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Experiment Number (2)

Liquid-Liquid Equilibrium

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Abstract:

This experiment's goal is to explore liquid-liquid equilibrium when a solute (Acetone) is dissolved in a polar solution (water) and then transferred due to the difference in the chemical potential transferred to a second immiscible or partially immiscible organic non-polar solvent (Toluene). Cloudiness (turbidity) was the indicator of the three components being in equilibrium. The solvent that is enriched in solute is referred to as an extract, as is the solvent that is enriched in feed liquid and modest concentrations of solute. But in this experiment, the acetone is only partially dissolved by the raffinate and extract. The ternary phase diagram was plotted using the equilibrium points observed from different compositions for the solute and both solvents.

main Results

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Results:

Table (1): Densities at 20 °C in g/ml.

Water density	Toluene density	Acetone density
0.9982	0.8669	0.7900

1-Ternary mutual solubility curve formation

A. For water rich phase

Table (2): Water rich phase results.

Acetone volume (ml)	Water volume (ml)	Toluene volume (ml)	Acetone mass (g)	Water mass (g)	Toluene mass (g)	Total mass (g)	Acetone fraction (by mass)	Water fraction (by mass)	Toluene fraction (by mass)
5.00	20.00	0.70	3.950	19.964	0.621	24.535	0.161	0.814	0.025
10.00	20.00	1.00	7.900	19.964	0.887	28.751	0.275	0.694	0.031
15.00	20.00	0.60	11.850	19.964	0.532	32.346	0.366	0.617	0.016
10.00	10.00	1.40	7.900	9.982	1.242	19.124	0.413	0.522	0.065
20.00	10.00	1.40	15.800	9.982	1.242	27.024	0.585	0.369	0.046
30.00	10.00	5.00	23.700	9.982	4.435	38.117	0.622	0.262	0.116

B. For organic solvent rich phase

Table (3): Organic rich phase results.

Acetone volume (ml)	Toluene volume (ml)	Water volume (ml)	Acetone mass (g)	Toluene mass (g)	Water mass (g)	Total mass (g)	Acetone fraction (by mass)	Toluene fraction (by mass)	Water fraction (by mass)
5.00	20.00	0.30	3.950	17.738	0.299	21.987	0.180	0.807	0.014
10.00	20.00	0.30	7.900	17.738	0.299	25.937	0.305	0.684	0.012
15.00	20.00	0.30	11.850	17.738	0.299	29.887	0.396	0.593	0.010
10.00	10.00	0.20	7.900	8.869	0.200	16.969	0.466	0.523	0.012
20.00	10.00	0.70	15.800	8.869	0.699	25.368	0.623	0.350	0.028
30.00	10.00	4.00	23.700	8.869	3.993	36.562	0.648	0.243	0.109

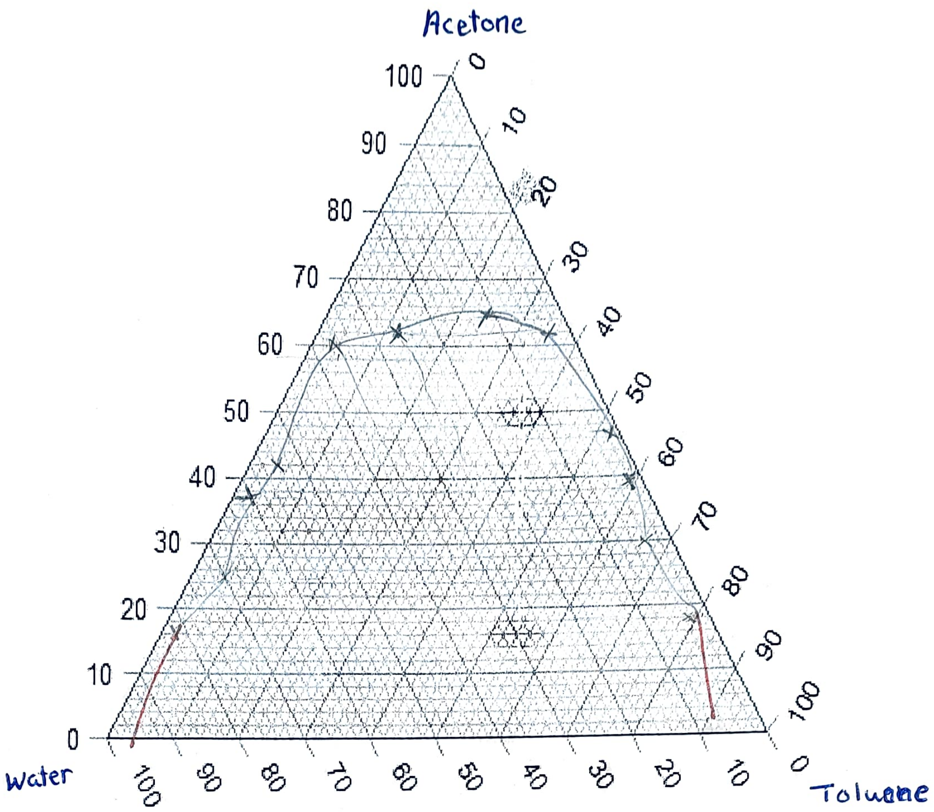

Figure (1): Mutual solubility curve for ternary system of acetone, water, and toluene.

2-Tie Line determination:

Table (4): Tie-Line determination data.

sample no.	water volume (ml)	Toluene volume (ml)	Acetone volume (ml)	water mass (g)	Toluene mass (g)	Acetone mass (g)	Total mass (g)	water mass fraction	Toluene mass fraction	Acetone mass fraction
1	18	20	13	17.968	17.338	10.270	45.576	0.394	0.380	0.225
2	26	17	8	25.953	14.737	6.320	47.011	0.552	0.313	0.134
3	19	29	3	18.966	25.140	2.370	46.476	0.408	0.541	0.051

Table (5): Tie-line composition and othmer-Tobias Correlation.

sample no.	RI of water layer	RI of toluene layer	Raffinate		Extract		othmer -Toblas correlation	
			Mass fraction of acetone in water layer	Mass fraction of water in water layer	Mass fraction of acetone in toluene layer	Mass fraction of toluene in toluene layer	log ((1-a)/a)	log ((1-b)/b)
1	1.3499	1.4709	0.226	0.774	0.21	0.79	-0.535	-0.575
2	1.348	1.481	0.197	0.803	0.137	0.863	-0.610	-0.799
3	1.3388	1.4893	0.05	0.95	0.075	0.925	-1.279	-1.091

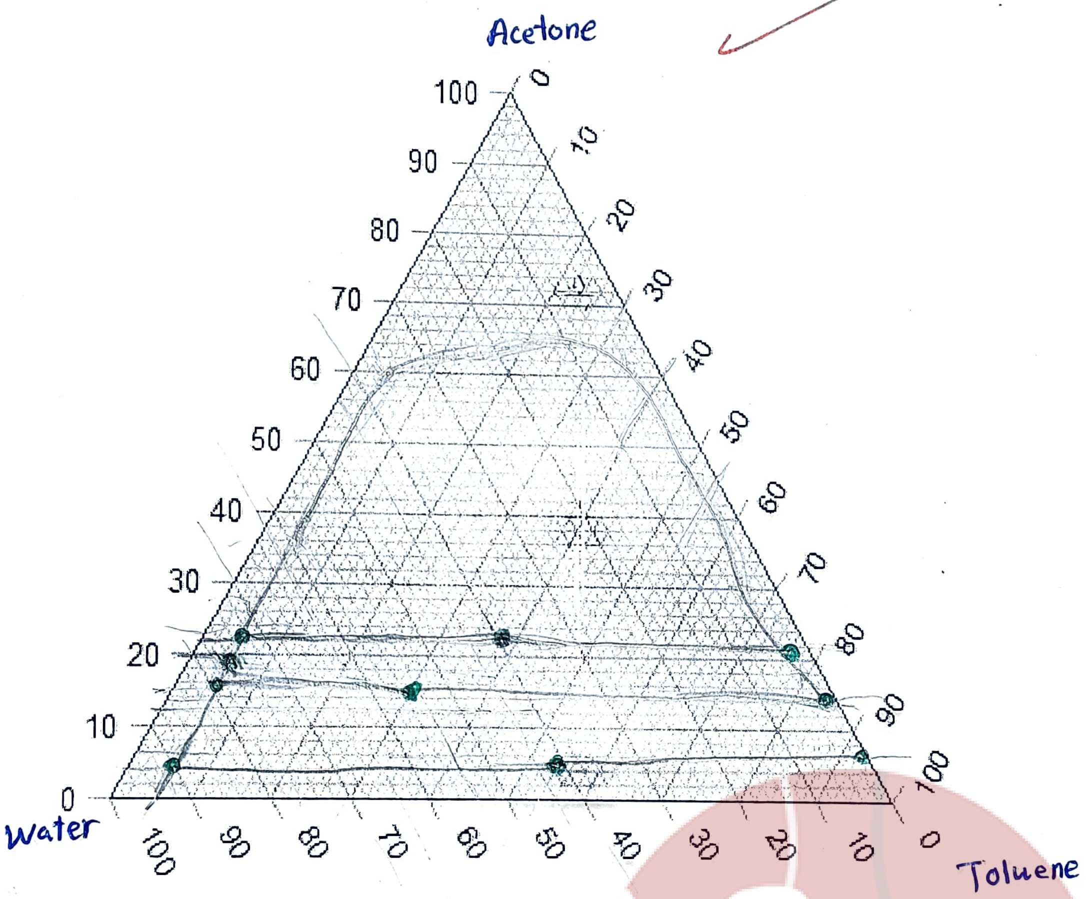

Figure (2): Ternary diagram of acetone, water, and toluene. Represent Tie-line.

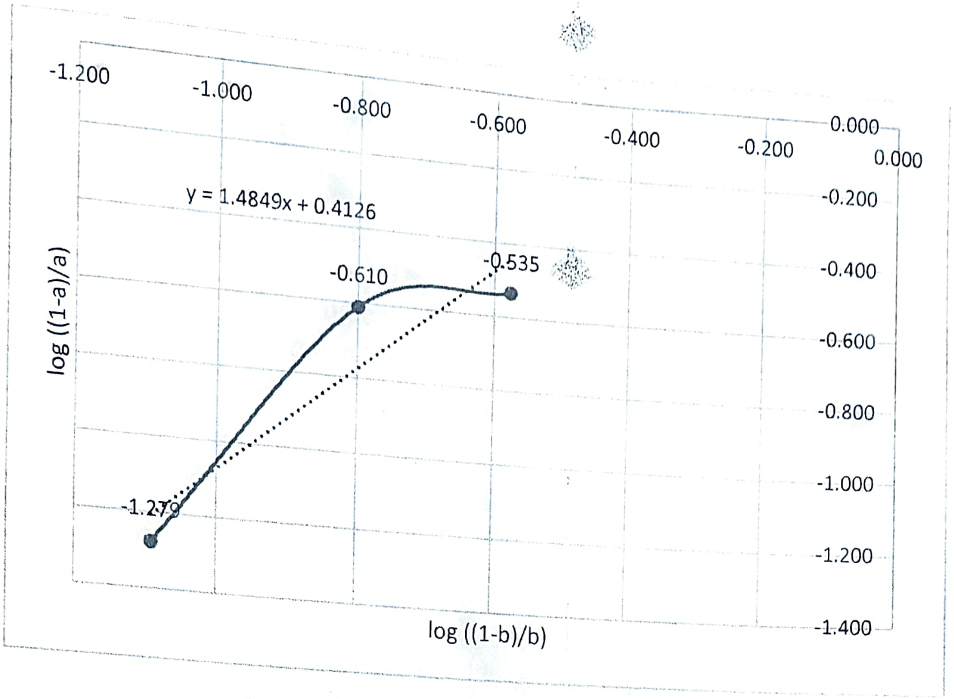

Figure (3): Othmer-Tobias Correlation.

Discussion

A phase diagram is a visual representation of a substance's physical states under various pressures, composition, and temperatures.

The main objective of using Phase diagrams is to illustrate the composition and the phase of a single or multiple component system. There are ternary diagrams, a sort of phase diagrams, which are triangular diagrams that depict the phase behavior of the three components of a combination under constant temperature and pressure. There are two varieties of ternary diagrams: equilateral triangles and the right triangle.

For this experiment, to represent all the equilibrium states between the extract and the raffinate layers after the separation process is carried out, ternary diagrams are used, an individual precise tie line is used to represent each state of equilibrium.

For a system made up of water, toluene, and acetone that is performed in a laboratory to gather the necessary experimental data, a ternary diagram is created. According to Table (2): Results for the water rich phase were obtained by taking six samples of the water rich phase and titrating them with toluene until cloudiness appears. This shows that the solution has changed from a one-phase to a two-phase region. The data is then plotted on a ternary diagram to create the water rich side of the solubility equilibrium curve. Comparatively, six samples of a solvent high in toluene were produced and titrated with water until turbidity was seen. The results are shown in Table (3). These findings were plotted to create the solvent side of the solubility equilibrium curve.

These two sides can be combined with a single line to generate a fully soluble line that divides the diagram into two regions: one phase and two phases. Figure (1): Acetone, water, and toluene ternary diagram, showing solubility equilibrium curve.

As you can clearly see, there is a variance in sample number six of the water-rich side (final row). This is a sign of an error, perhaps it is a personal error, that is in determining the cloudiness point or taking the correct reading from the burette.

The plait point should be determined, which is a point where the composition of the solvent-rich phase and the water-rich phase are equal, but it is challenging to identify due to sample composition range.

Using table (4): Tie-lines determination data lists the three samples, to generate the tie-lines. These samples represent a homogeneous mixture that, after settling in the cells, separated into two nonhomogeneous phases: water, acetone, and toluene, acetone. To determine the composition, the reflect index meter (RI) was used to evaluate the two phases.

refractive

As shown in Table (5): Tie-line composition and Othmer - Tobias Correlation. Three samples gave three tie-lines drawn as best fit for the three points that is mentioned before which is shown in Figure (2): Ternary diagram of acetone, water, and toluene, represents Tie-lines.

Figure (3): Othmer - Tobias Correlation, to check for linearity where slope = 1.484 and intercept =0.4126

Conclusion

The experimental phase diagram is affected by personal errors, To identify the plait point, numerous samples must be taken. Cloudiness and turbidity both represent the observable change from a one phase mixture to a two phase mixture.

The physical states of a substance at various pressures, compositions, and temperatures are depicted in a phase diagram.

It is evident that sample number six from the side with more water differs from the others (final row). This is a sign that the cloudiness point was calculated incorrectly or the burette was properly interpreted—possibly a personal error.

References

1- Manual Chemical Engineering Laboratory (2), Faculty of Engineering and Technology, Department of Chemical Engineering, The University of Jordan ,1/11/2022.

2- Seader, Henley and Roper, Separation Processes Principles -Chemical and Biochemical Operation, Chapter 4. John Wiley and Sons Inc., Third Edition ,1998.

3- Merck (2022). Pure Water Density Standard 1/11/2022 from https://macro.lsu.edu/howto/solvents/toluene.htm.

4- Toluene Solvent Properties , Acetone Solvent Properties https://www.sigmaaldrich.com/JO/en/product/sial/denwat1/11/2022.

Appendix

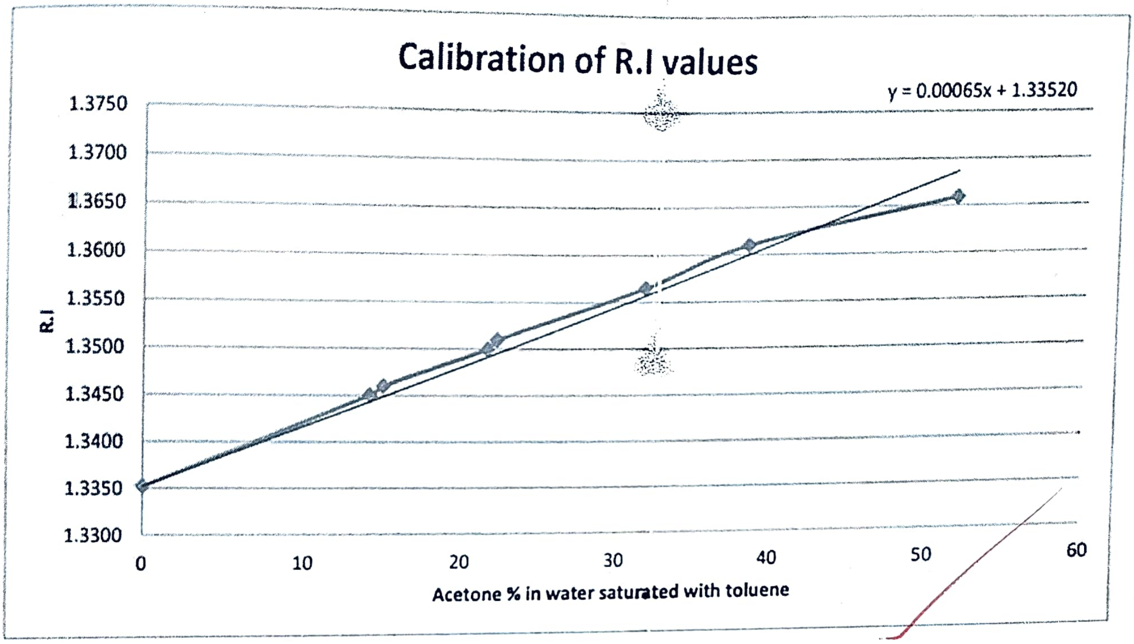

Figure (4): Wt % of Acetone in water saturated with Toluene Vs Refractive Index

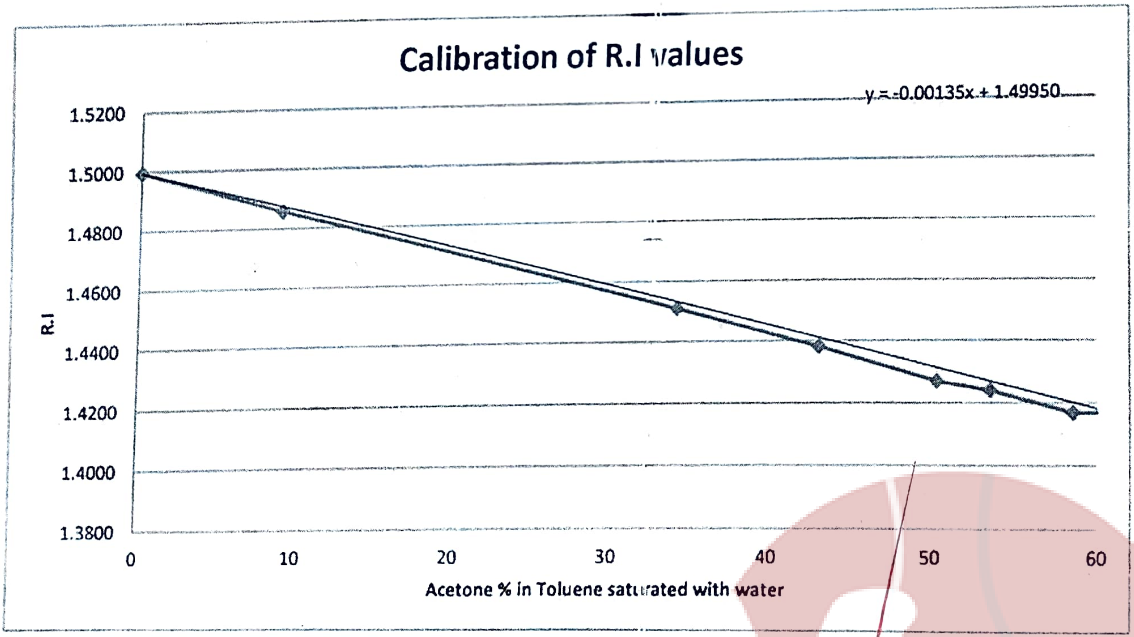

Figure (5): Wt % of Acetone in Toluene saturated with water Vs Refractive Index

Sample of calculation:

(The calculations for the first row of each table will be shown)

- ❖ Mass = Density * Volume
- ❖ Mass fraction = $\frac{\text{mass of component}}{\text{total mass}}$
- ❖ Total mass = water mass + toluene mass +acetone mass

1.Ternary mutual solubility curve formation

1. For water rich phase (from table 2):

➢ Volume of Water: 20.00 ml.

Density of Water at 20 °C: 0.9982 g/ml.

Mass of Water = 20.00 ml * 0.9982 $\frac{g}{ml}$ = 19.964 g.

➢ Volume of Acetone: 5.00 ml.

Density of Acetone at 20 °C: 0.7900 g/ml.

Mass of Acetone = 5.00 ml *0.7900 $\frac{g}{ml}$ = 3.95 g.

➢ Volume of Toluene: 0.70 ml.

Density of Toluene at 20 °C: 0.8669 g/ml.

Mass of Toluene = 0.70 ml * 0.8669 $\frac{g}{ml}$ = 0.607 g.

Total mass = 19.964 g + 3.95 g + 0.607 g = 24.521 g.

Water mass fraction = $\frac{19.964\ g}{24.521\ g}$ = 0.814

Acetone mass fraction = $\frac{3.95\ g}{24.521\ g}$ = 0.161

Toluene mass fraction = $\frac{0.607\ g}{24.521\ g}$ = 0.248

2. For Organic rich phase (from table 3):

➢ Volume of Water: 0.30 ml.

Density of Water at 20 °C: 0.9982 g/ml.

Mass of Water = 0.30 ml * 0.9982 $\frac{g}{ml}$ = 0.299 g.

- Volume of Acetone: 5.00 ml.

  Density of Acetone at 20 °C: 0.7900 g/ml.

  Mass of Acetone = 5.00 ml *0.7900 $\frac{g}{ml}$ = 3.95 g.

- Volume of Toluene: 20.00 ml.

  Density of Toluene at 20 °C: 0.8669 g/ml.

  Mass of Toluene = 20.00 ml * 0.8669 $\frac{g}{ml}$ = 17.338 g.

Total mass = 0.299 g + 3.95 g + 17.338 g = 21.587 g.

$$\text{Water mass fraction} = \frac{0.299\text{ g}}{21.587\text{ g}} = 0.014$$

$$\text{Acetone mass fraction} = \frac{3.95\text{ g}}{21.587\text{ g}} = 0.183$$

$$\text{Toluene mass fraction} = \frac{17.338\text{ g}}{21.587\text{ g}} = 0.803$$

2.Tie-line determination:

A. Tie-line determination (from table 4):

- Volume of Water: 18.00 ml.

  Density of Water at 20 °C: 0.9982 g/ml.

  Mass of Water = 18.00 ml * 0.9982 $\frac{g}{ml}$ = 17.968 g.

- Volume of Acetone: 13.00 ml.

  Density of Acetone at 20 °C: 0.7900 g/ml.

  Mass of Acetone = 13.00 ml *0.7900 $\frac{g}{ml}$ = 10.270 g.

- Volume of Toluene: 20.00 ml.

  Density of Toluene at 20 °C: 0.8669 g/ml.

  Mass of Toluene = 20.00 ml * 0.8669 $\frac{g}{ml}$ = 17.338 g.

Total mass = 17.968 + 17.338 + 10.27 = 45.576 g

Water mass fraction = $\frac{17.968\ g}{45.576\ g} = 0.394$

Acetone mass fraction = $\frac{10.27\ g}{45.576\ g} = 0.225$

Toluene mass fraction = $\frac{17.338\ g}{45.576\ g} = 0.380$

B. Othmer – Tobias correlation (from table 5):

1. Finding (a) value:

RI of water layer =1.3499

From calibration curve of RI (values from figure (4)), the calibration equation y = 0.00065x + 1.33520

Mass fraction of acetone in water saturated with toluene, x= $\frac{(y-1.33520)}{0.065}$, applying this:

$$x=\frac{(1.3499-1.33520)}{0.065} = 0.226$$

Since we need to find the weight fraction of the solvent in the extract phase:

mass fraction of water in water layer =1 - mass fraction of acetone =1 – 0.226 = 0.774

2. Finding (b) value:

RI of toluene layer =1.4709

From calibration curve of RI (values from figure (5)), the calibration equation y = -0.00135x + 1.49950

Mass fraction of acetone in toluene saturated with water, x= $\frac{(y-1.4995)}{-0.135}$, applying this:

$$x=\frac{(1.4709-1.4995)}{-0.135} = 0.21$$

Since we need to find the weight fraction of the carrier liquid in the raffinate phase:

Mass fraction of toluene in toluene layer =1 - mass fraction of acetone = 1- 0.21 = 0.79

3. Checking reliability by Othmer -Tobias correlation

The reliability of equilibrium data for any system may be tested by applying the Othmer-Tobias correlation. This correlation states that:

$$\log\frac{(1-a)}{a} = n\log\frac{(1-b)}{b} + s$$

there is a linear relation between the values of ($\log\frac{(1-a)}{a}$) and ($\log\frac{(1-b)}{b}$) ,where (a) represents water fraction in water layer, while (b) represents toluene fraction in toluene layer.

As sample of calculation, x, and f(x) can be found as below:

- x= $\log(\frac{(1-b)}{b}) = \log(\frac{(1-0.774)}{0.774})$ = -0.535
- F(x)= $\log(\frac{(1-a)}{a}) = \log(\frac{(1-0.79)}{0.79})$ = -0.575
- The same calculation has been applied on the other points and plotted to find the linear expression for the resulting line.

سدة يحى نور ليلى تالين

Liquid-Liquid Equilibrium Data Sheet

Tie-Lines Determination:

Volume of water (ml)	Volume of Toluene (ml)	Volume of Acetone (ml)	RI of water layer	RI of Toluene layer
20	15	15		
→ 18	20	13	1.3499	1.4709
→ 26	17	8	1.3480	1.4810
20	25	5		
→ 19	29	3	1.3388	1.4893.

Solubility curve

A. Water rich phase:

Volume of Acetone (ml)	Volume of water (ml)	Volume of Toluene (ml)
5	20	0.70
10	20	1.00
15	20	0.60
10	10	1.40
20	10	1.40
30	10	5.00

B. Organic solvent rich phase:

Volume of Acetone (ml)	Volume of Toluene (ml)	Volume of water (ml)
5	20	0.30
10	20	0.30
15	20	0.30
10	10	0.20
20	10	0.70
30	10	4.00

Instructor signature:	
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