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CHARACTERISATION AND PREPARATION OF ADSORBENT FROM *MERETRIX CASTA* SEASHELLS FOR THE REMOVAL OF LEAD FROM AQUOEUS SOLUTION

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ABSTRACT

The present study aims to investigate the adsorption efficiency of seashell powder derived particularly from *Meretrix casta* species on the removal of lead from synthetic wastewater under laboratory conditions. The collected seashells were thoroughly cleaned with tap water followed by deionized water to remove dirt and any contaminants attached to it. It was then ground and sieved to obtain finer adsorbent particles. Physical characteristics of seashell powder were studied by SEM. The functional groups in the adsorbent were analysed using FTIR spectroscopy and compounds were analysed by XRD. A feasibility study was conducted to make sure the adsorption ability of seashell powder. The optimum values of pH and Contact time are identified experimentally.

Keywords used- adsorption, seashells, Meretrix casta, SEM, FTIR, XRD

I. INTRODUCTION

Pb(II) is one of those heavy metals which can cause a lot of problems towards a human being and environment. The major sources of lead pollution are the battery industry, auto exhaust, ammunition and the ceramic glass industries (Suleman Qaiser et al. 2008). Lead poisoning, which causes usually by long term exposure to lead, can cause severe damage to the inner organs such as kidney, liver, nervous and reproductive systems in human beings. High exposure can cause nephrotoxic effects and long term exposure can lead to bone damage (Sarifah Fauziah Syed Draman et al. 2015). The permissible level of lead in drinking water is 0.05 mg/L as per the Environmental Protection Agency (EPA) (Joseph T. Nwabanne et al. 2012), but that by Bureau of Indian Standards (BIS) is 0.01 mg/L (IS 10500:2012). It has been reported that adults absorb 5-15% of lead and retain about 5% of it. Presence of lead more than 0.5 - 0.8 μg /ml into blood can cause many abnormalities such as mental retardation, hepatitis, reduction in the production of haemoglobin (Achla Kaushal et al. 2017).

There are several methods available for the removal of lead from water, one which is precipitation. Lead precipitates usually in the form of lead hydroxide $Pb(OH)_2$ or lead carbonate ($PbCO_3$), and the amount of lead precipitated depends on the quantity of carbonate in the water or added to it, as well as the treatment pH. Lead-containing wastewater can be treated by hydroxyapatite/chitosan nanostructures. Solid-phase extraction of lead by covalent attachment of a lead-selective macrocyclic sequestering agent onto the surface of silica gel is also possible and which is a relatively new technique. Adsorption is a widely used simple and cheap method to remove lead (Sadiq Danladi Attahir et al. 2016). *Meretrix casta*, a bivalve mollusk, is a backwater clam species. These occur abundantly in estuaries and backwaters of the east and west coast of India. The study says about the adsorption of lead from aqueous solution by using *Meretrix casta* seashells as adsorbent without any pretreatment. It is very simple and cheap method of water purification.

II. MATERIALS AND METHODS

A. Preparation of Adsorbent

M. Casta clam shells, which are widely available at the coastal area of Karnataka were collected from NITK Beach (Surathkal, Mangalore) (Figure 1). The shells were thoroughly cleaned by washing with tap water followed by deionized water to eliminate dirt and any contaminants attached to it. The shells were then dried in a hot air oven at temperature of 100^oC for 24 hrs. followed by ground using a ball mill and sieved to 100µm. The very fine adsorbent powder was stored in an air tight container at room temperature.

Figure 1: Meretrix casta seashells collected

Synthetic metal ion solution was prepared for conducting the adsorption study on *Meretrix casta* seashell. 0.1598g of $Pb(NO_3)_2$ powder was measured exactly and mixed with distilled water and made it up to 1000ml using a standard volumetric flask for obtaining 100ppm std. lead stock solution. Sample solutions were prepared by diluting it into different concentrations. For pH adjustment 0.2 N HCl and NaOH solutions were used. All the chemicals were analytical grade.

B. Instrumentation

A ball mill was used to grind the seashells into powder form and sieved using a 100µm sized nylon sieve. FTIR spectra was recorded by a Bruker (Alpha) spectrometer. The surface characteristics and morphology of the adsorbent were analyzed by JOEL JSM 6060 SEM. The JOEL diffractometer was used for the XRD crystallographic analysis. The scanning was made between 2^o and 80^o in 2θ with a step size of 0.01^o and a count time of 0.5 s $step^{-1}$. The quantitative analysis of lead was done by GBC 932 FAAS (Flame Atomic Absorption Spectroscope).

C. Biosorption process

Metal ion solutions are prepared from 100ppm stock solution. For metal biosorption, batch experiments are conducted. A known amount of seashell powder is added to 100ml Pb(II) solution in a 300ml BOD bottle and pH is adjusted using 0.2N HCl and NaOH. The optimum range of speed used in rotary shaker for the stirring purpose is in the range of 140-300rpm. After the contact period, the samples were filtered using a 0.2µ syringe filter and filtrates were tested using an AAS to check the lead content after the adsorption process.

Maximum biosorption capacity (q) and removal percentage (%R) was calculated by using the following equations:

$$q = \frac{Co-Ce}{V} m$$

(1)

$$\%R = \frac{Co-Ce}{Co} 100$$

(2)

where q is the biosorption capacity of the biosorbent (mg/g); m is the weight of the biosorbent (g); V is the volume of solution (L); %R is the removal percentage; and Co and Ce (mg/L) are the initial and equilibrium concentrations of the biosorbates in the solution, respectively.

III. RESULTS AND DISCUSSION

A. X-Ray Diffraction studies

XRD gives the structure, composition and physical properties of the material. X-ray diffraction provides an easy way to distinguish between the two polymorphs of $CaCO_3$: Calcite or Aragonite. X-Ray Diffraction crystallographic analysis was done by Bruker(Alpha) Diffractometer. The scanning was made between 20^{o} and 80^{o} in 2θ with a step size of 20^{o} and a count time of 0.5 s step^{-1}. The figure 2 shows the polymorph present is Aragonite. Well defined peaks are observed, hence the material under investigation is crystalline (A. Basker et al. 2014). The Aragonite 2θ peaks are 26.2°, 27.2°, 33.1°, 36.2°, 37.9°, 38.4°, 42.9°, 45.8°, 50.2°, 52.5°, and 52.9° (Kai-Yin Chong et al. 2014).

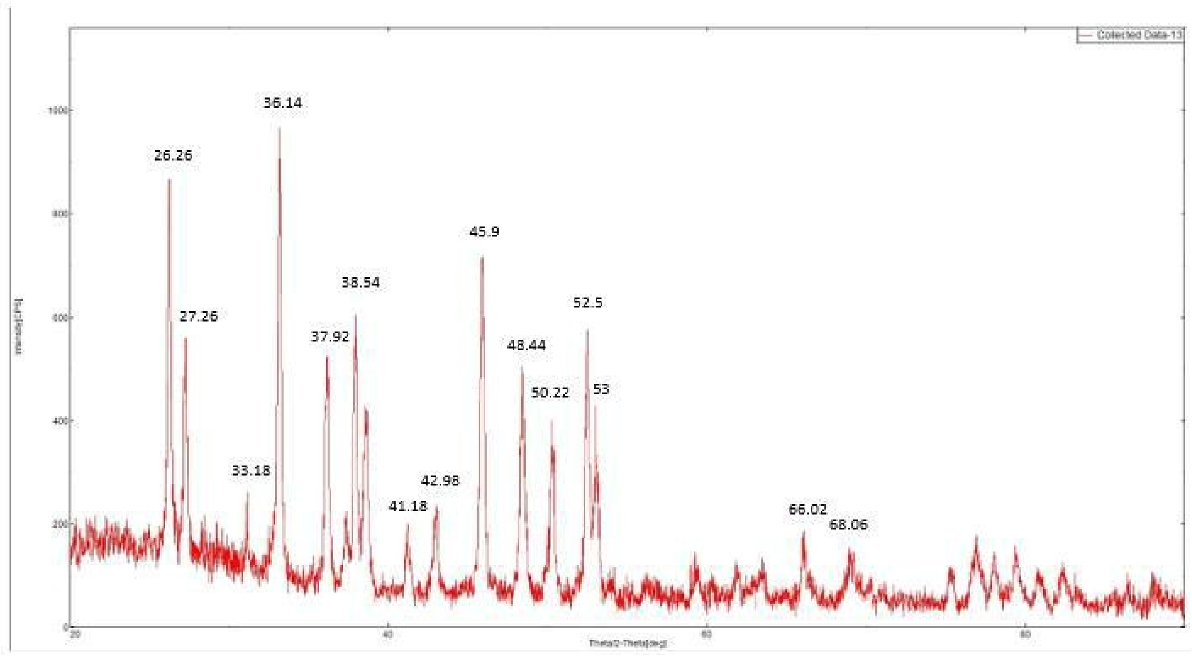

Figure 2: XRD result of the raw seashell

B. FTIR functional groups studies

Fourier-transform infrared spectroscopy (FTIR) is a technique used to obtain an infrared spectrum of absorption or emission of a solid. The FTIR spectral analysis is important to identify the characteristic functional groups on the surface of the adsorbent, which are responsible for adsorption and to decide if it is chemical or physical. IR analysis were performed by a Bruker (Alpha) spectrometer. The spectra over the range of 600-4000cm-1 were recorded by using the KBr pellets. The functional groups present according to the graph obtained are: Aragonite - 2522.43, 1487.81, 863.471, 712.569 cm-1, Calcite – 1785.28, 1082.83 cm-1, Hydroxyl (O-H) – 3268.27 cm-1, Carbonate group – 1644.98 cm-1, Phosphate (O-P-O) – 1082.83, 578.54 cm-1, Amine (N-H) – 3268.27 cm-1 (Daniek Suteu et al. 2011; Mike Masukume et al. 2014; Shamik Chowdhary et al. 2010). The

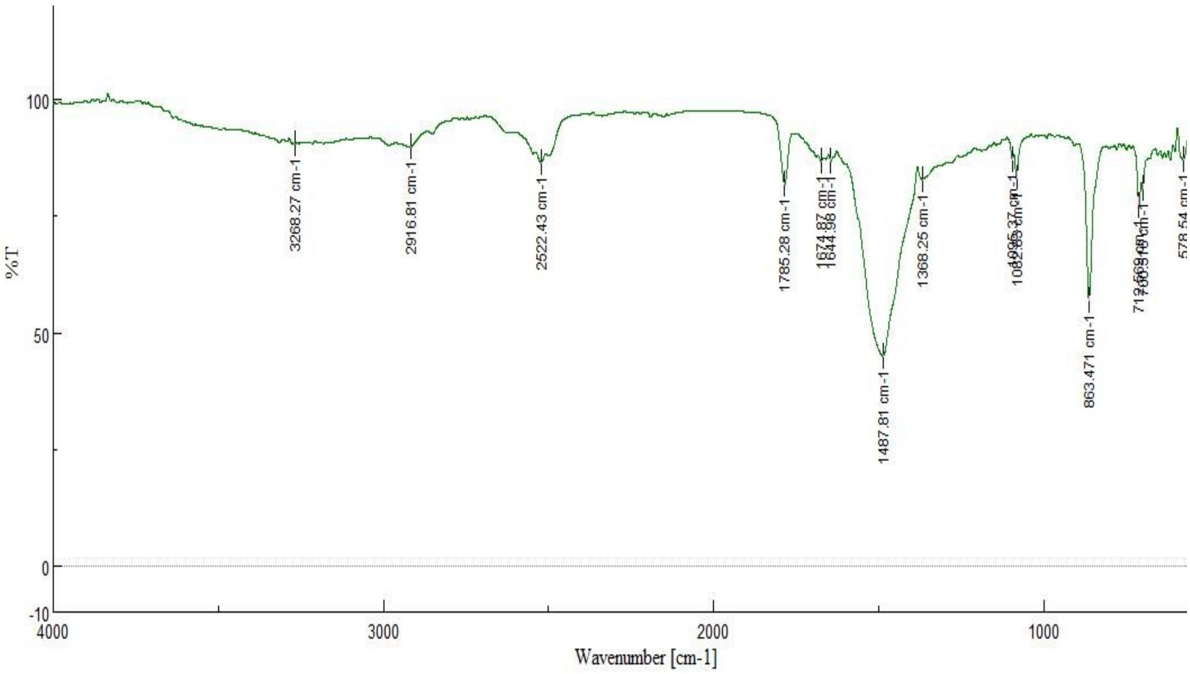

Figure 3: FTIR spectrum of raw seashell

C. SEM Morphology

The morphology and surface structure of the seashell powder before and after adsorption are analysed by Scanning Electron Microscopy.

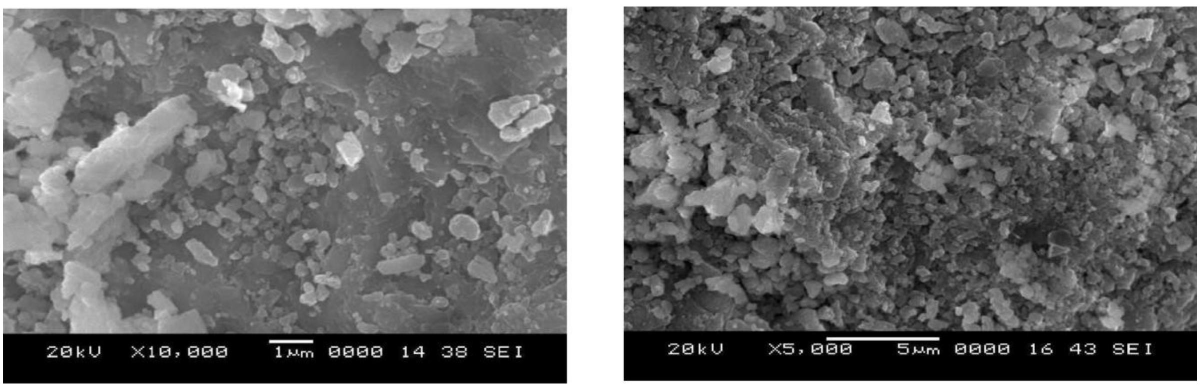

Figure 4: SEM images of seashell before (left) and after (right) adsorption

As obtained, the raw seashell powder has heterogeneous surface and many small granules attached to it, which provide greater surface area and enhance the adsorption process. The lead particles attached to the surface can be seen in the after adsorption picture (Figure 4).

D. Effect of pH on biosorption

pH is a very important parameter which can affect the mechanism of adsorption, charge of adsorbent, degree of ionization etc. Generally, the degree of adsorption will be lesser in the low pH ranges because of the protonation of biosorbent active sites in acidic media (Seda Karayünlü Bozbas et al. 2016). When pH increases the surface become more negative and it will encourage the sorption process. So it is supposed to increase the efficiency in higher pH ranges. But the solution precipitates at and above pH 6. Thus the desirable optimum value

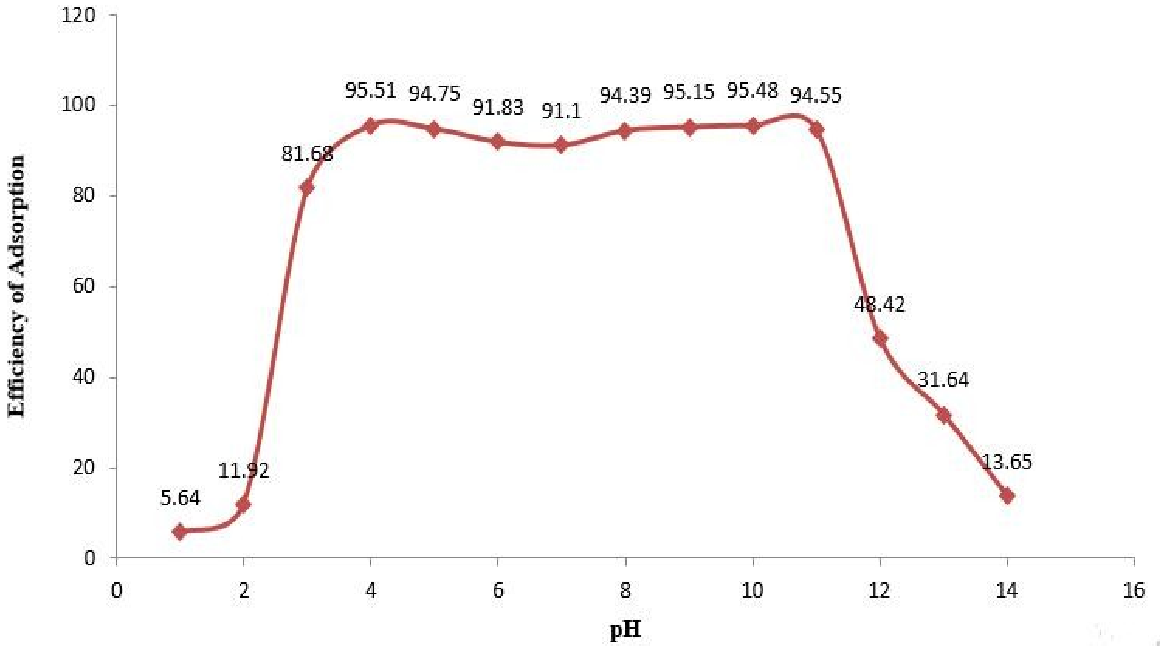

Figure 5: Variation of adsorption efficiency with the change in pH

of pH based on efficiency is fixed as 4. pH 1 to 14 solutions were adjusted by using 0.2N HCl and NaOH. Contact time, adsorbent dosage and adsorbate concentration were kept constant as 30min, 10mg and 10mg/l respectively. The results are presented in the Figure 5.

E. Effect of contact time on biosorption

The effect of contact time on adsorption efficiency is shown in the Figure 6. It can be seen that the biosorption efficiency increases with the increase in contact time initially. The maximum efficiency (100%) was obtained at

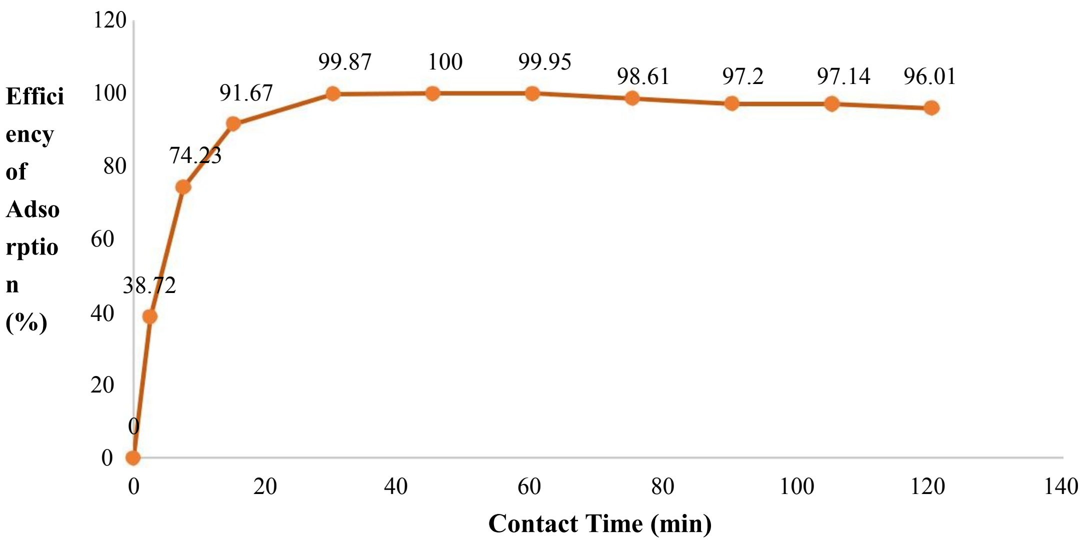

Figure 6: Variation of adsorption efficiency with the change in contact time

45 min. After that the lead content in the solution increases very minutely and so that the efficiency decreases. This can be due to the desorption process, as a small percentage of the lead might have attached through physisorption. For checking the effect of contact time, pH, adsorbent dosage and adsorbate concentration are fixed

as 4, 10mg and 10mg/l respectively. Contact times taken in to consideration are 2.5, 7.5, 15, 30, 45, 60, 75, 90, 105 and 120 min.

F. Effect of Adsorbate concentration to Adsorbent dosage ratio on biosorption

Adsorbate concentration to adsorbent dosage ratio (q) is taken as one of the main parameters to characterize the adsorption process. The experiments are carried out using different q values with pH and contact

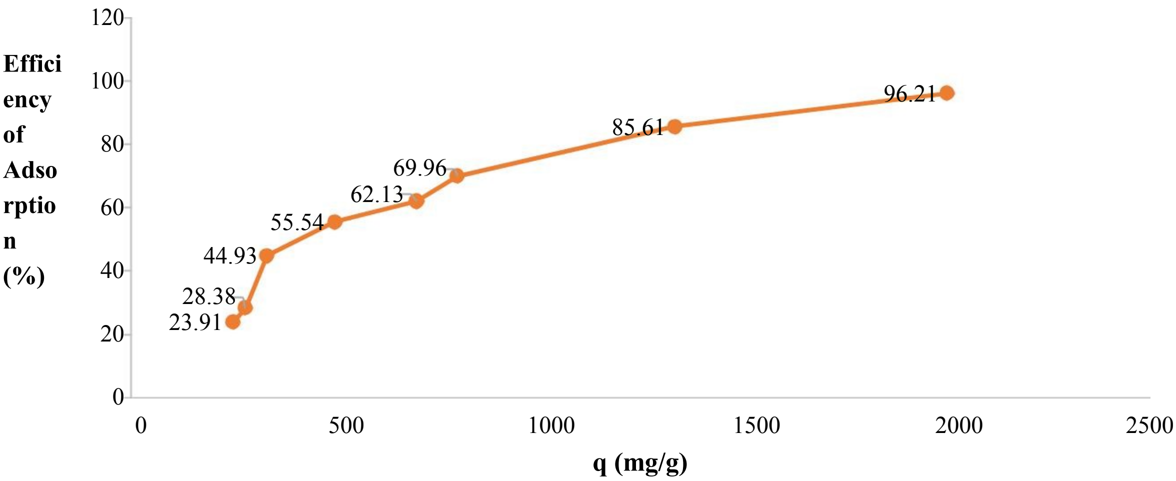

Figure 7: Variation of adsorption efficiency with the change in adsorbate concentration to adsorbent dosage ratio (q)

time kept constant as the optimum values obtained for both of them in the previous experiments. pH is taken as 4 and contact time as 45 min. q values considered are 250, 280, 333, 500, 700, 800, 1333 and 2000 mg/g. The results obtained are plotted in the Figure 7 graphically.

It is clear from the values that the maximum efficiency that can be achieved with optimum conditions is 96.21% at a *q* value of 2000. The efficiency is found to be increased with the increase in adsorbate to adsorbent ratio.

IV. CONCLUSION

The efficiency of adsorption of lead in aqueous solution by *Meretrix casta* seashells is found to be nearly 96%. Hence it is proved that seashell is a very effective adsorbant for lead. This type of seashells is abundantly available in Indian coastal regions and no activation is needed. So it is an easy and cost effective adsorption process and hence can be used in a very small scale in villages to large scale for water purification purpose. The optimum values obtained for greatest efficiency are pH 4, Contact time 45 min and Adsorbate concentration to Adsorbent dosage value 2000 mg/g.

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