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REMOVAL OF SURFACE ACTIVE AGENTS FROM A DILUTE SOLUTION BY ABSORPTIVE BUBBLE SEPERATION TECHNIQUE

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Abstract:

Adsorptive bubble separation technique, the generic name was first proposed by *Lemlich (1996).* This technique is based on differences in surface activity. Material, which may be *molecular, colloidal or macro* particulate in size is selectively adsorbed or attached at the surface of bubbles rising through the liquid and is thereby concentrated or *separated*. A substance, which is *not surface* active itself, can often be made effectively surface active union with or adherence to a *surface-active collector*. The substance so removed is termed as *colligend*. Here we studied Tetradecyl trimethyl ammonium bromide (TTAB), which is a cationic surfactant and Sodium lauryl sulfate (SLS), which is an anionic surfactant. We studied some characteristic properties of them i.e. performance criteria, %removal, Critical micelle concentration, relation with surface tension, surface excess concentration etc. We chose drugs like Gentamycin Sulphate, Neimycin Sulphate as a cationic surface active drugs, Pencillin-G as an anioinic surface active drug. The results showed that the Enrichment ration (Er) of TTAB is 44.67, which is maximum at 2mM, and it decreases as the concentration increases from 2 to 2.75mM. The recovery also decreases as concentration increases. The maximum recovery is 90 at 2mM. Reverse result shows when we used SLS. At the concentration 7mMit was found that the recovery and the Enrichment were 65.02 and 32.50 respectively. But at 7.75mM the recovery and the Enrichment were 49.51 and 24.75 respectively.

Key words: ABSM, TTAB, SLS.

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1. **Introduction:**

It is not always in *drug manufacture* unit that finished products sometimes fail to pass through the quality control steps of good manufacturing practices. In that situation, the *faulty product* is either discarded as waste or the active component is recovered from inert excipients of drug formulations by suitable low cost method. The *different conventional methods* used are solvent extraction, membrane separation, filtration, osmosis, distillation, precipitation, electrophoresis, chromatography etc. I would like to explore the possibilities of 'Foam Separation method' in Pharmaceutical field where conventional methods of separations are laden with many problems and inconveniences. This method also offers an important application in wastewater treatment and removal of toxic material in to prevent environmental pollution. This method is especially cost effective for separation of valuable materials from large volume of very dilute solutions. This foam separation method has already made formidable presence and proved its worth in many separation activities e.g. separation of ore, heavy metal ions from dilute aqueous solutions, separation of component of natural products. [1]

IMPORTANT FEATURES OF ADSORPTIVE BUBBLE COLUMN:-

This method offers some advantages like absence of moving parts eliminating need for seals, minimum maintenance, smaller floor space, large transfer surface per unit volume of column resulting in high mass transfer rate, low construction cost. At low gas velocity (<0.5cm/sec) the bubble diameter is found to be strong function of the orifice diameter and a weak function of the gas velocity in the in the orifice. At high gas velocity bubble diameter depends on gas flow rate. It is important to note the effect of the present of an electrolyte in solution on the bubble size. Because of the surface tension and the electrolyte and the electrostatic potential of the resultant ions at the gas-liquid interface smaller bubble are formed in the presence of electrolytes in the water. Bubble size depends on electrolyte concentration and type of electrolytes. [2]

PRINCIPLE:-

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 Gibb's equation as-

$$\frac{\tau}{C} = -\frac{1}{RT} \cdot \frac{dy}{dC} \qquad [3]$$

T is the surface excess of the adsorbed solute (i.e. the concentration at the surface g.mole/cm2), C is the bulk equilibrium concentration, and can be considered a distribution factor, Υ is the surface tension , 'R' is the gas constant, and T is the absolute temperature. The value of $\frac{dy}{dC}$ may be readily determined from the slope of Υ-C plot. Better separation would always occur below the Critical Micell*e* Concentration. [4]

2. **MATERIALS AND METHOD:-**

- Penicillin-G was gifted by Standard Pharmaceutical.
- Gentamycin and Neomycin were gifted by Greenco Biological Pvt Ltd

- Sodium lauryl sulphate Ar & Tetradecyl trimethyl ammonium bromide were purchased from E.Merck (India) Limited
- Foam fractionation glass column was fabricated by local glass blower.
- Stalagnometer was purchased from local manufacture.
- Beckman- DU 64 Spectrophotometer, Hitachi Ltd, Tokyo, Japan, Model 200-20
- Digital pH meter (Model MK-VI)- SYSTRONICS.

2.1. EXPERIMENTAL PROCEDURE:

The surfactant, Sodium lauryl sulphate (SLS) & Tetradecyl trimethyl ammonium bromide (TTAB/Cetrimide) form stable foam were adsorbed on the foam-bubble interface. As the foam ascends the column, the liquid present in the inter bubble space decreases due to drainage of liquid downward, while surfactant concentrate in the interface of gas bubble and liquid. And then its concentration was measured by titremetric assay method or UV absorbance using a spectrophotometer or HPLC method.[5]

2.2. FOR ASSAY OF TTAB:

Produce 100ml. Transfer 25ml of chloroform, 10ml of 0.1M sodium hydroxide and 10ml of freshly prepared 5% w/v potassium iodide. Shake well and discard the chloroform layer. Shake with three quantities chloroform with 10ml and add 40ml of dilute HCl. Titrate with 0.05M potassium iodide until deep brown color is almost discarded. Each ml of 0.05 M potassium iodate equivalent to 0.03364gm of Cetrimide ($C_{17}H_{38}B$) [6]

2.3. FOR ASSAY OF SLS:

Produce 1000ml with water. To 20ml add 15ml chloroform, 10ml dilute sulphuric acid and 1mldimethylyellow oracet blueB solution. Shake well. Titrate with 0.004M benzethonium chloride until the chloroform layer becomes green. Each ml of 0.004M benzethonium chloride is equivalent to 0.001154gm of sodium alkyl sulphate ($C_{12}H_{25}NaO_4S$).[7]

2.4. PERFORMANCE CRITERIA: [8]

The following performance parameters were determined for each experiment:

- Enrichment ratio (ER):
  It is the ratio of drug concentration in the foamate (Cf) and the drug concentration in the initial feed solution(Ci).
- Separation Ratio (SR):
  It is the ratio of drug concentration in the foamate (Cf) and the drug concentration in the residual solution (Cr).
- Percentage Ratio (PR):
  It is the percentage of the ratio of amount of the mass of drug in the foamate and the mass of drug in the initial feed solution.

3. Results and Discussions:

TABLE 1. REMOVAL OF TETRADECYL TRIMETHYL AMMONIUM BROMIDE (TTAB) WITH TIME, (AT 35 ⍰C, WITH SUPERFICIAL GAS VELOCITY 0.03CM/S AT pH 7):CONC OF TTAB = 2M

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	67.28	20.18	52.14	0.61	77.50	38.75	126.63	77.50
60	2	98	67.28	20.18	53.15	0.61	79.62	39.81	130.09	79.01
90	2	98	67.28	20.18	54.53	0.61	81.64	40.82	132.43	81.05
120	2	98	67.28	23.54	55.60	0.71	82.64	41.32	115.75	82.65
150	2	98	67.28	23.54	56.51	0.71	83.99	41.99	117.63	84.00
180	2	98	67.28	25.91	58.01	0.78	86.22	43.11	109.92	86.23
210	2	98	67.28	26.91	58.70	0.81	87.24	43.62	106.92	87.23
240	2	98	67.28	30.10	59.85	0.91	88.95	44.67	97.43	90.00

Table 2. CONC OF TTAB = 2.25M

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	3	97	75.69	23.29	53.82	0.71	53.33	23.70	74.69	71.11
60	3	97	75.69	23.29	55.50	0.71	53.99	24.41	75.62	7333
90	3	98	75.69	23.76	57.18	0.72	56.66	25.18	78.51	75.55
120	3	97	75.69	23.53	60.00	0.71	59.45	26.42	83.26	79.28
150	3	97	75.69	23.42	60.55	0.71	6.00	26.66	83.56	80.00
180	3	97	75.69	23.29	64.33	0.71	63.74	28.33	89.28	85.00
210	3	97	75.69	23.49	66.03	0.72	65.43	29.08	90.88	87.25

(Time =θ, Foam volume = Vf, Residual volume =Vr, Foam amount =F, Residual amount= R, Residual concentration =Cr, Foamate concentration =Cf, Enrichment ratio= Er, Separation ratio= Sr, Percentage recovery =% Rp).

TABLE 3. REMOVAL OF TETRADECYL TRIMETHYL AMMONIUM BROMIDE (TTAB) WITH TIME
(AT 35⍰C, WITH SUPERFICIAL GAS VELOCITY .03cm/s at pH 7):CONC OF TTAB = 2.5mM

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	84.10	23.54	50.46	0.71	75.00	30.00	105.04	60.00
60	2	98	84.10	23.54	53.82	0.71	80.00	32.00	112.04	64.00

90	2	98	84.10	23.23	55.50	0.63	82.49	32.99	107.83	65.99
120	2	98	84.10	26.91	57.18	0.71	85.00	34.00	104.14	68.00
150	2	98	84.10	26.91	60.55	0.88	85.00	34.00	104.14	71.99
180	2	98	84.10	27.10	62.35	0.91	87.00	34.80	104.69	75.89
210	2	98	84.10	28.00	67.11	0.91	99.74	39.89	117.48	79.80
240	2	98	84.10	29.30	70.57	0.91	104.89	41.95	112.66	84.02

Table 4. CONC OF TTAB =2.75Mm

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	92.51	23.54	47.36	0.71	70.39	25.59	98.59	51.20
60	2	98	92.51	23.54	53.82	0.71	80.00	29.09	112.09	58.18
90	2	98	92.51	2.18	58.87	.63	87.49	31.81	138.43	63.63
120	2	98	92.51	23.54	60.55	0.71	89.90	32.69	125.91	65.45
150	2	98	92.51	29.12	63.91	0.88	94.99	34.54	107.94	69.08
180	2	98	92.51	30.10	67.99	0.91	101.05	36.74	110.67	73.50
210	2	98	92.51	30.27	71.99	0.91	107.00	38.90	116.55	77.82
240	2	98	92.51	30.27	75.85	0.91	112.75	41.00	122.82	82.00

(Time =θ, Foam volume = Vf, Residual volume =Vr, Foam amount =F, Residual amount= R, Residual concentration =Cr, Foamate concentration =Cf, Enrichment ratio= Er, Separation ratio= Sr, Percentage recovery =% Rp).

TABLE 5. REMOVAL OF SODIUM LAURYL SULPHATE (SLS) WITH TIME (At 35□C WITH SUPERFICIAL GAS VELOCITY 0.03CM/S AT pH 7, CONC OF SLS= 7.75 mM

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	223.49	25.51	89.39	0.90	154.98	19.99	172.20	40.00
60	2	98	223.49	25.82	92.14	0.91	159.75	20.61	175.54	41.23
90	2	98	223.49	26.01	94.98	0.92	164.67	21.24	178.98	42.50
120	2	98	223.49	27.00	99.02	0.95	171.68	22.15	180.71	44.31
150	2	98	223.49	27.91	101.28	0.98	175.68	22.65	179.18	45.32

180	2	98	223.49	28.10	103.96	0.99	180.24	23.25	182.06	46.52
210	2	98	223.49	28.20	105.55	0.99	183.00	23.61	184.84	47.23
240	2	98	223.49	29.00	110.64	1.02	191.83	24.75	188.06	49.51

Table 6. CONC OF SLS 7.50Mm

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	216.28	25.71	91.91	0.90	159.35	21.24	177.05	42.50
60	2	98	216.28	25.99	95.18	0.91	165.02	22.00	181.34	44.01
90	2	98	216.28	26.23	97.86	0.92	169.67	22.62	184.42	45.31
120	2	98	216.28	27.00	100.59	0.95	174.40	23.25	183.57	46.51
150	2	98	216.28	28.12	102.59	0.99	178.08	23.74	179.87	47.49
180	2	98	216.28	28.85	107.08	1.02	185.65	24.75	182.00	49.51
210	2	98	216.28	29.13	108.16	1.03	187.53	25.00	182.06	50.01
240	2	98	216.28	29.92	112.46	1.05	194.98	25.93	185.69	52.00

(Time =θ, Foam volume = Vf, Residual volume =Vr, Foam amount =F, Residual amount= R, Residual concentration =Cr, Foamate concentration =Cf, Enrichment ratio= Er, Separation ratio= Sr, Percentage recovery =% Rp).

TABLE 7. REMOVAL OF SODIUM LAURYL SULPHATE (SLS) WITH TIME (At 35□C WITH SUPERFICIAL GAS VELOCITY 0.03CM/S AT pH 7) CONC OF SLS =7.25mM

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	209.07	29.23	105.60	1.03	183.09.	25.25	177.25	50.51
60	2	98	209.07	29.91	109.82	1.05	190.40	26.26	180.95	52.53
90	2	98	209.07	30.12	114.97	1.06	199.33	27.49	188.04	55.01
120	2	98	209.07	30.81	120.23	1.09	208.45	28.75	191.23	57.51
150	2	98	209.07	31.01	125.50	1.09	217.59	30.01	199.62	60.03
180	2	98	209.07	31.81	130.68	1.12	220.00	30.34	196.42	62.51
210	2	98	209.07	32.00	139.03	1.13	224.05	30.90	198.27	65.02

Table 8. CONC OF SLS =7.00mM

θMIN	Vf	Vr	FEED mg	R mg	F mg	Cr mM	Cf mM	Er mM	Sr mM	%Rp
30	2	98	201.86	28.91	106.03	1.02	183.83	26.26	180.22	52.53
60	2	98	201.86	29.01	11.04	1.02	192.52	27.50	188.74	55.01
90	2	98	201.86	29.58	116.08	1.04	201.26	28.75	193.51	57.51
120	2	98	201.86	29.91	119.27	1.05	206.79	29.54	196.94	59.90
150	2	98	201.86	30.31	123.19	1.06	213.58	30.51	201.49	61.03
180	2	98	201.86	31.01	126.26	1.09	218.91	31.27	20083	62.55
210	2	98	201.86	31.85	131.24	1.12	227.54	32.50	203.16	65.02

(Time =θ, Foam volume = Vf, Residual volume =Vr, Foam amount =F, Residual amount= R, Residual concentration =Cr, Foamate concentration =Cf, Enrichment ratio= Er, Separation ratio= Sr, Percentage recovery =% Rp).

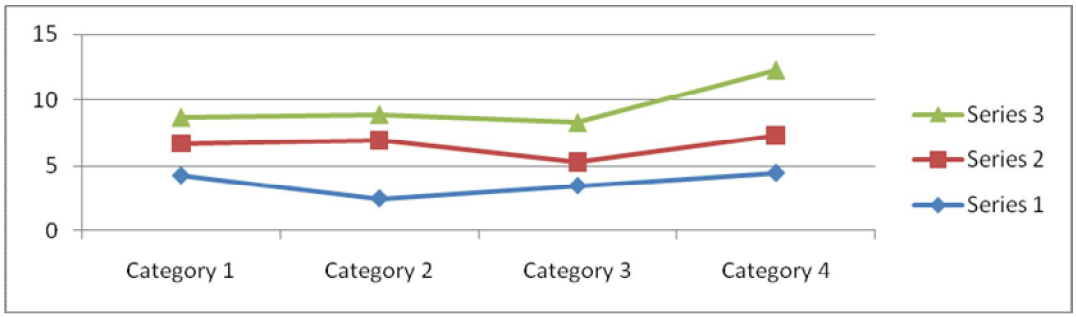

Fig 1. Removal of TTAB

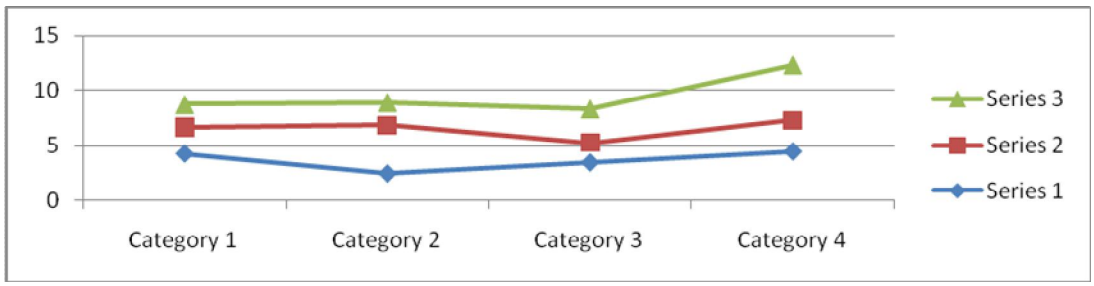

Fig2. Removal of SLS.

From the above table (1 to 8), and from the figures (1 and 2) it is shown that at the SGV .03 CM/S, and at pH 7, the enrichment factor is 44.67 and percentage recovery is 90 at 2mM. The enrichment and the recovery was found 29.08 and 87.25 respectively when the concentration of TTAB was 2.25mM. At 2.5mM the enrichment factor was 41.95 and the percentage recovery was 84.20. Those values were 41.00 and 82.00 respectively at 2.75mM. It was found that as the concentration was increased from 2 to 2.75mM, the enrichment factor and the percentage recovery were decreased.

Similarly, in case of SLS recovery, when we choose the concentration from 7 to 7.75mM, different result shown. When the concentration was 7.75mM, the recovery and the enrichment of SLS were 49.51 and

24.75. but at the concentration of 7 those values were 65.02 and 32.50. So it is indicated the as the concentration decreases the recovery and enrichment were increased simultaneously, which is totally reversed from the result from TTAB recovery.

4. **Conclusion:**

'Foam fractionation' under adsorptive bubble separation method is a well known operation. Foam fractionation method is adopted in the present study to establish its feasibility in the recovery of surfactants at low cost. There is no report that Foam fractionation method is used in downstream processing like other methods for the recovery of biomolecules. There are several criteria like recovery time, purification factor, enrichment factor, operation expenditure, investment costs, running costs and environmental pollution which are to be considered before selecting a recovery method. Operation expenditure, investment costs, running costs and environmental pollution are higher in precipitation and chromatographic techniques. These are significantly lower in foam fractionation technique. But it is restricted to lower concentrated feed.

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