribution coefficient, k_D value of 1.9213 in carbon tetrachloride-water system with the dimerization constant, K of -30.7821. It has the ionization constant, α of 0.0149 in this medium. Ethylenediaminetetraacetic acid has the k_D value of 1.2186 in diethyl ether-water system and dimerization constant of -23.8972 with the ionization constant of 0.0687. The k_D of EDTA in n-hexane-water system is 0.2035 with the dimerization constant of 2329.7237 and ionization constant of 0.0005.

Conclusion: Distribution coefficient technique is used for solute-solvent extraction, and the beneficiation of minerals. A high distribution coefficient shows preferential solubility in the solvent that is involved and hence good separation. From the three systems used, carbon tetrachloride-water and diethyl ether-water systems are good for the analyses of Ethylenediaminetetraacetic acid. Carbon tetrachloride-water system is the best binary immiscible solvent, from the three systems investigated, for the extraction and purification of the acid. Good extraction and purification of EDTA in the recommended solvents will be done in several continuous steps.

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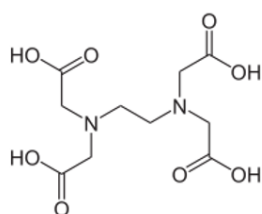
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Introduction

Ethylenediaminetetraacetic acid (EDTA) is an aminopolycarboxylic acid with the formula $[\text{CH}_2\text{N}(\text{CH}_2\text{CO}_2\text{H})_2]_2$. [www.m.wikipedia.org] Ethylenediaminetetraacetic acid forms the ion EDTA^{4-} . This ligand forms complexes with many metal ions, including Pb^{2+} . It is used as an antidote to lead poisoning. [www.ethylenediaminetetraacetic acid].



Structure of EDTA

It is used to bind to iron and calcium ions. It is a hexadentate chelating agent. In industry, EDTA is used to sequester metal ions in aqueous solution. It prevents metal ion impurities from modifying colours of dyed products in the textile industry. In the pulp and paper industry, it inhibits the ability of metal ions like Mn^{2+} , from catalyzing the disproportionation of hydrogen peroxide that is used in chlorine-free bleaching. It is added to food as a preservative/stabilizer to prevent catalytic oxidative discoloration caused by metal ions. In soft drinks that contains ascorbic acid and sodium benzoate, EDTA inhibits the formation of carcinogens like benzene. [www.analytice.com/en/analysis-of-ethylenediaminetetraacetic-acid-edta-cas-60-00-4-in-soils/] It is used to reduce water hardness, and to dissolve scale in both laundry and boiler applications. Cleaning solutions contain EDTA. Also cement industry uses EDTA. It is used in agriculture and hydroponics. In medicine, derivatives of EDTA are used in chelation therapy. It is used in dentistry. It is used as a preservative in ocular preparations and eye drops. It is used in nuclear medicine, in evaluating kidney function. EDTA is used in the analysis of blood. It is used in cosmetics, in laboratory

applications. The importance of EDTA touches all aspects of life. [www.m.wikipedia.org]

Due to the numerous applications of EDTA, questions have been raised to consider it as a persistent organic pollutant. The continued existence of EDTA poses serious issues in the environment. The degradation of EDTA is slow. It occurs abiotically in the presence of sunlight. The most important process for the elimination of EDTA from surface waters is direct photolysis at wavelengths below 400nm. In many industrial wastewater treatment plants, EDTA elimination is achieved at about 80% using microorganisms. In this process, both the backbone and acetyl groups are attacked. The most sensitive method of detecting and measuring EDTA in biological samples is selected reaction monitoring capillary electrophoresis mass spectrometry. EDTA has been measured in non-alcoholic beverages using high performance liquid chromatography (HPLC), at a level of 2.0 $\mu\text{g/ml}$. [www.analytice.com/en/analysis-of-ethylenediaminetetraacetic-acid-edta-cas-60-00-4-in-soils/]

Because of the versatile nature of EDTA in the industry, agriculture, and all aspects of life, many analyses are done on it always. These analyses are directed by the nature and scope of the studies. There have been analyses using the mass spectrometry in biological samples, the HPLC in abiotic systems, the use of microorganisms etc. In all these analyses, low levels of EDTA have been achieved and there have been significant disorientation of the molecule (EDTA) in many of the processes. In wastewater treatments that involve the use of microorganisms, both the EDTA backbone and the acetyl groups are attacked resulting to the production of the byproducts – ethylenediaminetriacetic acid (ED3A) and iminodiacetic acid (IDA). EDTA is not eliminated by sewage treatment plants. It is therefore released into rivers and lakes. Since EDTA is not retained by activated carbon filters, it can also contaminate drinking water [www.analytice.com/en/analysis-of-ethylenediaminetetraacetic-acid-edta-cas-60-00-4-in-soils/]

Analysis has proven a quantitative determination of EDTA in food [1]. EDTA in food samples was extracted with water by ultra-sonication. It was first converted to Fe (III) ions complex in the presence of Fe (III) ions and EDTA was separated on a reversed – phase column, and detected with ultraviolet detection 260 nm). This method allows determination of EDTA in foods at concentrations as low as 0.01 mmol/kg. Good recoveries 95.2 – 101%) were obtained by the standard addition method on four samples with high repeatability (RSD, 0.8 -3.4%). The method was successfully applied to the analysis of EDTA in carbonated drinks, jellies, canned beans, canned corn, and food supplements. Analysis of organic ligands such as EDTA is a necessary challenge due to their ability to increase the mobility of radionuclides and metal. Reactive desorption electrospray ionization mass spectrometry (reactive – DESI – MS) was used for direct analysis of EDTA on concrete samples. EDTA forms complexes and those with Fe (III) ions are among the most thermodynamically favored ones. This complexing capacity was used to improve the specific detection of EDTA directly on a concrete matrix by doping the solvent spray of DESI with a solution of FeCl_3 to selectively create the complex between EDTA and Fe (III) [2]. In the “Determination of Acid in Boiler Water by Liquid Chromatography”, tetra sodium ethylenediaminetetraacetate (Na_4EDTA) is an active ingredient in boiler treatment formulations, and it has been used for metal oxide solubilization and for maintenance of metal ions in sequestered state in solution. At a high temperature of 200°C, EDTA decomposes to form iminodiacetate and other degradation

products. It is necessary that a procedure be devised that would allow the determination of total EDTA in boiler water [3]. A mathematical formula that describes the efficiency of extraction of metals as chelates, from aqueous into organic phase was derived [spiral.imperial.ac.uk].

The analysis of EDTA in this research used the titration method as described by Onyeocha et al. in: “The Dimerization Effects of Some Solutes on the Partition Coefficient (k_D) in Binary Immiscible Solvents” and ‘Comparisons of the effects of solute interactions on partition coefficient, k_D , in selected binary immiscible solvents: a case of oxalic acid and succinic acid’ [4, 5]. In this method, a binary solvent was formed and the EDTA solute was dissolved in it. When the system reached equilibrium, the concentrations of EDTA in the two phases were calculated, and the partition coefficient of EDTA in the binary system was calculated from the concentrations. Solvent extraction is the process in which a compound transfers from one solvent to another owing to the difference in solubility or distribution coefficient between these two immiscible or slightly soluble solvents. Compared with other separation methods, it gives a better separation effect than chemical precipitation, and a higher degree of selectivity and faster mass transfer than the ion exchange method. Compared with distillation, solvent extraction has advantages such as low energy consumption, large production capacity, fast action, easy continuous operation and ease of automation [www.sciencedirect.com]. Solvent extraction is a process in which compounds are separated based on their relative solubilities. It involves using a solvent, a fluid that has the ability to dissolve another substance. It is used to separate hazardous contaminants from sludge and sediments as well [resource.temarry.com]. It is used across multiple industries. Solvent extraction and partitioning is a method used to separate compound or metal complexes based on their relative solubilities in two different immiscible liquids, usually water (polar) and an organic solvent (non-polar). There is the net transfer of one or more species from one liquid into another liquid phase, generally from aqueous to organic. The transfer is driven by chemical potential. When the transfer is complete, the overall system of chemical components that make up the solutes and the solvents are in a more stable configuration or lower energy. [www.en.wikipedia.org/wiki/liquid] It is a basic technique in chemical laboratories. It is performed with a variety of apparatus, from separator funnels to countercurrent distribution equipment [www.m.wikipedia.org]. A high distribution coefficient shows preferential solubility in the solvent and hence good separation. [www.sciencedirect.com].

In this work, Ethylenediaminetetraacetic acid was used as the solute. It was sourced from open market. The dissolution/distribution of EDTA in binary immiscible solvents was considered in the following liquids: water, carbon tetrachloride, diethyl ether and n-hexane for the formation of the binary immiscible solvents. EDTA dissolves in water; it does not dissolve in organic solvent except in the presence of a base [www.reddit.com>comment>EDTA]. The application of partition coefficient is a direct consequence of the laws of thermodynamics [6]. The driving force which is defined in the partition coefficient values shows the efficiency of the application of this principle. The use of partition coefficient technique to predict the mode of extraction has long been in practice in solvent-solute extraction [7]. This research has introduced a conscious study and novel beneficiation parameters, as in thermodynamics, to checkmate the efficiency of solute extraction in binary solvents. The findings herein are in accordance with the predictive parameters such as, Enthalpy,

H, and Gibb's Free Energy, G as in thermodynamics. These findings justify the statement that " k_D , is the driving force for solute extraction from binary solvents. This is in consistence with the predictive parameters in thermodynamics". The driving force which is defined by the partition coefficient values determines the efficiency of k_D application as described in this study. Systems left alone in nature run down to a stage where the observable properties that describe such systems become independent of time [6]. For example, water runs down a hill. This is because a property gradient is formed in the manner of position gradient. When a binary immiscible solvent is used as discussed in this work, efficiency of solvent extraction is enhanced. This is because a partition coefficient gradient is created with the solute in the binary immiscible solvents. Partition coefficient is the driving force of solute extraction in binary immiscible solvents.

The Solutes and Solvents

In this work, the Ethylenediaminetetraacetic acid and the other compounds, and solvents used are sourced from open market. The solvents used for the analyses in this research are water, carbon tetrachloride, n-hexane and diethylether.

Acid-Base Titration

The need for acid-base titration cannot be over emphasized. Strong acids and strong bases dissociate completely in solutions. The molarities of strong acids and strong bases directly show that the hydronium ion concentration $[H_3O^+]$, and the hydroxide ion concentration $[OH^-]$ can be calculated. Solutions of weak acids, such as Ethylenediaminetetraacetic acid and acetic acid, are different. The $[H_3O^+]$ and $[OH^-]$ cannot be calculated directly because all the molecules of the weak acids are not ionized or dissociated. This is the same with weak bases. The pH's of these solutions are measured experimentally by titration and the $[H_3O^+]$ and $[OH^-]$ are then calculated from the measured pH values [www. Ethylenediaminetetraacetic acid].

Determination of pH (titrations) using Indicators

An approximate value for the pH of a solution could be obtained using acid-base indicators. Acid-base indicators are compounds whose colours are sensitive to pH. The colours of these indicators change as the pHs of the solution change. Indicators are either weak acids or weak bases. In solution, a weak acid indicator undergoes the reaction:



(Where $In^{(-)}$ is the symbol of the anion part of the indicator) Thus, HIn and $In^{(-)}$, are different colours.

The Various Concentrations of Solute

Concentration is expressed as moles (n) of solute per liter of solvent. By considering the iodine solute dissolved in the carbon tetrachloride-water system as reported by Rastogi et. al.[6,8]

$$\frac{\text{concentration of } I_2 \text{ in } CCl_4 \text{ layer}}{\text{concentration of } I_2 \text{ in } H_2O \text{ layer}} = k_D \quad (2)$$

Concentration could also be expressed as mole fraction, then with the case of the iodine solute dissolved in the carbon tetrachloride-water system,

$$\frac{\text{mole fraction of } I_2 \text{ in } CCl_4 \text{ layer}}{\text{Mole fraction of } I_2 \text{ in } H_2O \text{ layer}} = k_D \quad (3)$$

The temperature, the nature of the solute, and the properties of the binary immiscible solvents affect the magnitude of the partition

coefficient [9, 10].

Thermodynamic Expression of k_D

When equilibrium is reached in the distribution of solute molecules in a binary immiscible solvent, the chemical potential (μ) of the solute in the two solvents is equal. [6]

$$\text{Thus, } \mu^A = \mu^B \quad (\text{at equilibrium}) \quad (4)$$

Generally;

$$\mu = \mu^\circ + RT \ln a \quad (5)$$

Where a = activity of the solute in the solution, R is the gas constant and T is the temperature.

Therefore, at equilibrium, equation (4) becomes;

$$\mu^\circ A + RT \ln(a)^A = \mu^\circ B + RT \ln(a)^B \quad (6)$$

Thus

$$\ln \frac{a^A}{a^B} = \frac{\mu^{0B} - \mu^{0A}}{RT} \quad (7)$$

The pure component at the standard state shows that,

$$\mu^{\circ A} = \mu^{\circ B} \quad (8)$$

Hence;

$$RT \ln(a)^A = RT \ln(a)^B \quad (9)$$

$$\text{Simplifying shows that } (a)^A = (a)^B \quad (10)$$

OR

Taking from equation 7,

$$\frac{a^A}{a^B} = \text{Exp} \left(\frac{\mu^{0B} - \mu^{0A}}{RT} \right) \quad (11)$$

μ° is a function of temperature only; therefore, the right hand side of equation (11) is constant at a given temperature [11, 12]. Thus,

$$\frac{a^A}{a^B} = \text{constant} = k_D \quad (12)$$

Further, μ° is independent of concentration, so k_D is also independent of concentration too.

Solute and Solvent Interactions: Solvent-Solvent Interaction

Consider the illustration,

Solvent [A] + solvent [B] \rightarrow miscible binary solution (AB)

Solvent [A] + solvent [B] \rightarrow immiscible binary solvent (A + B)

Solvent-solute interaction

Solvent [A] + solvent [B] + solute [C] \Rightarrow (i) solvent [A] + solute [C] + reactions

(ii) solvent [B] + solute [C] + no reactions

Solvent [A] + solute [C] \Rightarrow (i) Dimerization reaction

(ii) Ionization reaction

Partition Law for a Chemical Reaction System

Consider the equation,



In equation (13), the solute, X, (for example EDTA), disperses into the solvent, A, with the involvement of a chemical reaction to form the products C and D. The partition coefficient, k_D , shows the degree of the distribution of the solute in the binary solvent.

EDTA is a tetravalent molecule that is capable of forming a stable complex with divalent ions such as Ca^{2+} . [www.sciencedirect.com] Polyamide amines are obtained when Ethylenediaminetetraacetic acid and ethylene diamine condense, in solid state [13]. The formation of dimers, anhydrides, and ions in the binary immiscible solvents affects the distribution of solutes in the binary immiscible solvents.

When dimerization of the solute (X) occurs in one of the liquids, the dimerization reaction is represented by equation (14):

$$\frac{C_X^A}{C_X^{*B}} = k_D + 2k_D^2 K C_X^B \quad (14)$$

A plot of $\left(\frac{C_X^A}{C_X^{*B}}\right)$ vs C_X^{*B} is a straight line with intercept

k_D and a slope that is equal to $2k_D^2 K$

This will enable the calculation of dimerization constant, K. When ionization of the solute (X) occurs in one of the liquids, the ionization constant, α , is given by the equation (15).

Thus:

$$\frac{C_X^A}{C_X^{*B}} = k_D + (Kk_D)^{1/2} (C_X^{*B})^{-1/2} \quad (15)$$

A plot of $\frac{C_X^A}{C_X^{*B}}$ vs $\frac{1}{(C_X^{*B})^{1/2}}$ gives a straight line. The slope is

equal to $(Kk_D)^{1/2}$ from which K is calculated. K is the ionization symbolized as α .

When both dimerization and ionization reactions are taking place in solvents A and B respectively, equation (16) is applied.

$$\frac{C_X^A}{C_X^{*B}} = k_D(1-\alpha) + 2k_D^2 K(1-\alpha)^2 C_X^{*B} \quad (16)$$

Equations (14), (15) and (16) were applied in the analyses of the Tables and graphs as used in Onyeocha et al. and Rastogi et al. [4-5, 8].

Background of the Study

Among the numerous uses and applications of EDTA in life, this research considers the recovery of EDTA from contaminants when it is used to reduce water hardness, and to dissolve scale in both laundry and boiler applications, and in cleaning solutions. The interactions and dissolutions of EDTA with solvents at the temperature of 30° C and atmospheric pressure were studied. Changing concentrations of the solute within the solvents were analyzed. These analyses were done by titration method. The literature review on the properties and reactivities of EDTA were underscored. Its interactions in organic solvents and water

were studied. Characteristic reactions such as dimerization and ionization of the solute were marked. These reactions and the distribution of the molecular solute in the dissolving solvent are the outcome of the solvent-solute interactions. Interfering reactions like the characteristic dimerization and ionization reaction affect and lowers the partition coefficient of the solute in the binary immiscible solvents.

Brief Note

- Solute-solvent interaction gives rise to solubility of the solute in the solvent.
- In binary immiscible solvent, the greater the solute-solvent interaction, the greater solubility in the solvent in preference to another solvent with limited interaction.
- This principle is developed for use in solvent extraction of solutes.
- Temperature variation effects could also be applied in the analyses.

Statement of the Problem

Three binary solvents were proposed for the solute that was assessed. A good condition for the titration analyses was underscored. The availability of solutes and the solvents was checked for the materials. Realizing that most solutes are insoluble in organic solvents, immiscible binary solvents were prepared for the analyses in attempts to get appreciable k_D from the solute-solvent interactions.

Objective of the Study

The distribution coefficient values (k_D) for the solute in the respective binary immiscible solvents were determined. Evaluation of the values of k_D would establish the nature and type of solute-solvent and solvent-solvent interactions. Also, the degree of the interfering reactions like association, ionization and dimerization were assessed from the values of k_D . Most importantly, k_D values were used to determine the most appropriate solvents for the purification of the impure solute in the process of solvent extraction.

Justification of the Study

Solvent extraction is significant in the isolation of drugs, beneficiation of minerals, and separation of mixed minerals. Nature endowed man with important minerals, molecules, and compounds. Tetraoxosulphate (VI) acid (H_2SO_4) for example is a versatile inorganic solvent which is of great importance to man. Many organic compounds such as acetic acid, oxalic acid, succinic acid, and Ethylenediaminetetraacetic acid (EDTA), to mention a few are also of great importance, and have continued to be used for the benefit of man. The continued production of these materials in their pure states and applications, and recycling of these products have necessitated the research. This study employed the distribution coefficient technique in order to establish the binary solution(s) for the beneficiation of Ethylenediaminetetraacetic acid (EDTA). It will show the degree of the characteristic and interfering reactions of this solute in the respective binary immiscible solvents.

Scope of Study

A thorough literature review on EDTA, to underscore the physical properties, chemical properties and reactivates in the selected solvents that were used for the analyses was made. This would assist in appreciating the nature and type of molecular interactions EDTA would have in the solvents. The formulations of binary immiscible solvents were based on the properties of the solvents. The analyses were carried out using titrations, and the concentrations of the solute in the solvents were then calculated.

Methods

AnalaR Grade materials were used for the experiment [14]. The reagents were Ethylenediaminetetraacetic acid, carbon tetrachloride, diethyl ether, n-hexane, distilled water, 0.1 M sodium hydroxide solution. Model FA1204 drawell digital precision balance, with a serial number: FA-1505028 and origin 50 software for graphs were used for the measurements. The various solutions of the binary immiscible solvents were prepared to get the stock solutions as shown below [8, 15]. This method was used in the analyses of acetic and succinic acids in binary immiscible solvents, as described by Onyeocha et. al. [4-5] All measurements were done at 30°C and atmospheric pressure.

Preparation of Sodium Hydroxide Solution (0.1M NaOH)

4g of sodium hydroxide solid was weighed into 500ml of distilled water in a conical flask and the solution was formed as described by Onyeocha et. al., [4, 5].

Preparation of Phenolphthalein Solution

1g of phenolphthalein solid was measured into 50ml of 90% ethanol and 50ml of distilled water. The indicator solution was prepared as described by Onyeocha et al., for the titrations [4, 5].

Preparation of Binary Immiscible Solvents I and the Determination of Partition Coefficient, k_D from the Binary Immiscible Solvent, Method I

As described by Onyeocha et al., 25.00 ml of distilled water was measured using a clean measuring cylinder into a separating funnel [4, 5]. 25.00 ml of carbon tetrachloride was measured using another clean measuring cylinder, and was poured into the same separating funnel with the water. This mixture forms the binary immiscible solvent.

0.40 g of EDTA was measured out using weighing balance. It was weighed into a binary immiscible solvent system of 25.00 ml of water and 25.00 ml of carbon tetrachloride in a separating funnel. The separating funnel was shaken up for 15-20 minutes by hand, for proper mixing of the content. The system was allowed to stand for 60 minutes until equilibrium was reached and separation of the organic and aqueous layer achieved. The lower organic layer was separated by hand, by pouring it gradually into a clean beaker while the upper aqueous layer was separated into another clean beaker. These steps were repeated for 0.60 g, 0.80 g, and 1.0 g of EDTA to create four systems.

10.00 ml of the organic phase of the 0.4 g system was titrated with 0.10 mol sodium hydroxide per 1 liter of water, with phenolphthalein as the indicator. The titration was carried out until a pink color was obtained as the end point was reached. The volume of sodium hydroxide used was recorded. 10.00 ml of the aqueous phase was also titrated with 0.10 M sodium hydroxide again using phenolphthalein as the indicator until the end point was reached. Again, the volume of sodium hydroxide used was recorded. The experiment was repeated separately for the 0.60g,

0.80g, and 1.0g systems of EDTA acid. The concentrations of EDTA acid in the two phases were calculated.

Preparation of Binary Immiscible Solvents II and the Determination of Partition Coefficient, k_D from the Binary Immiscible Solvent

As recorded by Onyeocha et al., the method for preparation of binary immiscible solvents I (method I) was exactly repeated using diethyl ether and water instead of carbon tetrachloride and water, with 0.40g, 0.60g, 0.80g, and 1.0 g of EDTA acid respectively [4, 5]. The concentrations of EDTA acid in the two layers were also calculated.

Preparation of Binary Immiscible Solvents III and the Determination of Partition Coefficient, k_D from the Binary Immiscible Solvent

As reported by Onyeocha et al., the method for preparation of binary immiscible solvents I (method I) was exactly repeated using n-hexane and water instead of carbon tetrachloride and water, with 0.40 g, 0.60 g, 0.80 g, and 1.0 g of EDTA acid respectively [4, 5]. The concentrations of EDTA acid in the two layers were also calculated [16].

Summary on the Determination of Partition Coefficient

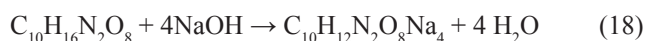
Method I was used to determine the partition coefficients of the acid in the binary immiscible solvents. Every system of each binary solvent has two phases, aqueous and organic, which were separated manually by pouring each partition gradually into a beaker. The partition coefficient is given by “concentration of the acid in the organic layer (layer A) / concentration of the acid in the aqueous layer (layer B)”. Taking the case of EDTA in n-hexane-water system, the partition coefficient is given as, “concentration of EDTA in n-hexane/concentration of EDTA acid in water”.

The equation:

$$\text{The equation } C_A V_A / C_B V_B = \text{Mole ratio} \quad (17)$$

Was used to calculate the concentration or the relative amount of the acid in the two solvents of the binary immiscible solvent systems.

The reaction of EDTA and NaOH gives:



Where the volume of base (NaOH) used for the titration = V_B

The concentration of the base, $C_B = 0.1M$

The volume of the acid (EDTA), = $V_A = 10ml$

The concentration of acid, C_A , was calculated.

The results from this research are shown in Tables 1 – 10 and Figures 1 – 9 below.

Table 1: Data for the partition coefficient (k_D) of EDTA in carbon tetrachloride-water at 30°C and atmospheric pressure

Mass of EDTA	Concentration in carbon tetrachloride (A) (C_X^A) (mole/litre)	Concentration in water (B) (C_X^B) (mole/litre)	$\frac{C_X^A}{C_X^B}$ (k_D)
0.4	0.0038	0.0038	1.0000
0.6	0.0038	0.0063	0.6032
0.8	0.0025	0.0025	1.0000
1.0	0.0025	0.0013	1.9231

Table 2: Data for the partition coefficient of EDTA in carbon tetrachloride-water at 30°C and atmospheric pressure for the plot: C_X^A/C_X^{*B} vs $1/(C_X^{*B})^{1/2}$ equation (15).

C_X^A/C_X^{*B}	$1/\sqrt{C_X^{*B}}$ (mole/litre) ^{-1/2}
1.0000	16.2338
0.6032	12.5945
1.0000	20.0000
1.9231	27.7008

Table 3: Data for the partition coefficient of EDTA in carbon tetrachloride-water at 30°C and atmospheric pressure for the plot $C_X^A/C_X^{*B}(1-\alpha)$ vs $C_X^{*B}(1-\alpha)$ equation (16)

C_X^A/C_X^{*B}	$C_X^A/C_X^{*B}(1-\alpha)$	C_X^{*B} (mole/litre)	$C_X^{*B}(1-\alpha)$ (mole/litre)
1.0000	0.9851	0.0038	0.0037
0.6032	0.5942	0.0063	0.0062
1.0000	0.9851	0.0025	0.0025
1.9231	1.8944	0.0013	0.0013

Table 4: Data for the partition coefficient (k_D) for EDTA in the binary solvents: diethyl ether-water at 30°C and atmospheric pressure

Mass of EDTA (g)	Concentration in diethyl ether (C_X^A) (mole/litre)	Concentration in water (C_X^{*B}) (mole/litre)	$\frac{C_X^A}{C_X^{*B}}(k_D)$
0.4	0.0013	0.0025	0.5200
0.6	0.0038	0.0025	1.5200
0.8	0.0038	0.0038	1.0000
1.0	0.0050	0.0100	0.5000

Table 5: Data for the partition coefficient of EDTA in diethyl ether-water at 30°C and atmospheric pressure, for the plot C_X^A/C_X^{*B} vs $1/(C_X^{*B})^{1/2}$ from equation (15)

C_X^A/C_X^{*B}	$1/\sqrt{C_X^{*B}}$ (mole/litre) ^{-1/2}
0.5200	20.0000
1.5200	20.0000
1.0000	16.2338
0.50000	10.0000

Table 6. Data for the partition coefficient of EDTA in diethyl ether-water at 30°C and atmospheric pressure for the plot of equation (16)

C_X^A/C_X^{*B}	$C_X^A/C_X^{*B}(1-\alpha)$	C_X^{*B} (mole/litre)	$C_X^{*B}(1-\alpha)$ (mole/litre)
0.5200	0.4843	0.0025	0.0023
1.5200	1.4156	0.0025	0.0023
1.0000	0.9313	0.0038	0.0035
0.5000	0.4657	0.0100	0.0093

Table 7. Data for the partition coefficient (k_D) for EDTA in n-hexane-water at 30°C and atmospheric pressure

Mass of EDTA (g)	C_X^A/C_X^{*B}	C_X^{*B} (mole/litre)
0.4	0.6500	0.0020
0.6	0.2778	0.0018
0.8	0.5333	0.0015
1.0	0.7222	0.0018

Table 8. Data for the partition coefficient of EDTA in n-hexane-water at 30°C and atmospheric pressure for the plot C_X^A/C_X^{*B} vs $1/(C_X^{*B})^{1/2}$ from equation (15)

C_X^A/C_X^{*B}	$1/\sqrt{C_X^{*B}}$ (mole/litre) ^{-1/2}
0.6500	22.3714
0.2778	23.5849
0.5333	25.8398
0.7222	23.5849

Table 9: Data for the partition coefficient of EDTA in n-hexane-water at 30°C and atmospheric pressure for the plot: $C_X^A/C_X^{*B}(1-\alpha)$ vs $C_X^{*B}(1-\alpha)$ equation (16)

C_X^A/C_X^{*B}	$C_X^A/C_X^{*B}(1-\alpha)$	C_X^{*B} (mole/litre)	$C_X^{*B}(1-\alpha)$ (mole/litre)
0.6500	0.6497	0.0020	0.0020
0.2778	0.2777	0.0018	0.0018
0.5333	0.5330	0.0015	0.0015
0.7222	0.7218	0.0018	0.0018

Table 10. The partition coefficient, k_D , dimerization constant, K , and ionization constant, α , for ethylenediaminetetraacetic acid (EDTA) in the binary immiscible solvents: carbon tetrachloride-water, diethyl ether-water and n-hexane-water respectively at 30°C and atmospheric pressure

EDTA	Distribution coefficient k_D	Dimerization constant, K	Ionization constant α	Association & ionization constant
Carbon tetrachloride-water	1.92128	-30.7821	0.0149	
Diethylether-water	1.21856	-23.8972	0.0687	-27.62
n-hexane-water	0.20346	2329.7237	0.0005	-2337.0768

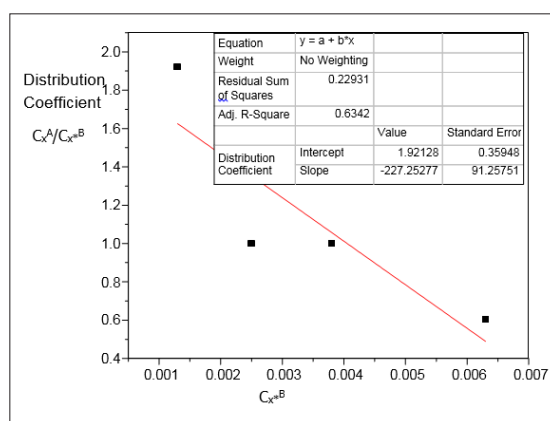


Figure 1: Plot for C_X^A/C_X^{*B} vs C_X^{*B} at 30°C and atmospheric pressure for EDTA in carbon tetrachloride-water, from equation (14).

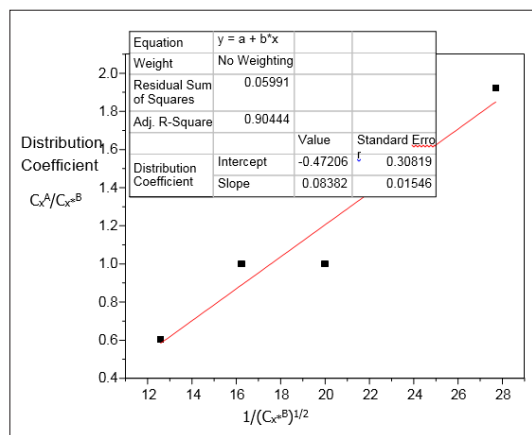


Figure 2: Plot of C_X^A/C_X^{*B} vs $1/(C_X^{*B})^{1/2}$ for the ionization of EDTA acid in water of carbon tetrachloride-water at 30°C and atmospheric pressure from equation (15).

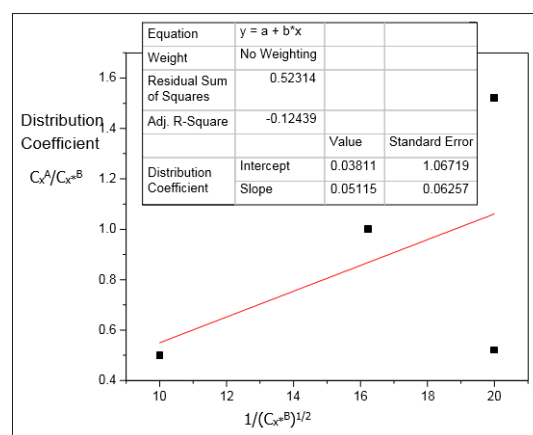


Figure 5: Plot for C_X^A/C_X^{*B} vs $1/(C_X^{*B})^{1/2}$ for the ionization of EDTA in water of diethyl ether-water at 30°C and atmospheric pressure, from equation (15).

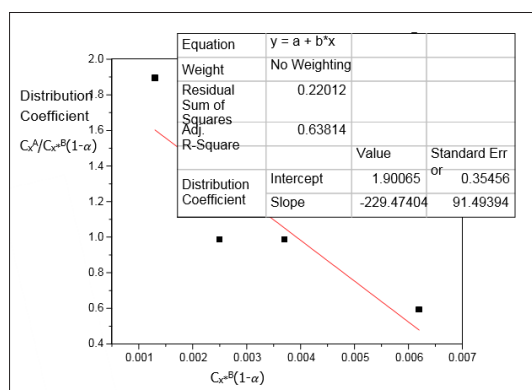


Figure 3: Plot for $C_X^A/C_X^{*B}(1-\alpha)$ vs $C_X^{*B}(1-\alpha)$ for the association and ionization of EDTA in carbon tetrachloride-water at 30 °C and atmospheric pressure

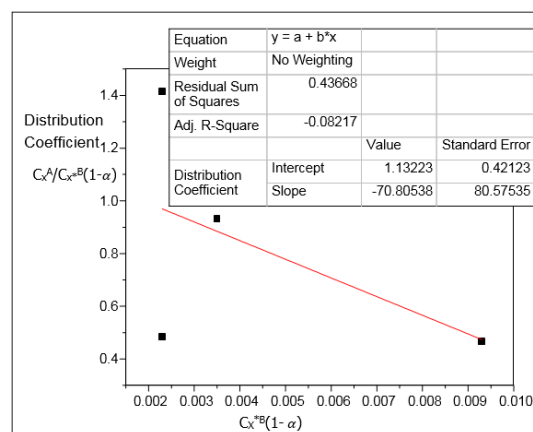


Figure 6: Plot for EDTA in diethyl ether-water at 30°C and atmospheric pressure for the plot $C_X^A/C_X^{*B}(1-\alpha)$ vs $C_X^{*B}(1-\alpha)$, equation (16).

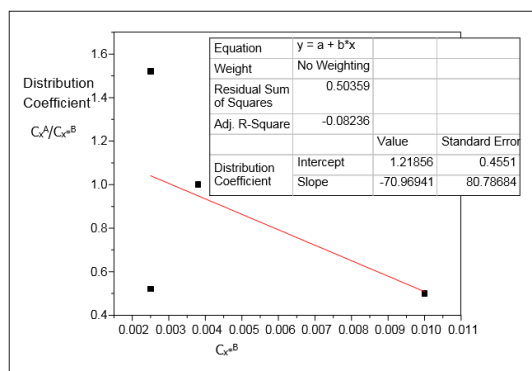


Figure 4: Plot for C_X^A/C_X^{*B} vs C_X^{*B} for EDTA in diethyl ether-water at 30°C and atmospheric pressure, from equation (14).

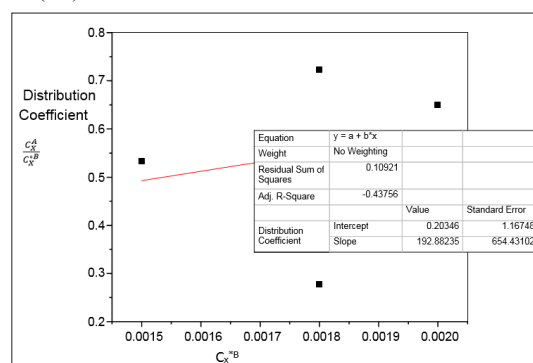


Figure 7: Plot of $\frac{C_X^A}{C_X^{*B}}$ against C_X^{*B} for EDTA in n-hexane-water at 30°C and atmospheric pressure.

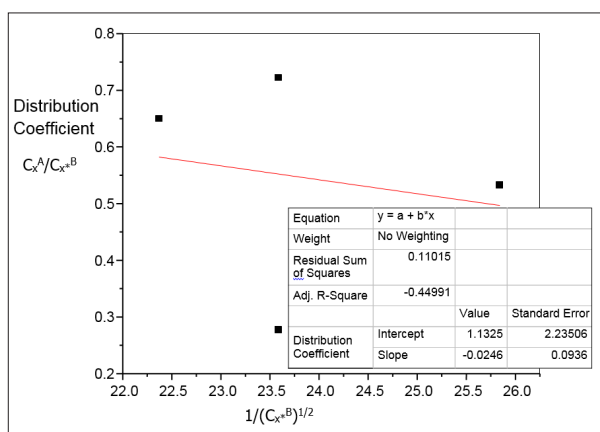


Figure 8: Plot for C_X^A/C_X^B vs $1/(C_X^B)^{1/2}$ for EDTA in n-hexane-water at 30°C and atmospheric pressure.

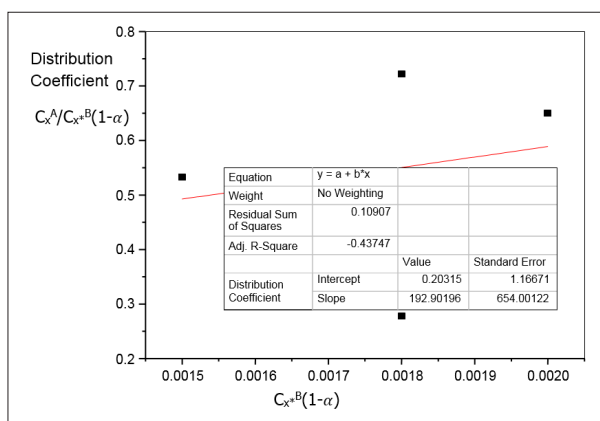


Figure 9: Plot for $C_X^A/C_X^B(1-\alpha)$ vs $C_X^B(1-\alpha)$ for EDTA in n-hexane-water at 30°C and atmospheric pressure.

Results and Discussion

The values of the partition coefficient (k_D) for ethylenediaminetetraacetic acid (EDTA) in carbon tetrachloride-water at 30°C and atmospheric pressure were calculated as shown in Table 1.

Partition coefficient reflects the solubility of a solute in the organic and aqueous layers. It is dependent on the solvent system used. EDTA has the appreciable partition coefficients, k_D , of 1.9213 in carbon tetrachloride-water, and k_D of 1.2186 in diethyl ether-water systems respectively. EDTA has the dimerization constant, K , of -30.7821, in carbon tetrachloride-water, and dimerization constant, K , of -23.8972 in diethylether-water systems respectively. Both carbon tetrachloride-water and diethyl ether-water systems are recommended for the analyses of EDTA. Carbon tetrachloride-water system is the best binary immiscible system for the analyses of EDTA from the three binary immiscible systems that are investigated.

Conclusion

The use of binary immiscible solvent in the extraction and recovery of Ethylenediaminetetraacetic acid (EDTA) is recommended for boiler treatment, and sewage treatment. It is cost effective and efficient. Both carbon tetrachloride-water and diethyl ether-water solvents are good. Carbon tetrachloride-water system has more appreciable partition coefficient and is therefore the best binary solvent. EDTA forms dimer in the organic solvents too. Dimers are pure forms of the constituting molecule.

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