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Research Article

Effect of Process Variables on Liquid- Liquid Extraction: Toluene-Acetone-Water System

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Abstract

Liquid extraction is a process used to separate fermentative organic products. Several factors like types and concentration of extractants, temperature, packing device are considered in the process and column designs for effective separation. The effect of packing device (Raschig rings, wire gauze rings and stainless steel rings) on the efficiency of liquid-liquid extraction of toluene-acetone-water system was evaluated at 298.15K and atmospheric pressure, using bench and pilot scales. were investigated. In the bench scale experiment, the Raschig rings has the highest transfer unit; HTU (162.52) and column efficiency (0.0062) with respect to extract solute concentration (0.0150) at ±1.8784% confidence limit. Extraction was not achieved in all the 4 stages involved in the pilot scale experiments, except situations when the feed and solvent flowrates were both maximum. The HTU was low compared to the bench scale, for all the packing materials, with Raschig rings having comparably lowest HTU. This portends the pilot scale has better column efficiency for all the packing materials used in the extraction process, with Raschig rings being the best materials for the pilot scale extraction process.

Keywords: Liquid-Liquid Extraction, Process Variables, Packing Rings, Toluene-Acetone-Water System, Pilot Scale, Efficiency.

1. Introduction

Liquid-liquid extraction process is one of the most widely used unit operation in the separation technology especially in chemical, biochemical and petroleum industries. As its name depicts, it is the separation of constituents of the homogeneous liquid mixture. It is done by introduction of a liquid solvent which is miscible or partially miscible with one of the components. To enhance separation of the homogeneous mixture, contact devices are inserted into the extraction column to create mild turbulence, thus allowing rapid mixing of the feed components and the solvent [1]. The design variables affecting the degree of separation vary with equipment. In packed column contactors, efficiency depends on type and size of packing, column diameter, packing depth and type of liquid distributor. The size, shape and material of packing are essential in the design of a packed column. The introduction of packing into the contacting column increases efficiency by decreasing back-mixing or vertical circulation in the continuous phase and increasing mass transfer through continuous coalescence and break up of drops in the dispersed phase [2]. The size of packing influences the height and diameter of column, pressure drop across the column and cost of packing. As the packing size increases, the cost per unit volume of packing and pressure drop per unit height of packing decrease. Thus, mass transfer efficiency increases [3,4].

Packings are broadly divided into two major categories; the broken solids and shaped packings. The broken solids are cheapest form of packings with size ranging from 10mm to 100mm based on the size of the extraction column. They have large interfacial area and are good corrosion resistant material. The shaped packings are the most common types used in chemical plants. They vary in shape (rings, saddles, gauze, protruded metal, helix, etc) and efficiency. Common examples of shaped ring packings are Raschig rings, pall rings and lessing rings. Examples of saddles packings are Berl saddles, intalox saddles and super intalox saddle. The highly efficient metallic gauze packings are made in small sizes (¼ inch or less) and in form of saddles and rings. The high efficiency exhibited by these packings is due to their small sizes and large contact surface area they provide. Also, their large open areas provide them with high capacity and low pressure characteristics. Generally, shaped packings give more effective surface area per unit volume [5,6]. The role and effect of packing in extraction column have been reported in many literatures [7-10]. They suggested that enhancement over and above was caused by a reduction

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in back mixing of the continuous phase, which is due to the increased mixing within the drop arising from distortion and/or coalescence and re-dispersion while passing through the packing. Hence, packing materials tends to have different effects on the efficiency of extraction of liquid-liquid mixture. This work is aimed at studying the effect of packings on extraction of acetone from toluene-acetone system, with water on in bench and pilot scales.

2. Experimental Section

2.1 Materials and Equipment

Abbe refractometer (model M.46) was used to determine refractive index and density measuring bottle was used for density determination [11-13]. The refractometer was fitted with thermo-prism surrounded by water jackets and the temperature of the prism is controlled within ± 0.002 oC by the circulation of cooling water in a controlled environment. The refractometer has a scale ranging from 1.300 to 1.700 with each scale division being equal to 0.001. The prism surfaces were cleaned with water before and after every measurement.

The density measuring bottle used was washed and dried before use. The dry density bottle was weighed before and after introduction of the equilibrium mixture.

2.2 Experimental Procedure

2.2.1 Determination of the Physical Properties of the Systems for Different Composition

The refractive index values of the binary mixtures at different mole fractions were determined while the refractive index and density of the ternary mixtures were determined at different equilibrium compositions using refractometer and density measuring devicebottle. For the binary systems, samples of differential composition varying from 0.1 to 1.0 moles of acetone were prepared using toluene as the second component. This was done by mixing pure component of each at different volumes using a measuring cylinder and burette. The composition (mole) of the mixtures were determined using the following equations:

$$\text{No of mole, } x = \frac{\dfrac{v_i \rho_i}{m_i}}{\sum_{i}^{n} \dfrac{v_i \rho_i}{m_i}} \qquad 1$$

where v = volume, m = molecular weight, n = no of component in the mixture and i = component 1, 2, or 3. The refractive index of these samples were measured and the samples kept in a cool environment to prevent evaporation. The experiment was performed at room temperature.

For the ternary system, the extractant, s, the solute, i and the carrier, j were mixed in a fixed proportion inside a conical flask placed in a water bath with the aid of a burette that contained the solute. The saturation points were first determined by adding small drops of component i to 20ml of solvent s obtained by the addition of a drop of carrier j to form a phase with s and vice-versa. The composition and physical properties of the two samples were calculated and measured respectively. After these, many samples of the heterogeneous system of component j and solvent s were prepared for different compositions between the two saturation points and were titrated with solute i until a homogeneous phase was formed, (i.e. titrated to turbidity). The refractive index and density values were then measured. Finally, the calculated equilibrium composition was plotted on a triangular graph to get the binodal or solubility curve [11-14].

2.2.2 Determination of Liquid-Liquid Equilibrium Data for the Ternary System

Different compositions of the ternary mixture measured into 30ml bottles were shaken vigorously and left for 24 hours to settle and attain equilibrium state. The compositions of the mixture are heterogeneous mixtures and were determined by randomly taking points under the binodal curve (that is, points were taken from the heterogeneous section of the binodal curves) and their composition measured. The refractive index values of resulting two phases after attainment of equilibrium were measured using the refractometer. The corresponding equilibrium homogeneous composition of each phase were determined from the plot of refractive index against composition for each system [11-14].

2.3 Liquid-Liquid Extraction Process

The extraction process was carried out on in bench and pilot scales using three different types of shaped packings mainly- Raschig ring, gauze ring and stainless steel ring - and having approximately same dimensions. Table 1 shows the volume of solvent used for corresponding feed sample on in bench scale. The total volume of both the feed and solvent samples used for pilot scale is 10 times the volume of the feed and solvent samples used for bench scale, though the flowrates of the feed and solvent for the two scales are relatively close in magnitude.

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Identification number	Feed (Acetone + Toluene)			Solvent (Water)
	Mole fraction of acetone	Mole fraction of toluene	Volume (cm^3) of feed used	Volume (cm^3) of solvent used
1C	0.154	0.846	180.0	20.0
2C	0.466	0.534	160.0	40.0
3C	0.897	0.103	140.0	60.0
4C	0.686	0.314	100.0	100.0

Table 1: Corresponding Solvent and Feed Volume (cm^3) in Bench Scale

2.3.1 Experimental Procedure for Bench Scale Process

The apparatus used for bench scale extraction is as shown in Figure 1. Clamped Funnel A and B are 250ml separating funnels, with 8 mm rubber tube connected to each outlet being used to join them at a T-junction that leads to a packed column. The column of length 450mm was packed to a height of 420mm with either Raschig rings, wire gauze rings or stainless steel rings. The packed column then leads to another 250ml separating funnel that separates the resulting mixture into raffinate and extract.

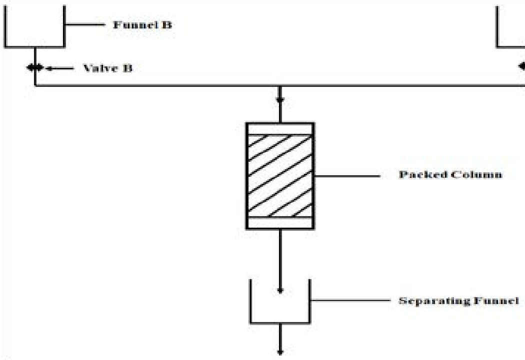

Figure 1: Liquid-liquid Extraction Schematic Diagram (Bench Scale)

A measured volume of extractant (water) was poured into a funnel B and corresponding volume of binary mixture, also measured was poured into a funnel A. These corresponding volumes were read from the binodal curve. The two valves (valve A and B) were opened simultaneously in order to control the rate of flow of the binary mixture and solvent into the packed column. The flowrate of each was taken. Also, the time taken for the ternary mixture to pass through the packed bed was determined. Finally, the refractive index values of the resulting raffinate and extract separated in the separating funnel were measured and recorded.

2.3.2 Experimental Procedure for the Pilot Process

The pilot scale liquid-liquid extraction process was carried out in a packed column fabricated and assembled in the Department of Chemical Engineering, Obafemi Awolowo University, Ile-Ife, Nigeria. The set-up is as shown in Figure 2. It includes two packed columns of height 85cm and 80cm diameter, one is suitable for cocurrent flow pattern and the other for counter current flow pattern. The column which is suitable for cocurrent was employed using wire gauze ring packing, Raschig ring packing and stainless steel ring packing at different times. Apart from the spherical vessel that contains one of the liquids at the top, there is a metal tank of volume 3,000cm3 placed on a plank on the same level with the vessel to maintain constant liquid head. It has a liquid pump, attached to the bottom tank, that supplies the liquid to the top spherical vessel. Finally, four 500ml conical flasks with cork were placed intermittently at the bottom of the packed column to collect samples after the extraction process.

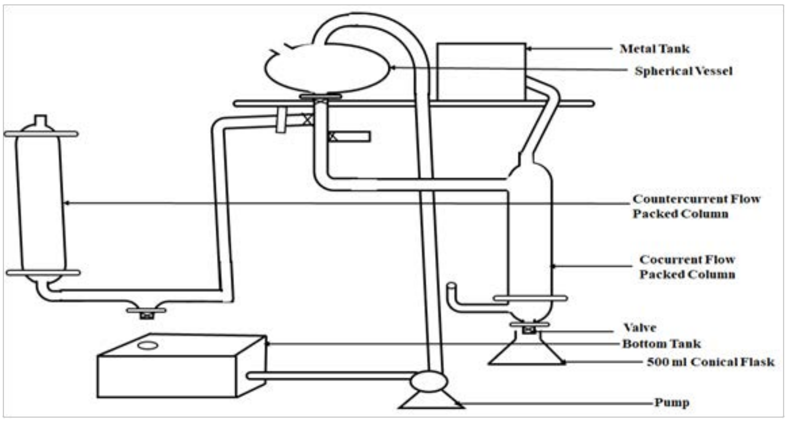

Figure 2: Liquid-Liquid Extraction Schematic Diagram (Pilot Scale)

Clean water was pumped into the spherical vessel to wash down any dirt or residual chemical in the column, all lines were drained and all valves were closed after drainage. The spherical vessel and metal tank were charged with measured volumes of feed (binary mixture) and solvent (extractant) respectively, and the two inlet valves were opened at the same time for the feed and solvent to run and mix through the bed. The time taken for the feed and solvent to flow through the valves to the packed column was recorded. Thereafter, the resulting extraction mixture was collected into the four 500ml conical flasks (representing 4 stages) and allowed to settle. These was done for four different feed compositions using varying volumes of solvent and the equipment was shut-down before the packing in the column was changed.

The process was shut down by pumping clean water through the spherical vessel to wash residual chemical from the column. All lines were drained totally and all valves were closed after drainage. Then, the pump was switched off.

2.3.3 Determination of Column Efficiency

The efficiency of each packed column used for the separation was determined by calculating the number of transfer unit, transfer coefficient and height of transfer unit as follows ([8]):

$$N_{TOR} = \int_{\mathrm{x((0)}}^{\mathrm{x(L)}} \frac{\mathrm{d}x}{x_i - x_i^*} \qquad 2$$

$$N_{TOR} = \frac{\mathrm{h}}{3}(f_0 + f_n + 4(f_1 + f_3 + ... + f_{n-1}) + 2(f_2 + f_4 + ... + f_{n-2}) \qquad 3$$

$$\text{where } f(x) = \frac{1}{x_i - x_i^*} \qquad 4$$

$$K_R\mathrm{a} = \frac{L_m N_{TOR}}{h} \qquad 5$$

$$\mathrm{H_{ODP} = HTU = L_m / K_R a} \qquad 6$$

The efficiency (experimental or predicted) of an extraction column based on dispersed phase is related to the height of the transfer unit as:

$$\text{Efficiency, } \mathrm{E_{ODP}} = \frac{1}{\mathrm{H_{ODP}}} \qquad 7$$

where the height of transfer unit HODP is related to number of transfer unit NODP and the column height H as:

$$\mathrm{H_{ODP}} = \frac{\mathrm{H}}{\mathrm{N_{ODP}}} \qquad 8$$

$$\mathrm{N_{ODP}} = \frac{\lambda}{\lambda - 1} \ln\lambda \frac{(1 - \mathrm{E_{ODP}})}{1 - \mathrm{E_{ODP}}} \qquad 9$$

where λ is the extraction factor (experimental), and is related to mass flowrates in dispersed phase (WD), continuous phase (WC) and solute distribution coefficient (m) as:

$$\lambda = \frac{\mathrm{W_C}}{\mathrm{mW_D}} \qquad 10$$

The degree of deviation of the predicted number of transfer unit from experimental, based on dispersed phase (NODP) is given by a confidence limit (C.L) determined as:

$$\mathrm{C.L} = \pm 2 \frac{\left(\sum (1 - \frac{\text{predicted value}}{\text{experimental value}})^2\right)^{0.5}}{\mathrm{N_2} - 1} \qquad 11$$

and N_2 is the number of value compared.

3. Results and Discussion

3.1 Properties of Systems

The data obtained for the properties of the systems are summarized in Table 2. The increase in the optical density (refractive index) with increasing concentration of toluene in the acetone-toluene-water system agrees with the predictions in the literature [15]. The differences in the extent by which various substances cause a decrease or increase in the speed of light

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as compared with its speed in air or vacuum (refractive index) depends on temperature, pressure, chemical composition, physiochemical constitution, homogeneity and purity of the substance [11-13]. It then implies that with all other factors constant, change in chemical composition or physicochemical constitution will result in change in the refractive index measurement of the substance. This trend observed in Table 2 might be due to the fact that toluene has a higher refractive index value than water and the more the concentration of toluene in the ternary mixture the higher the refractive index of the mixture [15].

Sample No	Water	Toluene	Acetone	Refractive index	Density g/cm3
1	1.0000	0.0000	0.0000	1.331	1.0000
2	0.2976	0.0595	0.6429	1.367	1.9345
3	0.2370	0.0948	0.6629	1.372	1.9255
4	0.2000	0.1200	0.6800	1.376	1.9255
5	0.1751	0.1401	0.6848	1.378	1.9164
6	0.1548	0.1548	0.6904	1.379	0.9074
7	0.1320	0.1650	0.7030	1.381	0.9074
8	0.1149	0.1916	0.6935	1.384	0.9029
9	0.0901	0.2252	0.6847	1.389	0.8939
10	0.0548	0.2740	0.6712	1.391	0.8848
11	0.0000	1.0000	0.1000	1.493	0.8700

Table 2: Variation of Refractive Index and Density with Volume Fraction of Toluene-Acetone-Water System

3.2 Liquid-Liquid Equilibrium Data of Toluene-Acetone-Water System

The liquid-liquid equilibrium data of the system is reported in Table 3. The data shows that an increase in the mass fraction of solute (acetone) in the feed increases the mass fraction of solute in the extract and raffinate layers. The separation factor was greater than unity (6.82 – 118.50) for all the feed ratio. This could be as a result of the greater immiscibility of water and toluene which gave large heterogeneous layer. Many literatures had reported toluene-acetone-water as a good liquid-liquid extraction system with large heterogeneous region [16]; [17]. Also, better distribution of acetone between the two phases was observed when the ratio of concentration of acetone to water in the inlet streams was less or equal to 0.32. However, the distribution of solute between the two phases (0.779 – 1.25) was not constant over the whole two-phase region.

Sample No	Feed (Mass fraction)			Extract (Water) layer			Raffinate (Toluene) layer			K
	Toluene	Acetone	Water	Toluene	Acetone, $y = Kx^*$	Water	Toluene	Acetone, x	Water	
1.	0.614	0.053	0.333	0.010	0.060	0.930	0.948	0.048	0.004	1.250
2	0.379	0.139	0.482	0.017	0.148	0.835	0.861	0.128	0.011	1.156
3	0.164	0.109	0.727	0.013	0.128	0.859	0.885	0.105	0.010	1.219
4	0.582	0.190	0.228	0.021	0.184	0.795	0.795	0.189	0.016	0.974
5	0.427	0.309	0.264	0.038	0.310	0.652	0.657	0.315	0.028	0.984
6	0.083	0.221	0.696	0.028	0.230	0.742	0.754	0.225	0.021	1.022
7	0.096	0.351	0.553	0.040	0.345	0.615	0.529	0.433	0.038	0.797
8	0.177	0.424	0.399	0.040	0.409	0.551	0.495	0.463	0.042	0.883
9	0.233	0.510	0.257	0.042	0.451	0.507	0.368	0.579	0.053	0.779
10	0.504	0.413	0.083	0.040	0.345	0.615	0.542	0.418	0.040	0.825

Table 3: Liquid-Liquid Equilibrium Data for Toluene- Acetone-Water System (Mass Fraction)

3.3 Effect of Packing Material on the Distribution of Acetone between Toluene and Water Phases

The effect of three different packing used for the extraction on the distribution of acetone are reported in Table 4. It was observed that extract solute concentration increase with increase in feed solute concentration. Treybal (2021) [8], and Schiebel and Karr (1950) [18] stated that increase in the solute concentration of feed sample increases the solute concentration in the resulting extract and raffinate phases, as observed in this work, for all the packing materials used. Stainless steel rings packing gave the highest extract solute concentration while the wire gauze rings packing gave the least extract solute concentration for the bench scale experiment. Wire gauze rings packing had the highest raffinate solute concentration (0.008 – 0.026), though the difference in magnitude was ere not much.

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Figure 3 shows the variation of separation factor with raffinate solute concentration using Raschig rings, wire gauze rings and stainless steel rings packing. There is decrease in separation factor as raffinate solute concentration increases, which is consistent with reported literatures ([11]; [12]; [13]. a, b). Wire gauze rings packing has the highest separation factors compared to other packing materials as a result of its tendency for bubble formation. This had been severally reported to be very efficient as contacting device for distillation and liquid-liquid extraction processes because it provides larger interfacial area for mixing and hence better extraction [19-21]. This is true in this work as it has the highest separation factor for the studied system. This implies that it provides the best mixing and avenue for greater mass transfer of solute (acetone) into the solvent (water). This could make the extraction of acetone from acetone-toluene using water, as solvent, feasible on large scale.

ID No	Raffinate Phase			Extract Phase			Distribution Coefficient, K
	W11	W21	W31	W13	W23	W33	
Raschig Rings							
1C	0.8740	0.1260	0.0000	0.0003	0.0150	0.9847	0.119
2C	0.6600	0.3400	0.0000	0.0013	0.0592	0.9395	0.174
3C	0.1000	0.5320	0.3680	0.0027	0.1750	0.8223	0.329
4C	0.6630	0.3370	0.0000	0.0014	0.1013	0.8973	0.300
Wire Gauze Rings							
1C	0.8920	0.1080	0.0000	0.0003	0.0150	0.9847	0.139
2C	0.6400	0.3600	0.0000	0.0014	0.0700	0.9286	0.194
3C	0.1090	0.5450	0.3460	0.0027	0.1750	0.8223	0.321
4C	0.6760	0.3240	0.0000	0.0012	0.1110	0.8878	0.343
Stainless Steel Rings							
1C	0.8950	0.1050	0.0000	0.0006	0.0230	0.9764	0.219
2C	0.6500	0.3500	0.0000	0.0014	0.0700	0.9286	0.200
3C	0.1000	0.5300	0.3700	0.0026	0.1874	0.8100	0.353
4C	0.6500	0.3500	0.0000	0.0012	0.1110	0.8878	0.317

Table 4: Composition of the Raffinate and Extract Phases using Raschig Rings, Wire Gauze Rings and Stainless-Steel Rings Packing for Acetone-Toluene-Water System in Bench Scale

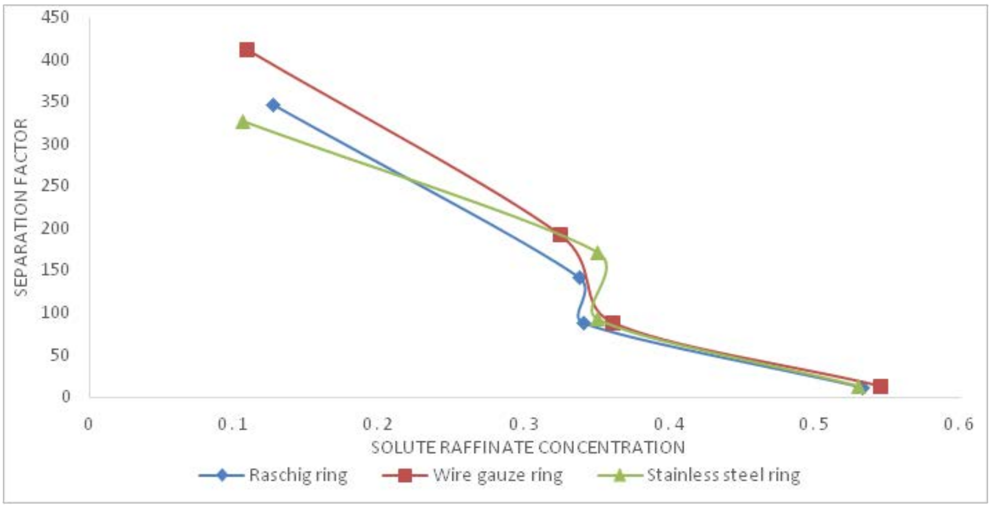

Figure 3: Variation of Separation Factor with Raffinate Solute Concentrations for Acetone-Toluene-Water System using Raschig Rings, Stainless Steel Rings and Wire Gauze Rings Packing

3.4 Effect of Packing Material on HTU and K_Ra of Extraction for Acetone-Toluene-Water System

The experimentally observed rate of mass transfer, K_Ra in extraction processes is often expressed with overall mass transfer coefficient [8]; [24]. The Raschig rings gave the highest values of overall mass transfer coefficient. This implies that Raschig rings is the most effective for transfer of solute (acetone) from toluene to solvent (water). Therefore, types of packing materials and feed solute concentration are among the factors that affect the height of transfer unit and overall mass transfer coefficient of extraction process. The observed dependency of HTU and K_Ra on concentration gradient ($x_i - x_i^*$) is in agreement with Fick's law that states that mass transfer rate is directly proportional to the concentration gradient and interfacial area

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across the direction of flow [25]. The variation of HTU and K_Ra with concentration gradient (driving force) is similar for the three types of packing. This implies that the trend of the variation of HTU and K_Ra with $x_i - x_i^*$ is independent of the type of packing in the extraction column. For the acetone-toluene-water system, HTU decreases with $x_i - x_i^*$ while K_Ra increases with $x_i - x_i^*$. The differences in the variation of HTU and K_Ra with $x_i - x_i^*$– for the three packing materials used in the study may be due to the interfacial phenomena. Thornton (2023) [7] stated that interfacial phenomena can predominantly affect the mass transfer coefficient of extraction, especially when the depth of the phases between which mass transfer takes place is large in comparison to the thickness of the liquid layer subjected to separation by interfacial movement of mass which is the case in this study.

ID No	Feed Flowrate	Solvent flowrate	Raffinate solute concentration x_i	Equilibrium concentration x_i	$x_i - x_i^*$	HTU	$K_R a$
Raschig Rings							
1C	0.3893	0.3720	0.1260	0.0630	0.0630	162.5226	0.0024
2C	0.3916	0.4133	0.3400	0.2200	0.1200	41.615	0.0094
3C	0.3945	0.4292	0.5320	0.4030	0.1290	14.627	0.0270
4C	0.3720	0.4044	0.3370	0.2175	0.1195	14.915	0.0249
Wire Gauze Rings							
1C	0.3893	0.3720	0.1080	0.0540	0.0540	94.620	0.0041
2C	0.3916	0.3445	0.3600	0.2375	0.1225	50.995	0.0077
3C	0.3945	0.4292	0.5450	–	–	–	–
4C	0.3720	0.4044	0.3240	0.2080	0.1160	14.667	0.0254
Stainless Steel Rings							
1C	0.3488	0.3720	0.1050	0.0520	0.0530	89.124	0.0039
2C	0.3306	0.4133	0.3500	0.2300	0.1200	46.257	0.0071
3C	0.3382	0.4650	0.5300	0.4020	0.1280	14.864	0.0227
4C	0.2906	0.3200	0.3500	0.2300	0.1200	15.970	0.0182

Table 5: Variation of HTU, K_Ra with Feed and Solvent Flowrates for Acetone-Toluene-Water System Using Raschig, Wire Gauze and Stainless-Steel Rings Packings

3.5 Effect of Packing Material on NTU and Efficiency of Extraction for Acetone-Toluene-Water System

The effect of packing materials on number of transfer unit and column efficiency using Raschig rings, wire gauze rings and stainless steel rings packing is reported in Table 6. The number of transfer unit, NTU decreases as extract solute concentration increases, though this was not constant over the whole phase region. The phenomenon is similar for all the packing materials used for the extraction process. The packing material only affects the magnitude of the NTU with respect to equilibrium solute concentration. This could be as a result of the differences in their interfacial area, voidage volume and flow capacity being offered by each during extraction process [26,27]. The Hartland-Mechleburg (1966) [28] solution of the axial dispersion model equation was used to predict experimental NTU values. The very low confidence-limit values of the predicted values for each packing materials shows the method is not suitable for predicting experimental NTU values.

The column efficiency decreases as extract solute concentration increases. This phenomenon is similar for all the packing materials employed for the extraction process. Raschig rings packing had the best column efficiency (0.7087), thus making it the most suitable packing material for this extraction process. This could be as a result of the wettability of Raschig rings materials used as contact device for the extraction process [29,30]. There is no much difference in the values obtained from wire gauze rings and stainless steel rings packing.

ID No	Extract solute concentration (y)	Equilibrium solute conc. y*	Ditribution coefficient, K	Extraction factor	Experimental NTU	Predicted NTU	Column Efficiency	Confidence Limit
Raschig rings								
1C	0.0150	0.0350	2.3330	0.4096	3.8300	0.0622	0.0062	±1.8784%
2C	0.0592	0.1170	1.9760	0.5342	0.1610	0.2490	0.0240	
3C	0.1750	0.2850	1.6290	0.6680	0.0708	0.0748	0.0684	
4C	0.1013	0.1900	1.8760	0.5841	1.6460	0.0737	0.0670	
Wire gauze rings								
1C	0.0150	0.0350	2.3330	0.4096	4.1780	0.0224	0.0106	±1.9602%
2C	0.0700	0.1390	1.9857	0.4431	1.4074	0.0202	0.0020	
3C	0.1750	0.2850	1.6286	0.6680	0.0000	0.0000	0.0000	
4C	0.1110	0.2020	1.8198	0.5973	1.7165	0.0741	0.0683	
Stainless steel rings								
1C	0.0230	0.0500	2.1739	0.4907	4.1932	0.0114	0.0112	±1.9654%
2C	0.0700	0.1390	1.9857	0.6295	1.4950	0.0222	0.0216	
3C	0.1870	0.2980	1.5936	0.8629	0.0847	0.0726	0.0673	
4C	0.1110	0.2020	1.8198	0.7034	1.4950	0.0677	0.0626	

Table 6: Variation of extract solute concentration with extraction factor, experimental and predicted NTU values and column efficiency for acetone-toluene-water system using Raschig, wire gauze and stainless steel rings packing

3.6 Effect of Scale Ip on Process Output Parameters (Extract Solute Concentration and Efficiency) for Acetone-Toluene-Water Ternary System

3.6.1 Distribution of Acetone Between the Two Phases

The process scale-up parameters are reported in Table 7. The scale up was done by using 10 times of the feed and solvent volumes used in bench scale, and the resulting extract and raffinate were collected in four equal batches of 500 cm^3. The extraction of acetone in all the satges was not feasible as there was no traces of acetone at the later stages for some packings. Stainless steel rings provided the best platform for the best acetone extraction (in 3 stages), followed by wire gauze rings (in 4 stages) especially when the feed solute concentration is higher. It can be observed that variation of distribution coefficient with feed solute concentration; and the performance of the three types of packings, are the same for both the bench and pilot scale extraction, but the magnitude of the variation differs. Higher values of distribution coefficient were obtained for bench scale process than for pilot scale process. This may be due to the stage system used. Also, the volumes and flowrates of the solvent and feed samples were different and this may affect the rate of extraction at each stage.

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ID No	Stage No	Raffinate Phase			Extract Phase			Distribution coefficient, K
		Toluene	Acetone	Water	Toluene	Acetone	Water	
1C	1	0.8883	0.0726	0.0391	0.0006	0.0017	0.9977	0.0234
	2	0.9051	0.0334	0.0615	0.0018	0.0211	0.9771	0.6317
2C	1	0.8072	0.1391	0.0537	0.0014	0.0120	0.9866	0.0863
	2	0.4815	0.3875	0.1308	0.0041	0.0586	0.9373	0.1512
3C	1	0.5323	0.3591	0.1086	0.0025	0.0328	0.9647	0.0913
	2	0.2134	0.5964	0.1902	0.0068	0.0377	0.9555	0.0632
4C	1	0.6474	0.2677	0.0849	0.0045	0.0655	0.9300	0.2447
	2	0.6395	0.2711	0.0894	0.0041	0.0586	0.9373	0.2162
	3	0.5323	0.3591	0.1086	0.0041	0.0586	0.9373	0.1632
	4	0.3067	0.5282	0.1651	0.0117	0.2023	0.7860	0.3830
1C	1	0.8386	0.1070	0.0544	0.0006	0.0017	0.9977	0.0158
2C	1	0.7521	0.1847	0.0632	0.0022	0.0277	0.9701	0.1500
	2	0.5689	0.3265	0.1046	0.0052	0.0701	0.9247	0.1215
	3	0.4816	0.3875	0.1309	0.0066	0.0859	0.9075	0.2217
3C	1	0.5554	0.3352	0.1094	0.0052	0.0701	0.9247	0.2091
	2	0.2104	0.6030	0.1866	0.0168	0.2467	0.7365	0.4091
4C	1	0.6084	0.2836	0.1080	0.0031	0.0402	0.9567	0.1417
	2	0.6036	0.2896	0.1068	0.0052	0.0701	0.9247	0.2421
	3	0.5554	0.3352	0.1096	0.0055	0.0773	0.9174	0.2306
	4	0.3026	0.5300	0.1674	0.0091	0.1229	0.8679	0.2305
1C	1	0.9569	0.0256	0.0175	0.0005	0.0017	0.9977	0.0664
	2	0.7672	0.1644	0.0684	0.0014	0.0120	0.9866	0.0728
2C	1	0.7913	0.1553	0.0534	0.0014	0.0120	0.9977	0.0773
3C	1	0.3661	0.4840	0.1499	0.0045	0.0655	0.9300	0.1353
4C	1	0.8261	0.1198	0.0541	0.0018	0.0211	0.9771	0.1701
	2	0.7035	0.2147	0.0818	0.0029	0.4453	0.9517	0.2110
	3	0.3256	0.5154	0.1591	0.0082	0.1145	0.8773	0.2222

Table 7: Composition of Raffinate and Extract Phase for Pilot Scale Process Using Raschig, Wire Gauze and Stainless-Steel Ring Packings

The variation of K_Ra and HTU with feed solute concentration, as shown in Table 8, is similar in trend and magnitude for bench scale and pilot scale. The resulting HTU values for pilot scale process are smaller than the resulting HTU values for bench scale process. Likewise, the resulting K_Ra values are higher for pilot scale processes than for bench scale process. This is in agreement with the observation of Azizi and Rezaeimanesh (2016) [31] and Minne et al, (2018) [32] that increase in the ratio of packing size to column diameter increases the rate of extraction (i.e mass transfer) though Verteressian and Fenske (1936) [33] observed that decrease in this ratio increases the mass transfer. This contradiction in observation was due to effect of the shape and void spaces of the packings used by Verteressian and Fenske (1936) [33] as compared to the more open chain and ring packing reported by Schultes (2003) [34

The dimensions of the pilot scale equipment were packing length: 59.5cm, and internal column diameter: 8.0cm. Also, the dimension of the bench scale equipment was packing length: 40.0cm, and internal diameter: 3.7cm. the same size of packings were used for both scales. The total volume of both the solvent and feed sample used for pilot scale is 10 times the total volume of both the solvent and feed sample used for bench scale process, though the flowrate of the solvent and feed sample for the two scales are relatively close in magnitude.

Treybal (2021) [8] observed that column efficiency increases with scale up, or at worst, remain constant This is evident in the extraction of Uranium into ion-exchange type solvent [35]; [36], where mass transfer was of great importance and internal diameter of liquid-liquid extraction column was successfully scaled up on this basis from 6 to 36 inches. In the absence of chemical reaction, column efficiency increases with scale up, as being shown in this work; the column efficiency for the pilot scale process is higher than the column efficiency for the bench scale process since HTU varies indirectly with efficiency, that is, the lower the HTU, the more efficient the equipment [37].

4. Conclusion

Bench and pilot scales were used for the liquid-liquid extraction of toluene-acetone-water system at 298.15 K. Three different packings - Raschig rings, wire gauze rings and stainless steel rings - were used for the operation, to increase extraction efficiency. In the bench scale experiment, the Raschig rings has the highest transfer unit; HTU (162.52) and comparably lower column efficiency (0.0062) with respect to extract solute concentration (0.0150) at ±1.8784% confidence limit. The HTU increases as solvent flowrates increases. In the pilot scale experiments, separation could not be achieved in all the four stages, but only when the feed and solvent flowrates were maximum. The HTU was low compared to the bench scale, for all the packing materials with Raschig rings having comparably lowest HTU. This portends the pilot scale has better column efficiency for all the packing materils used in the extraction process, with Raschig rings being the best materials for the pilot scale extraction process.

ID No	Feed Flow-rate	Solvent Flow-rate	Raffinate Solute Concentration (X_1)				Equilibrium Raffinate Solute Concentration, X_1^*				Concentration Gradient (X_1-X_1^*)				Overall Mass Transfer Coefficient, K_La				HTU			
			Stages																			
			1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
Raschig rings																						
1C	0.4116	0.5684	0.0726	0.0334	-	-	0.0340	0.0170	-	-	0.0386	0.0164	-	-	0.0102	0.0206	-	-	40.303	19.998	-	-
2C	0.4301	0.7958	0.1391	0.3875	-	-	0.0705	0.2625	-	-	0.0686	0.1250	-	-	0.0223	0.0044	-	-	19.310	97.032	-	-
3C	0.4581	0.6940	0.3591	0.5964	-	-	0.2371	-	-	-	0.1220	-	-	-	0.0385	-	-	-	11.886	-	-	-
4C	0.6459	0.7958	0.2677	0.2711	0.3591	0.5282	0.1620	0.1630	0.2371	0.4000	0.1057	0.1081	0.1220	0.1282	0.0372	0.0366	0.0274	0.0133	17.379	17.635	23.190	48.382
Wire gauze rings																						
1C	0.5029	0.5526	0.1070	-	-	-	0.0530	-	-	-	0.0540	-	-	-	0.0062	-	-	-	81.443	-	-	-
2C	0.5062	0.6169	0.1847	0.3265	0.3875	-	0.0975	0.2093	0.2625	-	0.0872	0.1172	0.1250	-	0.0211	0.0095	0.0052	-	23.923	53.200	97.032	-
3C	0.6055	0.5968	0.3352	0.6030	-	-	0.2165	-	-	-	0.1187	-	-	-	0.0453	-	-	-	13.365	-	-	-
4C	0.6459	0.7368	0.2836	0.2896	0.3352	0.5300	0.1740	0.1790	0.2165	0.4020	0.1096	0.1106	0.1187	0.1280	0.0350	0.0349	0.0302	0.0131	18.476	18.500	21.405	49.120
Stainless steel rings																						
1C	0.6282	0.6631	0.0256	0.1644	-	-	0.0135	0.0850	-	-	0.0121	0.0794	-	-	0.0933	0.0430	-	-	6.733	14.621	-	-
2C	0.5305	0.6417	0.1553	-	-	-	0.0810	-	-	-	0.0743	-	-	-	0.0509	-	-	-	10.420	-	-	-
3C	0.6055	0.6782	0.4840	-	-	-	0.3540	-	-	-	0.130	-	-	-	0.0651	-	-	-	9.305	-	-	-
4C	0.7652	0.6860	0.1198	0.2147	0.5154	-	0.0593	0.185	0.3865	-	0.0605	0.0962	0.1289	-	0.1253	0.1019	0.0310	-	6.107	7.509	22.154	-

Table 8: Variation of Raffinate Solute Concentration With (X1-X1*), HTU and Using Raschig, Wire Gauze and Stainless-Steel Rings Packings for Pilot Scale Process in Acetone-Toluene-Water System

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