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Studies on the Extraction of Mn (II) by Tri-capryl Amine Oxide

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Abstract— *Tri- Capryl amine Oxide (TCAO) in benzene has been used as extractant for the extraction of Mn (II) from aqueous acid solutions. The extractions are found quantitative from hydrochloric and sulphuric acid solutions and partial from nitric acid solutions. Various factors - acidity, metal ion, extractant concentration and influence of other ions etc. on the extraction process has been studied. Solvated extracted species is also suggested. Estimation of manganese in food and pharmaceutical samples has been achieved successfully.*

Keywords— *Extraction - Mn (II) - Tri- Capryl amine Oxide (TCAO) –Mineral acid- Pharmaceutical samples.*

I. INTRODUCTION

Solvent extraction of Mn (II) has been carried out by earlier workers to a major extent using phosphorus bonded extractants [1-7]. There are no reports available on the extraction of Mn (II) from amine oxides. Therefore, in the present communication includes extraction of Mn (II) by Tri-capryl amine Oxide (TCAO) from hydrochloric and sulphuric nitric acid solutions and the results obtained are presented.

II. MATERIALS AND METHODS

TCAO was synthesized by N- oxidation of Tricapryl amine using hydrogen peroxide as oxidant.[8]. A stock solution of 0.25 M TCAO (Fluka AG) in benzene was prepared and diluted as per the requirement. Mn (II) stock solution was prepared by dissolving 1.52 g of manganese sulphate monohydrate in 1lt double distilled water and was standardized complexometrically with standard EDTA. AnalaR grade reagents were used in the studies. Double distilled water was used throughout the course of investigations.

Mn (II) extraction:

Extraction of Mn (II) has been studied with different concentrations of the metal salt and mineral acid by shaking with equilibrating with an equal volume (10ml) of TACO in benzene (0.025M) pre-equilibrated with 0.1M mineral acid. After 10 minutes the two phases were separated after settlement. Mn (II) from the organic phase was stripped with 10ml of 0.1M HNO_3. The concentration of Mn (II) in both the phases was determined by AAS method.

III. RESULTS AND DISCUSSION

Variation of distribution ratio with concentration of the acid used (HCl and H_2SO_4) in this study is presented in Table 1. Distribution ratio (K_d) was found enhanced with increasing concentration of the acid up to 5.0 M acidity above which there was no change in extraction efficiency (remains unchanged) in HCl media. In sulphuric acid media, the distribution ratio found to increase up to 4.5M acidity followed by decrease in extraction. On the other hand maximum extraction efficiency was observed at 2.5M acidity (64.06).

Composition of the extracted species:

Composition of the extracted species has been determined by the extraction isotherm method [9] and distribution ratio method [10]. Ratio of Mn (II) to TCAO was found unity with all the acid systems in the extraction isotherm method. Variation of log K_d Vs. log TCAO from these acid solutions yielded straight lines with slope two in sulphuric acid solutions and unity in other/ hydrochloric acid solutions.

These mole ratios of Mn(II) and TCAO from these mineral acid media explains the extraction of metal ion by the salvation mechanism as mentioned below.

From hydrochloric and nitric acid solutions:

$$(TCAO)_{org} + Mn^{+2} + 2\,X^-_{aq} \rightleftharpoons MnX_2\,Cl_2.(TCAO)_{org.}$$

Where X= Cl^- or NO_3^-

From sulphuric acid solutions:

$$2(TCAO)_{org}{}^{+}\,Mn^{+2}{}_{aq} + SO_4{}^{2-}{}_{aq} \rightleftharpoons Mn\,SO_4.2(TCAO)_{org.}$$

Effect of stripping agents:

Stripping of Mn (II) from the organic phase with 20ml reagents of acetic, sulphuric and nitric acid solutions. 1.0 M HNO_3 solution was found quite effective. But Mn (II) could not be extracted in a single step. 99.5% of Mn (II) was stripped effectively from organic phase by shaking with equal volumes of 1.0 M HNO_3 for three times.

Variation of diluents

Several solvents with varying dielectric constants were tested as the diluents (Table -2). Quantitative extractions were achieved with benzene as diluent. More than 80% efficiency was obtained with carbon tetrachloride, hexane, toluene, cyclohexane and xylene. Nitrobenzene and n-heptane were found to be poor in extraction. Hence benzene was preferred as diluent throughout the study.

Analysis of Mn in various samples

Food and pharmaceutical samples were analyzed for the recovery of manganese to examine the validity of the method. Ten tablets were weighed and finely powdered in a mortar and pistle. An accurately weighed amount equivalent to one tablet was transferred quantitatively to 100-ml volumetric flask and then 50 ml of 0.01 M H_2SO_4 was added. The mixture was shaken well for about 15 min. The mixture was diluted by 0.01 M H_2SO_4 solution and then filtered by Whatman filter paper No.

40. The first portion of filtrate was discarded. The clear solution obtained was used as a stock sample solution and different aliquots of prepared solutions were diluted with 0.01 M H_2SO_4 to produce different concentrations. A 10.0 ml portion of the solution was extracted with 0.025 M TCAO solutions as per the procedure described earlier. The results are presented in Table-3.

TABLE 1. Variation of acidity with %extraction

Acid Molarity (M)	HCl	H_2SO_4	HNO_3
0.5	62.87	54.28	51.80
1.0	70.52	68.74	58.22
1.5	81.29	73.95	67.45
2.0	86.29	82.26	70.51
2.5	87.68	84.72	64.06
3.0	89.45	85.23	62.27
3.5	90.22	89.28	59.45
4.0	94.13	94.53	54.15
4.5	96.50	97.25	52.48
5.0	98.05	94.86	49.26
6.0	98.05	90.31	47.82
7.0	98.05	88.83	45.55

TABLE 2. Effect of diluents on extraction
[Mn (II)] = 1.03 x 10^{-4} M ; [H_2SO_4] = 9.0M; [TCAO] = 2.5 x 10^{-2} M

Diluent	Dielectric constant	% Extraction
Benzene	2.28	98.05
Chloroform	4.81	88.22
CCl_4	2.23	85.88
Cyclo hexane	2.0	82.35
n-hexane	1.89	80.29
Dichloro methane	8.08	80.15
n-heptane	1.92	73.63
Toluene	2.43	88.83
Nitrobenzene	34.82	71.42

TABLE 3. Estimation of Mn (II) in food and pharmaceutical samples

Sample	Mn (II) present (ppm)	Mn (II) found by extraction (ppm)	%
Ragi	3.00	2.85	95.02
Green gram	4.05	3.89	96.05
Ferrous fumerate(300mg)	114.10	112.66	98.73
Ferrous dextrin(50mg)	100.22	99.92	99.70

IV. CONCLUSION

The proposed method is very simple, rapid and selective. It requires less than 20 min. time to extract and estimate manganese content in natural food as well as pharmaceutical samples.

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