Separation of Cationic Surfactant using Adsorptive Bubble Separation Method

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ABSTRACT:

Separation of various chemical components from each other is often the most difficult step in analytical procedures. The problems attached to separation become further magnified when the species concentations are extremely low. A group of techniques that has proven useful especially in dilute solutions for separating and concentrating metallic as well as nonmetallic ions and complexes, proteins, microorganisms, particulates, etc. is the adsorptive bubble separation techniques.Minerals have indeed been treated using some of these techniques for decades. The success or these processes is primarily dependent upon differences in the natural surface activity of various species or particulates in the system or in their tendency to associate with surfactants. The efficiency of the process is determined by such variables as solution pH,ionic strength,concentration or various activating and depressing agents, and temperature.A proper control or variables offers an opportunity to separate a variety of metallic and nonmetallic species and particulates. The solvent sublation is very simple, effective and fast process for removal and separation of chemical compounds from dilute aqueous solutions and can be treated as preconcentration method in their analytical determination.In this paper the principles governing various foam separation techniques, are presented along with the recent results on the role of variables that can be controlled to achieve complete removal of species and particulates for analytical purposes.

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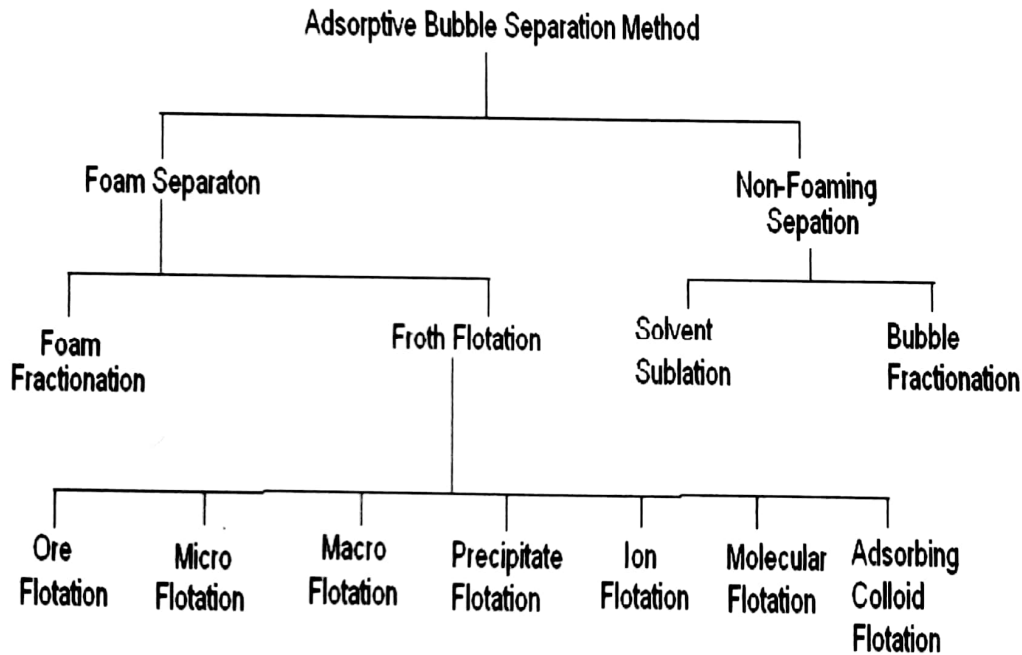E-mail: gmukhopadhyay8@gmail.com.

KEYWORDS:

Anionic Surfactant, Dilute Solution, Absorptive Bubble Surfactant.

1. INTRODUCTION[1]:

Adsorptive bubble separation technique, the generic name was first proposed by Lcmlieh[2] (1966). This technique is based on differences in surface activity. Material, which may be molecular colloidal,or macro particulate in size is selectively adsorbed or uttnched at the surface of bubbles rising through the liquid and is thereby concentrated or separated.A substance, ,, hich is not surface active itself. can often be made effectively surface active through union with or adherence to a surface-active collector. The substance sll removed is termed as colligend.

This extends Adsorptive Bubble Separation method applicable to a quite wide range of substances including ions, molecules, precipitate, active carbon, minerals, proteins and bacteria.

1.1 **Classification of Adsorptive Bubble Separation Method[4]:**

Adsorptive Bubble Separation Method

Foam Separaton

Non-Foaming Sepation

Foam Fractionation

Froth Flotation

Solvent Sublation

Bubble Fractionation

Ore Flotation | Micro Flotation | Macro Flotation | Precipitate Flotation | Ion Flotation | Molecular Flotation | Adsorbing Colloid Flotation

a) **Ore Flotation:** A special case of froth flotation of mineral particles from their ores.

b) **Macroflotation:** The separation of macroscopic particles by foaming specially colloid or microorganisms.

c) **Adsorbing colloid flotation:** The separation of dissolved materials by adsorbing an colloid particles which are surface active.

d) **Ion flotation:** The separation of surface-inactive ions by foaming with a sur- factant, which yields an insoluble product (called sublate), especially if removed as a scum.

e) **Molecular flotation:** The separation of surface-inactive molecules by foam- ing using a surfactant which yields an insoluble product.

f) **Precipitate flotation:** The process in which a precipitate is removed by a surfactant, which is not the precipitating agent.

No foaming adsorptive bubble separation techniques include bubble fractionation, and solvent sublation.

1.2 Mode of operation in Adsorptive Bubble Separation Method[4]:

   a) Simple Batch

   b) Simple Continuous Flow

   c) Continuous Flow Enriched by Reflux

   d) Continuous Flow Stripping

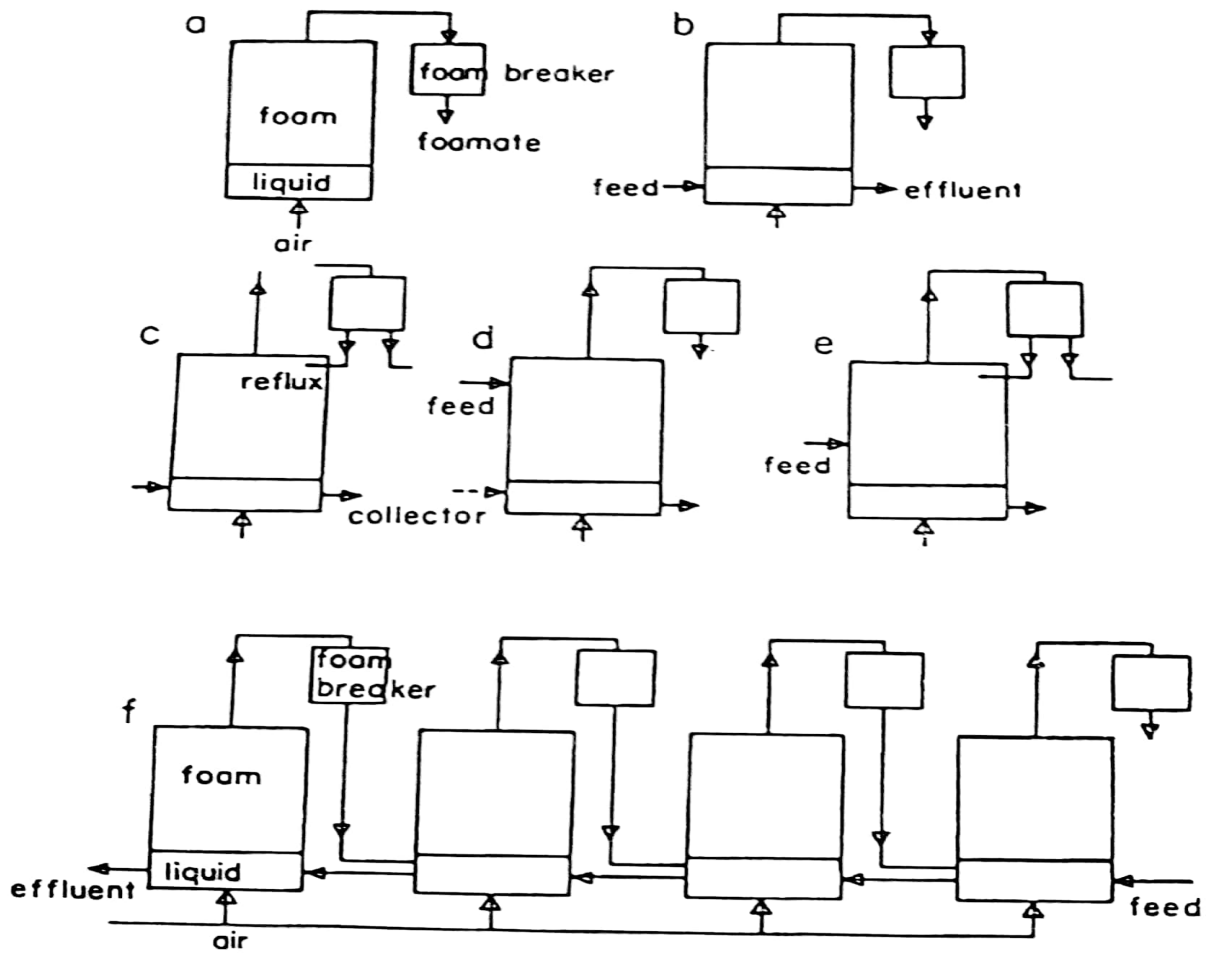   e) Combined Enriching and Stripping

f) Staged Operation

FIG. 1, Different types of mode of operation in ABSM

1.3 Principle[4]:

In Adsorptive bubble separation method,as described before,depends upon the differences in physico-chemical properties of particles.Under equilibrium conditions of dilute solutions adsorption of surface active species from bulk solution at a gas-liquid interface can be quantitatively described by the Gibbs' equation as ;-

$$\frac{\tau}{c} = -\frac{l}{RT}\frac{d\gamma}{dc}$$

'τ' is the surface excess of the adsorbed solute (i.e. concentration at the surface **g mole/cm²**),'C' is the bulk equilibrium concentration,and can be considered a distribution factor,'γ' is the surface tension,'R' is the gas constant,and 'T' is the absolute temperature.The value of (dy/dc) may be readily determined from the slope of γ^{-c} plot.Better separation would always occur below the Critical Micelle Concentration.

1.4 Application of ABSM:

a) Separation of heavy metals like uranium from aqueous solution

b) Recovery and separation of ore

c) Waste water purification by removing toxic inorganic like mercury,lead etc.

d) Removal of oxyions from solution

e) Foam fractionation for separation of enzymes, proteins of biological materials

f) Recovery of protein and microorganism from cultivation medium

g) Surfactant removal

h) Removal of active radio isotopes

i) Separation of fatty acids

j) Separation of organic dyes

k) Separation of protein from beet root juice and potato juice

l) Enrichment of plant proteins

m) Foam fractionation in protein skimming

n) Foam fractionation of bile acids

2. LITERATURE SURVEY:

In the year 1996 Daniel W. Armstrong[5] et al. and his coworkers worked on Foam Flotation Enrichment of Enantiomers. The chiral collectors used in his study are able to associate with analytes via Ligand exchange interaction, Hydrophobic complexation, and Hydrogen bonding interaction among others.Most of the enantiomeric ratio in the foam fractions were determined by HPLC.It can be concluded that effect of reflux equilibration time and concentration of the chiral collectors on the enantiomeric enrichment of D-N-t-BOC-alanine.The curve represent different concentration of the chiral collectors like 1%,0.1%,0.01% and 5% vs Enantiomeric excess% in the room temperature.

In the same year 1994 Borje Sellergreo[6] et al. worked on Direct Drug determination by selective sample enrichment on an Imprinted Polymer. Pentamidin (PAM), a drug used for the treatment of AIDS-related pneumonia, can selectively be removed from a dilute solution. At a physiological concentration (30nM) this gave an enrichment factor of 54 using a PAM selective polymer. Here chromatographic analysis is needed. PAM could be enriched and directly analyzed when present in low concentration in urine sample.

In the year 1995,Anand.K[7] et al.,studied the Kinetic adsorption of Lysozymes and Bovine Serum Albumin (BSA) at the air water interface from binary mixture.They showed that the increase in BSA concentration in bulk phase caused a decrease in the extent of adsorption of lysozymes and an exponential decrease in it's apparent diffusion coefficient,the lag time for it's adsorption,however decreased progressively with the increasing bulk concentration of BSA.The ratio of BSA to Lysozyme in the mixed monolayer increased with increasing in the ratio of BSA to Lysozyme in the bulk phase.However the unit cell dimensions occupied by BSA and Lysozyme in the mixed monolayer were the same as those in single component monolayer,suggesting that both BSA and Lysozyme adsorbed independently and the thermodynamic state of one protein was not modified by other protein in the interface.

In the year 1998,Armstrong[8],D.W. and his co-workers investigated the bubble fraction of enantiomers from solutions using molecularly imprinted polymers as collector. They investigated the enantiomer selectively of phenylalanine analide with molecularly imprinted polymer. The polymer was prepared by methacrylic acid approximately 17 mole % and 83 mole %. Ethylene glycol dimethacrylate. Both the molecularly imprinted polymers were used to test for an identified nonenantioselective adsorption.The foaming device used consist of cylindrical colume of 25cm in height, the top of the colume was removed by heating with a Bunsen burner, a ledge and approximately 1cm wide was then form at the top of this colume by heating and shaping the glass.

In the year 1998,Brown,A.K[9] et al.,investigated the foam separation of different protein mixture like β-casein:Lysozyme,BSA:Lysozyme and β-casein:BSA.The foaming device used for the investigation consists of glass,having height 0.51m,with a flat plate sparger of perforated ceramic plate 2cm in diameter with 20 evenly distributed 0.1cm diameter holes.Humidified N_2 gas was passed through the base of column via sparger at the rate of 600 cc/min,the foam height was 0.34m.The result revealed that the enrichment ratio in the foam was found to decreased when the concentrations of proteins in the feed solution was increased.The other process variables were gas flow rate,P^H,feed flow rate,column height,foam height,shape and size of flat sparger.

In 1999 Zhi-dong Chang[10] and coworkers studied on foam separation of tributyl phosphate from aqueous solutions. Tributyl phosphate(TBP) can be concentrated from dilute aqueous solution by foam separation with sodium dodecylsulfate (SDS) or cetyl trimethyl ammonium bromide(CTAB) as surfactant. The highest TBP concentration in the foam overflow from the separation column is about 5-7 times that of TBP in the aqueous feed. When the surfactant

concentration is low as just enough to maintain the foam stability, the TBP concentration in the foam has the higher enrichment under the experimental condition. Foam separation has been reported usually for two-component system. In this paper, a mathematical model is developed to describe the process of foam separation of tributyl phosphate(TBP) from aqueous solutions using sodium dodecyl sulphate(SDS) as surfactant. The model assumed that the bubbles are uniform in size and have regular dodecahedral structure. The solution in liquid pool has uniform concentration and the enrichment is completed in the course of film drainage. The model is calculated under different operating parameters.

In the year 2000, Schugerl[11] had studied the recovery of proteins and microorganisms from cultivation media by foam floatation method. He also presented a clear description of foaminess of proteins and microbial cell cultivation system in his paper. He investigated the recovery of Bovine serum Albumin (BSA), a globular protein from aqueous solution by foam flotation. The foaming device used consists of a thermostated column with 23mm internal diameter, 49cm bubbling liquid height and 30 cm from layer.

In the year 2001, Zaid S[12] and co-worker studied of the separation of proteins from multicomponent mixtures by a semi-batch foaming process. Their experiences was conducted to obtained the values of the average bubble size, gas holdup, interfacial area, the bulk phase concentration and the heat desorption (which determines the concentration of absorbed components) in the liquid solution using a 75mm diameter, 530mm long glass column fitted with stainless steel sparger for bubble generation. Here the mass transfer coefficient was determined from the analysis of concentration of foam and feed at times using the above mentioned parameters.

 In the year 2003, Darton[13] and his co-worker worked on the development of a multistage foam fractionation column. Here, surface-active materials stabilize foam by adsorption at the gas/liquid interface. In foam fractionation, the foam is condensed to give a 'foamate', liquid rich surfactant, which is extracted and concentrated on organic solute. The measured liquid composition were in good agreement with a model, which described the equilibrium using an adsorption isotherm, which makes a mass balance for each stage in the column.

In the year 2004 Backleh M[14] and his coworkers works on enrichment of the glycoalkaloids alpha-solanine and alpha-chaconine from potato juice by adsorptive bubble separation using a pH gradient. For the first time, Adsorptive Bubble Separation (ABS) could quantitatively enrich the solanidine alkaloids alpha-solanine and alpha-chaconine from potato juice with a pH gradient. The enrichment into the foam was influenced bt the pH value, bubble size and

gas flow rate. The efficiency was highest on using diluted samples with a concentration between 2 and 6 mg L(-1) of the alkaloids at pH 6.0. The experiments with a standard solution of each alkaloid confirmed that these substances could be quantitatively enriched into the 'spumat' without surface-active potato proteins. The transfer into the foam fraction under these conditions was similar to that from the aqueous potato extract.

In the year of 2005 Kazimierz Jurkiewicz[15] worked on the adsorptive bubble separation of zinc and cadmium cations from solution in the presence of ferric and aluminium hydroxides was carried out by means of Tween 80 (non-ionic surfactant) and sodium laurate and stearate (anionic surfactants). The mechanism of metal removal is different depending on the nature of the surfactant used. The removal of zinc cations by adsorbing colloid flotation is higher than that of cadmium cations. It increases with increase in the amount of hydroxide precipitate and the concentration of Tween 80. The removal of zinc cations by ion flotation is lower than that of cadmium cations. It does not change with increases in the hydroxide amount. It increases, however with increased sodium laurate or separate concentration.

In the year of 2005,K.Malysa[16] et al.,showed the influence of surface active substance on the bubble motion and collision with various interface.There are three distinct stages in the bubble motion: 1) a rapid acceleration 2) a maximum velocity value followed by it's monotonic decrease 3) attainment of terminal velocity.It's showed that the bubble terminal velocity decreases rapidly at low surfactant concentration.When the rising bubble arrive sat liquid/gas interface or liquid/solid interface there can be formed either foam or wetting film or three phase contact (TPC).Time scale of TPC formation is very short.

In the year 2006,Gerken et al. and in 2008,Qu et al. performed the foam fractional studies in continuous stripping mode and not in batch mode.

In the year 2012,Rujirawanich et al. investigated the effect of process and design parameters such as air flow rate,feed flow rate,feed concentration and column height on the separation of multicomponent systems.He applied the concept of competitive adsorption to it's study of separation of surfactant mixtures of Cationic Cetylpyridinium Chloride (CPC) and noniomic Triton X-100 using a continous multistage foam fractionation system.However the complexity of the system limited the depth of insight into the fundamentals of the process.

3.AIMS AND OBJECTIVES:

1. To study the enrichment and removal of Surface Active Agents and some Organic Compounds from a dilute solution by Adsorptive Bubble Separation Method.

2. To study the effect of following variables on the enrichment ratio,separation ratio and percentage of recovery of separation process:

- Concentration of drug
- Concentration of collector
- P^H of the feed solution
- Gas flow rate
- Column diameter
- Presence of electrolyte

4.DISCUSSION:

4.1 The Different Collectors used:

A) Surface Active Agents:

a) Cetyltrimethyl ammonium bromide

b) Sodium lauryl sulphate

c) N-heptyl-L- hydroxy proline

d) Dodecylbenzene sodium sulphonate

e) Lauryl pyridinium chloride

f) Sodium laurate

g) Octadecyltrimethyl ammonium chloride

h) Dodecylamine

i) Hexadecyltrimethyl ammonium bromide

j) Tetradecyltrimethyl ammonium bromide

B) Chiral Collector:

a) Cyclodextrine (e.g. Heptakis-2,3,6-tri-O-methyl-β-cyclodextrine)

b) Alkylated amino acids

c) Digitonin

d) Antibiotics (e.g. Vancomycin,Rifamycin B)

4.2 Factors Affecting the Efficiency of ABSM:

a) Concentration of Collector

b) Colligend Concentration

c) Collector-colligend ratio

d) Pulshed Addition of Collector

e) P^H

f) Ionic Strength

g) Temperature

i) Gas Flow Rate

j) Presence of Other Auxiliary Reagents

k) Surface Area of Bubbles

l) Foam Height

m) Foam Density

n) Foam Drainage

o) Equipment Design

4.3 Chain Length of the Surfactant: For reasons given earlier,an increase in length of the nonpolar part of the surfactant should lead to an increase in its adsorption at interfaces and therefore it's separation.Our results for alkylammonium acetates have in fact shown that these reagents adsorb at the solution/air interface in increasing quantities as the chain length is increased.The length of the hydrocarbon chain of the surfactant was found to affect froth flotation of materials in a similar manner.Hallimond tube flotation of quartz with alkylammonium acetates of varying chain length is shown in Fig.2 as a function of the surfactant concentration.It can be seen that the percent removal increases drastically on increasing the chain length of the surfactant.The chain length effect on flotation was ascribed to the tendency of the longer chains to associate into two dimensional aggregates called hemi-micelles at the solid–liquid interface.The driving force for this association is the cohesive interaction between the chains and therefore is dependent on the chain length.

4.4 Surfactant Concentration: It is also evident that the percent removal is strongly dependent on the concentration of the surfactant.This is not due to the dependence of physical properties or stability of foam since effects of such factors are eliminated in a Hallimond tube test.Rubin and co-workers,among others,have also observed dependence

of collector concentration on the precipitate flotation of copper species.They found a collector to colligend ratio of one to be necessary in their case to get nearly complete removal of the copper.Concentration of the collector was found to be even more critical if ion flotation is used instead of precipitate flotation.Rubin and Lapp have reported that while 100% removal of zinc species is possible using a collector to colligend ratio of 0.2 in the pH range of 8 to 11 where zinc hydroxide precipitates,almost no flotation is obtained at that ratio below pH 8 when zinc is present in dissolved ionic form.A larger quantity of collector was needed to remove the zinc completely under these conditions.

A great excess of collector has, however, been found to reduce the flotation

of minerals,precipitates, and ions.In the case of particulate flotation,this is sometimes due to a reduction in the size of the bubbles to such a level that the bubbles could not levitate the large number of particles that collect on them. Adsorption of a second layer of collector at higher concentration with an orientation opposite to that of the first layer or adsorption of micelles can also cause a decrease in flotation , but less likely to do so in most cases since only a small fraction of the surface needs to be hydrophobic for flotation to occur. The inhibitive effect of excess collector on ion flotation has been proposed by Davis and Sebba to be mainly due to possible crowding of the bubble surface by the collector ions themselves and the formation of micelles with consequent adsorption of colligend ions on the nonfloatable micelles.Both for ion flotation and precipitate flotation,an optimum collector to colligend ratio is reported to exist.One technique reported by Grieves et. al. for removing additional colligend from solution is by adding the collector in pulses instead of in one dose.

4.5 Solution p^H: The role of pH in determining the form of the species present in solution and thereby its flotation as ions or precipitates is evident in the work of Rubin and Lapp discussed earlier.The effect of pH on particulate separation is even more significant.In fact,it is primarily the proper choice of pH along with the type of collector that enables one to selectively float one type of particulate from another and thus obtain their separation.This effect of pH is illustrated in Fig.3 where flotation of calcite with an anionic and a cationic collector is given as a function of pH at two concentrations.The isoelectric point of calcite as measured by streaming potential is about p^H 8 to 9.5.It can be seen that significant flotation with an anionic collector is possible only below the isoelectric point where the particles are positively charged.Similarly flotation with a cationic collector is possible only above the isoelectric point where the particles are

negatively charged.For analytical puroposes,one is interested in determining how this material can be separated if mixed with another material,for example quartz.The isoelectric point of quartz is near pH 2.Between P^H 2 and 8 quartz is therefore negatively charged while calcite is positively charged.It is therefore possible to achieve a separation either by floating calcite with an anionic collector at,for example,pH 7 or by floating quartz with a cationic collector at that pH.It must be noted that this separation is likely to fail at much lower pH values since dissolved calcium species,for reasons given elsewhere,will activate the anionic flotation and depress the cationic flotation of quartz.
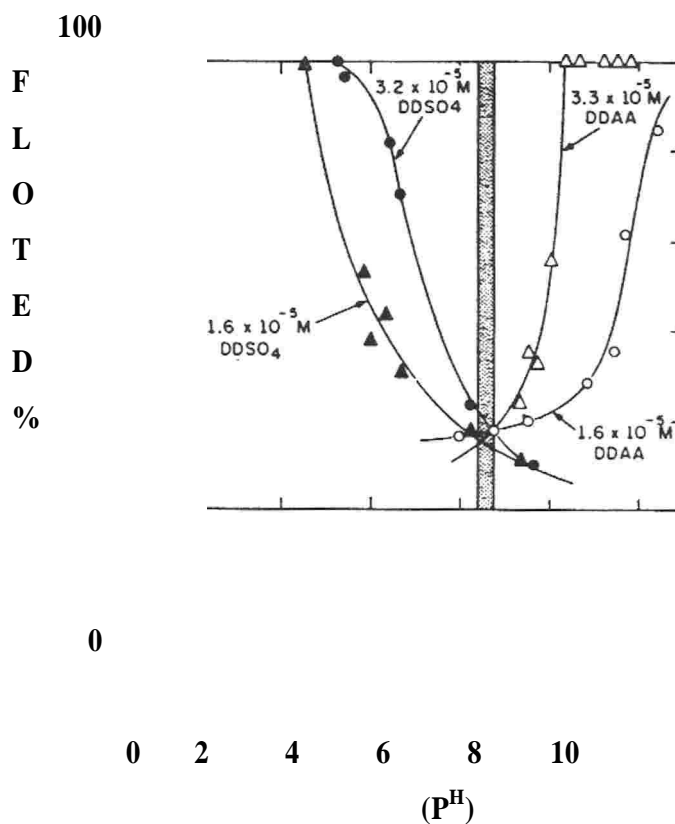

FIG. 3, The effect of p^H on the anionic and cationic flotation of Calcite

Control of pH can be similarly used for separation purposes with other foam techniques.As an example,distribution factors obtained by Karger et al. for mercury and iron in the presence of an amine are given in Fig. 4.These results suggest that the separation of mercury from iron can be obtained either by floating the former at higher pH values or the latter at lower pH values.

In addition to the above effects.pH also influences separation due to dependence of the collector hydrolysis on it.A typical example of this is the cationic flotation of quartz

inbasic solutions.Quartz is negatively charged above pH 2 and therefore it should be possible to float it with a cationic collector above this pH.In practice, however,one gets very little ftotation of quartz with dodccylammonium acetate above p^H 12.Above p^H 12,most of this collector is in its neutral molecular form and under such conditions it is apparently unable to adsorb on quartz and make it hydrophobic.
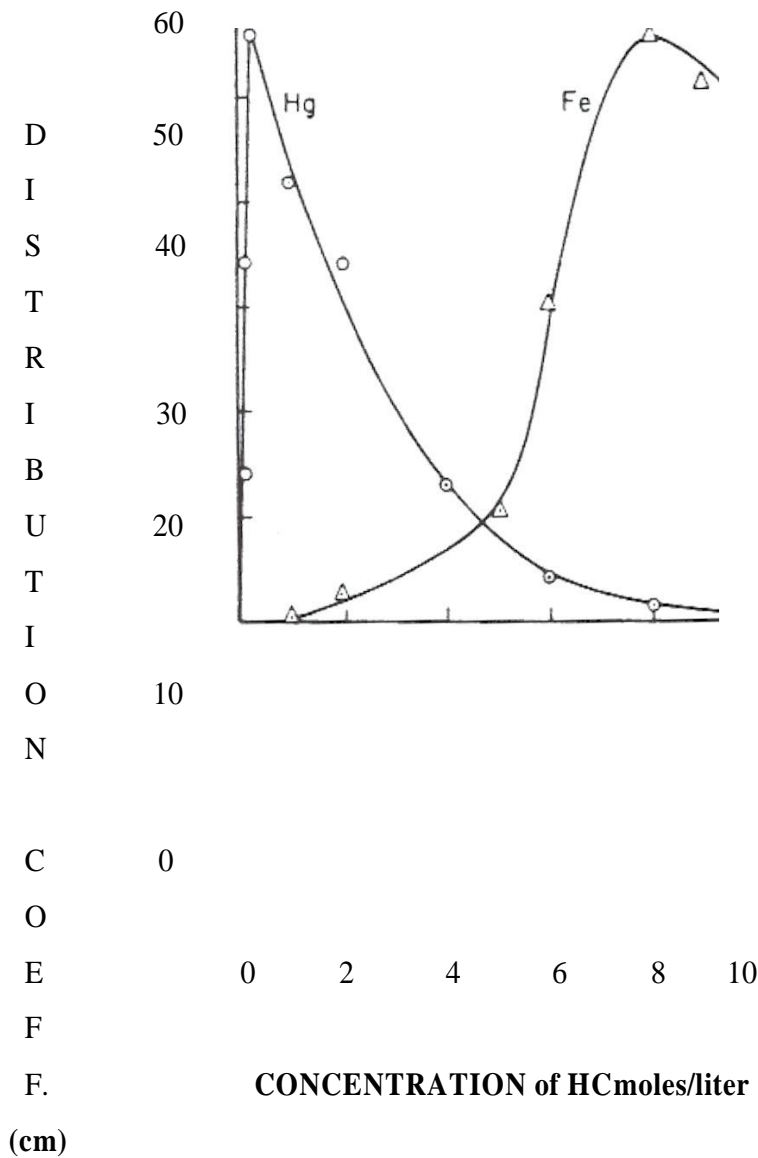

FIG. 4, Distribution coefficients for Fe and Hg as a function of HCI concentration in the presence of a cationic surfactant

Neutral molecules can,however,act as good collectors when present along with ionic surfactant species.Total adsorption of

the surfactant at the solid/solution interface and hence flotation in a system containing both ionic and neutral surfactant species appears from the experimental results to be higher than when the same amount of surfactant is present totally in one or the other form.This is suggested to be due to the fact that,if some of the species adsorbed on the solid are neutral,they can actually screen the repulsion between the charged heads of the adsorbed ions.Based on the same principle, Fuerstenau and Yamada were able to enhance flotation by adding long-chain alcohol along with the collector.

4.6 Ionic Strength: It is possible to float quartz with an amine collector above pH 2 because,as mentioned earlier,cationic aminium species adsorb clectrostatically on the negatively charged quartz.Such electrostatic adsorption of aminium ions will take place in competition with other ions that are similarly charged.A significant increase in concentration of nonsurfactivc cations will therefore decrease the adsorption of the cationic collector ions on the solid and hence also its flotation.Results obtained recently for the cationic flotation of quartz show this to be the case (Fig.5). Potassium nitrate thus acts as a depressant for the cationic flotation of quartz.Modi and Fuerstenau have observed similar effects of sodium chloride on the anionic flotation of alumina. When sodium sulfate was added to the system instead of sodium chloride,the depression of flotation was even larger.This larger effect of sulfate over that of chloride results from the tendency of the bivalent sulfate to strongly adsorb and compete with dodccylsulfate more than the monovalent chloride.The above effect can also be used to enhance the flotation of a particle that has a charge opposite to that of the collector.Modi and Fuerstenau were thus able to get complete flotation of the positively charged alumina at pH 6 using a cationic surfactant by adding sufficient sodium sulfate to get a concentration of 10^{-2} mole/ liter.Flotation of alumina occurs in the presence of bivalent sulfate ions because specifically adsorbing sulfate at such concentrations reverses the charge of the alumina particle to a negative sign and thus makes the adsorption of cationic surfactant possible.Similarly,negatively charged particles can be floated with anionic collectors if the particles are first modified by means of cations such as calcium and magnesium.These ions can often function most effectively in the pH range where they are in the hydrolyzed soluble form.Fuerstenau et al. studied the role of

iron,aluminum,lead,manganese,magnesium and calcium in the anionic flotation of quartz as a function of pH and found that each cation began to function as an activator as the metal ions began to hydrolyze and ceased functioning in that manner when the solution pH corresponded to that at which the metal hydroxides begin to precipitate.
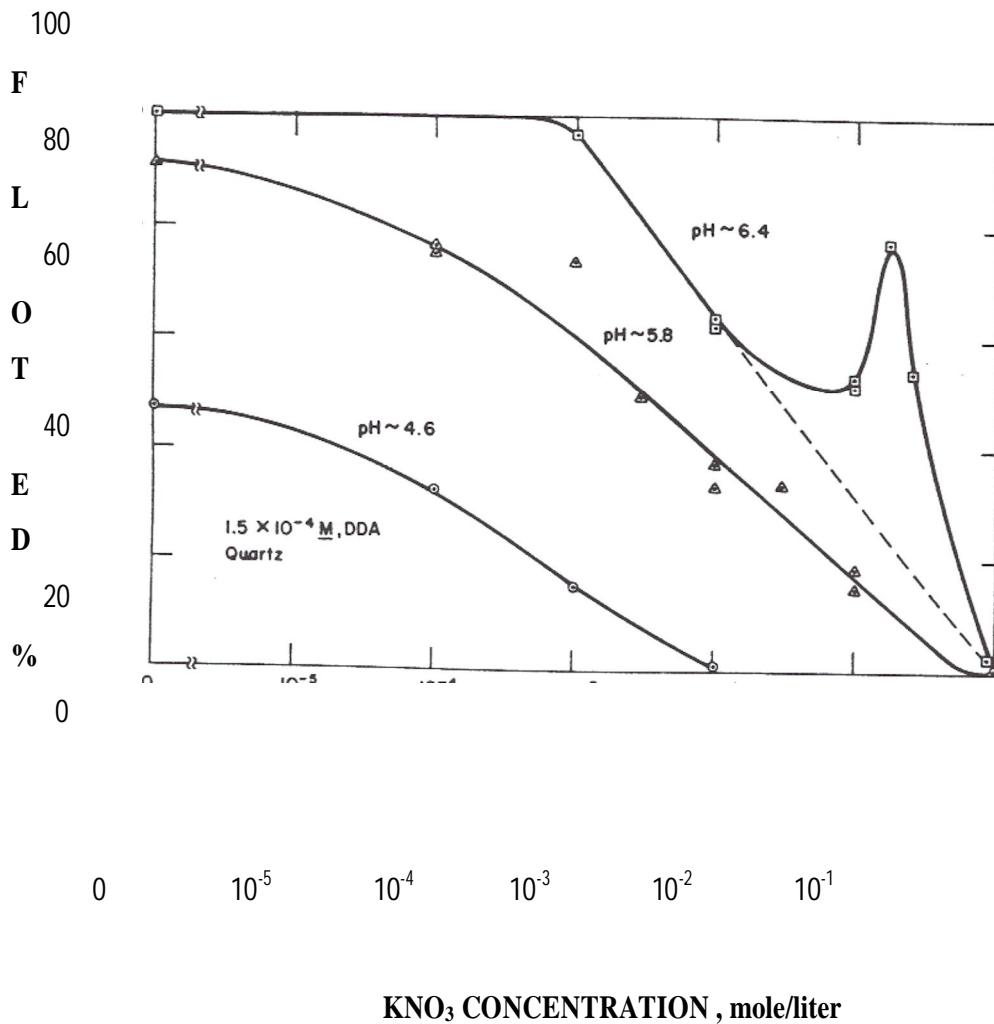

FIG. 5, Effect of ionic strength on the flotation of quartz with Dodecylammonium Acetate (DDA)

5. CONCLUSION:

Potential Areas of Application in Pharmaceutical Field:

1. Separation of drug components or purification of drugs from a mixture of components

2. Separation of enantiomeric drugs mixture

3. Separation of chemical constituents from plant source, e.g. salts of alkaloid mixture, separation from soap, and enrichment of active components by foam fractionation method

4. Enrichment of plant proteins with adsorptive foam separation method

5. Foam fractionation of fruit juice enzymes for example, brumelin from pineapple

6. Removal of drug components from wastewater.

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