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Studies on Pectin-Tin(IV) Phosphate: A New Biopolymer Doped Hybrid Ion Exchanger, An Efficacious Ion Exchanger in Water Purification Process

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Pectin doped tin(IV) phosphate was prepared as novel class of hybrid ion exchanger *i.e.* biopolymer based hybrid ion exchanger, which has been characterized by few physico-chemical characterization techniques such as FTIR analysis, SEM study, UV-vis spectrophotometry and elemental analysis. The characteristic parameters of novel ion exchange such as ion exchange capacity, concentration study, elution study, thermal stability, *etc*. were also reported. Adsorption study has been explored for few alkaline earths and some heavy or transition metal ions in several acidic media. Based on adsorption study, it has been found that the reported ion exchanger has shown enantioselectivity for cadmium(II) ions, being one among toxic metal ions. The antimicrobial activity of pectin based tin(IV) phosphate on few microorganisms has also been studied to prove the antimicrobial nature of the synthesized ion-exchange material.

Keywords: Hybrid ion exchanger, Pectin, Biopolymer, Tin(IV) phosphate, Ion exchange studies, Cadmium.

INTRODUCTION

Recently, hybrid ion exchangers [1-4] have opened a plethora of possibilities for chemists and researchers. Organic ion exchangers have been entirely reproducible in their ion exchange properties and depict chemical stability [4]. However, they mislay their ion exchange characteristics with respect to radiations and elevated temperatures. Inorganic ion-exchangers have been entrenched their place in ion exchange chemistry because of their tenaciousness on appearance of strong radiations and high up temperatures and their specificity for several metal ions [4]. Nonetheless, the primary disadvantage of these materials has been their chemical and mechanical stability. Further, they are very little reproducible in ion exchange conducts. However, the chemical and mechanical instability of these materials has proven to be their main drawback. Farther, they do not reveal substantial reproducibility in ion-exchange behaviour. The pitfalls and benefits of both, organic and inorganic ion exchangers have paved the way to investigate the properties of organic based inorganic or hybrid ion exchangers.

The search for increased chemical, thermal and mechanical strength, reproducibility in ion-exchange behaviour, as well as their selectivity towards metal ions, has been sparked by these hybrid ion-exchangers. A wealth of aforesaid materials [5-12] have been prepared by combining organic species whether it is monomer [5-10] or polymer [11,12] and inorganic species in the laboratories and they have proved their important place in analytical chemistry, evident by their promising ion-exchange conducts. Several authors [13-15] used surfactant media in order to explore ion exchange characteristics of few ion exchange materials and reported the enhanced ion exchange properties of these materials, which has led to synthesis of surfactant based hybrid ion exchangers [16-24].

Cadmium is the poisonous heavy metal, which builds up and is retained by the body, leading to kidney damage, lung cancer and demineralization of the bones [25]. Many products, including electroplating, storage batteries, vapour lamps and some solders, employ cadmium. Hence, before wastewater is discharged into freshwater bodies, the most essential objective is to eliminate harmful heavy metal pollutant ions since these ions can directly or indirectly cause a multitude of health issues in humans and aquatic animals [26].

Among other ion exchangers, due to their outstanding ion-exchange behaviour, tin-based cation exchangers have drawn a lot of attention [27,28]. Pectin, a soluble biopolymer, primarily used in food production and home cooking has been well known owing to its health benefits and nutritional values. Pectin has been used in the synthesis of cerium and thorium phosphates, fibrous ion exchangers [3,29] and antibacterial activity of pectin cerium iodate has been reported for bacteria, *E. coli* [29]. By virtue of properties of pectin and tin(IV) phosphate, efforts have been made to develop pectin doped tin(IV) phosphate.

Present work approaches synthesis and investigations on pectin doped tin(IV) phosphate (PSP), hybrid cation exchanger in which pectin has been doped into its layers. Studies include physico-chemical characterization by FTIR analysis, SEM study, UV-vis spectrophotometry and elemental analysis along with ion-exchange properties such as ion exchange capacity, concentration study, elution study, thermal stability and adsorption study for a few alkaline earths and heavy metal ions. According to the findings of this research, the prepared pectin doped tin(IV) phosphate (PSP) has a high degree of selectivity towards cadmium(II) ions. The antimicrobial activity of above synthesized material on few microorganisms has also been explored.

EXPERIMENTAL

Reagents and chemicals: From CDH, India, tin chloride ($SnCl_4·5H_2O$) was obtained. Both phosphoric acid (H_3PO_4) and pectin were purchased from Qualigens and E. Merck, India. All other substances were of Anala R grade.

Characterization: FTIR studies were performed on Nicolet iS5 FTIR spectrometer whereas UV-visible and SEM studies were conducted on double beam spectrophotometer, model-LMSPUV1900 and JEOL JSM 840, SM, respectively. Elemental analysis (carbon, hydrogen, nitrogen) was done by Heraeus Carlo Erba-1108 analyzer while tin and phosphorus were investigated by Inductively Coupled Plasma Atom Emission Spectrometer.

Preparation of the solutions, used: Solutions of tin(IV) chloride, phosphoric acid and pectin were made ready in demineralized water.

Synthesis of pectin doped tin(IV) phosphate: Several fractions of pectin doped tin(IV) phosphate were prepared with the addition of one volume of 0.3 M tin(IV) chloride solution to a mixture of 0.6 M H_3PO_4 and pectin (1:1) dropwise and the mixture was constantly stirred. The ensuing slurry was retained for about 24 h. Then, it was filtered and washed with demineralized water until pH ~4. It was dried and splintered into tiny granules by putting in demineralized water and transformed to H^+ by using 1 M of HNO_3 for about 24 h and at long last washed with demineralized water to take out profusion of acid, desiccated at 45 ºC and shifted to 50-70 mesh sized molecules. Eight samples were prepared by changing the pectin concentration. Considering that sample-5 had the greatest measured ion exchange capacity and chosen for further research. Table-1 depicts the samples synthesized with their corresponding ion exchange capacity.

Ion exchange capacity (IEC): By using a glass column with glass wool at the bottom and an internal diameter of less than 1 cm, the column technique was used to measure the ability of the material to exchange ions. The material in H^+ form (1 g) was used. As eluant, 250 mL of 1 M $NaNO_3$ solution were utilized at a flow rate of less than 0.5 mL/min. To calculate the total amount of H^+ ions emitted, the effluent was titrated against a standard alkali solution.

TABLE-1
PREPARATION OF DIFFERENT SAMPLES OF PECTIN-TIN(IV) PHOSPHATE

Number of sample	Pectin (%)	Na^+-ion exchange capacity (meq/dry g)
1	0	1.50
2	1.0	1.75
3	3.0	2.10
4	5.0	2.25
5	5.5	2.60
6	6.0	2.60
7	7.0	2.60
8	9.0	2.40

Concentration behaviour: Concentration behaviour was studied to determine the optimal concentration of an eluent. For that different concentration of eluent ($NaNO_3$) were passed in the columns of the synthesized material and, ion exchange capacities were calculated by column process [5].

Elution behaviour: By allowing various fractions of 10 mL of eluant ($NaNO_3$) to pass through the column during an elution study, to determine the volume of eluant required for full H^+ ion-exchange.

Thermal study: For thermal experiments, several 1.0 g fractions of hybrid ion-exchange material were exposed to temperatures varying from 45 to 700 ºC for 1 h in a muffle furnace and their ion exchange capabilities were then assessed using a column method after cooled to room temperature.

Adsorption study: The hybrid ion-exchanger (200 mg) was introduced to 20 mL of solution consisting of metal ions solution and acid solution in 1:9 ratio. For same mixture to reach equilibrium, it was held for 24 h. EDTA complexometric titrations were applied to quantify the concentration of metal ions prior to and afterwards reaching equilibrium. The cofficient distribution (K_d) value was calculated using following eqn.:

$$K_d = \frac{I - F}{F} \times \frac{V}{M}$$

where I and F are the initial and final metal ion concentrations; V is the total volume of solution (mL) and M is the weight of the exchange material (g).

Separations study: Using a column containing hybrid ion-exchanger material (2 g) with an internal diameter of 0.6 cm, several binary separations were performed. The mixture to be separated was put onto the column while it was being thoroughly washed with demineralized water at a flow rate of around 2-3 drops per min (0.15 mL min^{-1}). The separation was accomplished by eluting the column with a suitable solvent and the metal ions in the effluent were quantified using EDTA titrations.

Biological studies: The antibacterial activity and minimum inhibitory concentrations (MIC) of PSP against *Staph awrens*, *E. coli* ESS 2231, *Protect vulgaris* and *Aspergillus fumigatus* have been determined using the agar well-diffusion method.

RESULTS AND DISCUSSION

Ion exchange capacity (IEC): It was found that the newly synthesized hybrid ion-exchanger (PSP) has a greater ion exchange capacity (2.60 meq/dry g) (Table-2) by comparison to some earlier formed hybrid ion exchange materials [5-10, 17,18] (Table-3). Increased interlayer distances in tin(IV) phosphate are likely due to the intercalation of pectin, which consists of polar molecules, in the layers of tin(IV) phosphate.

TABLE-2
ION EXCHANGE CAPACITIES OF PECTIN-TIN(IV) PHOSPHATE FOR SOME METALS

Solutions of metal	Na^+-ion exchange capacity (meq/dry g)
Li(I) Cl	2.25
Na(I) NO_3	2.60
K(I) Cl	2.75
Mg(II) Cl	2.15
Ca(II) Cl	2.65
Sr(II) Cl	2.85
Ba(II) Cl	3.10

TABLE-3
COMPARATIVE STUDY OF ION EXCHANGE CAPACITY OF PECTIN-TIN(IV) PHOSPHATE IN COMPARISON TO OTHER HYBRID ION EXCHANGERS, PREPARED EARLIER

Hybrid ion exchangers	Na^+-ion exchange capacity (meq/dry g)
Pectin doped tin(IV) phosphate (Present work)	2.60
Acrylamide Th(IV) phosphate [5]	2.00
Pectin Ce(IV) phosphate [6]	1.78
Pectin Th(IV) phosphate [6]	2.15
Cellulose acetate Th(IV) phosphate [6]	1.70
Acrylonitrile Zr(IV) phosphate [6]	2.08
Acrylamide Sn(IV) phosphate [6]	2.10
n-Butyl acetate Ce(IV) Phosphate [10]	2.25
n-Butyl acetate Sn(IV) Phosphate [17]	2.20
n-Dodecyl pyridinium chloride Sn(IV) phosphate [18]	2.39

Concentration behaviour: The eluant concentration for the entire elution of H^+ ions from the ion-exchanger matrix was found to be 1 M (Table-4).

TABLE-4
CONCENTRATION STUDY OF PECTIN-TIN(IV) PHOSPHATE USING DIFFERENT ELUANT CONCENTRATION

Molar concentrations of $NaNO_3$	Na^+-ion exchange capacity (meq/dry g)
1/5	0.80
2/5	1.15
3/5	2.00
4/5	2.40
1	2.60
6/5	2.60

Elution behaviour: Fig. 1 show that only 160 mL of 1 M $NaNO_3$ is required for complete elution of H^+ ions from the exchanger matrix.

Thermal study: On heating the synthetic ion-exchanger from 45 to 600 ºC, a gradual decline in the ion-exchange capacity is observed (Table-5). According to the findings, the exchanger is quite stable up to 200 ºC, without losing much of its capacity. On heating upto 400 ºC it exhibits decrement by retaining 73.08% of its ion exchange capability. However, it begins to lose upto 50% of its capacity up to 500 ºC. Afterwards, it starts losing almost its ion exchange capacity by retaining sonly 19.23% of its ion exchange capacity at 600 ºC.

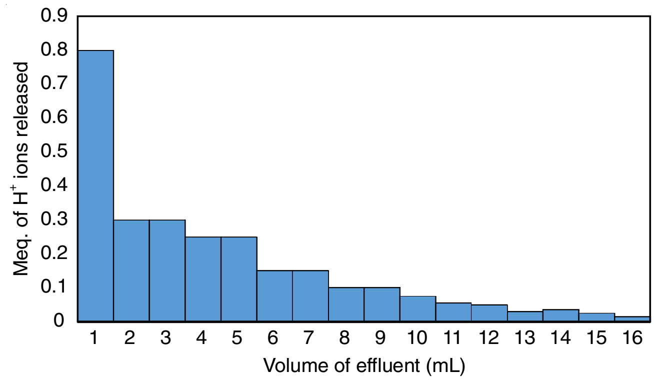

Fig. 1. Elution study of pectin-tin(IV) phosphate

TABLE-5
THERMAL STABILITY OF PECTIN-TIN(IV) PHOSPHATE AFTER HEATING AT VARIOUS TEMPERATURES FOR 1 h EACH

Temp. (°C)	Na^+-ion exchange capacity (meq/dry g)	Colour	Retention in ion exchange capacity (%)
45	2.60	Translucent white	100
100	2.50	Translucent white	96.15
200	2.45	Off white	94.23
300	2.00	Pale white	76.92
400	1.90	Pale white	73.08
500	1.30	Brownish white	50.00
600	0.50	Blackish white	19.23

Adsorption study: Adsorption studies of different metals on the synthesized PSP in different concentrations of HCl, HNO_3, $HClO_4$ and demineralized water is reported in Table-6, which reveals that the synthesized hybrid ion-exchanger have been specifically selective for Cd(II) ions.

Separations study: Based on specific selectivity of PSP, few binary separations have been explored on lab made samples in order to prove worth of the synthesized material in water pollution control. Results are depicted in Table-7.

IR studies: The matrix of synthetic hybrid ion exchange material is revealed by the IR analyses (Fig. 2) to contain metal-oxygen and metal-hydroxide linkages as well as external water molecules, phosphate groups and pectin components [30]. The peaks at 590.6 cm^{-1} and 1001.6 cm^{-1} confirmed the presence of phosphate group whereas the peaks at 1650.8 cm^{-1} and 3485.1 cm^{-1} depict the existence of the extracellular water molecules. The existence of pectin component in the synthesized exchange material is confirmed by the peaks at 1386.5, 1656.3, 2295.3, 2310.5 and 2936.5 cm^{-1}, which are due to aliphatic C-H, $–CH_2$ stretching and carbonyl groups [31].

Elemental analysis: The pectin-tin(IV) phosphate matrix of the hybrid ion exchanger is shown by the elemental analysis (Table-8) to contain pectin.

TABLE-6
ADSORPTION STUDY OF PECTIN-TIN(IV) PHOSPHATE FOR FEW ALKALINE EARTHS AND HEAVY METAL IONS

Metal ions	DMW	HCl			HNO_3			$HClO_4$		
		0.01 M	0.1 M	1 M	0.01 M	0.1 M	1 M	0.01 M	0.1 M	1 M
Mg^{2+}	571.43	571.43	422.22	327.27	487.50	327.27	261.54	422.22	370.00	261.54
Ca^{2+}	800	1025.00	542.86	400.00	650.00	462.50	350.00	800.00	650.00	350.00
Sr^{2+}	650	542.86	400.00	350.00	650.00	542.86	350.00	542.86	400.00	275.00
Ba^{2+}	760	975.00	514.29	437.50	760.00	514.29	437.50	514.29	330.00	330.00
Fe^{3+}	633.33	633.33	528.57	340.00	780.00	633.33	528.57	528.57	450.00	388.89
Mn^{2+}	557.14	666.67	475.00	411.11	557.14	360.00	360.00	411.11	318.18	228.57
Ni^{2+}	820	666.67	557.14	318.18	411.11	283.33	228.57	475.00	318.18	228.57
Co^{2+}	616.67	616.67	514.29	437.50	514.29	330.00	330.00	437.50	437.50	330.00
Cu^{2+}	840	683.33	571.43	327.27	571.43	327.27	261.54	487.50	422.22	261.54
Cd^{2+}	TA	TA	1050.00	666.67	2200.00	557.14	411.11	1433.30	1050.00	557.14
Hg^{2+}	683.33	571.43	571.43	261.54	840.00	422.22	327.27	487.50	261.54	261.54
Pb^{2+}	1025	800.00	650.00	542.86	650.00	350.00	275.00	800.00	542.86	400.00

TABLE-7
SEPARATIONS STUDY OF PECTIN-TIN(IV) PHOSPHATE

Separation conducted	Amount of metals loaded (mg)	Amount of metals found (mg)	Error (%)	Eluent	Volume of eluent used
Mg^{2+}-Cd^{2+}					
Mg^{2+}	3.8888	3.8888	0	0.1 M HNO_3	70
Cd^{2+}	17.9858	17.5948	-2.17	1 M $HClO_4$	60
Hg^{2+}-Cd^{2+}					
Hg^{2+}	32.0944	31.4115	-2.13	1 M NH_4Cl + 1 M HCl	70
Cd^{2+}	17.9858	17.5948	-2.17	1 M $HClO_4$	60
Ca^{2+}-Cd^{2+}					
Ca^{2+}	6.4125	6.1275	-4.44	1 M HCl	50
Cd^{2+}	17.9858	17.2038	-4.35	1 M $HClO_4$	60

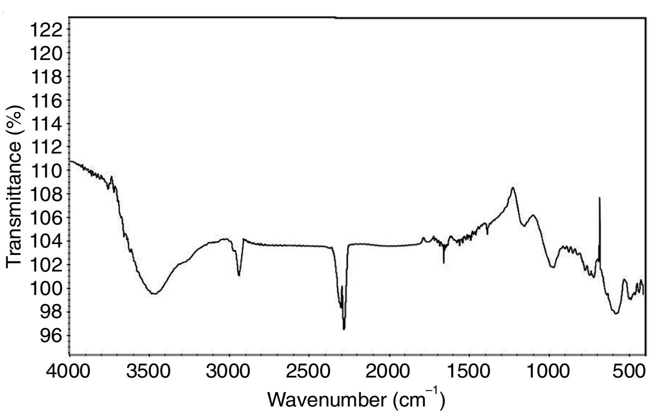

Fig. 2. FTIR spectra of pectin-tin(IV) phosphate

TABLE-8
ELEMENTAL DATA OF PECTIN-TIN(IV) PHOSPHATE

Components	% in 0.25 g of exchanger
Tin	40.30
Phosphorus	25.20
Carbon	8.6
Hydrogen	2.25

Scanning electron microscopy (SEM) study: Tin(IV) phosphate is not fibrous in its natural state however, it has been demonstrated to be fibrous in the context of a synthetic hybrid ion-exchanger as revealed by a SEM images (Fig. 3). The reason is attributed due to the intercalation of tin(IV) phosphate with pectin molecules.

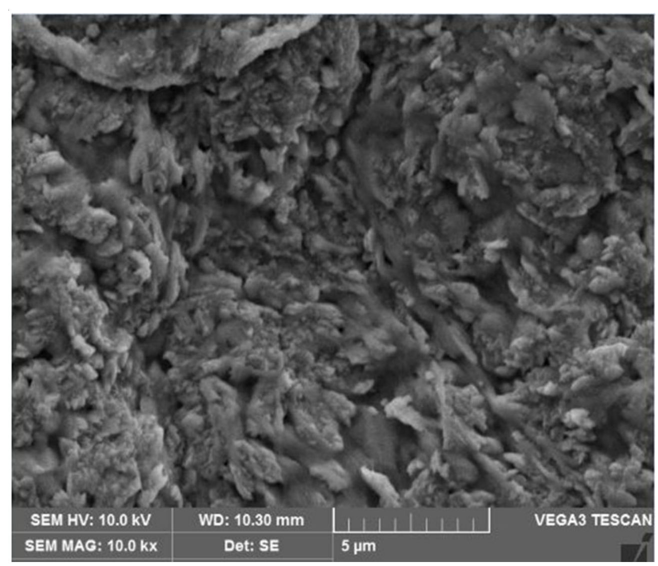

Fig. 3. SEM photograph of pectin-tin(IV) phosphate

XRD studies: The weakly crystalline character of the material is revealed by the XRD spectrum (Fig. 4), which was performed to determine the nature of the material in terms of solid state structure.

UV-visible studies: UV-visible spectra depicts a single peak which affirms that after doping of pectin we got a single product, *i.e.*, pectin-tin(IV) phosphate as a new hybrid ion exchanger, as shown in Fig. 5.

TABLE-9
ANTIBACTERIAL STUDY ON PECTIN-TIN(IV) PHOSPHATE

Conc. (µg/mL)	109	Conc. (µg/mL)	135	Conc. (µg/mL)	262	Conc. (µg/mL)	131
Staph awrens	20	*E. coli* ESS 2231	31	*Protect vulgaris*	25	*Aspergillus fumigatus*	22

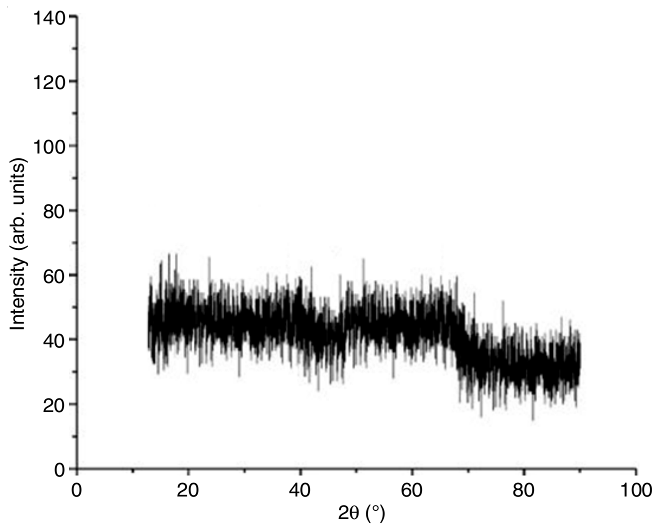

Fig. 4. X-ray diffraction pattern of pectin-tin(IV) phosphate

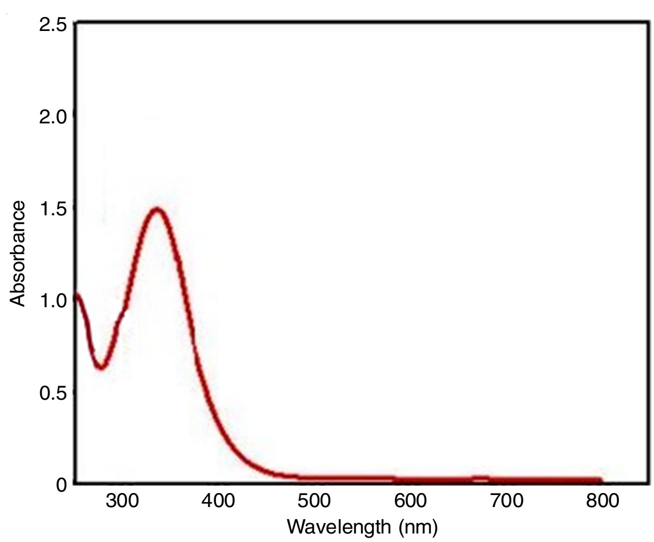

Fig. 5. UV-visible spectra of pectin-tin(IV) phosphate

Biological studies: The antibacterial activity of synthesized material against various bacteria like *Staph. awrens*, *E. coli* ESS 2231, *Protect vulgaris* and *Aspergillus fumigatus* have been reported in Table-9, which reveals the highest activity for *E. coli* ESS 2231.

Conclusion

The inclusion of pectin into tin(IV) phosphate matrix has increased the material's ability for ion exchange, which may be related to an increase in the distance between tin(IV) phosphate layers that allows for ion exchange. When compared to other reported hybrid ion exchangers, pectin doped tin(IV) phosphate (PSP) exhibited the highest ion exchange capacity. Additionally, the synthetic hybrid ion-exchanger is found to be more thermally stable, cadmium selective and has disinfectant properties which reveals its potential applications in water purification processes.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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