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Adsorptive Removal of Lead (II) Ions from Aqueous Media using Synthesized Zeolite and Fly Ash

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***Abstract*—Adsorption-based procedures offer efficient and economical solutions for the removal of toxic heavy metals from aqueous environments. Lead is one of the most toxic heavy metals present in industrial wastewater. In the present study, the adsorption performance of synthesized zeolite and fly ash toward Pb (II) ions was estimated using batch equilibrium experiments. Residual metal concentrations were calculated by atomic absorption spectroscopy (AAS). The adsorption equilibrium data were analyzed using Langmuir and Freundlich isotherm models. Zeolite consistently exhibited higher adsorption capacity than fly ash for all investigated metal ions. The adsorption process followed both Langmuir and Freundlich models, indicating monolayer adsorption on heterogeneous surfaces. The results demonstrate that synthesized zeolite is a promising adsorbent for heavy-metal remediation in aqueous systems.**

***Keywords*— Fly ash, Freundlich isotherm, Heavy metal adsorption, Langmuir isotherm, ZSM5.**

I. INTRODUCTION

Coal-fired power plants generate considerable amounts of fly ash (FA), creating significant environmental concerns. Because of its high silica and alumina content, FA has gained attention as a valuable precursor for the synthesis of composites, geopolymers, and zeolitic materials [1]. Zeolites produced from FA demonstrate wide-ranging applications, particularly in catalysis, membrane separation technologies, and adsorption systems [2].

The presence of heavy metal ions such as zinc, lead, and cadmium in water sources possess severe environmental and health risks due to their non-biodegradable and toxic nature [3]. Among various treatment techniques, adsorption has emerged as a highly effective and widely applied method owing to its simplicity, cost-effectiveness, and operational flexibility. Materials such as zeolites and industrial by-products like fly ash have attracted significant attention because of their surface activity, porosity, and ion-exchange capabilities.

Adsorption equilibrium studies using isotherm models provide appreciated insight into the nature of adsorbent–adsorbate interactions and surface characteristics.

The Langmuir model assumes monolayer adsorption on a homogeneous surface, whereas the Freundlich model describes adsorption on heterogeneous surfaces. In this study, the adsorption behaviour of Pb (II) ions on synthesized zeolite and fly ash was systematically investigated using AAS, with particular importance on equilibrium isotherm analysis.

II. MATERIALS METHODS

The main raw material, coal fly ash samples were collected from Deep Nagar Thermal Power Plant, Bhusawal. The samples contained both amorphous (mainly SiO2, Al2O3) and quartz and mullet. The unburnt carbon along with other volatile materials present in fly ash was removed by calcinations at high temperature for 2 hrs. Then 10g fly ash was taken and added with NaOH solution as 1:1.2 Proportions. The resultant mixture was stir at 300K for different time period. Then the mixture was filtered and washed and dried at 353K. [4-5].

III. CHARACTERIZATION

The diffractograms were recorded using Cu-Kα radiation at a scanning speed of 1.2 degree per min. The zeolite material is scanned in the 2θ range of 00 to 600. The XRD pattern was obtained at 30kV and 15mAon Philips (3710pw/1710) X-Ray Diffractometer system. The X-ray diffractograms of fly ash, ZSM5 (Fly Ash Zeolite FAZ) are shown in figures 1 and 2 respectively.

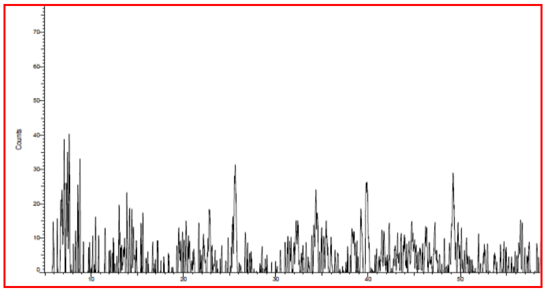

Figure 1. XRD of Fly Ash

Figure 1 illustrates the XRD pattern of raw fly ash, which is dominated by a broad diffuse hump accompanied by a few low-intensity diffraction peaks, indicating its largely amorphous character. The broad halo observed in the 2θ range of approximately 20–35° is associated with amorphous aluminosilicates glass phases, while the weak crystalline peaks can be ascribed to minor phases such as quartz and mullet. These features confirm that fly ash is primarily composed of poorly ordered aluminosilicates materials with limited crystallinity [6]

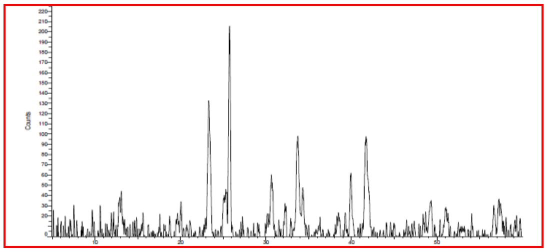

Figure 2 XRD of ZSM5

In contrast, the XRD pattern of the synthesized ZSM-5 zeolite shown in Figure 2 exhibits numerous sharp and intense diffraction peaks, evidencing the formation of a highly crystalline structure. Prominent reflections appearing at 2θ values around 7–9° and 22–25° are characteristic of the MFI framework of ZSM-5. Relative to fly ash, the disappearance of the amorphous hump and the development of well-defined diffraction peaks indicate a substantial structural transformation from an amorphous aluminosilicates precursor to an ordered zeolitic framework. Moreover, the absence of diffraction peaks corresponding to quartz or mullet suggests the effective conversion of fly ash constituents into phase-pure ZSM-5 zeolite.

Infrared spectrograms of fly ash and modified ZSM5 zeolite are shown in figure 3 and 4.These were recorded on Perkin-Elmer FT-IR spectrophotometer in the frequency range 400-4000cm-1 by using KBr pellet technique.

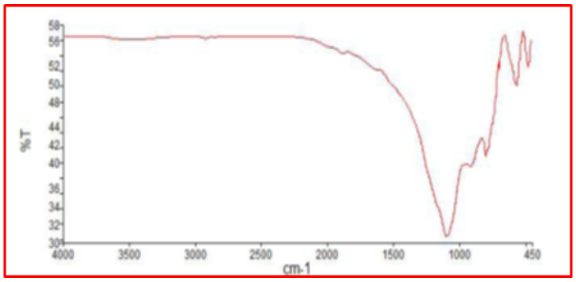

Fig. 3 IR Spectra of Fly Ash

Figure 3 shows the FTIR spectrum of raw fly ash, characterized by broad absorption bands typical of amorphous aluminosilicates materials. The broad band centred on 3400 cm^{-1} is attributed to the stretching vibrations of hydroxyl (–OH) groups associated with physically adsorbed moisture, while the band near 1630 cm^{-1} corresponds to the bending vibration of molecular water (H–O–H). A strong absorption band observed in the 1000–1100 cm^{-1} region is assigned to the asymmetric stretching vibrations of Si–O–Si and Si–O–Al bonds, confirming the presence of aluminosilicate glass phases [7]. Additional weak bands appearing below 800 cm^{-1} arise from symmetric stretching and bending vibrations of T–O bonds (T = Si or Al).

The FTIR spectrum of the synthesized ZSM-5 zeolite presented in Figure 4 exhibits sharper and well-resolved bands, indicating the formation of a well-ordered zeolitic framework. The broad absorption around 3400 cm^{-1} and the band near 1630 cm^{-1} are again associated with surface hydroxyl groups and adsorbed water molecules within the zeolite channels. A prominent band near 1100 cm^{-1} corresponds to the asymmetric stretching vibration of the Si–O–T (T = Si, Al) framework, which is characteristic of MFI-type zeolites. Notably, the distinct band observed in the 550–560 cm^{-1} region is attributed to the double five-membered ring (D5R) vibration, serving as a fingerprint feature of the ZSM-5 structure. Compared with fly ash, the emergence of this characteristic band and the increased sharpness of framework vibrations confirm the successful transformation of amorphous fly ash into crystalline ZSM-5 zeolite.

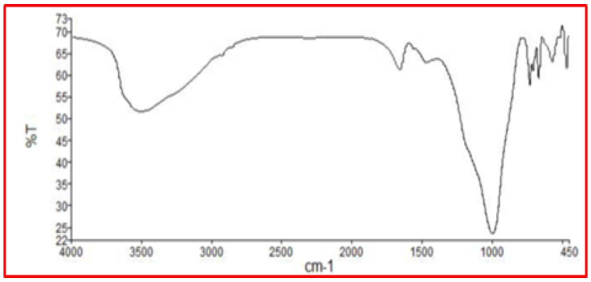

Fig. 4 IR Spectra of ZSM5

The scanning electron microscope (SEM) was recorded on LEO-Leica Stereo scan 440 Cambridge (U.K.) The magnification was observed between 5000X to 25000X. The EDAX analysis was done on Model Bruker, Quanta X-200, Germany. The SEM micrograph and EDAX spectra were recorded for Fly ash, ZSM5 zeolite is shown in figure 4 and 5 respectively.

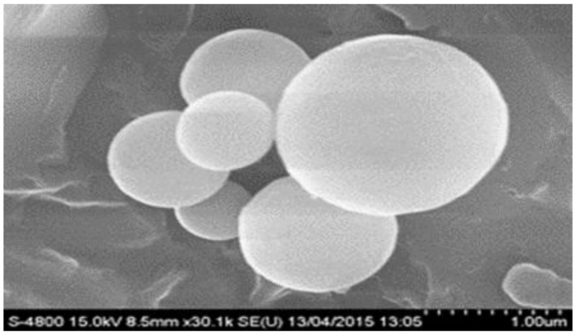

Fig. 5 SEM of Fly Ash

Figure 5 illustrates the SEM micrograph of raw fly ash, which is dominated by spherical particles with smooth surfaces and a wide range of particle sizes. These spherical features, commonly referred to as cenospheres, are characteristic of fly ash produced during high-temperature coal combustion. The particles exhibit dense and largely non-porous surfaces, reflecting their glassy aluminosilicate composition. The broad particle size distribution and the lack of distinct crystalline features further indicate the predominantly amorphous nature of the fly ash [8].

Fig. 6 SEM of ZSM5

Figure 6 shows the SEM micrograph of the synthesized ZSM-5 zeolite, which displays a markedly different morphology. The ZSM-5 sample is composed of agglomerated, elongated, rod-like, or coffin-shaped crystallites, typical of MFI-type zeolite structures. These particles possess rough and irregular surfaces with well-defined crystalline surfaces, suggesting effective nucleation and growth of the zeolitic framework.

Relative to fly ash, the disappearance of spherical particles and the development of well-defined crystalline morphologies highlight a pronounced morphological transformation during synthesis, confirming the successful conversion of amorphous fly ash into crystalline ZSM-5 zeolite.

Adsorption Property of Fly Ash and FAZ: Adsorption study of lead by zeolite and fly ash was carried out in the following manner. The metal concentration solution can be prepared using following formula

Metal (mg/Kg) = AAS reading (mg/l) × (Vol of digest, l)]/ (Amount of Sample, g)

Initially lead solutions are prepared as 2ppm, 5ppm, 10ppm 15ppm and 20ppm concentration. Then each sample is divided in three groups. First group is used as standard concentration. In second group 0.1g of zeolite is added and in third group 0.1 g of fly ash is added in each concentration respectively. The second and third sample is shaking time to time and kept for 1 hr. After that the second group and third group solutions are filtered and drop of 0.1N HNO3 is added in it. The adsorption study is carried out on atomic adsorption spectroscopy [9-10].

The adsorption isotherm data in figure 7 describe the uptake behavior of lead (Pb) ions onto the zeolite adsorbent. The experimental results were fitted using a linear relationship, Q=43.60, Ceq=3.917, with a coefficient of determination (R2) of 0.924. This linear equation serves as an empirical approximation within the investigated concentration range rather than representing a fundamental adsorption isotherm model.

Table 1
Adsorption of Lead on zeolite

Adsorption of lead on zeolite				
Ceq, mgs	**Q (mg/g)**	**Ceq/Q**	**lnCeq**	**lnQ**
0.1119	0.3927	0.28495	-2.19015	-0.93471
0.1294	1.46935	0.088066	-2.04485	0.38482
0.1473	3.61395	0.040759	-1.91528	1.284801
0.2113	5.3205	0.039714	-1.55448	1.671567
0.2326	5.91725	0.039309	-1.45844	1.777872

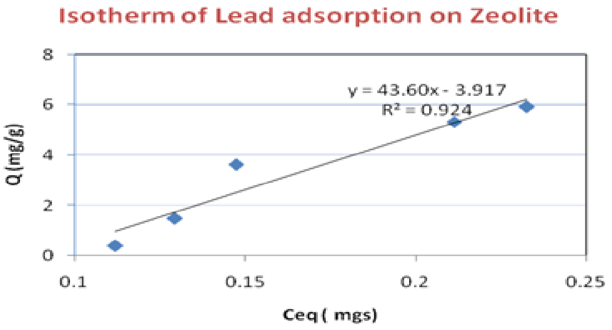

Fig 7 Isotherm of lead adsorption on zeolite

The relatively high slope value (43.60) indicates a strong affinity of the zeolite toward Pb ions, as the adsorption capacity Q (mg g^{-1}) increases markedly with increasing equilibrium concentration Ceq (mg L^{-1}). However, the reasonably high R2 value suggests a good, though not ideal, linear correlation under the experimental conditions, indicating that a nonlinear isotherm model would likely provide a more accurate description, particularly at low concentrations. Overall, the results confirm the effectiveness of zeolite for Pb removal, while the linear fit may be used as a practical, range-specific correlation for estimating adsorption capacity within the studied experimental limits.

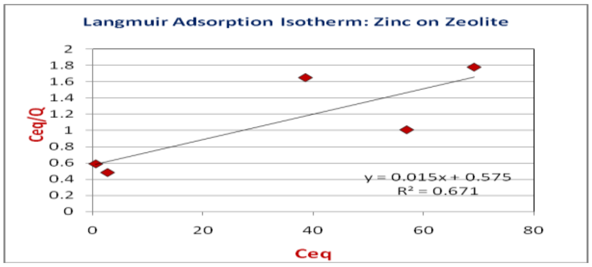

Fig 8 Langmuir adsorption isotherm: Lead on zeolite

The graph in figure 8 represents the Langmuir adsorption isotherm **for** Zinc ions on zeolite, where Qe (amount adsorbed per unit mass of adsorbent) is plotted against Ce (equilibrium concentration of Zinc ions). The straight-line nature of the plot indicates that the adsorption process approximately follows the Langmuir model, which assumes monolayer adsorption on a homogeneous surface with finite adsorption sites**.** The linear fit equation a positive relationship between Qe and Ce, meaning that adsorption increases with increasing metal ion concentration. The correlation coefficient R^2=0.671 shows a moderate fit, indicating that while Langmuir adsorption is significant, some deviation may occur due to surface heterogeneity or experimental uncertainties.

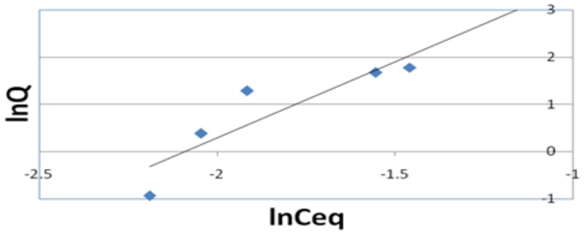

Fig 9 Freundlich adsorption isotherm: Lead on zeolite

Figure 9 presents the Freundlich adsorption isotherm for lead uptake on zeolite, plotted as the natural logarithm ofadsorption capacity, ln (Q), against the natural logarithm of equilibrium concentration, ln (Ceq).[11] The linear relationship observed in this log-log plot confirms that the adsorption process follows the Freundlich model, which is characteristic of heterogeneous surfaces with sites of varying energy. From the linear trend, the Freundlich constants can be derived: the adsorption capacity coefficient, KF, is approximately 2.72 mg/g, representing the adsorption strength at a unit concentration, and the intensity parameter, n, is about 1.83, indicating favorable adsorption over the studied range. A value of n>1 suggests that the binding of lead ions to the zeolite surface is energetically favorable and becomes more efficient as concentration increases. This behavior highlights the zeolite's suitability for lead removal applications, as the model provides a reliable empirical equation to estimate adsorption performance across different concentrations, supporting the design and optimization of treatment processes using zeolite-based adsorbents.

Table 2
Adsorption of lead on Fly Ash

Adsorption of lead on ash				
Ceq, mgs	Q (mg/g)	Ceq/Q	lnCeq	lnQ
0.2654	0.3159	0.84000	-1.32652	-1.15217
1.3881	0.975	1.42369	0.327936	-0.02532
1.952	2.7116	0.71987	0.668854	0.997539
2.1059	4.3732	0.48154	0.744743	1.475495
5.6835	3.1918	1.78065	1.737567	1.160585

Figure 10 shows the adsorption isotherm for lead removal using fly ash, with adsorption capacity (Q, mg/g) plotted against equilibrium concentration (Ceq, mg/L).

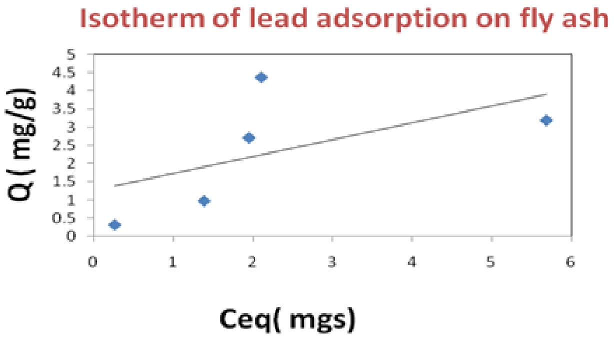

Fig 10 Isotherm of lead adsorption on Fly Ash

The isotherm exhibits a typical Langmuir-type (Type I) isotherm behaviour, indicating strong affinity between Pb^{2+} ions and the fly ash surface. A rapid increase in adsorption capacity at low Ceq reflects effective binding to available active sites, followed by a plateau at higher concentrations due to surface saturation and attainment of maximum adsorption capacity (Qmax). This behaviour suggests predominantly monolayer adsorption on finite active sites. Owing to its aluminosilicate and metal oxide constituents, fly ash provides suitable functional groups for lead uptake, demonstrating favourable and efficient adsorption performance and highlighting its potential as a low-cost, sustainable adsorbent for wastewater treatment applications.

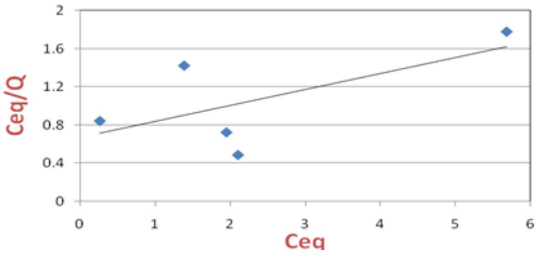

Fig. 11 Langmuir adsorption isotherm: Lead on Fly Ash

Figure 11 resents the Langmuir adsorption isotherm for lead adsorption onto fly ash, plotted as Ceq/Q versus equilibrium concentration (Ceq) [12-13]. The linear relationship observed indicates that the adsorption process follows the Langmuir model, suggesting monolayer coverage of Pb^{2+} ions on a homogeneous fly ash surface with a finite number of active sites. The positive slope reflects a finite maximum adsorption capacity, while the intercept corresponds to the Langmuir constant, representing the affinity between lead ions and the fly ash surface.

Despite minor data scatter, the overall linear trend confirms favourable adsorption behaviour, highlighting fly ash as an effective low-cost adsorbent for lead removal in aqueous systems.

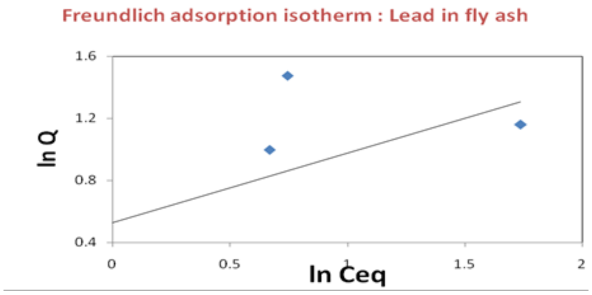

Fig. 12 Freundlich adsorption isotherm : Lead in Fly Ash

Figure 12 shows the Freundlich isotherm for lead adsorption on fly ash, plotted as ln Q versus ln Ceq. The linear trend confirms the Freundlich model is a good fit, indicating adsorption onto a heterogeneous surface with sites of different energies [14-16]. From the slope and intercept, the Freundlich parameters 1/n and KF can be determined; a value of $n > 1$ would reflect favorable adsorption. This behavior highlights the effectiveness of fly ash as a low-cost, multifunctional adsorbent for lead removal from water.

IV. CONCLUSION

The adsorption studies demonstrate that both fly ash and fly ash–derived zeolite (FAZ) are effective adsorbents for the removal of lead (Pb^{2+}) from aqueous solutions. Batch adsorption experiments carried out over a range of initial concentrations (2–20 ppm) reveal that zeolite exhibits a higher adsorption capacity and stronger affinity toward lead ions compared to raw fly ash, owing to its well-developed porous structure and higher density of active sites. The adsorption behavior of lead on zeolite is well described by the Freundlich isotherm, indicating heterogeneous surface adsorption with favorable interaction ($n > 1$), while the linear fit further confirms its strong uptake efficiency within the studied concentration range. In the case of fly ash, both Langmuir and Freundlich models adequately describe the adsorption process, suggesting predominantly monolayer adsorption on a finite number of active sites along with surface heterogeneity. The Langmuir analysis indicates favorable adsorption with a well-defined maximum capacity, highlighting the potential of fly ash as a low-cost and sustainable adsorbent.

Overall, the results confirm that zeolite derived from fly ash offers superior performance for lead removal, while raw fly ash itself remains a viable, economical alternative for wastewater treatment applications, contributing to both environmental remediation and beneficial utilization of industrial waste.

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