

## Synthesis and Characterisation of Ligand Free Cadmium Imprinted Polymer for Sensing of Cd(II) from Aqueous Solution

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### ABSTRACT

Cadmium(II)-imprinted polymer [Cd(II)-IIP] was synthesised without using the conventional complexing ligand to reduce unnecessary step for complexing Cd(II). During synthesis of Cd(II)-IIP, the complete removal of Cd(II) from the polymer was confirmed using Energy Dispersive Analysis of X-rays (EDAX) and Atomic Absorption Spectrophotometer (AAS). Synthesised polymers ([Cd(II)-IIP], non-imprinted polymer (NIP) and polymer with cadmium (CdP) were also characterised and compared. Scanning electron microscope (SEM) analysis of Cd(II)-IIP indicated the changes in surface morphology after imprinting, the polymer looked like a flaky material in comparison with NIP and CdP. BET surface area of Cd(II)-IIP (68 m<sup>2</sup>/g) was found to be more than that of NIP (24.6 m<sup>2</sup>/g) and CdP (17 m<sup>2</sup>/g). Cd(II)-IIP formed a complex with Cd(II) in the solution through carbonyl oxygen of the acrylamide as confirmed by FTIR. TGA analysis showed less char yield for Cd(II)-IIP than NIP. Cd(II)-IIP was further explored for the fabrication of ion selective electrode (ISE), which on binding with Cd(II) gave higher potential than NIP-ISE. Interference study was also performed using Cd(II)-IIP as such and also with fabricated ISE with Cd(II)-IIP using different cations such as Pb(II), Zn(II), Mn(II), Hg(II), and found that imprinted polymer showed more selectivity towards Cd(II) in aqueous solutions.

**Keywords:** Cadmium; Imprinted polymer; Selective recognition; Interference; Polymer.

### 1. INTRODUCTION

Cadmium is present in very minute quantity in environment. It can rise to maximum contamination level due to improper effluent disposal<sup>1</sup> or sabotaging of the environment. Cadmium is considered to be one of the most toxic metals, and causes severe health problems to living beings because it has long biological half-life<sup>2</sup>. Therefore, it is essential to make the water free from cadmium presence by means of its detection and subsequent removal.

A plethora of methods are available for the removal of metal ions from aqueous solutions which work on different principle such as ion exchange, solvent extraction, reverse osmosis, precipitation and adsorption<sup>3-4</sup>. Out of these technique, adsorption has a key role for the removal of Cd(II) from aqueous medium using various adsorbents such as molecularly imprinted polymer (MIPs), carbon, resins, bentonite etc. Among these, MIPs are important; because they have an ability to bind the analyte selectively<sup>5</sup>. Basically, MIPs are the synthesised polymers with predetermined imprinted sites with excellent thermal, chemical and mechanical stability properties. MIPs are employed for preconcentration<sup>6</sup>, solid phase extraction<sup>7</sup>, removal of toxicants<sup>8</sup>, liquid chromatography<sup>9</sup>, recognition of chemical warfare agent sulphur mustard and other species<sup>10-11</sup>.

Syntheses of the MIPs take place in three steps (a) precomplexation (b) co-polymerisation and (c) removal of

template molecule<sup>12</sup>. Metal ion imprinted polymers (MIIPs) are similar to MIPs but they recognise inorganic metal ions after ion imprinting<sup>13-16</sup>.

In the present study, Cd(II)-IIP has been synthesised without using a complexing ligand by using bulk polymerisation technique<sup>17</sup> which avoid the use of complexing ligand to make Cd-complex before synthesizing Cd-IIP. [Cd(II)-IIP] was also studied for its sensing behaviour by the fabrication of ion selective electrode (ISE) for Cd(II) ions.

### 2. MATERIALS AND METHODS

#### 2.1 Reagents and Chemicals

Acrylamide (Aldrich-USA), divinyl benzene (50 % Fluka-USA), 2,2-azobis isobutyronitrile (AIBN) (John Baker, USA), 2-methoxy ethanol (Rankem-India), cadmium chloride mono hydrate (Merck-India), thiourea (Aldrich-USA), hydrochloric acid (Qualigens-India), poly (vinyl chloride) (Aldrich-USA), 2-nitrophenyloctyl ether (NPOE) (Fluka-USA), and tetrahydrofuran (Aldrich-USA) of analytical grade and Millipore water (18 MΩcm at 25 °C) were used in this study.

#### 2.2 Instruments

Atomic absorption spectrophotometer (AAS) (GBC Avanta PM, Australia), TGA-2950 TA instruments USA, SAA (Autosorb 1C-AS1C-RGA2, Quanta Chrome, USA), Scanning electron microscope (SEM) and Energy Dispersive

X-ray Analysis (EDAX) ESEM-EDX (Quanta400-ESEM with EDAX-FEI, The Netherlands), Ultrasonicator (Yarco, India), Potentiostat/galvanostat with frequency response analyser (Autolab-302 with FRA, The Netherlands), Bruker tensor 27 FTIR (Germany) and Eutech instruments pH meter (pH-510, Singapore) were employed in this study.

### 2.3 Synthesis of Cd(II)-IIP, CdP and NIP

Cd(II)-IIP was synthesised by following the same procedure as described in our previous work for Cu(II)<sup>13</sup>. In brief, Cd(II)-IIP was synthesised by using three-step method based on free radical polymerisation technique. First step comprising of pre-complexation of acrylamide (10 mM) monomer and cadmium chloride (2.5 mM) in 2-methoxy ethanol followed by second step, addition of divinyl benzene (40 mM) as cross linker and 2,2'-azobis isobutyronitrile as free radical initiator, and then nitrogen gas was purged for 10 min in order to maintain inert atmosphere. Mixture was ultra-sonicated for a period of 1 h to achieve homogeneity after that it was kept in a vacuum oven at 60 °C for 24 h. In the third step, cadmium was removed from the polymer with the help of 0.1 M acidic thiourea. Subsequently, Cd(II)-IIP was washed with Millipore water for 4–5 times to make it free from acid then dried in oven at 65 °C for 2 h. Simultaneously, NIP was also synthesised following the same protocol but without adding cadmium chloride while CdP was ready even after 2<sup>nd</sup> step.

### 2.4 Characterisation of Cd(II)-IIP, Cd-P and NIP

Synthesised polymers such as Cd(II)-IIP, Cd-P and NIP were characterised using SEM, EDAX, TGA, surface area analyser, AAS and FTIR. Surface morphology of polymers was studied using SEM while elemental composition was studied by EDAX. FTIR study of polymers was performed by making pellet with KBr then scanned from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. EDAX and AAS were used for elemental analyses and for their removal study also. Thermal stability of Cd(II)-IIP, Cd-P and NIP was studied using TGA from 0 °C to 800 °C in N<sub>2</sub> atmosphere at heating ramp of 20 °C/min. Surface area of the synthesised Cd(II)-IIP, Cd-P and NIP were carried out after degassing the samples and carrying out the study under N<sub>2</sub> adsorption and desorption mode.

### 2.5 Recognition of Cd(II) by Cd(II)-IIP

Batch experiments were performed to recognise cadmium ion from aqueous solution having buffer range of pH 3 to pH 8 of phosphate-citric acid buffer (McIlvaine's buffer) using Cd(II)-IIP and also compared with the NIP. pH study revealed that pH 3.0 was the most suitable for maximum rebinding of Cd(II). Solution pH above 7.0 precipitates the cadmium ion in solution and also pH below 3.0 is not favorable for the binding of cadmium ion from solution as the potential change observed with ISE was negligible.

A solution of cadmium in the concentration range of 10 µg/mL (10 mL) was used for recognition study of Cd(II) from using 0.05g Cd(II)-IIP or NIP. Equilibrium time for each experiment was 4 h with stirring. AAS was used to determine the remaining concentration of Cd(II) in solution after rebinding with polymer. Adsorbed amount of cadmium was

determined by the difference in between initial concentration and the remaining concentration after adsorption. Imprinting factor of imprinted polymer was found 1.6. Cd complexed Cd(II)-IIP was regenerated for further use using 0.1 M acidic thiourea solution.

### 2.6 Interference Studies with Imprinted Polymer

Interferences study was performed to observe the effect of interferences on binding capacity of Cd(II)-IIP with Cd(II). This study was completed by the treatment of Cd(II)-IIP with single metal ion solution and also with binary solution where competitive adsorption of ions took place. For each experiment, contaminant concentration was 10 µg/mL even in binary mixture of the metal ions. The combinations of ions such as Mn(II)+Cd(II), Zn(II)+Cd(II), Pb(II)+Cd(II) and Hg(II)+Cd(II) were tested. In a batch experiment, 0.05 g of the Cd(II)-IIP was stirred with 10 mL solution of given concentration at room temperature. Treated sample was then filtered and aspirated in AAS for the estimation of metal ions.

### 2.7 Sensing of Cd(II) by Cd(II)-IIP

Ion Selective Electrode (ISE) based on Cd(II)-IIP or NIP were fabricated by applying the same method as described in earlier paper<sup>13</sup>. Where PVC membrane sensor was prepared first by suspending the polymer particles of Cd(II)-IIP (90 mg) or NIP (90 mg) in 0.25 ml of NPOE and mixed with the solution of THF (3.0 ml) containing 90 mg PVC. The mixture was sonicated to homogenise the solution then poured onto a glass plate and spreaded. After drying the membrane at room temperature, thickness was found to be 0.29 mm. This was cut into the shape of circle bigger than the diameter of hollow cylinder (20 mm dia.), where the membrane had to be fixed with adhesive. After fixing the membrane at one end, the tube was then filled with an internal filling solution of 10<sup>-3</sup> M of Cd(II) and then used these assembly for sensing purpose. The change in potential at the surface of membrane was measured using chrono-potentiometric method with respect of cadmium ion: Ag/AgCl/test solution/membrane/internal standard solution/Ag/AgCl<sup>13</sup>

Concentration of metal ion varying in range from 10<sup>-7</sup> to 10<sup>-3</sup> M in buffer solutions having various pH ranges were used for cadmium sensing and MIP-ISE sensing behavior also compared with that of NIP-ISE. These ISE did not require special condition to store and this can be kept in air when not in use. Even after six months, these were employed for Cd(II) sensing. Interference study of fabricated ISE was also studied with metal ions such as Mn(II), Zn(II), Pb(II) and Hg(II) in buffer solution of pH range from 7.0 to 3.0.

## 3. RESULTS AND DISCUSSION

### 3.1 Characterisation of Cd(II)-IIP, CdP and NIP

SEM images of NIP, Cd(II)-IIP and CdP are shown in Fig. 1. Here, NIP indicated rigid and smooth surface as shown in Fig. 1(a) while Cd(II)-IIP as in 1(b) showed flaky surface because of imprinted sites. CdP as mentioned in Fig. 1(c) was rough due to the presence of Cd(II) on surface and matrix.

Elemental composition of CdP and Cd(II)-IIP were determined using EDAX as given in Table 1 confirm the

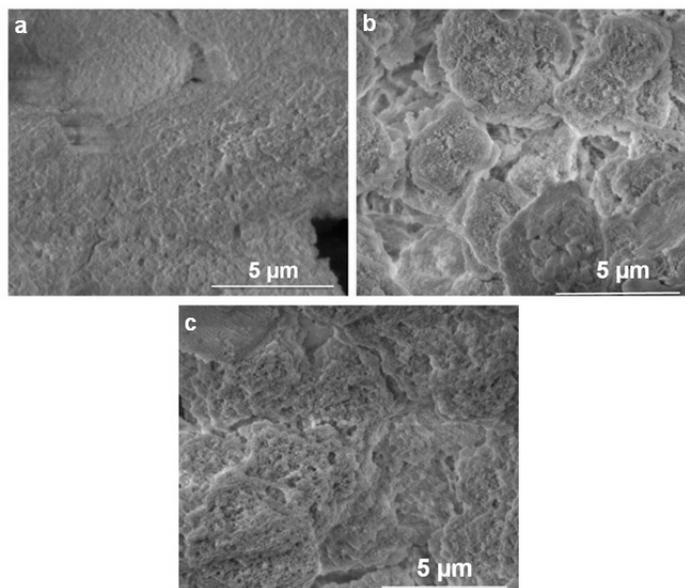


Figure 1. SEM image of (a) NIP (b) Cd(II)-IIP (c) CdP.

Table 1. Elemental composition analysis from EDAX: CdP and Cd(II)-IIP

Element	CdP (Wt %)	Cd(II)-IIP (Wt %)	CdP (At %)	Cd(II)-IIP (At %)
C K	57.92	77.97	63.78	81.92
N K	20.41	06.30	19.27	05.68
O K	19.93	15.73	16.48	12.41
Cl K	01.04		00.39	
Cd L	00.70		00.08	

complete removal of Cd(II) from the imprinted polymer. EDAX data for CdP as shown in Table 1 indicated that it was having Cd(II) while Cd(II)-IIP was free from Cd(II).

Thermogravimetric studies were also carried out to examine thermal stability of different polymers. Thermogram of Cd(II)-IIP in Fig. 2(c) showed similarity with the thermogram of NIP in Fig. 2(b) except that, it gave less char yield than NIP for the same temperature. Fig. 2(a) for CdP showed the different pattern than Cd(II)-IIP and NIP because of the presence of cadmium in polymer matrix. Therefore, thermogram of CdP had highest residue. The initial weight loss exhibited by Cd-IIP and NIP polymers were 3-4 per cent approximately at 205 °C, which clearly enumerated that polymers were stable up to this temperature.

BET surface area of Cd(II)-IIP, CdP and NIP were determined by N<sub>2</sub> adsorption desorption isotherms at liquid N<sub>2</sub> temperature, i.e., 77 K using SAA. Cd(II)-IIP got higher surface area (68 m<sup>2</sup>/g) compared to NIP (24.6 m<sup>2</sup>/g) and CdP (17 m<sup>2</sup>/g) due to Cd(II) imprinting in the polymer surfaces. Less surface area of CdP in comparison to Cd(II)-IIP and NIP was due to presence of Cd(II) in polymer which blocked the pores of polymer. Cd(II)-IIP and NIP were found to have 0.12 cm<sup>3</sup>/g and 0.01 cm<sup>3</sup>/g pore volume respectively.

FTIR study was used to establish the mechanism of

interaction in between carbonyl group of amide in acrylamide moiety and cadmium. Carbonyl group of acrylamide was observed at frequency 1673 cm<sup>-1</sup> while in CdP at 1668 cm<sup>-1</sup>. The shift of carbonyl frequency to the lower side confirmed the coordination of Cd with oxygen of carbonyl<sup>18</sup>. The IR of the

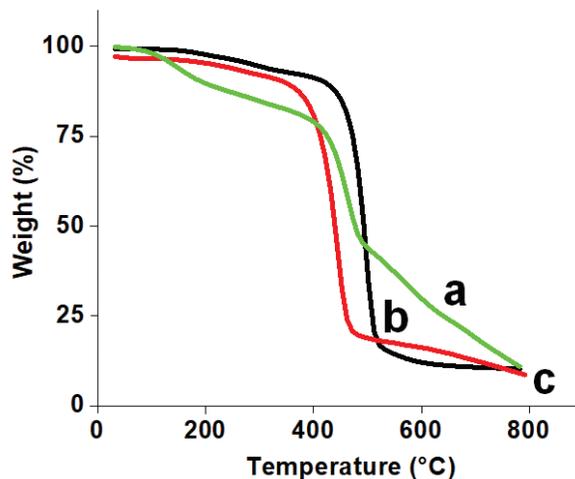


Figure 2. TGA curve for (a) CdP (b) NIP (c) Cd(II)-IIP.

washed Cd(P), i.e. after removal of Cd indicated the presence of carbonyl frequency at 1698 cm<sup>-1</sup> (Cd(II)-IIP), however, in NIP it was found to be at 1676 cm<sup>-1</sup>. This confirmed the complexation for making CdP, which is different than the NIP even after removal of cadmium as the carbonyl is observed in Cd(II)-IIP and NIP at different frequencies.

### 3.2 Recognition of Cd(II) by Cd(II)-IIP

Recognition study of Cd(II) from aqueous solution was performed using Cd(II)-IIP and found that Cd(II) was bound more with imprinted polymer because of the presence of cadmium imprinted sites than NIP. As mentioned in experimental section, 10 μg/mL Cd(II) solution (10 mL) was stirred with 0.05 g Cd(II)-IIP for 4 h to equilibrate with the material. The solution was filtered using Whatman filter paper No. 1, and then evaluated by AAS to determine the remaining concentrations of cadmium in solution. Generated data showed that uptake of Cd(II) was 9.4 μg/mL for Cd(II)-IIP while it was 6.0 μg/mL for NIP. It can be described due to the presence of imprinted sites in Cd (II)-IIP in which Cd(II) ions fit properly than NIP. The imprinting efficiency ( $\alpha$ ) of Cd(II)-IIP was calculated to be 1.6 and this is in agreement with earlier study<sup>13</sup>.

### 3.3 Interference Study for Cd(II)-IIP

Interference study with closely related metal ions such as Mn(II), Zn(II), Pb(II) and Hg(II) was also carried out in separate and in binary mixture solutions using Cd(II)-IIP. 1 per cent, 50 per cent, 53 per cent and 95 per cent removal of Mn(II), Zn(II), Pb(II) and Hg(II), respectively were achieved from their respective solution using Cd(II)-IIP. While in binary mixture of solution such as Mn(II)+Cd(II), Zn(II)+Cd(II), Pb(II)+Cd(II) and Hg(II)+Cd(II), cadmium competed with other ions in binding preference.

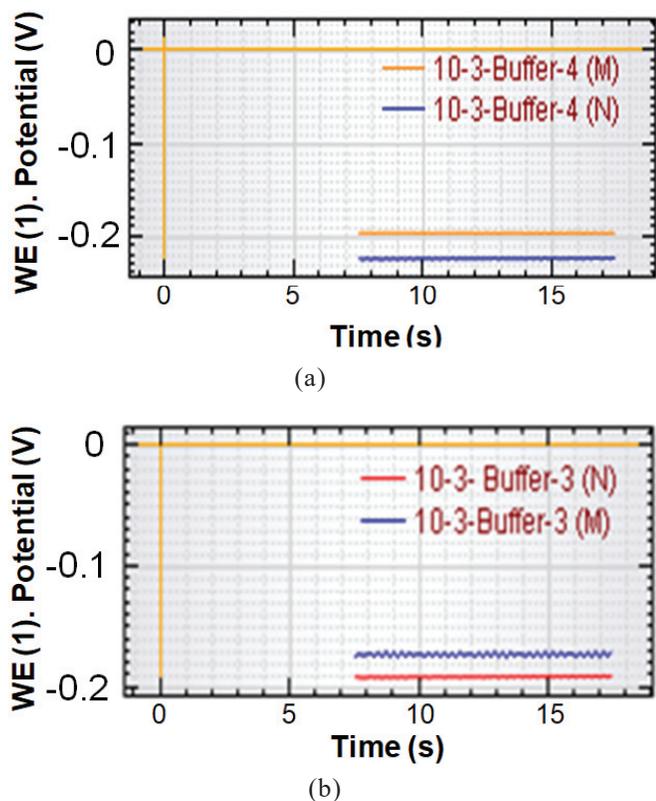


Figure 3. Effect of buffer pH 4 (a) and pH 3 (b) on sensing of cadmium ion by using Cd(II)-IIP and NIP.

### 3.4 Cd(II) Ion Sensing Using Cd(II)-IIP-ISE

Ion selective electrode (ISE) for cadmium sensing was explored and also compared with NIP-ISE. McIlvaine's buffer was used as described in section 2.5 which cover the pH range from 3.0 to 7.0. Chronopotentiometric study also revealed that buffer with pH 3.0 was suitable as indicated in Fig. 3. While, the buffer less than 3 pH gave less potential and more than 7 pH precipitated the metal ions in solution. Cd(II)-IIP-ISE always gave high potential because of maximum binding with Cd(II) from solution than NIP-ISE. An increment in potential was observed as increased the concentration of cadmium in solution ( $10^{-7}$  to  $10^{-3}$  M) as shown in Fig. 4.

Moreover, interference study was also carried out using ISE and found free from interferences and Cd(II) gave higher line of potential in comparison to Mn(II), Zn(II), Pb(II) and Hg(II) solution.

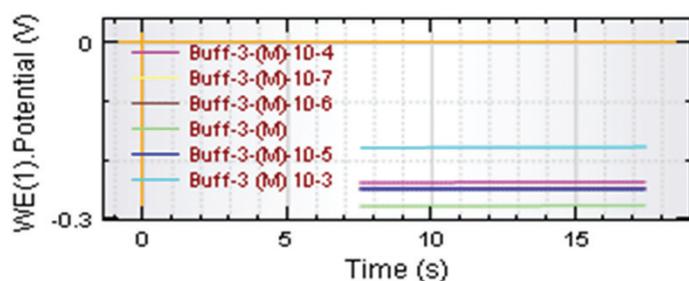


Figure 4. Effect of varying Cd(II) concentration ( $10^{-7}$  M to  $10^{-3}$  M) on Cd(II)-IIP.

## 4. CONCLUSIONS

Cd(II)-IIP was synthesised without a complexing ligand and compared with NIP. Recognition study of Cd(II)-IIP for cadmium ion showed its selective property with imprinting efficiency of 1.6. This behaviour of Cd(II)-IIP was due to the presence of imprinted sites which made the material spongy and floppy as presented by SEM image. High BET surface area of Cd(II)-IIP proved that it was more porous than NIP. FTIR characterisation of Cd(II)-IIP and cadmium complexed Cd(II)-IIP clearly indicated the bonding with metal through carbonyl oxygen in acrylamide moiety. Ion sensing study of Cd(II) imprinted polymer in the form of ISE showed more potential for Cd(II) than NIP in McIlvaine buffer of pH 3.0, at this pH, the potential response was very high. Chrono-potentiometry also enumerated that as the concentration of Cd(II) in solution increased the potential around the membrane also increased.

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