Low Cost, Non-conventional Adsorbents Neem leaves powder (NPL), Peepal leaves powder (PPL), Amla leaves powder (APL) used for Pb Removal

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Abstract: An investigation on removal of Pb^{2+} from waste water by use of powders of non conventional absorbents like Neem leaves (NLP), peepal leaves (PLP) and Amla leaves (ALP) at different adsorbent/metal ion ratios, pH, contact time, metal ion concentration was investigated by using batch method. During the investigation it was observed that maximum amount of lead adsorption occurs at pH: 6.0., temperature $30°C \pm 2°C$ The equilibrium of lead adsorption was attained in about 60 min. The adsorption depends upon the availability of number of active sites. De-sorption probably took place at around $40°C \pm 2°C$ and at higher temperature values. Powders of Neem leaves were found to be most effective adsorbents followed by the powders of Amla leaves and Peepal leaves.

I. INTRODUCTION

Lead (Pb2+) is an extremely toxic metal found in small fractions in earth's crust with an atomic no. 82 and molar mass 207.2 g/mol [Gordon et al., 2007]. The level of lead in the earth's crust is about 20 mg/kg. As per an estimate the concentration of lead is 0.02 m g/litre on the free surface of naturally available water. [Gavaghan & Helen]. Lead is unsafe for human health and also for the ecosystem [Verstraeten et al., 2008]. The primary sources of lead contamination are paints, leaded crystal, cigarette smoke, soldering by use of lead, children's toys, vinyl lunch boxes etc.

As per a study conducted in US it was estimated that 38 million US households are using lead contaminated paints. About 65% of the inhabitants of these households were found to be suffering from lead based hazards. [Toscano & Guilarte,2005].
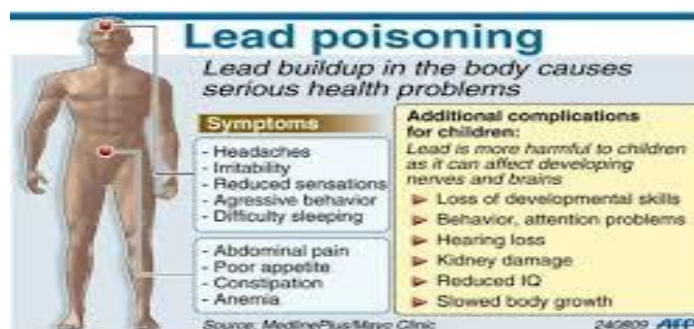
1.1Toxicity:

Lead causes toxic effect on human body and lead toxicity is known as plumbism. Impact of lead exposure in humans has been known to cause wide variety of health problems such as blood disorders and anaemia, it also causes rapid deterioration of nervous system. It also reduces fertility both in men and women. It may leads to failure of the kidney, Alzheimer disease. [Navas-Acien et al., 2007, Woodruff et al., 2008, Iqbal et al., 2008;Karthika et al., 2010].

1.2 Removal of toxic ions

The commonly used adsorbents of metal ions are coconut shells, tree bark, zeolites, lignin, mollusk shells, citrus peel, natural clay and microorganisms (bacteria, fungi and yeast) [O¨ ztu¨ rk, 2007; Guo et al., 2008;Wang and Peng, 2010; Kołodyn´ ska et al., 2012; Rao and Kashifuddin, 2012; Inagaki et al., 2010]. Lead affects several enzymatic processes involved in heme synthesis [Barbosa et al., 2005]

Lead may acts as a catalyst in Parkinson's disease and amyotrophic lateral sclerosis. It has been hypothesized that the lack of enzymes with hemoglobin may disrupt energy metabolism [Khan, A, 2008]. Environmental and domestic sources of Pb^{2+} are the most often seen causes for the above diseases, but with proper precautionary measures, it is possible to reduce the levels of risk associated with lead poisoning [Robert, 2008].

Today the main goal is to adopt appropriate methods and to develop suitable techniques and to investigate new substances either to prevent the pollution or to reduce it to low levels. Although the specific contaminants leading to pollution in water include a wide spectrum of chemicals but in present study, our aim is strictly focused on removal of toxic Pb^{2+}ions.

The objective of the present study is to develop a cost effective technology for the removal of cations. The conventional methods for the removal of the cations are: Evaporation [Singh and Chandel,2006], Ion exchange Membrane techniques [Qdias and Hassan, Moussa,2004], Reverse osmosis [Singh and Chandel, 2006], Electro-dialysis [Vasilyuk, S. L,2004]. Solvent based system [Borowiak-Resterna, 2010], Chemical precipitation [Benefield, L. D., Uluozlu, O. D, 2010].

Adsorption is the process which involves the transfer of a substance from a solution onto the surface of solid adsorbents by either chemical or physical attraction [Reynolds, T. D,1996] or more frequently by physical adsorptions which involves attraction due to electrical charge differences between the adsorbent and the adsorbate [Cheremishinoff,1978]. Chemical adsorption is the product of a reaction between adsorbent surface and the adsorbate. Adsorption involves transport of the soluble substances to the surface of the solid adsorbent, where the adsorbate then moves into the adsorbent pores

1.3 Low Cost Adsorbents

Low Cost Adsorbents like Azadirachta Indica (Neem leaves), Ficus Religiosa (Peepal leaves), Emblica Officinalis (Amla leaves) are used as adsorbents in this study**.** The above absorbent were studied and their potential to remove Pb^{2+} ions was investigated and the potential areas of applications are ultrafiltration, separation, environmental sciences and individual household water treatment plants.

II.   LITERATURE SURVEY

Industrial Pb^{2+} contaminated water acts as a potential hazard for the environment and it is the most important pollutant in surface and ground water [Brinza et al., 2009]. With the exponential increase in population, measures for controlling heavy metal ion emissions into the environment are essential [Mousavi et al., 2010 and Maximous, 2010] [M. Kafia and Surchi Shareef, 2011] have developed low cost adsorbents for Pb^{2+} from agricultural wastes like chalk powder, rice husk, sesame and sun flower plants residue and tea waste. Robert Brooks, 2010 has reported on removal of lead from contaminated water by using AAM-PRB (Alkali Ash Material Permeable Reactive Barrier. Pandhare et al, 2013 studied the effect of Neem leaves powder and activated carbon as an adsorbent. Gupta and Bhattacharya, 2005 used activated carbon developed from fertilizer waste for removal of $Pb^{2+,}$ Hg^{2+}, Cr^{4+}, and Cu^{2+}. Venkateswara Rao Surisetty et al., 2013 have investigated the aqueous solution on bio-sorption of lead. Pandhare Ghanshyam et al., 2013 studied on adsorption of lead from a stock solution using Neem leaves powder as adsorbent. Bhattacharyya Krishna and Arunima Sharma, 2004 has explored on Adsorption of Pb^{2} from aqueous solution by Azadirachta indica (Neem) leaf powder. Nagpal et al., 2010 in aqueous solutions has studied Equilibrium sorption studies for Pb2+ metal ions on three different biomasses obtained from the dried leaves of Terminalia Catappa, Dalbergia latifolia and Ficus benghalensis. Lovell Agwaramg et al., 2013 invested the process of removal of Lead from contaminated water using solid biomaterials: charcoal, coffee, tea, fishbone, and caffeine.

III. MATERIAL AND METHODS

3.1Preparation of Leaf Powder (NLP, PLP and ALP): leaves free from diseases were collected from trees. The leaves were washed with water to remove dust and soluble impurities and were allowed to dry at room temperature in shade. The 50 µm fractions of powdered leaves were separated and taken as the adsorbent. The powders after being dried for several hours at room temperature were preserved in glass bottles for use as adsorbents.

3.2 Method used for determination of Ions concentration:
Atomic absorption spectrophotometer AA-6300 SHIMADZU was used for determination of Pb^{2+} ion.

3.3 Characterization of the adsorbent:
Fourier transform infrared spectroscopy (FTIR) technique was used for the characterization of the adsorbents. Unloaded and loaded specimens were used to determine the wave numbers in the range of 4000 – 500 cm-1 using standard KBr diffusion method. Batch equilibration method was followed for the optimization process. The adsorption on all types of adsorbents was investigated, a fixed amount (1.0 gm) of absorbent was placed in a conical flask for the different cases. An ion(s) solution was then added to each adsorbent and was observed that Langmuri and Freundlich isotherm were achieved.
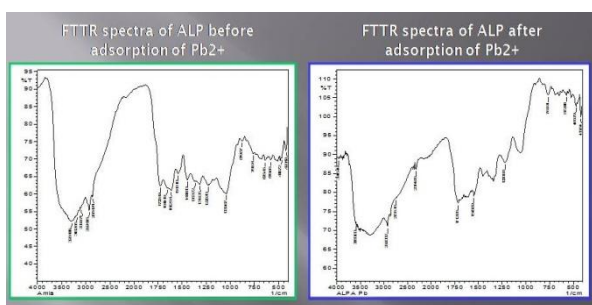
Fig. 1.0: FTIR spectra of NLP
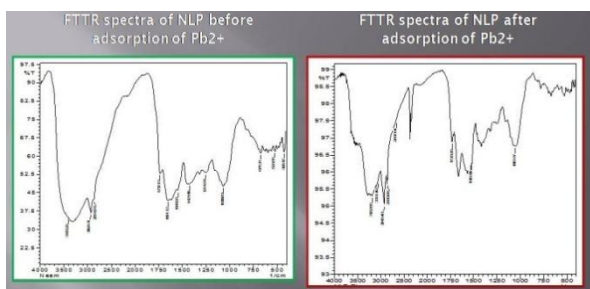
Fig. 2.0: FTTR spectra of PLP
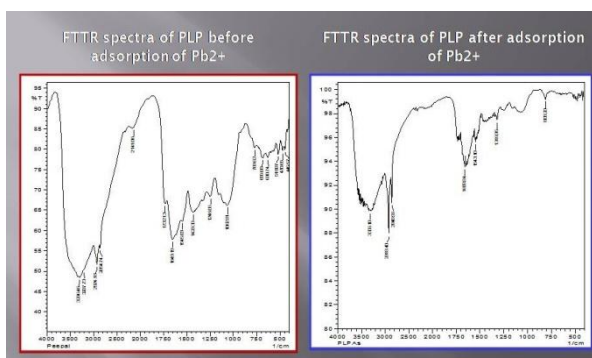
Fig. 3.0: FTTR spectra of ALP

Table: 1.0 Bands assigned to the surface functional groups of NLP, PLP and ALP before and after adsorption of Pb^{2+}

	Wave Number (cm^{-1})		
S. No.	**NLP, PLP and ALP(unloaded)**	**NLP, PLP and ALP (loaded) Pb^{2+}**	**Bond stretching**
1	3284.88 in case of NLP 3296.46, 3207.73 in case of PLP	3560.71, 3284.88 in case of NLP 3296.46 in case of PLP	-O-H in alcohols, acid, phenols and –N-H in amines and amides.

	3284.88 in case of ALP	3580.00 in case of ALP	
2	2924.18, 2854.74 in case of NLP, PLP and ALP	2918.40 in case of NLP 2918.40, 2848.96 in case of PLP 2920.32 in case of ALP	-C-H in $-CH_3$ and CH_2.
3	1722.49 in case of NLP and ALP 1732.13 in case of PLP	1728.28, 1662.69 in case of NLP 1737.92, 1651.12 in case of PLP 1712.85 in case of ALP	-C=O in carbonyl, carboxyl group and lactones.
4	1649.19, 1612.54,1543.10, 1442.80 in case of NLP and ALP 1545.03, 1429.30 in case of PLP	1552.75, 1417.73 in case of NLP 1543.10 in case of PLP 1548.89 in case of ALP	-C=C- in aromatic rings, -C=O in highly conjugated.
5	1319.35, 1039.67 in case of NLP and ALP 1062.81 in case of PLP	1319.35 in case of NLP 1039.67, 1037.74 in case of PLP	-C-O in alcohols, phenols, ethers, esters, acids, epoxides, lactones and carboxylic anhydrides.
6	879.57, 761.91, 638.46 in case of NLP and ALP 789.62, 686.62, 630.74 in case of PLP	669.39 in case of NLP 761.91 in case of ALP	-C-C- deformations and out of plane -C-H deformation in aromatic rings.

NLP, PLP and ALP were used for adsorption studies and were characterized by FTIR. The FTIR results for load and unloaded specimen are shown in Figs. 1.0, 2.0, and 3.0 respectively. In Table1.0 the peaks at 3560.71, 3284.88, 3269.45, 3182.65 cm-1 for NLP, 3306.10, 3296.46, 3207.73 cm-1 for PLP and 3524.06, 3338.89, 3294.53, 3284.88 cm-1 for ALP respectively, are the indicator of O-H (free) and N-H groups. The stretching of –OH groups bound to methyl group radicals presented in fig. 1.0, 2.0, 3.0 between 2924.18, 2918.40, 2854.74, 2852.40, 2850.88 cm-1 for NLP, 2918.40, 2854.74, 2848.96 cm-1 for PLP and 2928.40, 2924.18, 2854.74 cm-1 for ALP . The peaks located at 1728.28, 1722.49 cm-1 for NLP, 1737.92, 1732.13 cm-1 for PLP and 1722.491712.85, 1710.92 cm-1 for ALP respectively are the Characteristics of C=O group stretching from aldehyde and ketones. The appearance of OH group and C-O groups conforms the presence of carboxylic group in the adsorbents.

Therefore we concluded that the –OH, -NH, carbonyl, Carboxylic groups are important sorption sites. The FTIR spectrum of NLP, PLP and ALP adsorption of $Pb2+$ as shown in Fig. 1.0, 2.0, 3.0 respectively, reveals that the involvement of OH- and carboxyl groups in the adsorption.

3.4 Surface topography by SEM
The topography of the loaded and unloaded specimem were obtained using Scanning Electron Microscopy (SEM) and the results of the same are reflected in fig. 4.0, 5.0, and 6.0 respectively.

NLP (Neem Leaves Powder): The SEM images of Neem Leaves Powder unloaded and loaded with $Pb2+$ as shown in fig.4.0.We observed that the surface texture of Neem Leaves Powder (NLP) is rough. The rough texture of surface of the particles with quite big voids, throughout the material.

This indicates that particles are having some porosity, it is evident that active sites of adsorbents are covered due to the adsorption of constituent ions shows progressive changes in the surface particles with native adsorbents which depicts that the constituent ions have deposited as aggregates in the adsorbents.

PLP (Peepal Leaf Powder): The SEM image of PLP-plain indicates smoothness of the surface with a little even texture. It can be seen that adsorption of Pb2+ ions does not contribute towards change in surface texture as shown in fig. 5.0 and it is notable that surface looks smooth as compared to NLP.

ALP (Amla Leaf Powder): The SEM image of ALP - plain possess almost the same texture as that of PLP; however this sample has a little more roughness which is clearly visible in fig. 6.0. After the adsorption of Pb^2 ions it can be seen that these ions are well distributed at adsorption sites and cause measurable change.

Fig 4.0: Topography of NLP surface by SEM
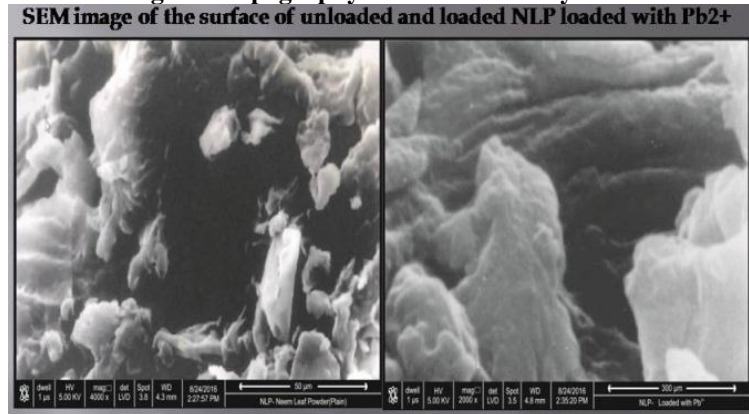
Fig 5.0 Topography of PLP surface by SEM
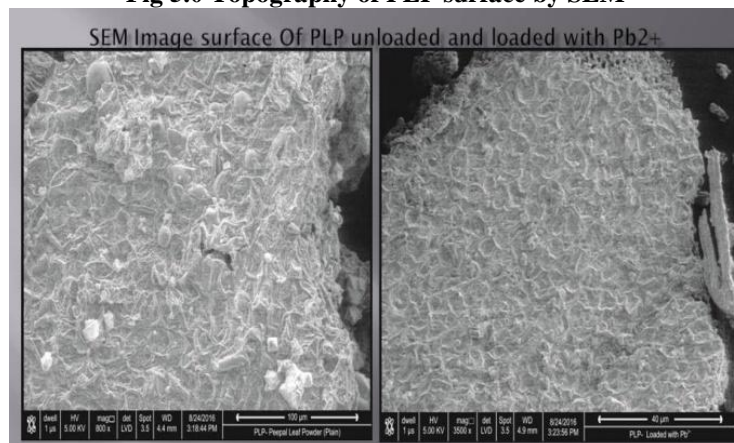
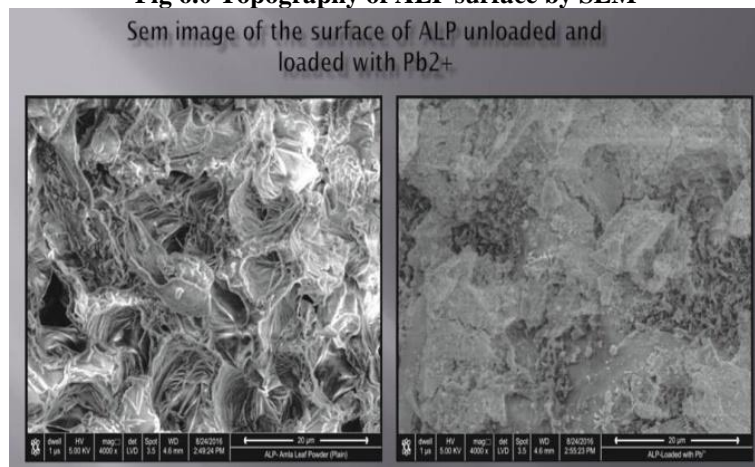**Fig 6.0 Topography of ALP surface by SEM**
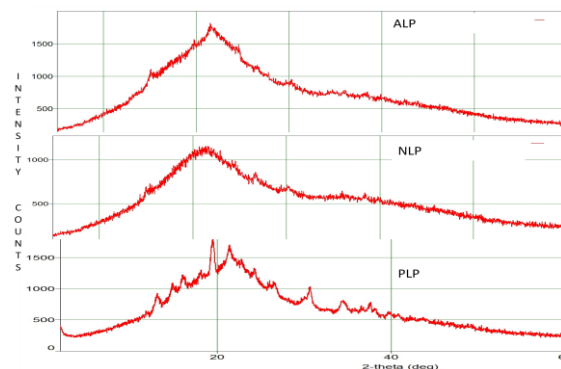
Fig. 7.0: Comparison of unloaded adsorbents

Fig. 8.0: Comparative XRD patterns after the adsorption of Pb2$^+$ on NLP, ALP, PLP surface.
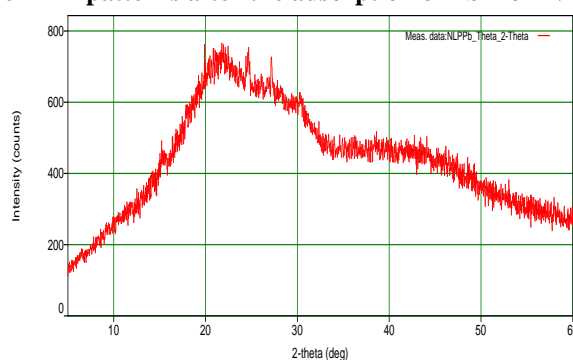

Fig. 9.0 : XRD patterns after the adsorption of Pb^{2+} of PLP
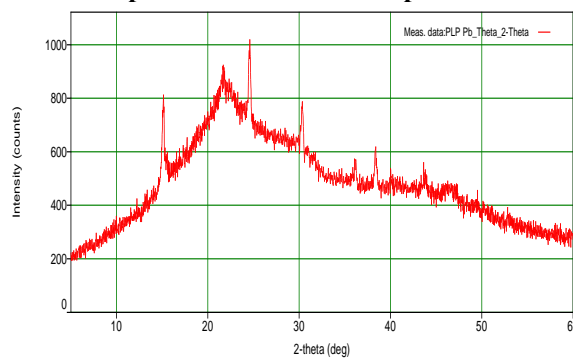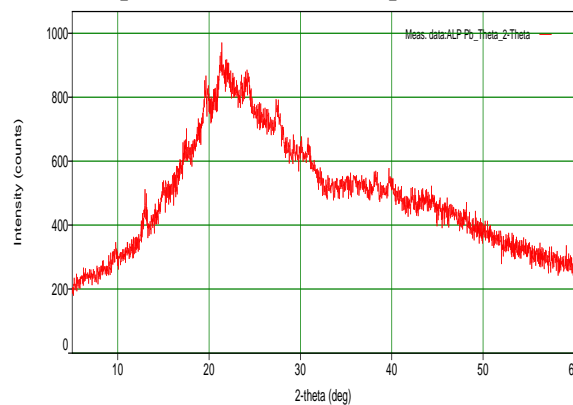

Fig. 10: XRD patterns after the adsorption of Pb^{2+} of ALP

XRD

Powder X-ray diffraction (PXRD) patterns were recorded using bench top X-ray diffractometer

The X-ray diffraction patterns of the leaf powders indicate amorphous nature with a broad peak centered on 20°, which is characteristic of polysaccharides (cellulose, starch, etc.).

However, PLP exhibited several crystalline peaks. The adsorption capacity was found to be of the order **NLP > ALP > PLP** as shown in fig 8.0, 9.0 and 10.0.

It can be inferred that the crystalline or ordered structure prevents adsorption of ions into the deeper layers, whereas amorphous structures allows penetration of ions into the adsorbent.

For ALP these peaks appeared as doublets, indicating interaction with functional groups also with the crystallization over the surface.

In conclusion an amorphous nature of the resultant material in XRD study indicated a fast adsorption due to stronger adsorption capacity.

Batch experiments were performed for adsorption as described earlier. Initial and final concentration of lead was measured by AAS (Atomic Absorption Spectrophotometer) with Hollow Cathode Lamp at 283.3 nm and acetylene - air flame AAS system and the results are presented below.

Table: 2 the adsorption batch experiments were conducted under the following conditions

| S. No.	Various conditions applied during batch experiment	
1.	Adsorbate lead solution	[0.1-0.5 mg/L]
2.	Adsorbents	(1) NLP(2) PLP (3) ALP
	pH	6.5
3.	Adsorbent dose	0.2 gm,0.4 gm, 0.6 gm, 0.8 gm, 1.0 gm
4.	Contact time	20 min., 40 min., 60 min., 80 min., 100 min.
5.	Temperature	20°± 2°C, 30°± 2°C, 40°± 2°C, 50°± 2°C
6.	Shaking time	1 hour
7.	Apparatus for determination of Pb^{2+} concentration	AAS (Atomic Absorption Spectrophotometer).

IV. RESULTS AND DISCUSSION

Effect of adsorbent dose:

The effect of NLP, PLP and ALP dosage on lead removal was studied, using 0.2-1.0 gm of them at initial lead concentration of 0.1mg/L, temperature**:** 30°± 2°C, pH: 6.5, contact time: 2hr., shaking time: 1hr. The results are summarized in figure 11.The percentage of lead removal increases from 7.4% to 85.2% with increase in contact dosage of NLP from 0.2 to 1.0 gm/50 mL, 10.6% to 81.5% with increase in contact dosage of PLP from 0.2 to 1.0 gm/50 mL, 12.4% to 83.6% with increase in contact dosage of and ALP from 0.2 to 1.0 gm/50 mL. This effect could be attributed to the increasing number of possible binding sites at high dosage of adsorbents. It was found that NLP, PLP and ALP all are efficient adsorbents. There was no significant variation in % removal by all adsorbents. A similar result was observed with other adsorbent by Reddy et al., 2010**.**
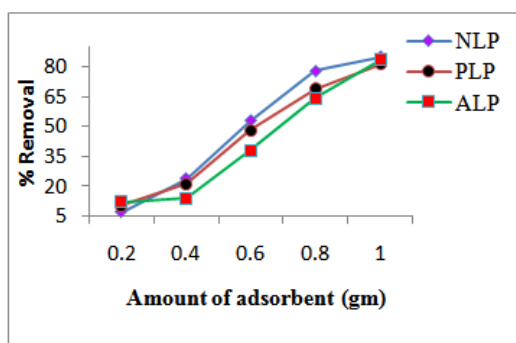
Figure 11 Effect of absorbent dose on lead ion removal

Effect of pH:. The pH level of the solution was varied between 2.0 to 12.0. The results are summarized in figure 12 at initial lead concentration: 0.1mg/L, adsorbent dose: 1gm/50mL, temperature: 30˚C ± 2˚C, contact time: 2hrs and shaking time: 1hr. it is observed that the % removal of lead is more dominant at pH – 6. The highest adsorption capacity 84.0% in case of NLP, 72.2% in case of PLP and 83.2% in case of ALP was obtained at pH = 6.0 then adsorption decreases with the increase of pH up to 10.0.

The adsorption of Pb^{2+} ion was found to be strongly dependent on the pH of solution. figure 12 depicts that the optimum pH for the adsorption of Pb^{2+}ion was about 6 which were rather acidic. At low pH levels there was excessive protonation of the active sites of NLP, PLP and ALP surfaces, the formation of bond between Pb^{2+} and active site showed a decreasing trend. At moderate levels of pH 3-6 the H+ ions are released from active sites and adsorbed amount of metal ions is generally found to increase.
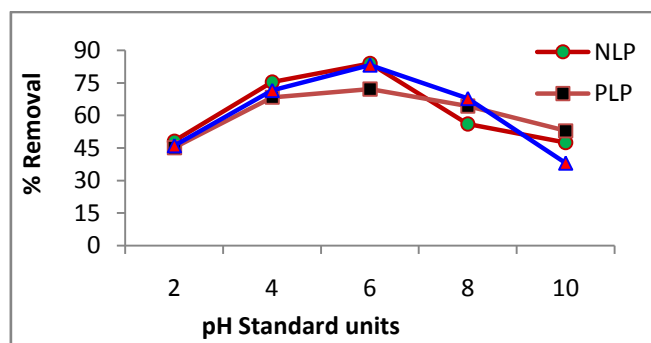
Fig. 12: Effect of pH on lead ion removal:

At higher pH (above-6) the precipitation is dominant or both ion exchange and aqueous metal hydroxide formation may become significant in the metal removal process. Similar results were reported by D.H.K. Reddy et al [232], Asandei, D. et al [24]. The adsorption capacity of adsorbents at all used pH values were as follows: NLP > ALP > PLP.

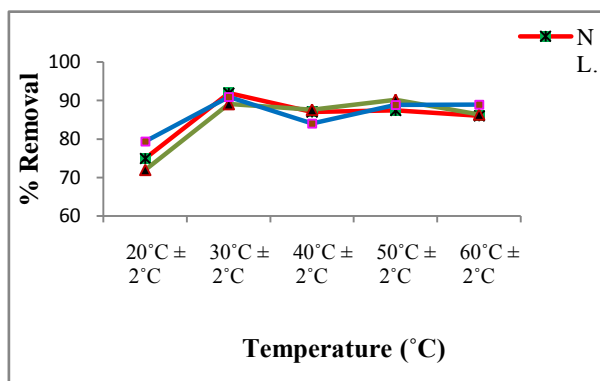**Effect of temperature on adsorption:**

The removal of lead from aqueous solution by NLP, PLP and ALP at various temperatures was studied by conducting batch experiments at 20˚C ± 2˚C, 30˚C ± 2˚C, 40˚C ± 2˚C and 50˚C ± 2˚C at initial lead concentration: 0.1 mg/L, adsorbent dose: 1gm/50 mL, pH: 6.5, contact time: 2hrs., shaking time: 1hr.

The results are summarized in figure 13**.** The results indicated that the adsorption increases with increasing temperature. The percentage of lead absorption was found to be maximum at 30˚C ± 2˚C The highest 92.0%, 89.1% and 91.0% lead were removed onto NLP, PLP and ALP at respectively.

This is due to the change in pore size which leads to intra particle diffusion of adsorbate. However when the temperature further increased, the lead adsorption decreased probably desorption took place.

It was observed that all adsorbents removed almost equal amount of lead. The results are in good agreement with Reddy et al., 2010.

Figure 13

**Fig. 13: Effect of different temperatures on lead ion removal**

Effect of contact time:
The effect of contact time on the adsorption process was studied in the time range of 20 to 120 minutes at initial lead concentration: 0.1mg/L, pH: 6.5 and temperature: 30˚C ± 2˚C with a fixed adsorbent dose: 1gm/50mL and shaking time: 1hr.
The results are summarized in figure 14. It was observed that an increase in contact time corresponds to an increase in adsorption. The adsorption increased rapidly during the first 60 mins. There after wards the adsorption remained constant.
Adsorption yield of 76.95% with NLP, 75.97% with PLP and 81.43% with ALP was attained in 60 minutes. Similar results were reported by several researchers [Yao, 2009; Zhang, 2005].

Figure 14
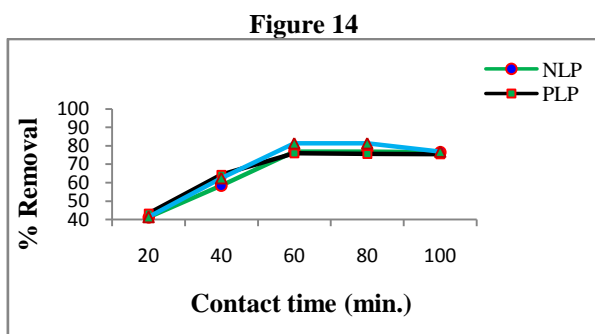
Fig. 14: Effect of contact time on lead ion removal.

Effect of initial lead ion concentration:
 Effect of concentration of lead solution was investigated by repeating experiments, using different initial concentration 0.1 mg/L to 0.5 mg/L of lead solution with a fixed adsorbent dose 1gm/50 mL, fixed temperature 30˚C ± 2˚C, fixed pH: 6.5, fixed contact time: 2hrs and shaking time: 1hr. The results are summarized in figure 14. It was observed that adsorption efficiency decreased as lead ion concentrations increased, indicating saturation.

78.18% removal of lead at 0.1 mg/L on NLP, 86.20% removal of lead at 0.1 mg/L on PLP and 87.20% removal of lead at 0.1 mg/L on ALP were observed. Since the number of active site are fixed, adsorption of lead decreases as the concentration of lead increase.
Such type of results has also been extensively reported in literature [Zhang and Banks, 2005; Zheng and Huang, 2011; Asandei, D.,2009].

Figure: 15
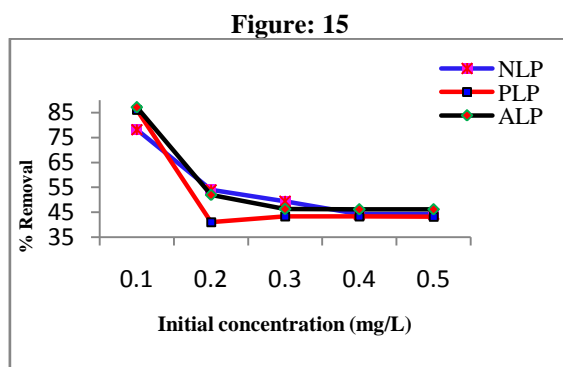
Fig. 15: Effect of different initial concentration on Lead removal

Adsorption isotherm:
 Langmuir isotherm
It assumes a monolayer adsorption onto a uniform adsorbent surface with energetically identical sorption sites [Giles, 1974]. The linear form of Langmuir isotherm is given by the following equation:

$$\frac{1}{q_e} = \frac{1}{Q_o} + \frac{1}{Q_o b}\frac{1}{C_e}$$

...... (1)

Where: Ce= equilibrium concentration of the adsorbate (mg/L)

qe = amount of adsorbate per unit mass of adsorbent (mg/g) Q_o and b are the Langmuir constants related to adsorption capacity and rate of adsorption respectively.
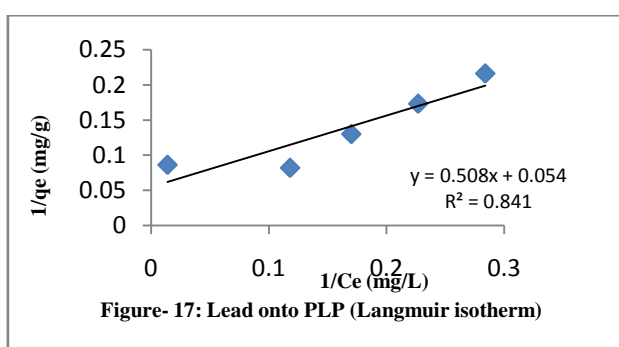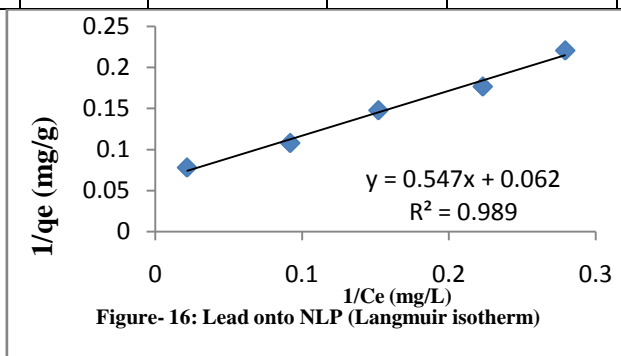
Freundlich isotherm It describes equilibrium on heterogeneous surface and hence does not assume mono layer capacity [Giles,1974]. The well known logarithmic form of the Freundlich isotherm is given by the following equation followed
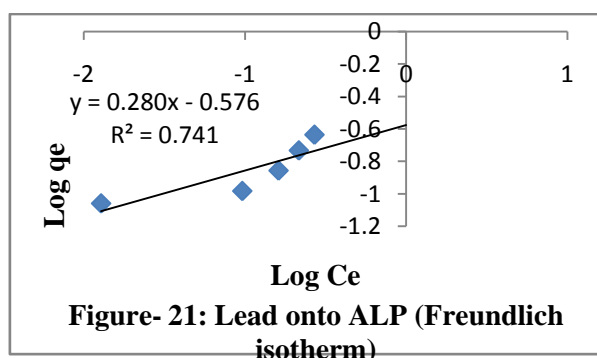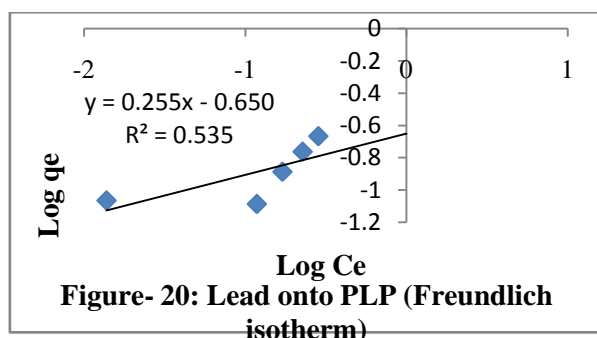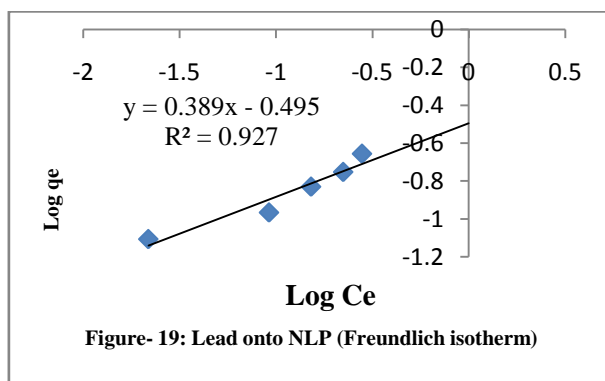
$$log\ qe = logKF + 1nlogCe$$

Where: Ce = equilibrium concentration of the adsorbate (mg/L)qe = amount of adsorbate per unit mass of adsorbent (mg/g) KF and n are the Freundlich constants.

Langmuir and Freundlich isotherm equations were **studied by plotting 1/ C_e versus $1/q_e$ and shown in figure-16, 17, 18, 19, 20 and 21 The adsorption isotherm parameters which were calculated from the slope and intercept of the linear plots using the linearized form of the Langmuir and Freundlich equations together with R^2 values are given in table 3**

Table No.: 3	Determination of coefficients for each model equation					
Isotherm Adsorbents	Langmuir			Freundlich		
	Parameters			Parameters		
	Q_0 (mg/g)	b (L/mg)	R^2	K_f (mg/g)/ (mg/L)$^{-1/n}$	n	R^2
NLP	16.120	0.113	**0.989**	0.310	2.570	**0.927**
PLP	18.519	0.107	**0.841**	0.223	4.444	**0.535**
ALP	15.873	0.110	**0.932**	0.265	3.571	**0.741**

Figure- 16: Lead onto NLP (Langmuir isotherm)

Figure- 17: Lead onto PLP (Langmuir isotherm)

Figure- 18 Lead onto ALP (Langmuir isotherm)

$y = 0.568x + 0.063$
$R^2 = 0.932$

$y = 0.389x - 0.495$
$R^2 = 0.927$

Figure- 19: Lead onto NLP (Freundlich isotherm)

$y = 0.255x - 0.650$
$R^2 = 0.535$

Figure- 20: Lead onto PLP (Freundlich isotherm)

$y = 0.280x - 0.576$
$R^2 = 0.741$

Figure- 21: Lead onto ALP (Freundlich isotherm)
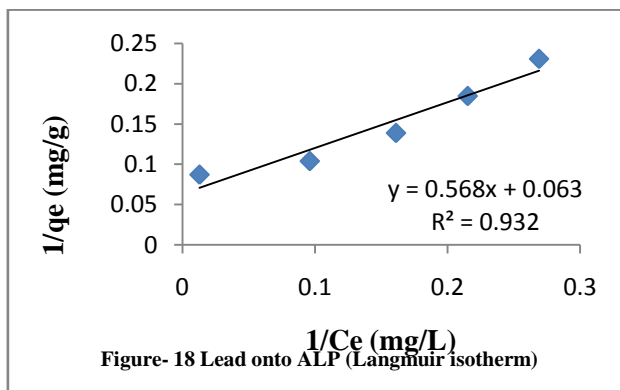
It is clear from regression coefficient (**R^2**) values that the Langmuir isotherm was better Freundlich isotherm model. In strict terms the Langmuir isotherm was originally developed for gas phase adsorption it is not surprising to see a poor correlation with the experimental results.

A favourable adsorption tends to have Langmuir isotherm (n) between 1 to 10 (= 9.2). Larger value of n, smaller of 1/n implies stronger interaction between the adsorbent and the adsorbate [Öztürk, N., Bektas, T. E.,2004. From table 3 , it can be seen that higher n values showing favourable adsorption of lead onto the NLP, PLP and ALP [Al Duri, B.;1996]**.**

V. CONCLUSION

As a result of this study we understand that the conventional processes are costly and are not energy efficient, a non conventional alternative for removal of lead by using the powders of Neem leaves (NPL), Peepal leaves (PPL), Amla leaves (APL) was investigatedFrom the investigation we understand the effect of different variables like temperature, pH, contact time, metal ion concentration and absorbent doses. Satisfactory results were obtained for a temperature of 30˚C ± 2˚C , pH: 6.0.,contact time 60 min and metal ion concentration below 1.0 mg/l.

The amount of lead adsorption increases with the increase of adsorbent concentration which indicates that adsorption depends upon the availability of active sites for lead, indicates that the adsorption depends upon the availability of active sites for lead but the desorption probably took place at about 40˚C ± 2˚C and higher temperature values. The equilibrium of lead adsorption was attained in about 60 min. The percent lead adsorption decreased with increased in lead concentrations. The experimental data were correlated reasonably well by Langmuir adsorption isotherm. So we concluded that lead can be removed satisfactory by leaf powders (NLP, PLP and ALP) and increasing order of lead adsorption on leaf powders was as follows.NLP > ALP > PLP

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