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Determination of sodium chloride in crude oil by a new liquid-liquid extraction apparatus in Gachsaran county oil wells

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ABSTRACT

A new automated Liquid-Liquid Extraction (LLE) apparatus have been designed and the applicability of this device was tested by the analysis of salt in crude oil of Gachsaran region in south of Iran. All of the results were compared with the standard test method. The advantages of this device include portability, water reservoir, cooling system, temperature controller and water level controller. The statistical test methods including t-test and F-test showed good accuracy and precision respectively in the analysis of sodium chloride in crude oils. The results showed that the mean of salt concentration as sodium chloride were 31.0 g m^{-3} with the relative standard deviation of (%RSD) 7.9 in Gachsaran County Oil Wells.

KEYWORDS

Sodium chloride;
Liquid-Liquid Extraction;
Gachsaran region.

INTRODUCTION

Many processes in chemical engineering require the separation of one or more of the components of a liquid mixture by treating the mixture with an immiscible solvent in which these components are preferentially soluble[1]. In some cases purification of a liquid may be the function of the process, in others the extraction of a dissolved component for subsequent processes may be the important aspect. An example of the former is the preparation of the pure organic liquids from products of the oil industry. Liquid-liquid extractions may also be used as energy saving processes by, for example, eliminating distillation stages. It is possible, of course that the substance of interest may be heat-sensitive anyway and that distillation is accordingly an unacceptable process[2].

When separation by distillation is ineffective or very difficult, liquid extraction is one of the main alternatives to consider. Close-boiling mixtures or substances that cannot withstand the temperature of the distillation, even under a vacuum, may often be separated from impurities by extraction, which utilizes chemical differences instead of vapor pressure differences. For example, penicillin is recovered from fermentation broth by extraction with a solvent such as butyl acetate. Another example for liquid extraction is recovering acetic acid from dilute aqueous solutions; distillation would be possible in this case, but the extraction step considerably reduces the amount of water to be distilled[3].

When either distillation or extraction may be used, the choice is usually distillation, in spite of the fact that heating and cooling are needed. In extraction the solvent must be recovered for reuse (usually by distillation), and the combined operation is more complicated and often more expensive than ordinary distillation without extraction. However, extraction does offer more flexibility in choice of operating conditions, since the type and the amount of solvent can be varied as well as the operating temperature[3]. In many problems, the choice between the methods should be based on a comparative study of both extraction and distillation.

In liquid-liquid extraction, as in gas absorption and distillation, two phases must be brought into contact to permit transfer of material and then be separated. Extraction equipment may be operated batchwise or continuous. The extract is the layer of solvent plus extracted solute and the raffinate is the layer from which solute has been removed. The extract may be lighter or heavier than the raffinate, and so the extract may be shown coming from top of the equipment in some cases and from the bottom in others. The operation may of course be repeated if more than one contact is required, but when the quantities involved are large and several contacts are needed, continuous flow becomes economical[3].

The rate at which a soluble component is transferred from one solvent to another will be dependent, among other things, on the area of the interface between the two immiscible liquids. Therefore it is very advantageous for this interface to be formed by droplets and films, the situation being analogous to that existing in packed distillation columns.

Analysis of the Salt (chloride) content of crude oil is important because this allows the oil to be evaluated for potential to corrode equipment and pipelines. Salt measurements are taken along the supply line from well head to Refinery, rapid sample determination allows prompt corrective action when unacceptable levels of salt are present.

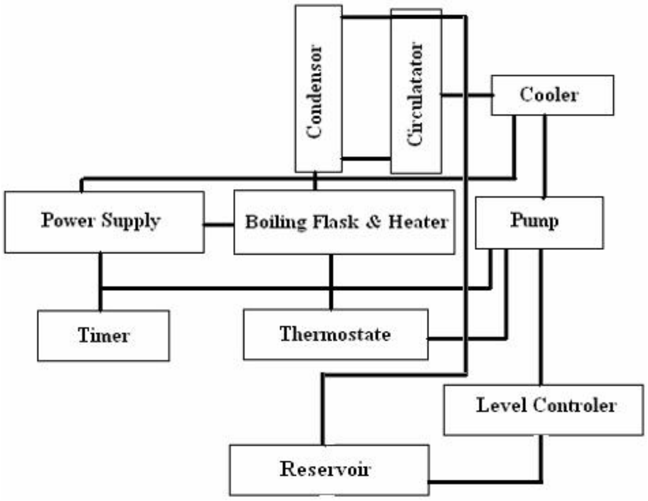

Figure 1 : Liquid-Liquid extraction apparatus.

In the present work a reliable and portable liquid-liquid extraction apparatus have constructed involving water reservoir, cooling system, temperature controller and water level controller (Figure 1). The applicability of the method was evaluated by the extraction of salt (sodium chloride) to aqueous phase and measured by the reversed titration using Volhard method.

EXPERIMENTAL

Chemicals

All the chemicals were of analytical grade and were purchased from Fluka unless stated otherwise. Doubly distilled water was used throughout.

A 5.0×10^{-2} mol L^{-1} potassium thiocyanate solution was prepared by dissolving 0.4859 g potassium thiocyanate (99.5 %) in water and the solution was diluted to 100 ml with water in a 100-ml volumetric flask.

Recommended procedure

According to the standard test method (ASTM 2038), an amount of 80 g of crude oil was poured into a 250 ml glass balloon and heated to 60 ^{o}C. Then 40 ml toluene was heated to the same temperature and added to the glass balloon slowly. After that 25 ml ethanol and 15 ml acetone added to the solution and boiled for 12 min. After cooling at ambient temperature, the aqueous layer was filtered and separated to a flask. About 100 ml of the filtered aqueous layer was separated and acidified with 5 ml nitric acid (65%). After removing of hydrogen sulphide completely, 100 ml amyl alcohol and 3 ml ferric alum added to the solution. Then, the concentration of salt as sodium chloride was determined base on the Volhard method using potassium thiocyanate as the titrant.

Reproducibility

To check the reproducibility of the new Liquid-Liquid extraction apparatus, analysis of salt in crude oil was repeated for five times (TABLE 1). As it is shown, the recommended apparatus and procedure has good reproducibility.

TABLE 1 : Replicate determination of sodium chloride in crude oil after extraction by liquid-liquid extraction.

No.	Sample	Date	Sodium chloride ($g\ m^{-3}$)	Mean ($g\ m^{-3}$)	RSD (%)
1	Crude oil	24-02-2010	33.0	31.0	7.9
2		25-02-2010	33.0		
3		26-02-2010	28.0		
4		27-02-2010	33.0		
5		28-02-2010	28.0		

Statistical tests

T-test

In order to compare the accuracy of the Liquid-Liquid extraction apparatus with older models, the analysis of sodium chloride in crude oil was replicate five times with each apparatus (TABLE 2). The results of the t-test ($t_{cal}<t_{crit}$) shows good agreement between two systems (TABLE 3).

TABLE 2 : Replicate determination of sodium chloride in a crude oil sample after extraction by two liquid-liquid extraction apparatuses.

Rows	Sample	Date	Sodium chloride/ gm^{-3}	
			System 1*	System 2**
1	Crude oil	31-01-2010	23.0	28.0
2		02-02-2010	28.0	23.0
3		03-02-2010	23.0	23.0
4		04-02-2010	37.0	35.0
5		05-02-2010	28.0	28.0

*** Older Liquid-Liquid extraction apparatus**

**** Constructed Liquid-Liquid extraction apparatus**

TABLE 3 : The results of t-test (with different source variances) between the results of sodium chloride analysis in crude oil by two different extraction apparatus.

Sources	Variable 1	Variable 2
Mean	25	25.4
Variance	9	9.8
Observations	5	5.0
Hypothesized Mean Difference	0	
df	8	
t Stat	0.21	
P(T<=t) one-tail	0.421	
t Critical one-tail	1.86	
P(T<=t) two-tail	0.84	
t Critical two-tail	2.31	

F-test

The F-test is designed to test if two population variances are equal. It does this by comparing the ratio of two variances. The results of F-test on the replicate sodium chloride analyses (TABLE 4) showed a good precision between two extraction apparatuses ($F_{cal}<F_{crit}$).

TABLE 4 : The results of F-test (confidence level 95%) for comparing the precision of two extraction apparatuses.

Sources	Variable 1	Variable 2
Mean	25	25.4
Variance	9	9.8
Observations	5	5
df	4	4
F	1.19	
P(F<=f) one-tail	0.05	
F Critical one-tail	6.39	

CONCLUSION

In this research, a Liquid-Liquid extraction apparatus have designed with the advantages including water reservoir, cooling system, temperature controller and water level controller. Because of the water circulation in condenser, it is a powerful instrument as a portable extractor.

The applicability of this system have evaluated in the salt analysis of crude oil in Gachsaran Oil & Gas Producing Company. The statistical tests such as t-test and F-test show good precision and accuracy in comparison with older models.

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