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RESEARCH ARTICLE

A Novel Technique for Extraction of phenol from the industrial Waste water by using York- Scheible column

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Abstract

Phenol is an example of a highly toxic chemical substance. The maximum amount sanctioned by the World Health Organization in drinking water is 0.001 mg/L. This component is found in large quantities in the effluents of different petrochemical factories and is regularly released into the rivers. Therefore there is great need for elimination phenol from the water, on the other hand, phenol constitutes a valuable component in the production of various chemicals and could be reclaimed from the water to great advantage. amongst one of the best and cheapest method for the separation of phenol from the industrial effulent is the liquid -liquid extraction by the York-scheible column with tolune as the extracting solvent.

Introduction

Presence of phenolic compounds even at low concentration in the industrial waste water adversely affects aquatic as well as human life directly or indirectly when disposed off to public sewage, river or surface water. Sometimes these form complex compounds with metal ions, discharged from other industries, which are more carcinogenic in nature than the phenolic compounds. The toxicity imparted by phenolic compounds is responsible for health hazards and dangerous to aquatic life. Different conventional processes generally being used to remove phenol are: solvent extraction, incineration, chemical oxidation, freecultured biological degradation, adsorption and membrane separation. However, depending upon the quality and quantity of waste water, the treatment methods are to be chosen. At lower concentration of phenol, all the above mentioned techniques will not be suitable to treat the waste water; in addition these become uneconomical for its industrial scale-up operation. Application of liquid liquid extraction method by using York-scehible column technique in the treatment of waste water containing phenolic compounds.

Literature survey:

VARIOUS METHODS FOR PHENOL REMOVAL:

1) Polymerization :

2) Electro coagulation

3) Adsorption and Ion exchange

4) Membrane based separation

A Novel technique for the phenol separation By using York-Schible Colunm with the Toluene as a Extracting Solvent

York-scheibel column:

The primary objective of this Paper will be to determine the amount of phenol extracted by the toluene using York-Scheibel column.

Theory:

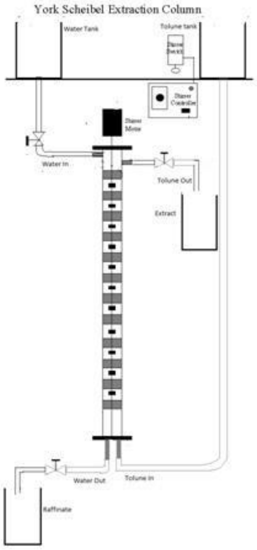
The York Scheibel Column Falis in the Class of Gravity Operated Extractors With Mechanical agitation. The Presentation of Agitationb And Calcining Section in york scheibel column yield higher extraction Efficiency. The Conventional layer paked column or spray column employed for liquid -liquid extraction. In This column Uniform dispersion in each of mixing chambers eliminates all possibilities of channelling. This Column Consistes of alternate calcining and mixing section with a controlling lacated shaft upon which mounted the agitators with blades.

The particular points of interest included:

(l) product purity as a function of feed ratio at various agitator speeds,

(2) the column efficiency as a function of agitator speed at various feed ratios,

(3) the column efficiency as a function of feed ratio at various agitator speeds,
(4) the column efficiency as a function of total column throughput at various agitator speeds, and
(5) the overall mass transfer coefficients as a function of total column throughput at various agitator speeds.



PROCEDURE:

Preliminary: Prepare a 2 Molar phenol Solution in water. For 5 Liters of solution:

1. Measure out 1 L tap water (DI water is not necessary) in a 2 L Nalgene container

2. Add 188 gm of phenol

3. Mix/ShakeWell

Start Up:

- Check to see that all the drainage valves are closed.

- Place the prepared phenol solution in the feed tank.

- Fill the Toluene feed tank with Toluene.

- Check to be sure the top water vent valve is open.

- Fill the column slowly with the water/Phenol solution by turning on the left motor with the breaker on the right power panel. Set flow rate on left by adjusting the Valve opening adjustment.

- Close the Toluene out when the liquid level reaches the top left (Toluene out) nozzle. (This will force water out the bottom of the column and allow the interface to form at the top)

- Allow the interface to form between the top mesh and the top left nozzle (Toluene out). The interface appears as an immiscible layer between and water with droplets of Toluene accumulating at this interface.

- Once the interface is formed in the desired location, open the Toluene out nozzle slowly until there are Toluene and water flowrates out of the column. Small adjustments should be made in order to keep the interface constant.

- Set the stirrer speed to a setting of dial on the top right of the lab equipment panel. Make sure the motor is powered with the top center switch on the right power panel.

- Allow the column to run until steady state is achieved (about ½ hour). While waiting for steady state, adjustments may be made to the Toluene out Nozzle to keep the interface at the desired level (table 1).

Table 1: Nozzle settings

Run #	**Nozzle setting**			**Flow Rate (mL/min)**		**Rotor Speed Setting**
	Toluene inlet	*H_2O w/phenol Inlet*	*Toluene Flow out*	*Toluene*	*H_2O w/phenol Inlet*	
1	39	29	~39	19.78	30.54	5
2	32	29	~32	10.675	30.54	5

Determination of Phenol in given solution:
Here We Use The Titration Method For determination of phenol. We Use Iodometric method for determinatoion of phenol (using 0.1 N $Na_2S_2O_3$)

Appartus-IodineFlask, Measuing Cylinder, Burette 100 ml volumetric flask, 10 ml pipette

Chemicals- Given Solution of phenol , Brominating solution, Conc. HCL, 10% KI , 0.1 N $Na_2S_2O_3$, Starch Solution.

Theory: Brominating solution is the solution containing 1:5 mixture of $KBrO_3$ and KB_r. This Solution on treatment with conc HCL Liberates bromine.

$KB_rO_3 + 5KB_r + 6\ HCL \longrightarrow 6\ KCL + 3H_2O + 3Br_2$

Phenol molecule has two ortho and one para positions activated for electrophilic substitution by bromine atoms.

In the experiment a known excess of Br_2 (in the form of brominating solution) is added in the phenol. The Unreacted amount of Br_2 is found out by back titration against standard $Na_2S_2O_3$ Solution.

The total amount of Bromine added for bromination , is found out in terms of volume of $Na_2S_2O_3$ Solution in the titration called as blank titration.

$Br_2 + 2\ KI \longrightarrow 2\ KB_2 + I_2$

$I_2 + 2Na_2S_2O_3 \longrightarrow 2\ NaI + Na_2S_4O_6$

Procedure:

Part 1: Back Titration:

- Dilute the given phenol solution with distlled water , up to the 100 ml mark , in the volumetric flask, shake welkl the solution.
- Fill the burette with $Na_2S_2O_3$ Solution.
- Pipette out 10 ml of diluted phenol in an iodine flask and 20 ml of bromaniting solution with the help of pipette.
- Stopper the flask immediately after adding about 5 ml conc HCL with the measuring cylinder, round shake the mixture for 3 minutes.
- Add about 10 ml of KI Solution on the mouth of iodine flask and slowly open the stoper, then wash the stopper and mouth with little water.
- Titrates this reddish coloured mixtures against $Na_2S_2O_3$ Solution , till the colour changes to yellow.
- Add 1ml (10 drops) of starch solution as indicator into the mixture. The mixture turns dark blue.
- Continue to titrate the dark blue colored mixture till the colour disapperes (mixture at this end point contains colorless solution along along white ppt of 2,4,6-tribrophenol) Note the end point reading.
- Repeat the titration.

Part 2 - Blank Titration:

- Fill the burette with $Na_2S_2O_3$ Solution.
- Pipette out 20 ml Brominating solution solution (now no phenol solution) in to the iodine flask. Add about 5 ml of HCL and stopper the flask immediately. Then add 10 ml of Solution on the mouth of iodine flask and slowly open the stoper, then wash the stopper and mouth with little water.
- Titrate the dark red reaction mixture against $Na_2S_2O_3$ Solution, the mixture becomes yellow.
- Now add 1 ml(10 drops) of starch indicator which turns yellow solution to dark blue.

- Continue to Titrate the mixture till the blue colour disappeares, note the burette reading.
- Repeat this blank titration.

Observation:

- Back Titration :

Burette= 0.1 N $Na_2S_2O_3$

Pipette= 10 ml diluted phenol solution + 20 ml brominating solution Indicator= starch solution.

End point = blue colour disappears.

Sr. No	Burette readings in ml	Average burette reading
1	85	89.66 ml
2	89	
3	95	

- Black Titration :

Burette = 0.1N $Na_2S_2O_3$ pipette= 20 ml brominating solution. Indicator= Starch Solution

End point= blue to colorless.

Sr. No	Burette readings in ml	Average burette reading ml
1	90	94.33 ml
2	95	
3	98	

Calculation:

Volume of 0.1N $Na_2S_2O_3$ corresponding to consumed Br_2 for bromination = (blank titration reading - back titration reading) = (X-Y)= 4.67ml

As 10 ml diluted phenol corresponds to (X-Y) ml of $Na_2S_2O_3$ = 4.67 ml

Therefor 100 ml diluted phenol corresponds to 10(X-Y) ml= 46.7 of $Na_2S_2O_3$

$1\ C_6H_5OH = 3Br_2 = 3I_2 = 6\ Na_2S_2O_3$
(94) (248)

This means,

6 ml 1 N $Na_2S_2O_3$ = 94 mg of phenol

$$\text{ml } 0.1\text{ N } Na_2S_2O_3 = \frac{10(X-Y)\times 0.1\times 94}{6}$$

=.73.163. mg of phenol in given solution.

Result

The Taken extract Phenolic Solution contains 73.163 mg of phenol.

As the 73.163 mg of phenol goes in to the extract phase, there for percentage of phenol in raffinate phase can be calculated as Original phenol in the Water-phenol solution= phenol in extract phase + phenol in raffinate phase. There for,94 mg of phenol in water phase

- 73.169 mg of phenol in the extract phase= 20.831 mg of phenol in raffinate phase.

Mass transfer calculation:

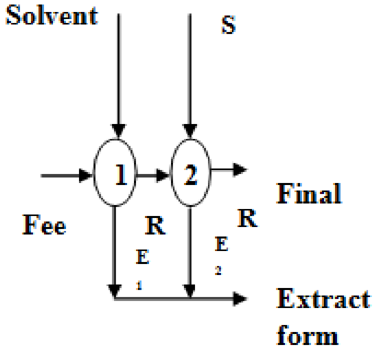
Material balance:

F- Mass of Feed
R_1- Mass of Raffinate phase
E_1- Mass of Extract Phase

X_F- Mass fraction of phenol in feed
Y_s- Mass fraction of phenol in solvent
X_1- Mass fraction of phenol in raffinate
Y_1-- Mass fraction of phenol in Extract
Making a total mass balance , we have

$F + S_1 = E_1 + R_1 = M_1$
Where M1= Mass of Extract and raffinate

Phenol calculation in single stage opration:



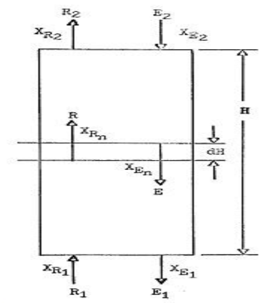


Fig. Counter current flow of Extract and raffinate phases

Material balance Calculation:

$$M = R_1 + E_2$$

$$MX_M = R_1X_{R_1} + E_2X_{E_2}$$

$$X_M = \frac{R_1X_{R_1} + E_2X_{E_2}}{M}$$

The calculated composition of the mixing point X_{M1}, can then be located on a line connecting $X_{R!}$ and X_{E2};on the ternary diagram. If only steady state conditions are assumed during the collection of operating data, material balances including the exit stream flovn-ates and compositions must yield the same value of M and $X_{M!}$ As,

$$M = R_2 + E_1$$

$$MX_M = R_2X_{R_2} + E_1X_{E_1}$$

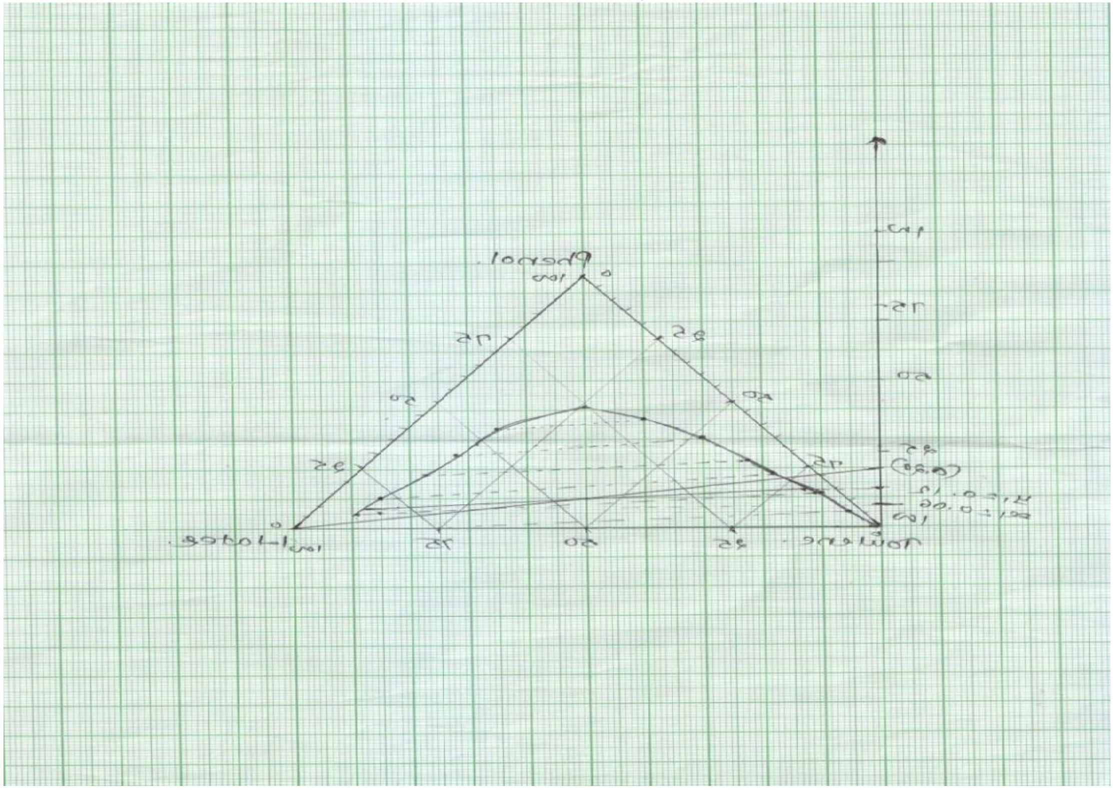
$$X_M = \frac{R_2X_{R_2} + E_1X_{E_1}}{M}$$

The liquid-liquid Extraction data of Water-Phenol-Toluene System is given in the following table:

	Toluene phase			Water phase		
	phenol	water	Toluene	phenol	water	Toluene
1	98	5	92.5	85	30	59
2	95	10	87.5	82	40	53
3	92	18	76	72	50	46
4	90	20	72	54	66	34
5	89	22	64	38	72	33
	85	30	59	27	77	31

Ternary plot for phenol water system

Water- Phenol-Toluene system



Ternary Plot from Observation

Calculation for the Exract and raffinate phase:

F = 0.5 kg

X_f = 0.2
Solvent in the feed S = 500 ml Phenol in the solvent = 0

Final value of phenol in the raffinate phase = 5.04 mg
To find the number of stages required to reduce the concentration of phenol in raffinate phase,

Overall material Balance:

$F + S_1 = E_1 + R_1 = M_1$

Overall phenol balance:

$FX_f + SY_s = E_1 + R_1 = M_1X_{m1}$

As there is no phenol contains in solvent Y_s= 0

$$X_{m_1} = \frac{FXf+Sys}{M1}$$

$$X_{m1} = \frac{500\times0.2 + 500\times0}{1000}$$

$$X_{m1} = 0.1$$

Show the point X_{m1} on the feed line FS_1

Draw the line through X_{m1} which is parallel to nearest tie line.
Find the points y_1 and x_1 on the plot which gives the concentration of phenol in Extract and Raffinate Phase respectively.

X_1= 0.06 Y_1= 0.12

$E_1 + R_1 = M_1$

$E_1y1 + R_1x_1 = M_1x_{m1}$

$E_10.12 + (500 - E_1)= 1000$
$E_1 = 666.66$
$R_1 = 333.33$
Phenol extracted by first calculation is 73.154 mg.

Power requirement calculation:

The power input to the agitator shaft where as calculated from the speed and torque and was found to vary as the cube of the speed. The type of agitator used by Scheibel as found to have a general correlation for the power consumption of ,

$$N_{Re} = \frac{\rho N D a^2}{\mu}$$

Where,

ρ – density of dispered phase 0.982 kg/m^3 N

- Speed Of Agitator =5RPS

Da- Diameter of Agitator 0.02 m

μ –Viscosity Of Mixture 2Cp

$$N_{Re} = \frac{\rho N D a^2}{\mu}$$

$$= \frac{982.5\times5\times9\times10^{-6}}{2\times10^{-3}}$$

$$=22.108$$

From The Power Curve Graph:

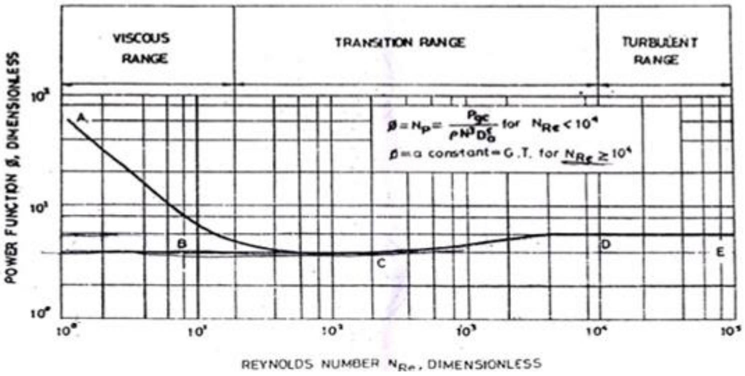


Fig 5- Power Curve Graph

From Power Graph Value Of Power Factor is 5.

Calculation Of Power Requirement

$$p = \frac{\emptyset \rho N^3 Da^5}{g_c}$$

Where as

D - is the outside diameter of the agitator is 3 mm=0.003m

ρ –average density in the mixing section based on one-third holdup of the dispersed phase is 7.23 lb / ft^3=982 kg/m^3

N-is the speed of the agitator is 5 rev/sec,

g- is the gravitational constant of 32.2 ft/sec=9.81 m/s^2

P is the power input per agitator in ft-lb^/sec. =watt

$\emptyset$- is the power factor which is 5 from the power curve

$$= \frac{5\times982.5\times5^3\times0.003^5}{9.81}$$

$= 1.5210^{-8}$ hp= 1.13410^{-8} kwat

$=1.13410^{-5}$ watt

CONCLUSION

The Great advantage of this project is due to emulsification the droplets are breaks down in the very small size particles and the rate of relation increase rigorously.

-Also due to very less amount of Toluene can be extract more than 85% of phenol within two stage operation,

-The Power requirement is too much lower for experimental performance.

This study led to the following conclusions on the performance of the York-Scheibel column with the Water-Phenol-Phenol system used.

1. Agitator speed had no effect on product purity up to the Optimum practical purity observed,

2. Column efficiency decreased with increasing agitator speed.

Recommendation For Further Study.

The most obvious area for investigation with the same ternary system would be at lower agitator speeds, since efficiencies were found to decrease as agitator speed was increased. Studies of other salting-out systems could also provide information about similarities in dehydration ability or performance characteristics. Eventually it might be possible to obtain performance-oriented correlations for salting-out systems, or for all extraction systems, involving the use of York-Scheibel extraction columns. If ternary diagrams were available at higher temperatures, the effect of preheating the extract feed to permit the use of higher salt concentrations could be investigated. The salt feed concentrations and the column temperature could be controlled to eliminate salt crystal deposits that would clog the system. Another area in which a great deal of work could be done is in computerized calculation procedures. Curve fitting the ternary equilibrium data could lead to of the number of theoretical transfer units and mass transfer coefficients, thereby eliminating the tedium normally associated with such calculations. complete analytical solution

Fig.Experimental Setup of York-Scheibel Column

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