

EVALUATION OF PB(II) REMOVAL FROM AQUEOUS MEDIUM BY BENTONITE AND ACTIVATED CHARCOAL

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ABSTRACT

Lead concentration in water and air may be attributed to geochemical reactions and anthropogenic activities too. The present paper discussed the strategy of removal of lead from aqueous medium using bentonite and activated charcoal. The batch experiments were done for different initial concentrations at different pH values and time intervals with a view to knowing the optimum condition. Bentonite was characterized by X-ray diffraction (XRD), Brunauer Emmett Teller (BET), Fourier transform infrared spectroscopy (FTIR), and Thermogravimetric analysis (TGA). A mixture of bentonite and activated charcoal in a ratio of 1:1 was also used for the adsorption of lead from an aqueous medium. The residual concentration of lead was determined using inductively coupled plasma-atomic emission spectroscopy (ICP-AES) at 220.353nm and 261.418nm. Results demonstrated that the residual concentration of lead was less than 2(μg/L) or 2ppb. The adsorptive removal can be attributed to high surface area of bentonite and cation exchange capacity of bentonite.

Keywords: Bentonite, Adsorption, Lead and Activated Charcoal, Surface Area.

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INTRODUCTION

Lead(II), Cadmium(II), and Chromium(VI) are the most common toxic metals that can cause kidney failure, depression, mental disorders, and even cancer after prolonged intake. Important anthropogenic sources of lead in water are dyes, pigments, wastewater, oil processing, and industries.^{1,2} According to WHO, the permissible limit of Pb(II) is 0.01 ppm above which its intake causes adverse health effects. Volcanic rocks have sulfide mineralization containing Pb, Cd, Hg, etc. Pb(II) is immobile on the surface and soil contains lead as well.³ Lead has four stable isotopes such as ²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb, and ²⁰⁸Pb obtained as the end product of all the decay series. Soil is the best sink of all these lead isotopes due to isotopic transfer.⁴ Chemical precipitation, ion exchange, reverse osmosis, and adsorption are a few mentioned techniques used for the removal of Pb(II) from an aqueous medium. Solid agriculture wastes such as maize-stem powder, and rice husk remove lead(II) from aqueous medium.⁵ Citrus nobilis peel, orange peel, oil cakes, and several medicinal plants also have the potential to remove Pb(II) from an aqueous medium. Cynodondactylon, aquatic weeds, herbs, and lemon grass remove heavy metals from aqueous medium.^{6,7,8} Amongst all the methods, adsorption has been used very frequently.^{9,10} Clay minerals such as bentonite have opened up a new class of adsorbents for heavy metals.^{11,12} Bentonite minerals characterized by XRD, FTIR, TGA, and BET, are hydrated alumina phyllosilicates with exchangeable cations.^{13,14,15} Owing to high cation exchange capacity and adsorption potential, bentonite mineral has emerged as an eco-friendly alternative for the removal of heavy metals, and arsenic also.^{16,17,18} Bentonite consisting of montmorillonite possesses high surface area, intercalation properties, and chemical properties of swelling, dispersion, bonding, plasticizing, and suspending.¹⁹ Replacement of ions such as Si⁴⁺ by Al³⁺ or of octahedral Al³⁺ or Fe²⁺ produces a deficit of positive charge and thus generates excess negative charge within the layer.²⁰ The negative charge is balanced by cations which are held on the surface of flakes giving rise to cation exchange capacity in the range of 70 to 100meq/100g of clay.²¹ The percentage of oxides of silica and alumina ranges from 45.32 to 61.46 and, 13.04 to 19.2 respectively with minor oxides of Ti, Be, W, Sn, Sr, and Zn present in traces.²² Removal of heavy metals may be attributed to the cation exchange capacity and large surface area of the bentonite mineral. Lead concentration may be known by the U.V. double-beam spectrophotometer. But here more latest technique of Induced coupled plasma-atomic emission spectroscopy (ICP-AES) has been

utilized for the most accurate results up to the ppb level. The residual concentration of lead has been determined at 220.353 nm and 261.418nm. Effects of initial concentration, pH, time interval, and adsorbent doses have been studied to know the adsorption process.

EXPERIMENTAL

Materials and Method

A bentonite sample from Barmer (Rajasthan) was ground into a 300 mesh sieve and a mixture with active charcoal was prepared in a ratio of 1:1. 100 ml of 5ppm Pb(II) solution was treated with 1g bentonite charcoal mixture up to 15, 30, 45, 60, and 90 minutes at a pH of 2, 4, and 6.23 respectively. The same experiment was repeated with an initial concentration of 10ppm Pb (II) solution. Different weights of biomass, time interval, and pH have been chosen for the study to find out the optimum condition of adsorption. Pb(II) concentration is determined from Induced coupled plasma-atomic emission spectroscopy (ICP-AES) model: (ULTIMA-2, Make: Horiba Jobin Yvon, France) at a wavelength of 220.353nm and 261.418nm respectively at ppb level. BC and B stand for bentonite-charcoal mixture and bentonite respectively. 10ppm and 5ppm Pb (II) solutions were prepared from a stock solution of 100ppm Pb (II) by dissolving Pb (NO₃)₂ in water. pH was adjusted by the addition of N/10 HCl and N/10 NaOH. Bentonite is characterized by TGA, DTA, FTIR, and XRD (D₈ Diffractometer). The BET surface was measured by N₂ adsorption on bentonite powder at an analysis bath temperature of -195.846°C by micromeritics 3 flex version 4.05 serial H 941 unit part 2.

RESULTS AND DISCUSSION

Analysis of Pb(II) done by ICP-AES showed that the residual concentration of Pb(II) after treatment with bentonite -charcoal mixture at 220.353nm was 0.004 ppm and 0.012 ppm at 261.418nm taking the initial concentration of 10ppm Pb(II) at a pH of 6.23. With an initial concentration of 5ppm Pb(II) solution, the residual concentration of Pb(II) at a pH of 2 is 0.003 ppm at 261.418nm (Table-1).

Table-1: Residual Concentrations of Pb(II) at Different Initial Concentrations and pH after Treatment with Bentonite-Charcoal Mixture (1:1)

Sample no.	Initial concentration	pH value	Time	Pb (µg/L) 220.353nm	Pb (µg/L) 261.418
BC1	10ppm	6.23	15 minutes	4	12
BC2	10ppm	6.23	30 minutes	< 2	< 2
BC3	10ppm	6.23	45 minutes	< 2	< 2
BC4	10ppm	6.23	60 minutes	< 2	< 2
BC5	10ppm	6.23	90 minutes	< 2	< 2
BC6	10ppm	4.00	15 minutes	< 2	< 2
BC7	10ppm	4.00	30 minutes	< 2	< 2
BC8	10ppm	4.00	45 minutes	< 2	< 2
BC9	10ppm	4.00	60 minutes	< 2	< 2
BC10	10ppm	4.00	90 minutes	< 2	< 2
BC11	10ppm	8.35	15 minutes	< 2	< 2
BC12	10ppm	8.35	30 minutes	< 2	< 2
BC13	10ppm	8.35	45 minutes	< 2	< 2
BC14	10ppm	8.35	60 minutes	< 2	< 2
BC15	10ppm	8.35	90 minutes	< 2	< 2
BC16	5ppm	8.35	15 minutes	< 2	< 2
BC17	5ppm	8.35	30 minutes	< 2	< 2
BC18	5ppm	8.35	45 minutes	< 2	< 2
BC19	5ppm	8.35	60 minutes	< 2	< 2
BC20	5ppm	8.35	90 minutes	< 2	< 2
BC21	5ppm	2.00	15 minutes	< 2	3
BC22	5ppm	2.00	30 minutes	< 2	< 2
BC23	5ppm	2.00	45 minutes	< 2	< 2
BC24	5ppm	2.00	60 minutes	< 2	< 2

BC25	5ppm	2.00	90 minutes	< 2	< 2
BC26	5ppm	8.09	15 minutes	< 2	< 2
BC27	5ppm	8.09	30 minutes	< 2	< 2
BC28	5ppm	8.09	45 minutes	< 2	< 2
BC29	5ppm	8.09	60 minutes	< 2	< 2
BC30	5ppm	8.09	90 minutes	< 2	< 2

Table-2: Residual Concentrations of Pb (II) after Treatment with Bentonite at Different Time Intervals

Sample no.	Initial concentration	pH value	Time	Pb ($\mu\text{g/L}$) 220.353nm	Pb ($\mu\text{g/L}$) 261.418nm
B1	5ppm	8.00	15 minutes	<2	<2
B2	5ppm	8.00	30 minutes	<2	<2
B3	5ppm	8.00	45 minutes	<2	<2
B4	5ppm	8.00	60 minutes	<2	<2
B5	5ppm	8.00	90 minutes	<2	4
B6	5ppm	2.00	15 minutes	<2	<2
B7	5ppm	2.00	30 minutes	<2	17
B8	5ppm	2.00	45 minutes	<2	<2
B9	5ppm	2.00	60 minutes	<2	<2
B10	5ppm	2.00	90 minutes	<2	<2

Characterization

The Barmer bentonite BB2 presented a total weight loss of 16.3817% between 100°C and 900°C. The first weight loss is attributed to hygroscopicity, the second loss to loss of water composition from the bentonite, and the third to dehydroxylation of the silicate lattice. Thus, a total weight loss of 16.3817% agrees with the standard value of DTA, and TGA for bentonite (Fig.-1a,b). The endothermic peak also characterizes bentonite.

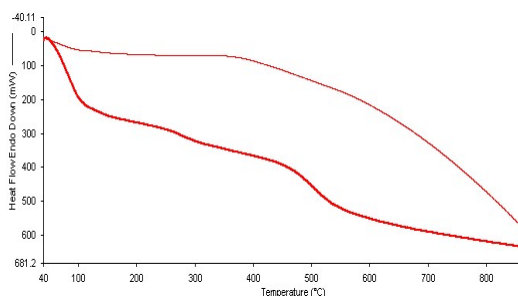


Fig.-1(a): DTA of Bentonite(BB2)

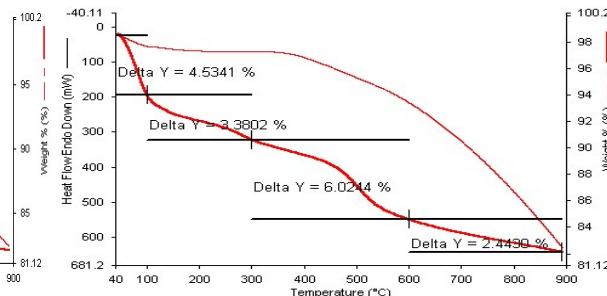


Fig.-1(b): TGA of Bentonite(BB2)

Fourier Transformed Infrared Spectroscopy (FTIR) shows the stretching frequency of -OH at 3620.34cm^{-1} and Si-O-Si linkage stretching vibration at 1635.67cm^{-1} (Fig.-2). The peak at 911.29cm^{-1} shows the Al-O bond.²³

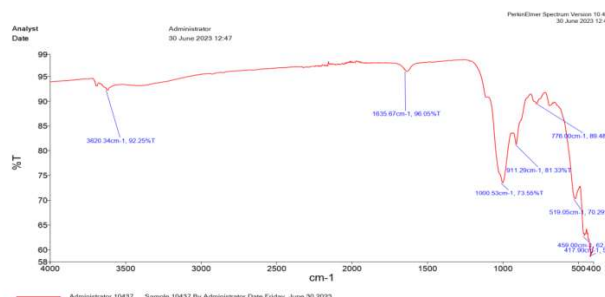


Fig.-2: FTIR of Bentonite

XRD analysis: The X-ray diffraction pattern of the bentonite sample has been shown in the figure indicating a maximum intensity peak at 26° 2 theta (degree). The XRD pattern confirms the presence of montmorillonite unit (Fig.-3)

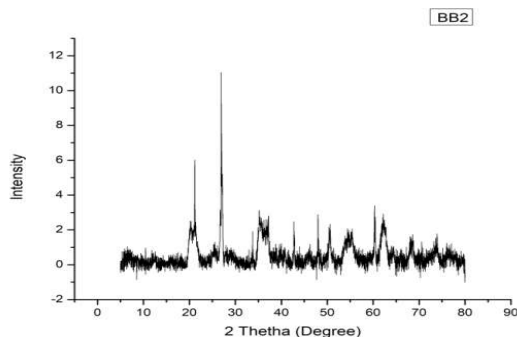


Fig.-3: XRD of Bentonite

Surface Area Analysis

The nitrogen adsorption method indicated a surface area of $157.7719 \text{ m}^2/\text{g}$ for BB2 bentonite. BJH adsorption surface area of BB2 is $146.5264 \text{ m}^2/\text{g}$. Adsorption pore diameter 4V/A by BET is $58.152 \text{ \AA}$ indicating the mesoporous nature of the powdered bentonite. Graph of V/V_m versus P/P_0 gives BET isotherm plot.

SEM Analysis

SEM analysis of bentonite and bentonite-charcoal mixture 1:1 analysis has been done to ascertain the surface morphology at different magnifications and wavelengths ranging from 1nm to 200nm. Figures 4(a and b) show the surface morphology of bentonite. The images before and after adsorption clearly confirmed the adsorption of lead on the surface (Fig.-5a,b, and c). Figures-6 and 7 show the EDAX of BC13 and BC21 respectively whereas Fig.-8 shows the EDAX of activated charcoal.

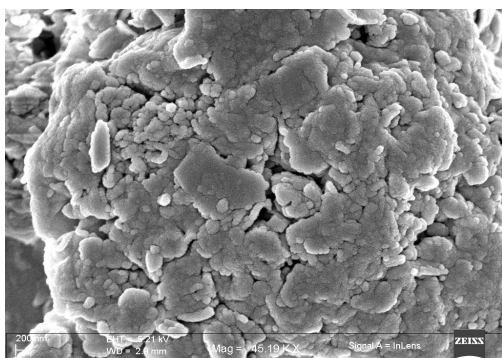


Fig.-4(a): SEM image of BB2 at 45.19KX

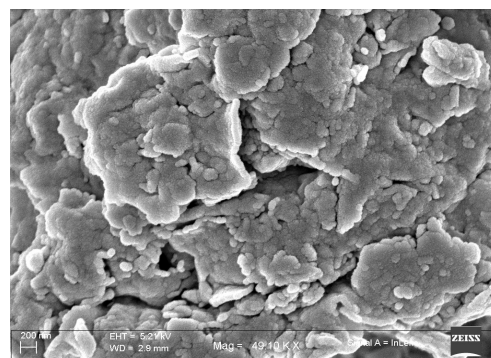


Fig.-4(b): SEM image of BB2 at 49.10KX



Fig.-5(a): SEM image of BC Mixture at 56.73 KX before Adsorption

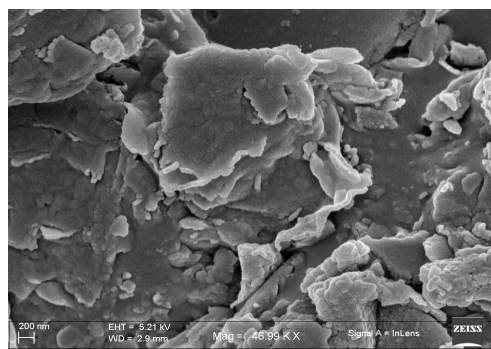


Fig.-5(b): SEM image of BC13 at 46.99KX after Adsorption

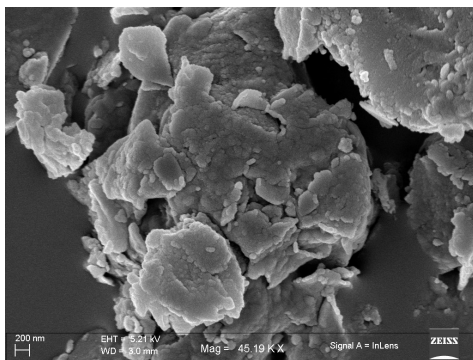


Fig.-5(c): SEM Image of BC21 at 45.19 KX after Adsorption

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 5

Standard :
C CaCO₃ 1-Jun-1999 12:00 AM
O SiO₂ 1-Jun-1999 12:00 AM
Al Al₂O₃ 1-Jun-1999 12:00 AM
Si SiO₂ 1-Jun-1999 12:00 AM

Elem... Weight% Atomic%

C K	72.48	79.46
O K	21.46	17.66
Al K	1.80	0.88
Si K	4.26	2.00

Totals 100.00

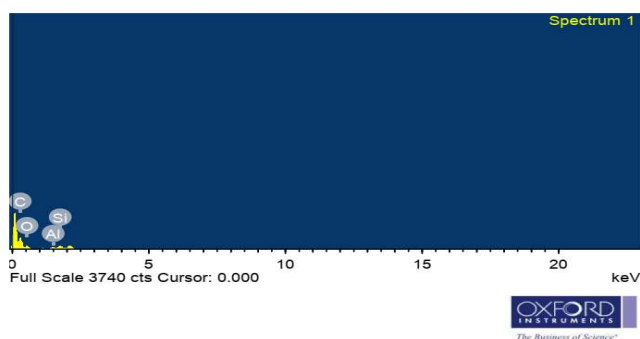


Fig.-6: EDAX of BC13

Spectrum processing :
Peak possibly omitted : 6.400 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard :
C CaCO₃ 1-Jun-1999 12:00 AM
O SiO₂ 1-Jun-1999 12:00 AM
Al Al₂O₃ 1-Jun-1999 12:00 AM
Si SiO₂ 1-Jun-1999 12:00 AM

Elem... Weight% Atomic%

C K	65.26	73.52
O K	26.65	22.54
Al K	2.61	1.31
Si K	5.48	2.64

Totals 100.00

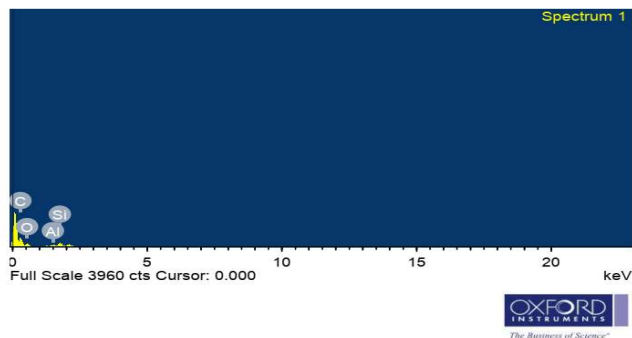


Fig.-7: EDAX of BC21

Spectrum processing :
Peak possibly omitted : 2.068 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

Standard :
C CaCO₃ 1-Jun-1999 12:00 AM

Elem... Weight% Atomic%

C K	100.00	100.00
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Totals 100.00

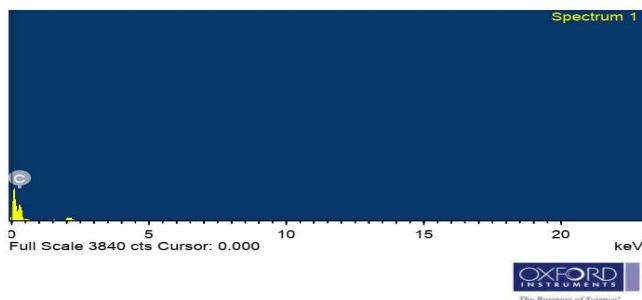


Fig.-8: EDAX of Activated Charcoal

CONCLUSION

Bentonite and bentonite charcoal mixture both have been established as an eco-friendly and low cost adsorbent of Pb(II) from aqueous medium.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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