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# Synthesis of a Mixed Cellulose Ether-Ester for the Removal of Pb(II) from Aqueous Solution

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## Abstract

A sorbent (HEC-Adip-Na) was prepared by the saponification of hydroxyethylcellulose (HEC) and adipic anhydride and then saponification with $NaHCO_3$ to remove the Pb(II) from aqueous solution. The sorption experiments were done in batches and it was found that the uptake of targeted metal ions was mainly reliant on pH, sorbent dose, metal ions concentration, contact

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time and temperature. The highest Pb(II) removal was took place at pH 5.5, sorbent dose 50 mg, metal ions concentration 80 mg/L, contact time 30 min, and temperature 298 K.

***Keywords:*** Chemisorption, Esterification, Hydroxyethylcellulose, Sorbent dose, Temperature

## 1. Introduction

The heavy metals that pollute water have been spreading day by day all over the globe as a result of industrial revolution, urbanization, human activities *etc*., and hence disorder the health structure of the living organisms [1]. Divalent lead Pb(II) is one of the heavy metals that have numerous commercial applications especially in making corrosion free alloys and battery precursors. In addition to this, their usage is also in the production of commercial and household items (Word Health Organization (WHO) established a standard of the maximum limit of this heavy metals in drinking water. Above this level, the lead is toxic to living organisms [2]. Therefore, Pb(II) is supposed to ought to be under control use in order to balance the health.

According to literature, there are a number of means in which Pb(II) can be removed out of polluted water. The latter are such as coagulation-flocculation, membrane filtration, solvent extraction, centrifugation, reverse osmosis, electrochemical processes, adsorption, *etc*. [3, 4]. However, these are not an economical method and carries other risks associated with synthetic sorbents. Still in mind the value of methodology, cost effectiveness, health gains, recontaminations, and efficiency of sorbents, it is thus necessary to come up with more efficient sorbents that are less hazardous to health and generable nature [5-24].

Hydroxyethylcellulose (HEC) is a cellulose derivative which is soluble in water. It is widely applied as stabilizer in soaps, thickener in industry, pharmaceutical excipient binder and biocompatible. Literature has indicated that HEC can be chemically functionalized with succinic anhydride and made water insoluble. Thus, HEC chemically modified to form HEC-succinate sodium salt and HEC-adipate sodium salt (HEC-Adip-Na) was found as a supersorbent to take up cadmium ions from aqueous solutions [25].

It is against this backdrop that we have proposed to study the performance of the HEC-Adip-Na to adsorb Pb(II) from aqueous solution, depending on pH, sorbent dose, starting concentration of metal ions, contact time and temperature.

## 2. Materials and methods

### 2.1. Materials

Hydroxyethylcellulose (Natrosol, HE10K, Belgium) was bought locally. The adipic anhydride was Sigma-Aldrich, USA. N,N-dimethylacetamide, 4-dimethylaminopyridine, NaOH, HCl, $HNO_3$, $NaHCO_3$ were purchased at Alfa Aesar, Germany. The rest of the reagents and chemicals were of Riedel-de-Haen, Germany. The glassware was washed with deionized water (DW) and batch sorption experiments were carried out.

### 2.2. Synthesis of sorbent

The HEC was dried in an oven at 110°C and allowed to dry up to a time of 5 h. The obtained dried polymer was transformed into a biosorbent, *i.e.*, HEC-Adip-Na. The initial step entailed the addition of 10 g (36 mmol) of HEC in a solvent *N*,*N*-dimethylacetamide (DMAc) after 120 min of stirring at 80°C. The mixture was added to the obtained clear solution with adipic anhydride (AAn, 59.6 g, 216 mmol) and the reaction was further proceeded during another 18

h. The amount of 4-dimethylaminopyridine (DMAP, 100mg catalytically) was also included. The content of flask was removed, allowed to cool at 298 K, and precipitated with diethyl ether (250 mL). The precipitates were filtered and washed with diethyl ether once more to decrease the unreacted portions of adipic acid or adipic anhydride (in case of occurrence). This was dried in a vacuum oven at 50°C. The dried HEC-Adip was stirred indicating 298 K with a saturated aq. $NaHCO_3$ for 3 h. The mix was then left to settle, filtered, dried and stored.

**2.3. Sorption experiments**

Sorption experiments were conducted with the aim of removing Pb(II) in aqueous solutions. To that end, experiments were conducted in batches and the effects of different factors were researched. Before proceeding to study the impact of operational factors, standard aqueous solutions (1000mg/L, 500 mL) were prepared through shaking reagent $PbCl_2.2H_2O$ in DW individually. Solutions were stirred and filtered after 60 min in order to homogenize the mixture. Where necessary, dilutions were done. Standard acidic (1.0 M HCl) and basic (1.0 M NaOH) solutions were made and adjusted in destined range to the pH of all the tested Pb(II) solutions respectively. A certain volume of sorbent HEC-Adip-Na (50 mg) was measured and placed in the conical flasks with solutions of the tested metals, *i.e.*, 50 ml of solutions and then shaken in incubator to mix properly. The solutions were then stirred with 30 min 298K and filtered. To find the concentration of the metals loaded onto HEC-Adip-Na and left behind in the aqueous solution in the term of equilibrium sorption capacity (qe), percentage uptake (%)

respectively, the filtrate of Pb(II) based solutions were centrifuged and irradiated simultaneously by FAAS, then in terms of the mass of sorbent taken (mg) (Eqs. 1 and 2):

$$q_e = \frac{C_i - C_e}{m} \times V \quad (1)$$

$$Percentage\ uptake = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

**2.3.1. Effect of pH**

A suspension of 50 mg of HEC-Adip-Na in 100 mL of 80 mg/L of Pb(II) was prepared and influence of pH was examined by choosing initial pH of 1-7 of all the metals under test. The other conditions/procedure of sorption were kept to their optimum, *i.e.*, temperature 298K, 200 rpm of agitation speed, and 30 min contact time.

**2.3.2. Response to first concentration.**

The concentration effect of targeted metals was observed by placing an optimal concentration of HEC-Adip-Na (50 mg) into solutions at 298K temperature, 200 rpm speed, 5.5 pH in the case of Pb(II) with varying concentration of the desired metals concentration, *i.e.*, 40-160mg/L of Pb(II). The Pb(II) was determined on FAAS.

**2.3.3. Effect of sorbent dosage**

This was taken advantage of to maximize the lowest dosage of HEC-Adip-Na which was needed to extract the maximized portion of Pb(II) in aqueous effluents. In this case, 100 mL of DW water was taken in pre-cleaned/washed conical flasks. The doses of HEC-Adip-Na was added in various amounts, *i.e.*, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 mg. All other sorption parameters, including concentration = 80 mg/L, pH = 5.5, and temperature = 298 K, agitating time = 30 min, and speed = 200 rpm were kept and values measured on FAAS.

### 2.3.4. Effect of contact time

The experiments were carried out in order to examine the optimal contact period between sorbent and sorbate and uptake-kinetics of Pb(II) on to HEC-Adip-Na. To this end, sorbent (50 mg) and a metal based-aqueous solution (80 mL) mixture was prepared and left to mix in a continuous form in a shaking incubator. The shaking duration was preset (5-120 min). Metal ions concentration, pH, temperature, and agitation rate kept constant as in the experiments described above.

### 2.3.5. Effect of temperature

It is highly important to observe the nature of sorption process by studying thermodynamic of sorption phenomenon. To this end, solutions were made and executed on FAAS similarly described above. Nevertheless, temperature was varied between 298 K and 338 K and sorption capacities calculated at point of equilibrium using Eqs. (1 and 2).

## 3. Results and discussion

### 3.1. Effect of pH

Sorbent are not identical at various pHs of aqueous solutions to respond against heavy metal ions. Thus, the pH effect was researched to trace the aptitude of HEC-Adip-Na to Pb(II) uptake in pH 2-7. It was noted that the metals that were adsorbed to HEC-Adip-Na faster at acid pH i.e. less than or at pH up to 5.5. But the sorption rate was slow as far as pH was up to 3.0 and then rapidly increased to 5.5 with Pb(II). Subsequently pHs, there occurred a decrease in the removal of said metal ions according to the results shown in Fig. 1. This could be attributed to the fact that at lower pHs the availability of large numbers of protons H(I) in aqueous solutions protonated the sodic functionality of HEC-Adip-Na as a result of exchange of H(I) ions with Na(I) ions and re-deprotonated it back into the carboxylic acid groups, i.e., basic pH carboxylic acid groups of HEC-Adip that has low sorption affinity because of fewer number of exchangeable sites whereas at higher pHs, *i.e.*, basic Conversely, since the pHZPC of this novel sorbent was determined as 5.5, thus at pH above $pH_{ZPC}$ it carries negative charge on its surface and provides stronger attraction to positively charged metal ions.

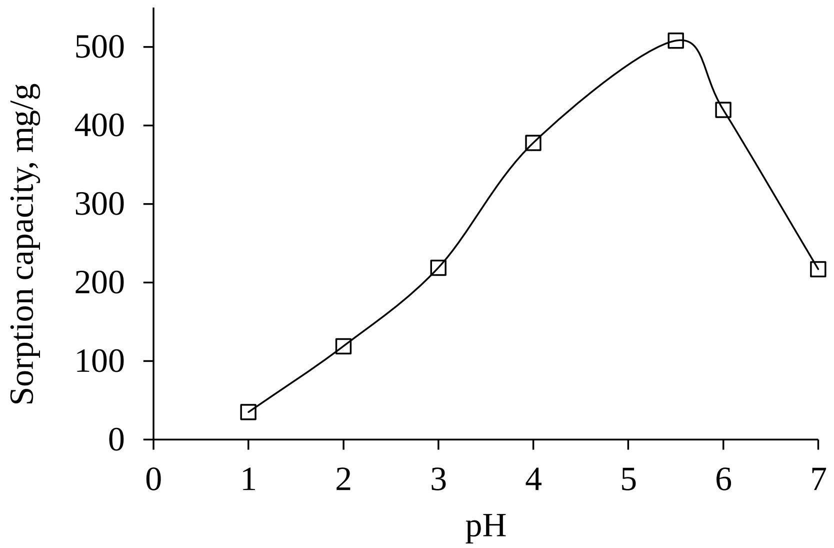


**Fig. 1.** Effect of pH

**3.2. Initial concentration effect**

Initial concentration of Pb(II) ions was examined to determine the potential of sorption of HEC-Adip-Na. Sorption capacities (qe, mg/g) versus initial metal ion concentration (Ci, mg/L) were plotted (Fig. 2). Clearly, based on the graph, HEC-Adip-Na absorb more and more metal ions in aqueous solutions with the increase of concentration of Pb(II) from 40-80. At the concentration of 80mg/L sorption equilibrium was achieved (Fig. 3). The rationale of this trend is that in the initial stage of sorption process, all sites of the surface of sorbent were naked and accessible to uptake of sorbed metal ions and therefore more uptake occurred at the beginning. Nonetheless, as more metal ions were added into the aqueous solution, the positions of the sorbent on the surface of the sorbent were occupied by these metal ions and very small proportions of active sites were remaining to allow the attachment of additional metal ions. Therefore, the sorption rate became persistent [26].

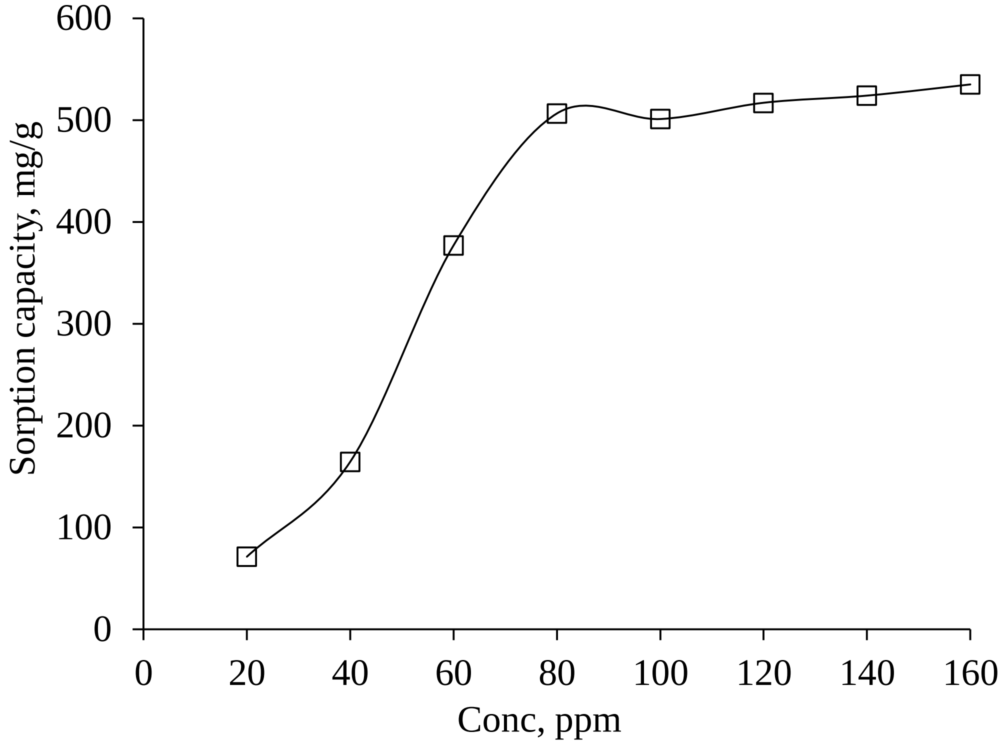


**Fig. 2.** Effect of metal ion concentration

### 3.3. Effect of sorbent dosage

The quantity of sorbent in aqueous solution also has a significant influence on the sorption process by modifying the quantity of the surface area of sorbent. Therefore, to explore the influence of sorbent dosage on Pb(II) removal, we conducted the experiment using 30, 40, 50, 60, 70, 80, 90, and 100 mg of HEC-Adip-Na to demonstrate that it is considered that as the dosage of the sorbent increases between 30-50 mg, the Pb(II) are quickly removed and at 50 mg of sorbent maximum quantities of the two metals The growth in the sorption capacities is attributed to the growth in surface area. However, at an optimal concentration, *i.e.*, 50 mg of sorbent, the reduction of sorption capacities could have taken place because of the saturation of metal pieces on the sorbent surface and hence decreased the amount of exchangeable sites on the surface of the sorbent [27, 28].

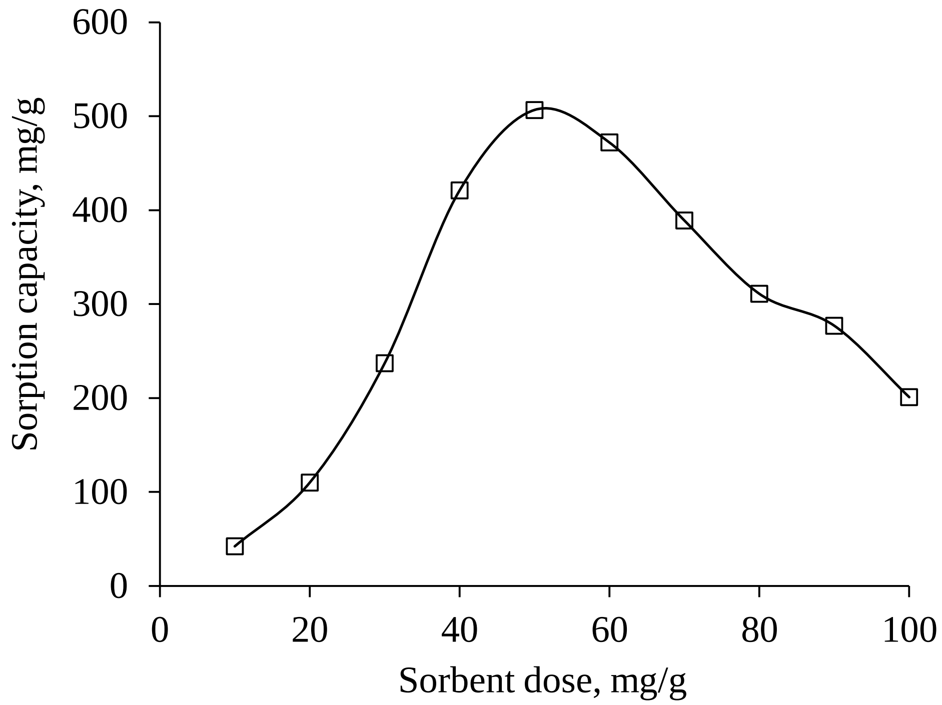


**Fig. 3.** Effect of sorbent dose

### 3.4. Effect of contact time

To identify the equilibrium time at which sorbent will be able to absorb maximum metal ions from aqueous environment the sorption capacities were studied with respect to time of contact. To this end, sorption tests were conducted by varying time of contact (5-120 min) between sorbent and sorbate and all the other conditions were kept at pre-optimized level. It was also noted that initially the rate of sorption process of metals was rapid and this is because there were more adipate functionality conjugated with sodium on the surface of HEC-Adip-Na. Therefore, there are increased opportunities of Pb(II) ion exchange with Na(I) ions because of the presence of more adipate moieties than because of the increased uptake of the metal cations. Although beyond a certain optimum the slope of curves became close to being parallel to the x-axis denoting time indicating that the maximum quantity of Pb(II) has been eliminated by HEC-Adip-Na on aqueous solution in the first 30 min of sorption process (Fig. 4).

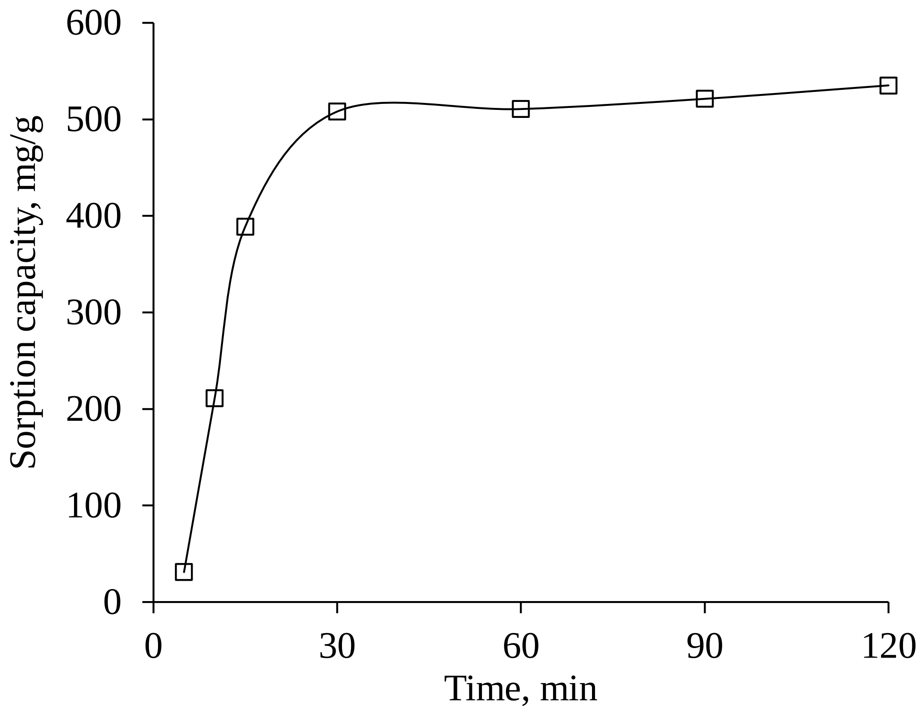


**Fig. 4.** Effect of interaction time

### 3.5. Effect of temperature

Temperature dependence of both spontaneous and non-spontaneous mode and thermodynamics of Pb(II) sorption on HEC-Adip-Na, *i.e.*, 298 K to 338 K with pH of 5.5 using a dosage of 50 mg of sorbent, Pb(III) at 80 mg/L was studied on a time of 30 min and results (Fig. 5) optimal sorbing temperature at 298 K was used. This temperature was therefore chosen to carry out all other experiments of sorption in batches. Such a reduction in sorption capacity with rise in temperature may be because of the decreasing of sorbent-sorbate forces of interactions that may be caused by subsequent increase in the mobility of metal ions of sorbate with rising temperature.

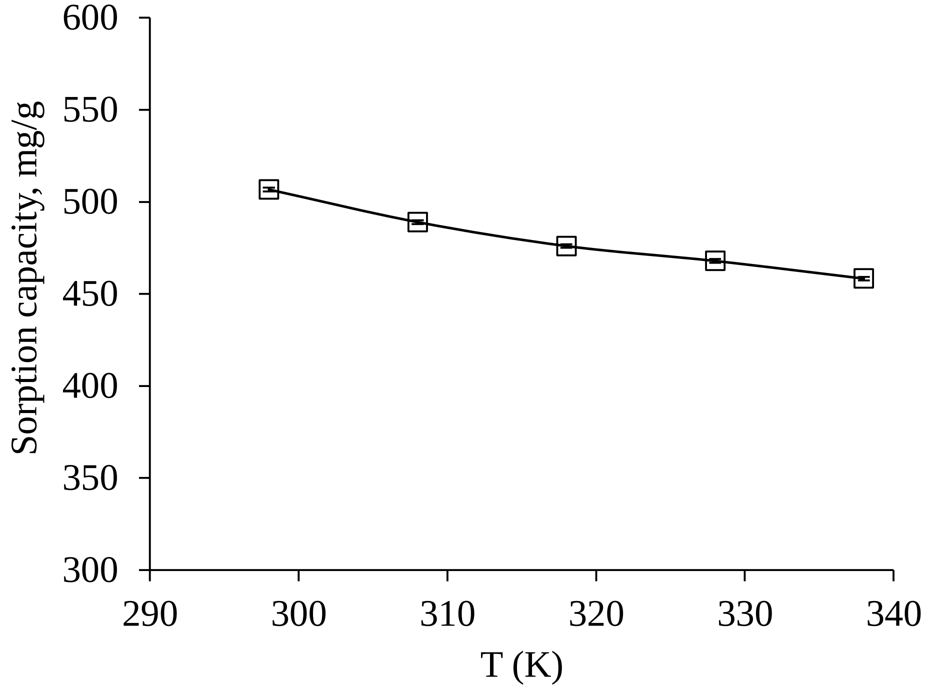


**Fig. 5.** Effect of temperature

## 4. Conclusion

Super selective HEC-adip-Na was made and used in uptake of Pb(II) in an aqueous solution. Optimum removal has been achieved under the following conditions: maximum pH of 5.5, sorbent dose 50 mg, starting concentration of metal ions = 80 mg/L, contact time 30 min and temperature 298 K. Morally, HEC-Adip-Na may be applied as safe and excellent sorbent to desalinate water.

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