

## Inorganic and Composite Ion Exchange Materials and their Applications

(Review)

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### ABSTRACT

Article describes applications of several groups of inorganic and composite ion exchange materials. Zeolites, acidic salts, hydrous oxides, metal ferrocyanides, heteropolyacids and composite organic/inorganic ion exchangers are reviewed.

**Keywords:** inorganic materials, composites, ion exchangers

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### INTRODUCTION

In science, analytical chemistry is an ancient branch, yet may be regarded as one of the youngest science with the growing global awareness in health hazards and environmental pollution, it has played a key role to unveil the causes. So the scientists all over the world are paying great attention to analytical chemistry.

Analytical Chemistry is basically concerned with the determination of chemical composition of matter. The aspects covered by modern analytical chemistry are identification of a substance, the elucidation of its structure, separation of different elements and synthesis of ion exchangers. Separation has very important applications in various fields namely medicine, agriculture and environmental analysis.

Separation is basically a pre-treatment method which, usually precedes any quantitative or qualitative analysis. Separation involves both classical and modern techniques. The examples of the classical methods are precipitation and distillation. The general methods of separation include distillation, extraction, precipitation, crystallization, dialysis, diffusion etc.

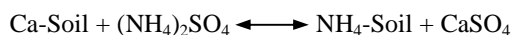
The most modern and versatile techniques used for the purpose of separations are chromatography, electrophoresis and ion exchange chromatography. Ion exchange

chromatography has emerged as a most versatile and standard analytical tool.

Ion exchange is basically a process of nature occurring throughout the ages from even before the dawn of human civilization. The phenomenon of ion exchange is not of a recent origin. The earliest of the references were found in Holy Bible, which says 'Moses' succeeded in preparing drinking water from brackish water by an ion exchange method [1]. Aristotle [2] stated that the seawater loses part of its salt content when percolated through certain sands. In 1623, Francis Bacon and Hales described a method for removing salts by filtration and desalination from sea water. In 1790, Lowritz purified sugar beet juice by passing it through charcoal.

In the beginning of 19th century, chemists were quite aware about ion exchange and were busy in new researches. Gazzari (1819) discovered that clay retained dissolved fertilizer particle. In 1826, Sprengel stated that the humus frees certain acids from soils. Fuchs (1833) pointed out that the lime frees potassium and sodium from some clay. By middle of 19th century sufficient experimental observations and information had been collected but principle of ion exchange had not yet been discovered. Thompson 1845, [3] and Way [4] in 1850 laid the foundation of ion exchange by base exchange

in soil. They observed that when soils are treated with ammonium salts, ammonium ions are taken up by the soil and an equivalent amount of calcium and magnesium ions are released. During 1850-55 the agrochemist Way demonstrated the following mechanism to be one of the ion exchange method involving the complex silicates present in the soil. As described by Way the process observed by the Thompson could be formulated:



The ion exchange process is reversible and aluminosilicates (zeolites) are responsible for the exchange in soil, established by Eichorn (1850) [5]. The first synthetic aluminum based ion exchanger was prepared by Harms and Rumpler [6] in 1903 to purify the beet syrup. According to Lamberg and Wiegner [7, 8], the materials responsible for the phenomenon were mainly clays, zeolites, gluconites and humic acids. Lamberg also obtained analcite ( $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ) by leaching the mineral leucite ( $\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ ) with solution of sodium chloride and found that, this transformation could be reversed by treating analcite with a solution of potassium chloride, thereby exchanging  $\text{Na}^+$  again by  $\text{K}^+$ .

The first application of synthetic zeolite for collection and separation of ammonia from urine was made by Follin and Bell [9]. Gans [10] succeeded in utilizing the synthetic aluminium silicates ion exchangers for industrial purpose like softening of water and also for treating sugar solutions. Due to the limitations in the applications of neutral and synthetic silicates in various industrial applications and in an attempt to meet the demands of the industries, Adam and Holms [11] in 1935 laid the foundation of organic exchangers when they observed that the crushed phonograph records exhibit ion exchange properties. This led to investigators to develop synthetic ion exchange resins. These resins were developed and improved by the former I.G. Farben industries in Germany followed by the manufacturers in U.S.A. and U.K, which proved very effective for separations, recoveries, the ionization catalysis etc.

Although inorganic ion exchanger materials were first to be recognized, they lost their utility after the discovery of organic resins. However, revival of the interest in these materials took place in the 20th century, because of their use in the field of nuclear research. At that time there was need of some new materials

that were stable at high temperatures and in presence of intense radioactive radiations.

Kraus et al. [12, 13] at Oak Ridge national laboratory and C.B Amphlett [14, 15] in United Kingdom did the excellent work on these materials in the initial stages. The work up to 1963 has been summarized by Amphlett [16] in the classical book 'Inorganic Ion exchangers'. The later work up to 1970 has been condensed by Pekarek and Vesely [17], Clearfield [18, 19], Alberti [20, 21] and Watton [22, 23] have also worked on different aspects of synthetic inorganic ion exchangers, In India Qureshi and co-workers have prepared a large number of such inorganic material and studied their ion exchange behavior during the last 15 years. Other groups that were engaged in the field of research and whose work is significant interest are Anil K. De at Shantiniketan and Tandon at Rorkee. The few important uses of these inorganic ion exchangers are:

- Separations of metal ions
- Separations of organic compounds
- Removal of wastes and air pollutants
- Preparation of ion-selective electrodes
- Preparation of artificial kidney machines
- Preparation of fuel cells

The analytical importance of the synthetic inorganic ion exchangers is now firmly established. The review of ion exchange in analytical chemistry for the year 1970 includes the significant statement; the obvious advances in the last two years are in the area of inorganic ion exchangers. Even today this statement is almost equally true. Synthetic ion exchanger may be classified in the following categories:

- 1) Synthetic zeolites
- 2) Polybasic acid salts
- 3) Hydrous oxides
- 4) Metal ferrocyanides
- 5) Insoluble ion exchange materials
- 6) Heteropolyacids

### 1 SYNTHETIC ZEOLITES

Zeolites were the first inorganic materials to be used for the removal of waste effluents at large scale. Zeolites are crystalline aluminosilicate based materials and can be prepared as micro-crystalline powders, pellets or beads. The main advantages of synthetic zeolites when compared with naturally occurring zeolites are that, they can be engineered with a wide

variety of chemical properties and pore sizes, and they are stable at high temperatures but have some limitations too.

The main limitations of synthetic zeolites are:

- Relatively high cost compared to natural zeolites
- Limited chemical stability at extreme pH ranges (either high or low)
- Ion specificity is susceptible to interference from similarly sized ions

The actual processing capacities obtained with zeolites are lower than their maximum capacities since the bed is changed at the early stages of breakthrough and because the waste streams usually contain other ions that will occupy some of the exchange sites and therefore reduce the processing capacity. Synthetic silica based ion exchangers were produced for technical purpose by fusing soda, potassium carbonate, feldspar and kaolinite (Schmaltz permutite) and later from aluminum sulphate solution containing sodium silicate by precipitation with sodium hydroxide solution (Gel permutite). Since then, artificial crystalline zeolites have also been successfully synthesized.

The advantageous properties of the crystalline silicate based ion exchanger over modern synthetic resin based ion exchangers are as follows:

- Less sensitive to higher temperatures
- Stiff and uniform structure
- More selective and suitable for separation of ions on the basis of their different sizes

Some of them are now also used as molecular or ionic sieves. In India, a systematic investigation has been carried out to evaluate the performance of locally available synthetic zeolites for the removal of cesium, strontium and thorium from solution [24-26]. The zeolites after exchange with cesium, strontium or thorium, were thermally treated to fix the ions successfully in the same matrix [27].

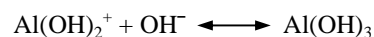
## 2 POLYBASIC ACIDS SALTS

Acidic salts of multivalent metals formed by mixing acidic oxides of metals belonging to IV, V and VI groups of the periodic table. Acid salts of the quadrivalent metals are most studied groups of this class. They are extremely insoluble. Their composition is non-

stoichiometric and depends on the condition under which they are precipitated. The materials which have been so far synthesized includes the phosphates, arsenates, molybdates, tungstates, antimonates, silicates, vanadates, and tellurates of zirconium, titanium, thorium, tin, cerium, chromium, iron, niobium, tantalum etc.

## 3 HYDROUS OXIDES

The hydrous oxides of some metal ions have also been the well-established materials for ion exchange purpose. Freshly precipitated trivalent metal oxides are very effective in this respect, e.g. hydrous ferric oxides and ferric hydroxides readily absorb alkaline earth cations according to the law of mass action [28, 29], other bivalent cations [30] being absorbed above pH 7. In this process, the alkali metals and alkaline earths are adsorbed on the surface and are readily eluted while more highly charged cations Ce(III), Y(III), Pm(III), Ru(IV) are sorbed in bulk and eluted only with difficulty [31]. The ion exchangers of this class show an amphoteric behavior depending upon the pH of the solution. The process can be described by the following equilibria [32].



Zinc oxide also shows amphoteric exchange properties [33]. Mixed oxides can be prepared in which second cations of higher charge than the parent cation is introduced into the structure. The resulting net positive charge is being balanced by the presence of anions other than oxides and hydroxides. Example of such materials include  $\text{Zn(OH)}_2$  in which  $\text{Zn}^{2+}$  is partly replaced by  $\text{Al}^{3+}$  and  $\text{Al(OH)}_3$  containing  $\text{Si}^{4+}$ ,  $\text{Ti}^{4+}$  or  $\text{Zr}^{4+}$ . The general formulae  $\text{Zn}_{n-1}\text{Al}_n(\text{OH})_2\text{X}_n$  and  $\text{Al}_{n-1}\text{M}_n(\text{OH})_3\text{X}_{n-1}$ , where  $\text{M}^{4+}$  is a tetravalent oxide and X is a monovalent anion. Quadrivalent metal oxides are also commonly used as inorganic ion exchangers such as  $\text{SnO}_2$ ,  $\text{SiO}_2$ ,  $\text{ThO}_2$  and  $\text{ZrO}_2$ . Actually these materials do not possess simple oxide formula as given above unless they are ignited at a high temperature. They are found to contain varying amount of water, which is not present as water of hydration since on heating, it is lost continuously over a range of temperature. Consequently these oxides are usually described as hydrous oxides. Inoue et al. [34-36] and A.K. De et al. [37-40] have done important work on hydrous oxides.

#### 4 METAL FERROCYANIDES

Insoluble metal ferrocyanides can also be used as inorganic ion exchangers. They are also known as scavengers for alkali metals. They are easily prepared and useful in the separation of radioactive wastes and fissionable materials [41] with less damage to radiation than their organic counter parts. Baetsley et al. [42] studied ferrocyanide molybdate and determined its structure by X-ray studies. They also used molybdenum and tungsten ferrocyanides for the separation of Cs-137 and Sr-90 from fissionable products in acidic medium.

Amine based metals ferrocyanides have also received attention. They were first introduced by Hahn and Clein [43], who prepared a cobalt amine ferrocyanide. Ferric hydroxides also have fast growing applications in the separation of metal ions and it has been used as an adsorbent for the removal of arsenic from natural water [44].

#### 5 INSOLUBLE ION EXCHANGE MATERIALS

Various insoluble ion exchanging materials are also of interest. A large number of such compounds have been prepared. These materials have been prepared by precipitation from metal salt solution with Na<sub>2</sub>S or H<sub>2</sub>S. The ion exchange properties of insoluble sulphides (e.g. Ag<sub>2</sub>S, SnS, CuS, PbS, FeS, NiS, As<sub>2</sub>S<sub>3</sub>, Sb<sub>2</sub>S<sub>3</sub>) have been investigated. The sulphides are selective towards cations forming insoluble sulphides. The exchange reaction occurs through metathetical reactions in which the metal of sulphide is displaced by appropriate ion from the solution. Quantitative sorption of Tl<sup>+</sup>, Ni<sup>2+</sup>, Co<sup>2+</sup>, Mn<sup>2+</sup>, Cu<sup>2+</sup> and Pb<sup>2+</sup> have been reported on ZnS, CdS and PbS [45], Uranium on PbS [46], separation of Cu<sup>2+</sup> from Zn<sup>2+</sup> and Cd<sup>2+</sup> on SnS [47] and noble metal on CuS [48, 49]. Some of the important two component ion exchangers with their composition, ion exchange capacity and selectivity of metal ions have been reported in table 1.1.

#### 6 HETEROPOLYACIDS

Heteropolyacid salts can be used as inorganic ion exchangers. This group of exchangers is derived from 12-heteropolyacids of general formula H<sub>n</sub>XY<sub>12</sub>O<sub>40</sub>.nH<sub>2</sub>O where X may be P, As, Si, B or Ce and Y may be one of the elements such as Mo, W or V. The heteropoly compounds especially those of 12-molybdo compounds are quite strong oxidizing agents. The exchangers of this type are stable in

moderately concentrated acid. However they dissolve in the solution of alkali.

The heteropolyacids exhibit high affinity to heavy alkali metals, thorium and silver. The size of univalent ions of these elements is suitable for their retention in the crystal lattice of heteropolyacids. Apart from the heteropolyacid salts, many other substances like mixed salts have also been synthesized and studied in detail for ion exchange properties. It has been found that double salts or mixed salts of the metal ions possess ion exchange properties different from that of simple salts. Usually they show superiority over simple salts mainly in three aspects. They are more thermally and chemically stable, secondly they are selective in nature and their ion exchange capacities are higher as compared to their simple salts. It is with this view, attention has been given to synthesize and to investigate ion exchange properties of these class of ion exchangers. Some mixed salts or double salts prepared earlier have been reported in table 1.2.

In order to characterize a new substance as an inorganic ion exchanger, its utility in various fields and its limitation, the following properties may be studied as per given order of preference.

- Ion exchange capacity
- Chemical and thermal stability
- Composition
- pH titration
- Structural studies
- Selectivity
- Analytical applications

Besides the applications, inorganic ion exchangers have some limitations:

- Relatively low exchange capacities
- Relatively low mechanical durability
- Non-controllable pore size
- Clay minerals tend to peptize (i.e. convert to a colloidal form)
- Zeolites are difficult to size mechanically
- Partial decomposition in acids or alkalis
- Limited chemical stability in many solutions
- Need for a chemical or thermal pretreatment (especially those with a very low salt content)

**Table 1.1** Two-component ion exchange materials with their properties

S. No.	Ion exchange Materials	Type	Composition	Empirical Formula	IEC (meq g <sup>-1</sup> )	Selectivity	References
1	Aluminium antimonate	Amorphous	Al/Sb=4.20	-	1.14	Ag <sup>+</sup> , UO <sub>2</sub> <sup>2+</sup> , Ba <sup>2+</sup>	50
2	Aluminium oxide	Crystalline	-	-	-	-	51
3	Aluminium vanadate	Amorphous	-	(Al <sub>2</sub> O <sub>3</sub> ) <sub>n</sub> .(V <sub>2</sub> O <sub>5</sub> ) <sub>n</sub>	-	-	52
4	Aluminium silicate	Amorphous	-	-	-	-	53
5	Aluminium tripolyphosphate	Amorphous	Al/P=0.50-0.66	-	2.50	-	54
6	Aluminium metatungstate	-	-	-	-	-	55
7	Antimonic acid	Amorphous	-	Sb <sub>2</sub> O <sub>5</sub> .4H <sub>2</sub> O	1.28	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Ta <sup>5+</sup>	56
8	Antimony molbdate	-	-	-	-	-	57
9	Antimony phosphate	-	-	Sb(HPO <sub>4</sub> ) <sub>2</sub> .H <sub>2</sub> O	-	Cd <sup>2+</sup> , Hg <sup>2+</sup>	58
10	Antimony silicate	Crystalline	-	Sb <sub>2</sub> O <sub>5</sub> (H <sub>2</sub> SiO <sub>3</sub> ) <sub>6</sub> .nH <sub>2</sub> O	-	Rh <sup>+</sup>	59
11	Antimony ferrocyanide	Amorphous	-	-	-	Sr <sup>2+</sup>	60
12	Antimony sulphide (Hydrous)	-	-	-	-	-	61
13	Bismuth tungstate	Amorphous	Bi/W=0.50	-	0.50	Pb <sup>2+</sup>	62
14	Bismuth nitrate	Amorphous	-	-	-	-	63
15	Bismuth tellurate	Amorphous	Te/Bi=0.70	Bi <sub>4</sub> (H <sub>2</sub> TeO <sub>6</sub> ) <sub>3</sub> .nH <sub>2</sub> O	3.20	-	64
16	Bismuth silicate	Amorphous	-	-	3.30	-	65
17	Cerium vanadate	Amorphous	-	CeO <sub>2</sub> .2V <sub>2</sub> O <sub>5</sub> .4H <sub>2</sub> O	-	-	66
18	Cerium antimonate	Amorphous	Sb/Ce=0.30	-	1.23	Hg <sup>2+</sup> , Cu <sup>2+</sup> , Tl <sup>+</sup>	67
19	Cerium arsenate	Crystalline	As/Ce=2.0	Ce(HAsO <sub>4</sub> ) <sub>2</sub> .2H <sub>2</sub> O	4.30	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Cs <sup>+</sup>	68
20	Cerium molybdate	Amorphous	Mo/Ce=2.3, 8.2	-	0.96	Pb <sup>2+</sup>	69
21	Cerium oxide	Amorphous	H <sub>2</sub> O/CeO <sub>2</sub> =3.0	-	0.99	Cu <sup>2+</sup>	68
22	Cerium oxide (Hydrous)	Crystalline	CeO <sub>2</sub> /H <sub>2</sub> O=2.50	-	-	-	70
23	Cerium tellurite	Amorphous	-	-	-	-	71
24	Cerium tungstate	Amorphous	Ce/W=0.49	-	-	Al <sup>3+</sup> , Hg <sup>+</sup>	72
25	Cobalt antimonate	Amorphous	Co/Sb=1.3	-	0.89	Bi <sup>3+</sup>	73
26	Cobalt ferrocyanide	Amorphous	-	-	-	Ag <sup>+</sup>	74
27	Copper ferrocyanide	Amorphous	-	-	-	-	75
28	Copper phosphate	Amorphous	-	-	-	-	76
29	Chromium phosphate	Amorphous	P/Cr=0.6-1.0	Cr <sub>2</sub> O <sub>2</sub> HPO <sub>4</sub> Cr <sub>2</sub> O <sub>3</sub> (HPO <sub>4</sub> ) <sub>2</sub>	5.90	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Rb <sup>+</sup> , Cs <sup>+</sup>	77
30	Chromium arsenate	Amorphous	As/Cr=1.98	Cr <sub>2</sub> O <sub>3</sub> (H <sub>3</sub> AsO <sub>4</sub> ) <sub>4</sub> .3H <sub>2</sub> O	0.63	Zr <sup>4+</sup> , Hf <sup>4+</sup>	78
31	Chromium molybdate	Amorphous	Mo/Cr=1.90	Cr <sub>2</sub> O <sub>3</sub> (H <sub>2</sub> MoO <sub>4</sub> ) <sub>4</sub> .8H <sub>2</sub> O	0.34	Pb <sup>2+</sup> , Ca <sup>2+</sup>	79
32	Chromium tungstate	Amorphous	W/Cr=1.92	Cr <sub>2</sub> O <sub>3</sub> (H <sub>2</sub> WO <sub>4</sub> ) <sub>4</sub> .11H <sub>2</sub> O	0.02	Th <sup>4+</sup> , Hf <sup>4+</sup>	80
33	Chromium antimonite	Amorphous	Sb/Cr=2.95	Cr <sub>2</sub> O <sub>3</sub> .3Sb <sub>2</sub> O <sub>5</sub> .22H <sub>2</sub> O	0.42	Co <sup>2+</sup> , Pb <sup>2+</sup>	81
34	Chromium tellurate	Amorphous	Te/Cr=0.20	-	-	-	82
35	Chromium ferrocyanide	Amorphous	Cr/Fe=0.33	K <sub>6</sub> Cr <sub>2</sub> [Fe(CN) <sub>6</sub> ] <sub>3</sub> .16H <sub>2</sub> O	2.65	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Ca <sup>2+</sup>	83
36	Ferric phosphate	Amorphous	P/Fe=2.00	FeH(HPO <sub>4</sub> ) <sub>4</sub> .nH <sub>2</sub> O	0.77	Pb <sup>2+</sup> , Eu <sup>3+</sup> , Ca <sup>3+</sup>	84
37	Ferric arsenate	Amorphous	As/Fe=1.33	-	0.80	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup>	85
38	Ferric silicate	Amorphous	-	-	0.20-1.60	Zn <sup>2+</sup> , Cd <sup>2+</sup> , Cr <sup>3+</sup> , Al <sup>3+</sup>	86
39	Ferric antimonate	Amorphous	Sb/Fe=2.40	-	0.80	Cd <sup>2+</sup>	87
40	Ferric tungstate	Amorphous	Fe/W=1.0	-	0.84	Ce <sup>4+</sup>	88
41	Ferric ferrocyanide	Amorphous	-	-	3.60	Cs <sup>+</sup>	59

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42	Germanium (IV) phosphate	Crystalline	-	Ge(HPO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	7.80	-	89
43	Hafnium phosphate	Crystalline	-	Hf(HPO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	4.17	-	90
44	Hafnium arsenate	Amorphous	-	HfAsO <sub>4</sub>	-	-	91
45	Lead phosphate	Crystalline	-	Pb(HPO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	4.79	-	89
46	Lead tungstate	Amorphous	W/Pb=2.50	-	1.00	Cu <sup>2+</sup>	92
47	Lead strontium phosphate	Amorphous	-	-	-	-	93
48	Lead ferrocyanide	Amorphous	-	Pb <sub>2</sub> [Fe(CN) <sub>6</sub> ]	-	Cs <sup>+</sup> , Co <sup>2+</sup>	94
49	Lanthanum tellurate	Amorphous	La/Te=0.66	La <sub>2</sub> (TeO <sub>4</sub> ) <sub>3</sub>	1.61	-	95
50	Lanthanum oxalate	Amorphous	-	-	-	-	96
51	Niobium phosphate	Amorphous	-	-	-	-	97
52	Niobium arsenate	Amorphous	Nb/As=1.96	-	1.06	Cd <sup>2+</sup> , Mn <sup>2+</sup> , Al <sup>3+</sup>	98
53	Niobium vanadate	Amorphous	-	-	-	Ce <sup>4+</sup> , Eu <sup>3+</sup>	99
54	Niobium Antimonate	Semi-Crystalline	Nb/Sb=1.40	-	1.10	Mg <sup>2+</sup>	100
55	Niobium pentaoxide	Amorphous	-	-	-	-	101
56	Stannic arsenate	Amorphous	Sn/As=1.84	-	0.79-0.94	Pb <sup>2+</sup> , Al <sup>3+</sup> , Fe <sup>3+</sup> , Ga <sup>3+</sup> , In <sup>3+</sup>	102
57	Stannic phosphate	Amorphous	P/Sn=1.25	SnO <sub>2</sub> ·0.62P <sub>2</sub> O <sub>5</sub> ·nH <sub>2</sub> O	1.20-1.44	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Rb <sup>+</sup> , Cs <sup>+</sup>	103
58	Stannic tungstate	Amorphous	Sn/W=1.30	-	0.58	Ba <sup>2+</sup> , Ca <sup>2+</sup> , Sr <sup>2+</sup> , Mn <sup>2+</sup> , Ni <sup>2+</sup> , Co <sup>2+</sup>	104
59	Stannic antimonate	Amorphous	Sb/Sn=1.0	SnO <sub>2</sub> ·Sb <sub>2</sub> O <sub>5</sub> ·nH <sub>2</sub> O	0.75	Co <sup>2+</sup> , Ni <sup>2+</sup>	105
60	Stannic oxide	Amorphous	-	SnO <sub>2</sub> ·xH <sub>2</sub> O	1.98	Cu <sup>2+</sup> , Zn <sup>2+</sup> , Co <sup>2+</sup> , Fe <sup>3+</sup> , Ni <sup>2+</sup> , Mn <sup>2+</sup>	106
61	Stannic molybdate	Amorphous	Sn/Mo=1.0	-	1.00	Pb <sup>2+</sup>	107
62	Stannic silicate	Amorphous	Sn/Si=4.0	-	-	Cu <sup>2+</sup> , Pb <sup>2+</sup> , Cr <sup>3+</sup>	108
63	Stannic selenite	Amorphous	Sn/Se=1.33	(SnO <sub>4</sub> )(OH) <sub>2</sub> (SeO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O	0.75	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , La <sup>3+</sup>	109
64	Stannic vanadate	Amorphous	Sn/V=1.0	[(SnOH) <sub>3</sub> V <sub>3</sub> O <sub>9</sub> ·4H <sub>2</sub> O] <sub>n</sub>	0.85	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup>	110
65	Stannic ferrocyanide	Amorphous	Sn/Fe=3.0	[(SnO) <sub>3</sub> (OH) <sub>3</sub> H <sub>4</sub> Fe(CN) <sub>6</sub> ·3H <sub>2</sub> O] <sub>n</sub>	0.85	Na <sup>+</sup> , K <sup>+</sup> , Ba <sup>2+</sup>	111
66	Tantalum pentaoxide	Amorphous	-	-	-	-	112
67	Tantalum phosphate	Amorphous	-	-	-	-	113
68	Tantalum arsenate	Amorphous	Ta/As=2.80	-	1.09	Na <sup>+</sup> , K <sup>+</sup> , Ba <sup>2+</sup>	114
69	Tantalum antimonate	Amorphous	Ta/Sb=1.30	-	0.99	NH <sub>4</sub> <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Vo <sup>2+</sup>	115
70	Tantalum selenite	Amorphous	Se/Ta=1.5	-	1.19	Fe <sup>3+</sup> , Ba <sup>2+</sup>	116
71	Tantalum tungstate	Amorphous	-	-	0.84	K <sup>+</sup>	117
72	Thorium Phosphate	Crystalline	Th/PO <sub>4</sub> =0.50	Th(HPO <sub>4</sub> ) <sub>2</sub> ·2H <sub>2</sub> O	0.77	Ca <sup>2+</sup> , Sr <sup>2+</sup> , Ba <sup>2+</sup>	118
73	Thorium tungstate	Amorphous	Th/W=2.0	Th(OH) <sub>2</sub> (HWO <sub>4</sub> ) <sub>2</sub> ·nH <sub>2</sub> O	0.46	Na <sup>+</sup> , K <sup>+</sup> , Cs <sup>+</sup> , Bi <sup>3+</sup> , Hg <sup>2+</sup> , Vo <sup>2+</sup>	119
74	Thorium antimonate	Amorphous	Sb/Th=3.65	-	0.32	Cu <sup>2+</sup> , Pb <sup>2+</sup>	120
75	Thorium oxide	Amorphous	-	Th(OH) <sub>n</sub> ·nH <sub>2</sub> O	2.00	Na <sup>+</sup> , Rb <sup>+</sup> , Ca <sup>2+</sup> , Sr <sup>2+</sup>	121
76	Thorium arsenate	Crystalline	As/Th=1.53	Th(HAsO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	0.20	Li <sup>+</sup>	122
77	Thorium molybdate	Amorphous	Th/Mo=0.5	-	0.75	Pb <sup>2+</sup> , Fe <sup>3+</sup> , Zr <sup>4+</sup>	123
78	Titanium arsenate	Amorphous	-	Ti(HAsO <sub>4</sub> ) <sub>2</sub> ·2.5H <sub>2</sub> O TiO <sub>2</sub> ·0.5 As <sub>2</sub> O <sub>5</sub> ·nH <sub>2</sub> O	1.00	Ba <sup>2+</sup> , Sr <sup>2+</sup> , Cd <sup>2+</sup> , Zn <sup>2+</sup> , Cu <sup>2+</sup> , Pb <sup>2+</sup>	124
79	Titanium antimonite	Amorphous	Sb/Ti=1.0-1.36	-	0.70	Mg <sup>2+</sup> , VO <sup>2+</sup> , Zn <sup>2+</sup> and rare earths	125
80	Titanium tungstate	Amorphous	Ti/W=0.80	-	0.40-0.76	Cs <sup>+</sup> , Ca <sup>2+</sup> , Cr <sup>3+</sup>	126
81	Titanium oxide	Amorphous	-	TiO(OH) <sub>2</sub> ·nH <sub>2</sub> O	2.00	Rb <sup>+</sup> , Cs <sup>+</sup> , Co <sup>2+</sup>	121
82	Titanium molybdate	Amorphous	Mo/Ti=0.5-2.0	-	0.80-1.60	Na <sup>+</sup> , K <sup>+</sup> , Ba <sup>2+</sup> , Pb <sup>2+</sup>	127
83	Titanium selenite	Amorphous	Ti/Se=1.39	-	0.78	Cd <sup>2+</sup>	128
84	Titanium vanadate	Amorphous	V/Ti=4.0	Ti <sub>3</sub> (V <sub>3</sub> O <sub>9</sub> ·1.5 H <sub>2</sub> O) <sub>4</sub>	0.68	Sr <sup>2+</sup>	129
85	Titanium tellurate	Amorphous	Te/Ti=2.06	-	-	-	82
86	Titanium ferrocyanide	Amorphous	Fe/Ti=2.00	-	1.40	Cs <sup>+</sup>	130

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87	Titanium silicate	Amorphous	Ti/Si=1.0	-	3.62	Li <sup>+</sup> , Na <sup>+</sup> , K <sup>+</sup> , Pb <sup>2+</sup> , Cr <sup>3+</sup>	131
88	Zirconium antimonate	Amorphous	-	ZrO <sub>2</sub> .Sb <sub>2</sub> O <sub>5</sub> .nH <sub>2</sub> O	-	Na <sup>+</sup> , K <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> , Rb <sup>+</sup> , Cs <sup>+</sup> , Li <sup>+</sup>	132
89	Zirconium arsenate	Amorphous	As/Zr=1.53-1.96	ZrO <sub>2</sub> .84As <sub>2</sub> O <sub>5</sub> .nH <sub>2</sub> O	4.30	Na <sup>+</sup> , K <sup>+</sup> , Cs <sup>+</sup>	133
90	Zirconium ferrocyanide	Amorphous	Fe/Zr=0.63	-	0.96	NH <sub>4</sub> <sup>+</sup> , Li <sup>+</sup> , Na <sup>+</sup> , Zn <sup>2+</sup>	134
91	Zirconium molybdate	Amorphous	Zr/Mo= 0.5-2.0	-	2.18	-	135
92	Zirconium oxide	Amorphous	-	ZrO <sub>2</sub> .4.7H <sub>2</sub> O	1.09	Ca <sup>2+</sup> , Ba <sup>2+</sup>	136
93	Zirconium pyrophosphate	Amorphous	P/Zr=2.5-2.8	-	-	Cu <sup>2+</sup> , Ni <sup>2+</sup> , Ba <sup>2+</sup> , Ca <sup>2+</sup> , Mg <sup>2+</sup> , Fe <sup>3+</sup>	137
94	Zirconium hypophosphate	Amorphous	Zr/P=0.57	-	-	Multivalent metals	138, 139
95	Zirconium selenite	Amorphous	Zr/Se=1.23	-	0.48	Ag <sup>+</sup> , Cu <sup>2+</sup> , Au <sup>2+</sup>	140
96	Zirconium silicate	Amorphous	Zr/Si=0.5	-	3.18	Ag <sup>+</sup> , Ca <sup>2+</sup> , Cu <sup>2+</sup> , Cr <sup>3+</sup> , Th <sup>4+</sup>	141, 142
97	Zirconium tellurate	Amorphous	-	Zr(H <sub>2</sub> TeO <sub>6</sub> ).4H <sub>2</sub> O	2.80		143, 144
98	Zirconium oxalate	Crystalline	-	Zr(OH)C <sub>2</sub> O <sub>4</sub> H	2.50	Alkali metals	145
99	Zirconium tungstate	Amorphous	Zr/W=1.0-0.44	-	-	Alkali metals	146
100	Zirconium polyphosphate	Amorphous	-	-	-	Na <sup>+</sup> , K <sup>+</sup> , Li <sup>+</sup> , Cs <sup>+</sup> , (NH <sub>4</sub> <sup>+</sup> form) Ba <sup>2+</sup> , Co <sup>2+</sup> , Cu <sup>2+</sup> , Fe <sup>3+</sup> (H <sup>+</sup> form)	147, 148
101	Zinc silicate	Amorphous	Si/Zn=1.25	-	2.00	-	149
102	Zinc ferro-cyanide	Amorphous	Zn/Fe=1.98	Zn <sub>2</sub> Fe(CN) <sub>6</sub>	6.10	-	150
103	Zinc phosphate	Amorphous	-	-	-	-	151

**Table 1.2** Three-component ion exchange materials with their properties

S. No.	Ion exchange Materials	Type	Composition	Empirical Formula	IEC (meq g <sup>-1</sup> )	Selectivity	References
1	Amine tin hexacyano ferrate	-		-	-	-	152
2	Ammonium dodeca molybdoantimonate	Crystalline		(NH <sub>4</sub> ) <sub>3</sub> (Mo <sub>12</sub> SbO <sub>40</sub> ).11H <sub>2</sub> O	-	-	153
3	Ammonium molybdophosphate	Crystalline		(NH <sub>4</sub> ) <sub>3</sub> PMo <sub>12</sub> O <sub>40</sub>	1.57	-	154
4	Ammonium tungstophosphate	Crystalline		(NH <sub>4</sub> ) <sub>3</sub> PW <sub>12</sub> O <sub>40</sub>	0.66	-	155
5	Anilinium zirconium phosphate	-		(ZrO <sub>2</sub> ) <sub>2</sub> (C <sub>6</sub> H <sub>5</sub> NH <sub>2</sub> ) <sub>2</sub> .HPO <sub>4</sub> .3.7H <sub>2</sub> O	1.87	Co <sup>2+</sup> , Zn <sup>2+</sup> , Cd <sup>2+</sup> , Hg <sup>2+</sup>	156
6	Anilinium tin phosphate	-		-	-	-	157
7	Chromium arsenophosphate	Amorphous	Cr:As:P=2:1:1	[Cr <sub>2</sub> O <sub>3</sub> .H <sub>3</sub> AsO <sub>4</sub> .H <sub>3</sub> PO <sub>4</sub> ].nH <sub>2</sub> O	-	K <sup>+</sup>	158
8	Cesium zirconium phosphate	-		-	-	-	159
9	Chromium arsenosilicate	Amorphous		2 Cr <sub>2</sub> O <sub>3</sub> .2.5 As <sub>2</sub> O <sub>5</sub> . 3 SiO <sub>2</sub> .nH <sub>2</sub> O	-	K <sup>+</sup>	160
10	Cerium phosphosilicate	Amorphous	Ce:Si:P=2:5:4	(CeO <sub>2</sub> ) <sub>2</sub> (SiO <sub>2</sub> ) <sub>3</sub> (H <sub>3</sub> PO <sub>4</sub> ) <sub>4</sub> .nH <sub>2</sub> O	-	-	161
11	Dipotassium tri-zinc hexa cyano ferrate	-		-	-	-	162
12	Nickel aluminosilicate	Amorphous		-	-	-	163
13	Stannic arsenoborate	Amorphous		-	0.99	-	164
14	Stannic arsenosilicate	-		-	-	-	165
15	Stannic boratomolybdate	Amorphous	Sn:B:Mo=1:1:1	-	1.12	Zr <sup>4+</sup> , Th <sup>4+</sup>	164
16	Stannic boratophosphate	Amorphous		-	1.10	-	166
17	Stannic boratosulphate	Amorphous		-	0.55	-	164
18	Stannic boratotungstate	Amorphous		-	1.15	-	164
19	Stannic iodophosphate	-		-	-	-	167
20	Stannic molybdophosphate	-	Sn:Mo:P=1:0.33:2	-	-	-	168
21	Tin(IV) arsenosilicate	-		-	-	Amino acid	169
22	Tin(IV) vanadopyro-phosphate	Micro-crystalline		-	3.17	-	170
23	Tin(IV) tungstovanado-phosphate	-		-	-	-	171
24	Tin(IV) tungstoselenate	Crystalline	Sn/Se/W=7:1:18	-	1.43	Ba <sup>2+</sup>	172
25	Titanium tungstophosphate	-		-	-	-	173
26	Titanium phosphate ammonium tungstophosphate	-		-	-	-	174
27	Titanium tungstoarsenate	-		-	-	-	175
28	Titanium arsenosilicate	-		-	-	Pb <sup>2+</sup>	176
29	Titanium vanadophosphate	-		-	-	-	177
30	Titanium tungstovanado-phosphate	-		-	-	Amines	178
31	Titanium phosphosilicate	Amorphous		-	-	Zr <sup>4+</sup> , Nb <sup>5+</sup> , Cs <sup>+</sup>	179
32	Titanium aluminium silicate	-		-	-	Pb <sup>2+</sup>	180
33	Titanium molydophosphate	-		-	-	-	181
34	Titanium phosphate ammonium phosphomolybdate	-		-	-	-	182
35	Titanium (IV) tungstosilicate	Crystalline		-	0.44	-	183
36	Zirconium hydrogen arsenate hydrogen phosphate	Amorphous		Zr(HAsO <sub>4</sub> )(HPO <sub>4</sub> ).H <sub>2</sub> O	-	-	184
37	Zirconium iodooxalate	Amorphous	Zr:IO <sub>3</sub> :C <sub>2</sub> O <sub>4</sub> H=2:1:3	(ZrO) <sub>2</sub> (IO <sub>3</sub> )(HC <sub>2</sub> O <sub>4</sub> ) <sub>3</sub> .nH <sub>2</sub> O	-	-	185



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38	Zirconium alumino pyrophosphate	-		-	-	-	186
39	Zirconium arsenophosphate	Amorphous	Zr:As:P=2:1:1	(ZrO <sub>2</sub> )(H <sub>3</sub> AsO <sub>4</sub> )(H <sub>3</sub> PO <sub>4</sub> ).nH <sub>2</sub> O	-	Rb <sup>+</sup> , Ag <sup>+</sup> , Tl <sup>+</sup>	187, 188
40	Zirconium arsenosilicate	Crystalline		-	-	Hg <sup>2+</sup>	189
41	Zirconium phosphoantimonate	-		-	-	-	190
42	Zirconium phosphoborate	-	Zr:P:B=1:1:1	-	-	-	191
43	Zirconium phthalophosphate	-		-	-	-	192
44	Zirconium vanadophosphate	-		-	-	Li <sup>+</sup> , Na <sup>+</sup> , NH <sub>4</sub> <sup>+</sup>	134
45	Zirconium sulphosalicylate	Semi-crystalline		-	-	-	193, 194
46	Zirconium phosphosilicate	Amorphous		-	2.2	Cs <sup>+</sup>	195, 196
47	Zirconium molybdophosphate	-		ZrH <sub>2</sub> MoO <sub>2</sub> .xH <sub>2</sub> O	1.6	-	197
48	Zirconium(IV) selenophosphate	-		(ZrO) <sub>5</sub> (OH) <sub>4</sub> (HSeO <sub>3</sub> ) <sub>2</sub> (H <sub>2</sub> PO <sub>4</sub> ).2H <sub>2</sub> O	-	-	198
49	Zirconium(IV) iodomolybdate	-		-	-	Hg <sup>2+</sup>	199
50	Zirconium(IV) iodophosphate	-		-	1.78	-	200
51	Zirconium titanium phosphate	Crystalline	Zr/Ti=3.25	Zr <sub>x</sub> Ti <sub>1-x</sub> (HPO <sub>4</sub> ) <sub>2</sub> .H <sub>2</sub> O	6.87	-	201-206
52	Zirconium molybdovanadate	-	Zr:V:Mo=1.68:1:0.08 8		-	Li <sup>+</sup> , Na <sup>+</sup>	207
53	Zirconium tungstoarsenate	Amorphous		-	-	UO <sub>2</sub> <sup>2+</sup> , Cs <sup>+</sup> , Tl <sup>+</sup>	208
54	Zirconium(IV) tungstophosphate	-	Zr:W:P= 18.8%:25.4%:4.3%		1.00	Pb <sup>2+</sup>	209, 210
55	Zirconium cerium phosphate	-		-	-	-	211
56	Zirconium(IV) 4-amino 3-hydroxy naphthalene sulphonate	-		-	-	Hg <sup>2+</sup>	212
57	Zirconium bis (monoocetyl) phosphate	Crystalline		-	-	-	213
58	Zirconium(IV) iodovanadate	Amorphous	Zr/IO <sub>3</sub> /VO <sub>3</sub> =2:1:3	(ZrO <sub>2</sub> )(IO <sub>3</sub> )(V <sub>5</sub> O <sub>9</sub> )	4.20	Electron exchanger	214
59	Zirconium(IV) tungsto-molybdate	Amorphous		(ZrO)(OH) <sub>2</sub> (H <sub>2</sub> WO <sub>4</sub> ) <sub>4</sub> (H <sub>2</sub> MoO <sub>4</sub> ) <sub>3</sub> . 8H <sub>2</sub> O	2.40		215
60	Acryl amide zirconium phosphate	Crystalline		-	2.26	-	216

## COMPOSITES

The main advantages of synthetic organic ion exchange resins are their high capacity, wide applicability, wide versatility and low cost relative to some synthetic inorganic materials. But they also have some limitations.

The main limitations are their limited radiation and thermal stabilities. At a total absorbed radiation dose of  $10^9$  to  $10^{10}$  rads most organic resins will exhibit a severe reduction in their ion exchange capacity (a 10 to 100% capacity loss), due to physical degradation at both the molecular and macroscopic level. The conversion of inorganic ion exchange materials has been taking place into composite ion exchange materials is the latest development in this discipline.

Sol-gel derived composite materials have found numerous applications in the areas of chemistry, biochemistry, engineering, and material science [217]. The 'organic-inorganic' hybrid materials prepared via the sol-gel technique have attracted significant attention in the literature [218]. The combination of organic and inorganic precursors yields hybrid materials that have mechanical properties not present in the pure materials. Organic-inorganic composite ion exchange materials show the improvement in its granulometric properties that makes them more suitable for the application in column operations. The binding of organic polymer also introduces the better mechanical properties in the end product, i.e. composite ion exchange materials.

More recently, some organic-inorganic composite ion exchange materials have been developed. Khan et al. have reported polypyrrole-Th(IV) phosphate [219], polyaniline-Sn(IV) arsenophosphate [220] and polystyrene-Zr(IV) tungstophosphate [221] used for the selective separation of  $Pb^{2+}$ ,  $Cd^{2+}$ ,  $Hg^{2+}$  respectively, and ion exchange kinetics of  $M^{2+}$ - $H^+$  exchange and adsorption of pesticide [222] have also carried out on these materials. Beena Pandit et al. have synthesized such type of ion exchange materials, i.e. o-chlorophenol Zr(IV) tungstate and p-chlorophenol Zr(IV) tungstate [223]. Styrene supported Zr(IV) phosphate hybrid material [224], and fibrous ion exchange materials such as polymethyl methacrylate, polyacrylonitrile, styrene and pectin based Ce(IV) phosphate, Th(IV) phosphate and Zr(IV) phosphate [225] have been reported by Varshney et al. have numerous analytical applications. These materials can be used as ion exchanger membranes and electrodes.

Polyacrylonitrile fibers and zeolite composites have also been reported in literature. Polyaniline-Zr(IV) tungstophosphate has been synthesized by Gupta et al. [226], which was used for the selective separation of  $La^{3+}$  and  $UO_2^{2+}$ . Chanda et al. reported polyacrylic acid coated  $SiO_2$  as a new ion exchange material. A polymeric/inorganic hybrid sorbent has also been used for arsenic removal [227].

Ion exchangers find applications in a wide variety of industrial, domestic, governmental and laboratory operations. The composite ion exchangers show some better granulometric properties that facilitates its stability in column operations especially for separation, filtration and preconcentration of ionic species. The column operation suitability makes it more convenient in regeneration of exhausted beds also. These hybrid ion exchangers have good ion exchange capacity, high stability, reproducibility and selectivity for heavy metal ions indicating its useful environmental applications. In general these materials have their applications in the following disciplines:

- Water softening [228, 229]
- Separation and preconcentration of metal ions [230]
- Nuclear separations [231]
- Catalysis [232]
- Redox system [233]
- Electrodialysis [234]
- Hydrometallurgy [235]
- Effluent treatment [236]
- Ion exchange fibers [237-239]
- Ion-selective electrodes [219, 240]

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Ion exchange can be defined as the reversible exchange of ions between a liquid phase and a solid phase (ion-exchange resin) which is not accompanied by any radical change in the solid structure. From: Principles of Fermentation Technology (Third Edition), 2017.

Related terms: Also, the basic principles of fixed and fluidized beds systems and their application in ion exchange process are presented. The application of the ion exchange is constantly developed along with the development of the worldwide economy branches. Ion-exchange membranes have recently been widely used in battery applications. Iron-chromium, one of the first developed redox flow batteries, used ion-exchange membranes from Ionics Inc. (Series CD1L). Ion exchange refers to the interaction of ionic species in aqueous solutions with adsorbent solid materials. It is distinguished from conventional Adsorption by the nature and morphology of the adsorbent material which in most cases is either a dynamic polymer matrix or an inorganic structure containing exchangeable functional groups. All modern ion exchange resins are polymeric structures, generally based either on styrene or an acrylic matrix. Inorganic materials are either naturally-occurring or synthetic materials. Typical examples are natural and synthetic zeolites; insoluble salts, e.g., hydrous zirconium phosphate; heteropolyacids, e.g., ammonium phosphomolybdate; natural clays, e.g., montmorillonite.

**Ion Exchange Selectivity. Inorganic and Composite Ion Exchange Materials and their Applications. (Review). Mu.**

Some of the important two component ion exchangers with their composition, ion exchange capacity and selectivity of metal ions have been reported in table 1.1.

**6 HETEROPOLYACIDS** Heteropolyacid salts can be used as inorganic ion exchangers. These exchangers are also called "Acidic ion exchange" materials, because their negative charges result from the ionization of acidic group. Anionic exchangers have positively charged groups that will attract negatively charged anions. These are also called "Basic ion exchange" materials. Then these particles are analyzed spectroscopically.

**Applications of ion exchange chromatography.** An important use of ion-exchange chromatography is in the routine analysis of amino acid mixtures. The 20 principal amino acids from blood serum or from the hydrolysis of proteins are separated and used in clinical diagnosis. Inorganic ions also can be separated by ion-exchange chromatography. Limitations of ion exchange chromatography. Only charged molecules can be separated.