

Study on swelling characteristics of newly prepared Zirconium Anthra Arsenate

1.Dr.Mukesh Srivastava

Associate Professor, Department of Chemistry,

Bipin Bihari College, Jhansi-UP

2.Dr.Anil Kumar

Associate Professor

Bipin Bihari College, Jhansi-up

3. Dr.DK Agarwal

Associate Professor

Bipin Bihari College, Jhansi-up

Abstract

Inorganic ion exchangers behave quite differently from the point of view of their swelling characteristics. Swelling in water is quite pronounced in clay minerals used as exchangers, but in synthetic products from Zr or another group of four elements, the swelling characteristics are quite different. Though the prepared product is hydrated to a great extent when they are dried, they lose much of their water except those molecules which are linked to the structure more strongly. If they again put in water, they do not absorb water to some extent. Though they show swelling in many cases up to ~100%, this effect is negligibly small for samples heated to higher temperatures. Zirconium phthalate and Zirconium phthalophosphate have been shown to give swelling of about 103-116% respectively. In general Zirconium Phosphate products are denser and do not swell considerably. The newly prepared exchanger has also been studied for its swelling characteristics.

Introduction

After the discovery of fact that the insoluble compound obtained from zirconium salts and phosphoric acid can be used to separate uranium and plutonium from fission products (1, 2). Zirconium phosphate becomes a model ion exchanger. The stability of this compound towards ionizing radiation, high temperature, and many common chemical reagents has made an important product which is of great use chemical processing contaminated moderator or cooling in atomic reactor. To improve its ion exchanging properties a (3) tried to prepare an exchanger of citric acid, similarly, phthalic anhydride was used to prepare zirconium phthalophosphate (4), similar to zirconium phosphate, arsenate, tungstate or molybdate have also been prepared. some zirconium-based exchangers have been reported (5-9), In this zirconium arsenate has been taken in place of phosphate, due to conditions of preparation for zirconium arsenate are similar to those of zirconium phosphate (10,11).

Carboxylic acid, anthranilic acid is used to prepare the proposed exchanger, Abbreviation used for Zirconium Anthra Arsenate, ZrAnAs,

The introduction of certain carboxylic groups into the network structure of zirconium arsenate can improve the crystallinity of the compound. Improvement of the ion exchange capacity of the prepared material is also expected. Hence attempts have been made to prepare a new ion exchanging material from zirconium salt in presence of anthranilic acid. Thus anthranilic acid has been chosen to precipitate zirconium arsenate gel in their presence separately, thus some zirconium-based three-component ion exchanger has been prepared. Bi-functional cation exchanger, Zirconium Anthra Arsenate, ZrAnAs have been prepared from Zirconyl nitrate, Sodium Arsenate, and Anthranilic acid in different molecular ratios.

The swelling character of the exchanger throws some light on the density of structure and hence also on their ion exchanging power, so the effect of swelling in an aqueous and alcoholic medium in different proportions was studied. **Experimental**

1. Preparation of Zirconium (IV) salt, organic acid, and sodium arsenate solutions

:

A. Zirconium (IV) salt solution was prepared by taking 7.6 gm. of Zirconyl Nitrate in 56.00 ml of 1.0N nitric acid. The solution so obtained was more than 0.5M in strength.

B. Sodium arsenate solution was prepared by dissolving 22.00 gm. of sodium arsenate in 200 ml. of water.

C. Anthranilic acid was dissolved in 2.00N sodium hydroxide to give a solution of concentration slightly greater than 1.00M.

2. Procedure for preparing the new exchangers

Preparation of exchanger with zirconium salt: anthranilic acid: Arsenate in ratio, 1:1:1. In zirconyl nitrate solution (solution A) was taken in 400 ml beaker added solution of organic anthranilic acid (solution C) with stirring. This formed a thick white precipitate of the complexed zirconium compound. It was then quickly poured into a 50.00 ml solution of sodium arsenate (solution B) taken in another 400 ml beaker with a magnetically driven stirring device. The mixture was stirred vigorously using the magnetic stirrer for about half an hour. The precipitate so obtained filtered and washed many times with distilled water. The filtrate was tested for the presence of nitrate ions and washing was continued till the precipitate was free from nitrate ions. The precipitate was then dried in an electric oven at $100 \pm 0.1^\circ\text{C}$. This provides an amorphous gel-type exchanger. This product after drying was again submerged in water and grounded. It was then filtered and dried again at $100 \pm 0.1^\circ\text{C}$.

3. Swelling characteristics of exchanger

A fixed volume of the exchanger was filled in the specially prepared glass cylinders. Then the required solvent was added to cover the resin completely. It was added in sufficient excess to allow complete swelling for the material. The air bubbles were removed by tapping these cylinders.

The cylinders so filled were kept overnight for the exchanger to swell completely. After this, the volume changes were again read off and noted. The swelling was calculated as a percentage change in the volume of the exchanger. The swelling study was carried out in water 100%, water-ethanol (50-50%), water-ethanol (25-75%), and ethanol 100%.

Table 1: Swelling characteristics ZrAnAs in Water- Ethanol medium

SN	Solvent Composition	Resin Volume before adding solvent, ml	Resin Volume after adding solvent, ml	Swelling Percentage
1	Water 100%,	0.84	0.85	+1.19
2	Water- Ethanol (50-50%),	0.83	0.81	-2.40
3	Water- Ethanol (25-75%),	0.85	0.83	-2.35
4	Ethanol 100%.	0.82	0.80	-2.44

+ Swelling represents actual Swelling of the exchanger and

- Swelling represents a contraction of the exchanger

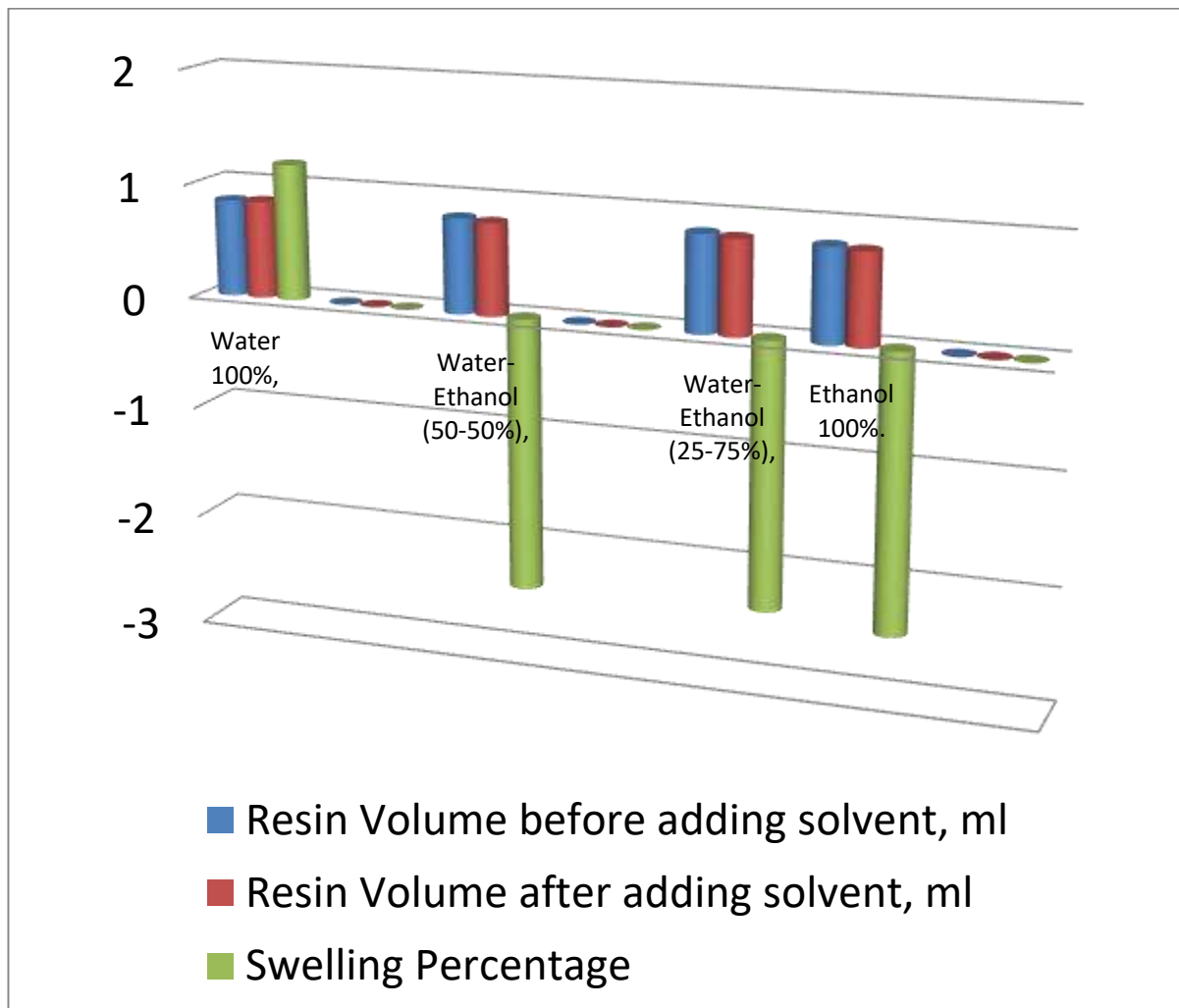


Fig 1: Comparison of swelling characteristics ZrAnAs in Water- Ethanol medium

Result and discussion

Showing swelling of resin in water 100%, from 0.84 to 0.85 i.e.+1.19%, Showing contraction of resin in Water- Ethanol (50-50%) from 0.83 to 0.81, in Water-Ethanol (25-75%) from 0.85 to 0.83 and in Ethanol 100% from 0.82 to 0.80 Comparative data are given in Table-1. The swelling has been shown as a positive change, while a decrease in volume has been shown as a negative change and a comparison of swelling and contraction of resin is shown in Fig 1. Very little swelling in water shows a quite denser structure of the ZrAnAs.

Conclusion

1. The prepared exchanger shows very little swelling +1.19% in water.
2. In Ethanol 100%, the exchanger contracts -2.44 a bit rather than swelling.
3. In mixed water – Ethanol solutions there is contraction rather than swelling.

References

1. C.B. Amphlett et.al, J. Inorg. and Nuclear chem. 6,220,236 (1958)
2. C.B. Amphlett, Intern. Cong. peaceful uses of Atomic Energy, 2nd Cong. Geneva 28,63 (1958)
3. H.S. Thompson, J. Roy. Agr. Soc. Eng. 11,68 (1850)
4. J.F. Way; J. Roy. Agr. Soc. Eng. 11,313(1850),13,123(1852)
5. J. Lamberg, J. deut. Geol, Ges., 22,355(1870),28,519(1876)
6. G. Wiegner, J. Land wirtsch. 60,111,197(1912)
7. F. Harm and A., Rumpler, 5th Intern. Congr. Pure and Appl. Chem. p.59 (1903)
8. R. Gaus. Jahrl. Preuss. Geol. Landesanstalt, Berlin, 26179(1905), German patent 174097 (1906)
9. C.B. Amphlett, L.A. McDonald and M.J. Redman. Chem and Ind. p.1314,(1956)
10. K.A. Kraus and H.O. Phillips, J. amer. Chem. Soc. 78,694(1956)
11. K.A. Kraus and H.O. Phillips, T.A. Carlson and J.S. Johnson, Intern. Cong. peaceful uses of Atomic Energy, 2nd Cong. Geneva 28,3 (1958)
12. E. Merz, Z. Electro, chem., 63,288(1959)
13. K.A. Kraus and H.O. Phillips, J. amer. Chem. Soc. 78,249(1956)
14. E.M. Larsen, W.C. Fernebie and U. Quill, Ind. Eng. Chem. 15,512 (1949)
15. C.B. Amphlett, U.S. patent No. 3,056,647(1962)
16. L. Baetsle and J. Pelsmaekers; J. inorg. and Nuclear chem. 21, 124(1961)
17. M. Michael and D.W. Fetcher, Trans. Am. Nucl. Soc. 3(1)(1960)
18. G.H. Nancollas and R. Peterson, J. Inorg. Nucl. chem. 22,259(1960)
19. E.M. Larsen and D.R. Vissars, J. Phys, Chem. 64,1732(1960)
20. V. Vesely and V. Pekarek, J. Inorg. Nucl. chem. 25,697(1963)

21. N.Michael, W.D.fletcher, D.E.Croucher, and M.J. Bell; Westing House Report, CVNA-135(1961)
22. C.B.Amphlett,G.H.Nancollas and T.Williams,Chem.Ind.London, pp.292 (1959)
23. I.J, Gal and O.S.Gal Proc.Second Intern.Cong. Peaceful uses of Atomic Energy, United Nations, Geneva,28,24(1958)
24. A.Clearfield, A.Oskarsson and C.Oskorssow,low exchange and membranes,1,91(1972)
25. A.Clearfield, G.D.Smith, and B.Hammond, J.Inorg. Nucl. Chem. 30,277 (1978)
26. E.Torracca,U.Coustantino and M.A.Hassucci, J.Chromag.,30,584 (1967)
27. G.alberti and E.Torracca, J.Inorg. Nucl.Chem.30,3075 (1968)
28. M.Qurashi and S.A.Nabi, J.Inorg. Nucl.Chem.32,2059 (1970)
29. D.Vissers, Ph.D.Thesis, University of Wisconsin, Madison(1959)
30. W.A.Cilley, Ph.D.Thesis, University of Wisconsin, Madison (1963)
31. E.M.Larsen and W.A.Cilley, J.Inorg. Nucl.Chem.30,287 (1968)
32. G.G.Rocco, J.R.,Wiener, and J.P.Cali, Physical science Research, AFCRL 64-1018(1964)
33. Gangadwala, J. et al. , Ind. Eng. Chem. Res. 42, 2146-55(2003)
34. Gonzalez Soto, E. et al., Analytical Letters 28, 2699-2718(1995)
35. Iyer, S.T. et al., Reactive a. Functional Polymers 29, 51-7(1996)
36. A technical Document issued by the intern atomic energy agency, Vienna (1986)