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Effect of Diluent on the Extraction of Oxoethanoic (Glyoxylic) Acid by *N*,*N*-Dioctyloctan-1-amine (TOA)

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ABSTRACT: The extractability of aqueous solutions of oxoethanoic acid by *N*,*N*-dioctyloctan-1-amine (TOA) in various diluents, including isoamyl alcohol, octan-1-ol, nonan-1-ol, decan-1-ol, methyl ethyl ketone, diisobuthyl ketone, hexan-2-one, toluene, kerosene, and *n*-hexane has been investigated. Batch extraction results at 25 °C have been calculated and reported as distribution coefficients (K_D), loading factors (Z), and extraction efficiencies (E). With these solvents, using TOA has been found to be very effective ($K_D \gg 1$). Decan-1-ol was found to be the most effective diluent with the highest value of K_D (13.43) at 1.37 $\text{mol}\cdot\text{kg}^{-1}$ concentration of TOA. The results of the liquid−liquid equilibrium measurements were correlated with a linear solvation energy relationship (LSER) model that takes into account physical interactions as well as with modified Freundlich and Langmuir equations. The experimental results for the oxoethanoic acid extraction are compared to the model results.

1. INTRODUCTION

Oxoethanoic acid is present in the body as the primary precursor of oxalic acid and as an intermediate in the conversion of glycolic acid to glycine. The metabolic pathway for conversion of fats to carbohydrates in certain microorganisms and plants is called the glyoxylate cycle. Glyoxylates (any salt, anion, or ester of oxoethanoic acid) are important in biochemical research. Pure oxoethanoic acid is a liquid with melting point of −93 °C and boiling point of 111 °C. Oxoethanoic acid is formed in commerce by oxidation of glycolic acid or by ozonolysis (oxidation) and reduction of maleic acid.[1] Therefore, it is essential to purify oxoethanoic acid from aqueous solution. In this study, the reactive extraction separation technique was used for recovery of oxoethanoic acid. Reactive extraction is an efficient, economical, and environmentally friendly method for the recovery of organic acids from aqueous solution. Furthermore, it is applicable in the recovery of acids from waste streams, facilitation of processes through separation of acidic products or byproducts, recovery of acidic products, and so forth. Extractive recovery of carboxylic acids from dilute aqueous solutions has received increasing attention.[2−5] Some studies of oxoethanoic acid extraction from aqueous solution using various extractants can be found in the literature. Li et al.[6] reported the extraction of oxoethanoic acid from aqueous solution using a trialkylphosphine oxide (phosphor-bonded) extractant. Bauer et al.[7] investigated selective extraction of oxoethanoic acid from its aqueous mixture with glyoxal using aliphatic or cycloaliphatic alcohols containing 4 to 8 carbon atoms. Extraction of glycolic acid and oxoethanoic acid from aqueous solution using trialkylphosphine oxide in methyl isobutyl ketone and *N*,*N*-dioctyloctan-1-amine (trioctylamine, TOA) in 1-octanol was studied by Qin et al.[8]

The main objective of the research described in this paper is to go further into the study of the extraction of oxoethanoic acid from aqueous solutions using TOA in order to gain a better understanding of the extraction equilibria of the acids. Extraction equilibria of oxoethanoic acid using TOA in various diluents (alcohols, ketones, and inert diluents) were investigated, and linear solvation energy relationship (LSER), modified Freundlich, and modified Langmuir models of the equilibria of oxoethanoic acid reactive extraction have been satisfactorily applied to the experimental results.

2. MATERIALS AND METHODS

2.1. Chemicals. TOA ($M = 353.67\ \text{g}\cdot\text{mol}^{-1}$), oxoethanoic acid, and the diluents were purchased from the Merck Company. All of the chemicals were used without further purification. The mass-fraction purities of the solvents all were greater than 0.98.

2.2. Analysis Methods. An aqueous solution of oxoethanoic acid with an initial concentration of 0.93 $\text{mol}\cdot\text{kg}^{-1}$ (0.08 in mass) was prepared. Extraction experiments involved shaking equal volumes (20 mL) of the aqueous and organic phases for 6 h in a temperature-controlled shaker at 25 °C and 40 rpm speed followed by settling of the mixture for at least 2 h at the same temperature in an incubator. It was found in preliminary tests that 6 h was sufficient time for the attainment of equilibrium. The pH of the aqueous phase was measured using a pH meter. The aqueous-phase acid concentration was determined by titration with 0.1 N NaOH. The relative uncertainty of the aqueous-phase determination did not exceed 3 %. The acid content in the organic phase was determined using mass balance.

3. RESULTS AND DISCUSSION

3.1. Equilibrium Results. *3.1.1. Complex-Formation and Distribution Coefficients.* The intent of carboxylic acids is to form dimers in the organic phase because of their intermolecular hydrogen bonding.[5] Complex formation via the interfacial reaction is represented by eq 1:

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Table 1. Results of Reactive Extraction of Oxoethanoic Acid by TOA in Various Diluents

	$\overline{C}_S^{\text{initial}}$ [a]	$\overline{C}_{HA}$ [b]				E [f]
diluent	mol·kg^{-1}	mol·kg^{-1}	K_D^{model} [c]	K_D [d]	Z [e]	%
isoamyl alcohol	0.23	0.34	0.51	0.59	1.50	36.91
	0.46	0.45	0.97	0.93	0.98	48.26
	0.69	0.57	1.72	1.60	0.83	61.61
	0.91	0.67	2.56	2.59	0.73	72.12
	1.14	0.74	3.78	3.93	0.65	79.71
	1.37	0.82	7.82	7.68	0.60	88.48
octan-1-ol	0.23	0.33	0.50	0.55	1.44	35.42
	0.46	0.43	0.81	0.85	0.93	45.81
	0.69	0.56	1.63	1.52	0.82	60.27
	0.91	0.68	2.52	2.74	0.74	73.26
	1.14	0.80	6.21	6.07	0.70	85.86
	1.37	0.84	9.77	9.84	0.61	90.77
nonan-1-ol	0.23	0.21	0.22	0.29	0.92	22.57
	0.46	0.40	0.96	0.75	0.87	42.71
	0.69	0.49	1.25	1.14	0.72	53.27
	0.91	0.61	1.79	1.95	0.67	66.13
	1.14	0.73	3.88	3.60	0.64	78.24
	1.37	0.84	9.65	9.70	0.61	90.66
decan-1-ol	0.23	0.38	0.63	0.69	1.66	40.95
	0.46	0.53	1.35	1.31	1.15	56.79
	0.69	0.64	2.22	2.26	0.94	69.36
	0.91	0.74	3.64	3.88	0.81	79.52
	1.14	0.80	6.25	6.36	0.70	86.41
	1.37	0.86	13.75	13.43	0.63	93.07
kerosene	0.23	0.34		0.58	1.49	36.57
	0.46	0.42		0.83	0.92	45.30
	0.69	0.57		1.60	0.83	61.49
	0.91	0.65		2.37	0.71	70.32
	1.14	0.78		5.12	0.68	83.65
	1.37	0.85		10.36	0.62	91.20
n-hexane	0.23	0.25		0.36	1.08	26.69
	0.46	0.37		0.67	0.82	40.23
	0.69	0.42		0.82	0.61	45.12
	0.91	0.56		1.54	0.61	60.56
	1.14	0.65		2.38	0.57	70.37
	1.37	0.80		6.11	0.58	85.94
toluene	0.23	0.15		0.19	0.64	15.80
	0.46	0.34		0.57	0.74	36.32
	0.69	0.41		0.79	0.60	44.06
	0.91	0.54		1.38	0.59	58.02
	1.14	0.62		1.97	0.54	66.33
	1.37	0.73		3.68	0.53	78.64
DIBK	0.23	0.42		0.83	1.85	45.49
	0.46	0.51		1.23	1.12	55.22
	0.69	0.52		1.30	0.76	56.49
	0.91	0.64		2.24	0.70	69.13
	1.14	0.70		3.13	0.62	75.78
	1.37	0.84		9.42	0.61	90.40
hexan-2-one	0.23	0.09		0.11	0.39	9.58
	0.46	0.26		0.38	0.56	27.48
	0.69	0.38		0.70	0.56	41.02
	0.91	0.51		1.22	0.56	54.90
	1.14	0.63		2.11	0.55	67.82
	1.37	0.76		4.50	0.55	81.83
MEK	0.23	0.15		0.19	0.66	16.16
	0.46	0.29		0.46	0.64	31.54
	0.69	0.40		0.77	0.59	43.39
	0.91	0.42		0.83	0.46	45.47
	1.14	0.56		1.55	0.49	60.79
	1.37	0.71		3.26	0.52	76.54

[a] Initial concentration of TOA in the organic phase. [b] Equilibrium concentration of oxoethanoic acid in the organic phase. [c] Distribution coefficient calculated using the LSER model. [d] Distribution coefficient. [e] Loading factor. [f] Extraction efficiency.

$$qHA(aq) + pS(org) \rightleftarrows HA_qS_p(org) \tag{1}$$

in which HA is the undissociated acid molecule, S is the amine molecule, and p and q are the stoichiometric coefficients. As the oxoethanoic acid−TOA complex forms, it is rapidly extracted into the organic phase.

The distribution coefficient is defined as the ratio of the concentrations of the organic acid in the organic and aqueous phases. The experimentally accessible distribution coefficient (K_D) is given by

$$K_D = \frac{\overline{C}_{HA}}{C_{HA,\text{total}}} = \frac{\overline{C}_{HA_qS_p}}{C_{HA} + C_{A^-}} \tag{2}$$

where $\overline{C}_{HA}$ is the total concentration of the acid in the organic phase, $C_{HA,\text{total}}$ is the total concentration of acid in the aqueous phase, $\overline{C}_{HA_qS_p}$ is the concentration of HA_qS_p complexes in the organic phase, C_{HA} is the concentration of undissociated acid in the aqueous phase, and C_{A^-} is the concentration of A^-, the conjugate base of the acid, in the aqueous phase. In all of the equations herein, overbars refer to the organic phase.

Table 1 presents the experimental results of liquid−liquid phase equilibrium investigations for the distribution of oxoethanoic acid. The TOA concentration in the initial organic solution was (0.23, 0.46, 0.69, 0.91, 1.14, or 1.37) mol·kg^{-1} in isoamyl alcohol, octan-1-ol, nonan-1-ol, decan-1-ol, methyl ethyl ketone (MEK), diisobutyl ketone (DIBK), hexan-2-one, toluene, kerosene, or *n*-hexane as the diluent. The oxoethanoic acid concentration in the initial aqueous phase was 0.93 mol·kg^{-1}. Figure 1 demonstrates the influence of TOA in the organic phase on the distribution coefficient of oxoethanoic acid. It was observed from the experiments that the oxoethanoic acid concentration in the organic phase varied between (0.09 and 0.86) mol·kg^{-1} after the extraction. The distribution coefficient of oxoethanoic acid in that range was between (0.19 and 13.43) in the 10 diluents. The maximum value, K_D = 13.43, was obtained for decan-1-ol at a TOA concentration of 1.37 mol·kg^{-1}. This high value can be explained by the formation of acid−amine complexes, which are affected by the diluents in different ways. Solvation of the complex by the diluent is a critical factor in the extraction of acid. The interactions between the complex and diluent can be divided into general solvation interactions and specific interactions of the diluent with the complex. Polar diluents have been

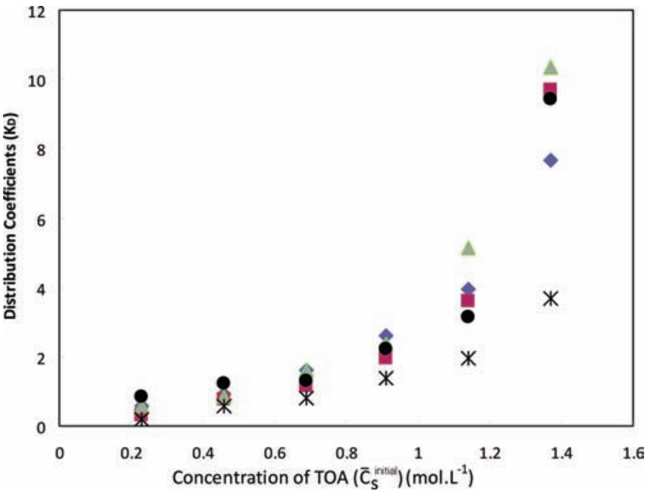

Figure 1. Plots of distribution coefficient (K_D) against the initial concentration of TOA ($\overline{C}_S^{\text{initial}}$): ●, DIBK; ■, nonan-1-ol; ▲, kerosene; ∗, toluene; ◆, isoamyl alcohol.

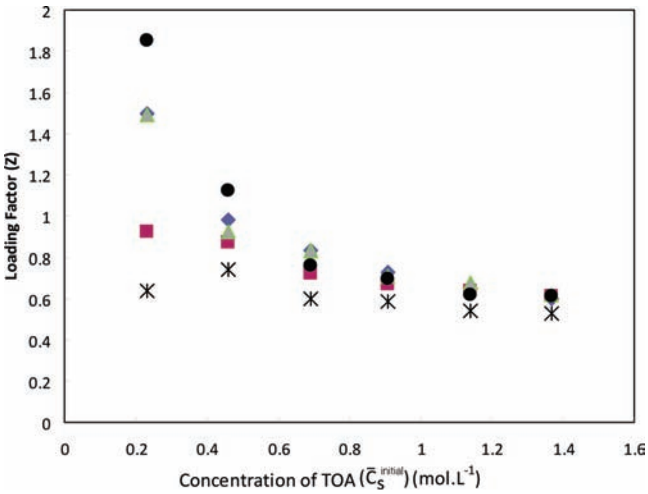

Figure 2. Plots of loading factor (Z) against the initial concentration of TOA ($\overline{C}_S^{\text{initial}}$): ●, DIBK; ■, nonan-1-ol; ▲, kerosene; ∗, toluene; ◆, isoamyl alcohol.

shown to be more convenient diluents than inert (nonpolar) ones because of their larger distribution coefficients. The large difference among the values of the complexation constant for the extraction of oxoethanoic acid by TOA with various diluents indicates that solvation of the complex in different diluents is a critical factor in acid extraction.

3.1.2. Loading Factors and Extraction Efficiencies. The loading factor for the extractant (Z) is defined as the total concentration of acid in the organic phase divided by the total concentration of amine in the organic phase:

$$Z = \frac{\overline{C}_{\text{HA}}}{\overline{C}_{\text{S}}^{\text{initial}}} \tag{3}$$

where $\overline{C}_{\text{HA}}$ is the total concentration of oxoethanoic acid in the organic phase and $\overline{C}_{\text{S}}^{\text{initial}}$ is initial amine concentration in the organic phase. The concentration of TOA in the organic phase ($\overline{C}_{\text{S}}$) can be expressed as

$$\overline{C}_{\text{S}} = \overline{C}_{\text{S}}^{\text{initial}} - p\overline{C}_{\text{HA}_q\text{S}_p} \tag{4}$$

Table 1 shows the effect of TOA concentration on loading. The loading curve in Figure 2 is a plot of the loading factor Z against the TOA concentration. In this work, the loading factor increased with decreasing concentration of TOA for all of the solvent mixtures. Overloading (i.e., $Z > 1$) indicates that complexes with more than one acid per amine were formed. Overloading was observed especially at the amine concentration of 0.23 mol·kg^{-1} for all of the diluents except toluene, MEK, and hexan-2-one in this study.

The extraction efficiency is defined by the following equation:

$$E = \frac{K_{\text{D}}}{1 + K_{\text{D}}} \times 100\ \% \tag{5}$$

It can be seen from Table 1 and Figure 3 that a considerable amount of oxoethanoic acid is removed from aqueous solutions by TOA. The amount of acid removed strongly depends on the concentration of TOA and the diluting solvent. The maximum removal of oxoethanoic acid was 93.07 % for a 1.37 mol·kg^{-1} initial concentration of TOA in decan-1-ol as the diluent. The acid concentration of the organic phase at equilibrium ($\overline{C}_{\text{HA}}$) increased from (0.38 to 0.86) mol·kg^{-1} when the concentration of TOA increased from (0.23 to 1.37) mol·kg^{-1} in decan-1-ol. For the other diluents, the extraction efficiency ranged between (76.54 and 91.20) % at an initial TOA concentration of 1.37 mol·kg^{-1}.

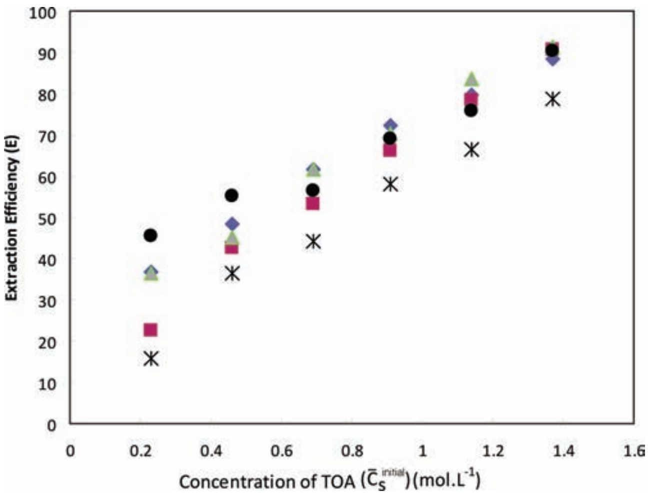

Figure 3. Plots of extraction efficiency (E) against the initial concentration of TOA ($\overline{C}_S^{\text{initial}}$): ●, DIBK; ■, nonan-1-ol; ▲, kerosene; ∗, toluene; ◆, isoamyl alcohol.

3.2. Model Results.

3.2.1. LSER Model Equation. The linear solvation energy relationship (LSER) was initially developed by Kamlet and co-workers.[9] They demonstrated that many types of chemical properties (designated as XYZ), such as aqueous solubility, octanol−water partition coefficient,[10] HPLC capacity factors using a number of mobile and stationary phases,[11] and toxicity to a variety of species,[12] depend on solute−solvent interactions. The model can be expressed in terms of an equation containing three simple and conceptually explicit types of terms:

$$\begin{aligned} \text{XYZ} &= \text{cavity term} + \text{dipolar term} \\ &\quad + \text{hydrogen-bonding terms} \end{aligned} \tag{6}$$

In the LSER model, a chemical property (XYZ) is related to molecular structure through the energy required to surround a

Table 2. Solvatochromic Parameters for Alcohols Used in This Study[9,13]

solvent	π^{*a}	δ^b	β^c	α^d
isoamyl alcohol	0.40	0	0.84	0.84
octan-1-ol	0.40	0	0.81	0.77
nonan-1-ol	0.40	0	0.81	0.75
decan-1-ol	0.40	0	0.81	0.72

[a] Dipolar polarizability parameter. [b] Nonspecific dielectric interaction parameter. [c] Hydrogen-bonding parameter for the solvent as the HBA and the solute as the HBD. [d] Hydrogen-bonding parameter for the solvent as the HBD and the solute as the HBA.

Table 3. Values of the Parameters in the LSER, Modified Freundlich, and Modified Langmuir Equations for Oxoethanoic Acid Extraction by TOA Along with Values of the Correlation Coefficient (R^2) for the Fits

LSER Equation					
$\ln K_D^0$	s	d	a	b	R^2
0.536	−41.854	0	27.162	−3.742	0.95

Modified Freundlich Equation			
alcohol	K_F	n	R^2
isoamyl alcohol	4120	0.493	0.995
octan-1-ol	3673	0.539	0.993
nonan-1-ol	3374	0.740	0.981
decan-1-ol	3625	0.449	0.989

Modified Langmuir Equation			
alcohol	K_L	Q_0	R^2
isoamyl alcohol	−957.9	−0.00098	0.9399
octan-1-ol	−1052.32	−0.00082	0.8855
nonan-1-ol	−1708.6	−0.00037	0.8485
decan-1-ol	−795.887	−0.00113	0.9887

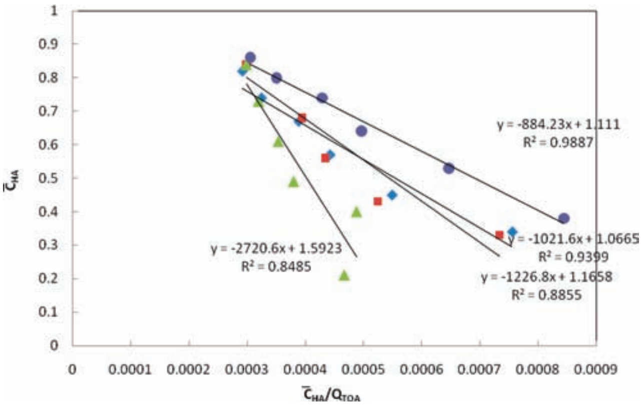

Figure 4. Plots of the modified Langmuir equation for oxoethanoic acid extraction by TOA: ●, decan-1-ol; ■, octan-1-ol; ▲, nonan-1-ol; ◆, isoamyl alcohol.

solute with solvent molecules and the energy gained or lost through formation of electrostatic and hydrogen bonds between the chemical and the medium to stabilize this solvent-molecule cavity and keep the compound inside it. Chemical properties dependent on this solute−solvent interaction are the result of the contribution from the component groups that form the molecule. The energy terms for the component groups then make up the four energy terms for the molecule used in the following predictive equation (for XYZ = K_D):

$$\ln K_D = \ln K_D^0 + s(\pi^* + d\delta) + b\beta + a\alpha \tag{7}$$

In eq 7, the dipolar polarizability parameter π^* represents the exoergic effects of solute−solvent dipole−dipole and dipole−induced-dipole interactions, and δ is a measure of the molecule's ability to stabilize a neighboring charge or dipole through nonspecific dielectric interactions. The hydrogen-bonding parameters α and β represent the exoergic effects of solvent−solute hydrogen bonding involving the solvent as the hydrogen-bond donor (HBD) acid and the solvent as the hydrogen-bond acceptor (HBA) base, respectively. The quantities $\ln K_D^0$, s, d, a, and b in eq 7 are regression coefficients. The values of the distribution coefficient were subjected to regression analysis according to eq 7 using the values of the solvatochromic parameters for the solvents from Table 2;[9,13] the regression was performed using the computer program SPSS version 14.0,[14] and the values of the regression coefficients are presented in Table 3 and eq 8:

$$\ln K_D = 0.536 - 41.854(\pi^* + 0\cdot\delta) + 27.162\beta - 3.742\alpha \tag{8}$$

Calculated values of K_D obtained from the LSER regression are presented in the K_D^{model} column in Table 1.

To assess the suitability of the fit, the root-mean-square deviation (RMSD) value was calculated using the differences between the experimental data (K_D) and the predictions of the LSER model (K_D^{model}) according to the following equation:

$$\text{RMSD} = \sqrt{\frac{1}{N}\sum_{i=1}^{n}\left(K_D - K_D^{\text{model}}\right)^2} \tag{9}$$

where N is the number of experimental data values. The RMSD of the LSER model was determined to be 0.94, which shows that all of the predicted distribution coefficients agree well with each other and that the agreement between the predicted and measured values is acceptable, considering the experimental uncertainty.

3.2.2. Modified Langmuir and Freundlich Equations. The extraction of liquid molecules or solutes in a solution by reactive extractants and interactions of interfacial area are an important phenomenon in physical chemistry, surface science, and industry. The plotting of isotherms is important to obtain an equation that represents the results and could be used for design purposes. These isotherms depend on the specific surface area of the extractant mixture, the nature of the extractant, and the acidity of the medium. The Langmuir equation has been modified for an extractant surface containing a finite number of identical complex sites. The model assumes uniform energies of extraction for interactions in both phases. When an extraction process is carried out at constant temperature, the amount extracted, Q, can be quantitatively described by the Langmuir equation. Poposka et al.[15] have studied the modified Langmuir equation for reactive extraction.

Our modified Langmuir equation is represented by the following equation:

$$\frac{\overline{C}_{HA}}{Q_{TOA}} = \frac{K_1}{Q_0} + \frac{\overline{C}_{HA}}{Q_0} \tag{10}$$

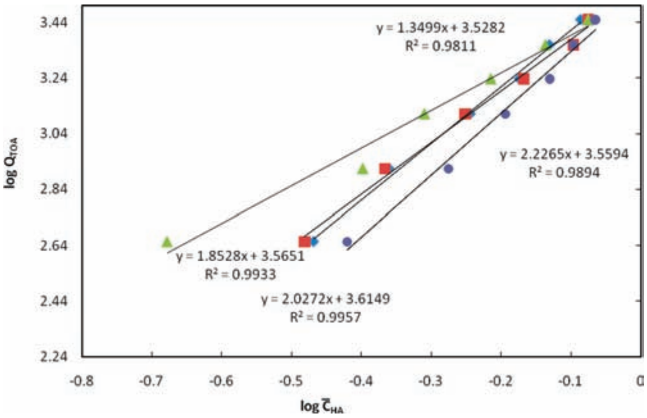

Figure 5. Plots of the modified Freundlich equation for oxoethanoic acid extraction by TOA: ●, decan-1-ol; ■, octan-1-ol; ▲, nonan-1-ol; ◆, isoamyl alcohol.

In eq 10, Q_{TOA} is the amount of extractant (amine), K_1 is the reciprocal of the Langmuir constant (K_L), and Q_0 is a constant obtained from linear regression. When a plot of $\overline{C}_{HA}/Q_{TOA}$ against $\overline{C}_{HA}$ gives a straight line, the applicability of the Langmuir isotherm is demonstrated, and the values of Q_0 and K_1 at different concentrations can be determined from the slope and intercept of the plot, respectively.[16] The modified Langmuir parameters are very useful in predicting extraction capacities and for incorporating a mass-transfer relationship. To verify the Langmuir adsorption isotherm, the ratios $\overline{C}_{HA}/Q_{TOA}$ were plotted against the equilibrium organic-phase acid concentration $\overline{C}_{HA}$ for various diluents (Figure 4). The values of the Langmuir parameters K_L and Q_0 were calculated graphically and are given in Table 3.

The Freundlich equation is used for heterogeneous-surface extractions in which the energy term in the Langmuir equation varies as a function of the surface coverage strictly as a result of variations in the heat of extraction. The modified Freundlich equation takes the following form:

$$Q_{TOA} = K_F \overline{C}_{HA}{}^{1/n} \qquad (11)$$

Taking logarithms of both sides in eq 11 linearizes the equation, enabling the exponent n and the constant K_F to be determined:[17]

$$\log_{10} Q_{TOA} = \log K_F + \frac{1}{n} \log \overline{C}_{HA} \qquad (12)$$

To verify the validity of modified Freundlich equation, log Q_{TOA} was plotted against log $\overline{C}_{HA}$ for various diluents (Figure 5). The linear plots of log Q_{TOA} against log $\overline{C}_{HA}$ indicate the applicability of the modified Freundlich equation. The values of the Freundlich parameters are given in Table 3. The results show that all of the experimental data for the alcohols obey the modified equations well.

4. CONCLUSION

The reactive extraction of oxoethanoic acid using *N*,*N*-dioctyloctan-1-amine dissolved in 10 diluents was investigated. Distribution coefficients, loading factors, and extraction efficiencies for this extraction system were obtained. The LSER model was applied to obtain distribution coefficients for all of the diluents. Decan-1-ol was found to be the most effective diluent, with the highest value of the extraction efficiency (93 %) at a TOA concentration of 1.37 mol·kg^{-1}. The LSER model values showed a good regression to the experimental data, with an R^2 value of 0.95. Modified Freundlich and Langmuir equations were also fitted to the equilibrium data. It was determined that the Freundlich equation is more suitable than the Langmuir equation with respect to the experimental data for alcohols.

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