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Green Zinc Oxide Nanoparticle Ingrained on Activated Silica for the Removal of As(III) from aqueous solution using *Ocimum sanctum* and *Azadirachta indica*

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Abstract: Proficient protocol for the fabrication of benign adsorbent with Zinc oxide nanoparticles ingrained on activated silica (ZnO- NPs-AS) exclusive of calcinations was urbanized by green synthesis method by aqueous leaf extract of *Ocimum sanctum* and *Azadirachta indica*. The progression involved the use of zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$) and sodium hydroxide (NaOH) as a antecedent and aqueous extract of plants as a solvent with assorted roles as promoter, stabilizer and template for synthesis of zinc oxide nanoparticle. Adsorption performance of benign adsorbents was applied to Freundlich, Langmuir, Tempkin and BET isotherm which afford important information on the surface properties of the adsorbent and its affinity for adsorbate. Data correctly fits Langmuir isotherm than Freundlich, Tempkin and BET isotherm proving monolayer and homogenous surface of adsorption with R^2 nearly 0.992 and 0.974. In this study pseudo second order model fitted better (R^2=0.982 and 0.906) when compared with first order kinetic model. Therefore the adsorption data in the present study supported chemisorptions. The linearity of the plots also showed the validity of pseudo-second order model. The sequence of adsorption of adsorbent follows ZnO-NPs-AS-*Os*> ZnO-NPs-AS-*AZI*. The morphology and size of the adsorbent were characterized by Scanning Electron Microscope (SEM) for its morphology, shape and Energy Dispersive X-Ray analysis (EDX) for the elemental constitution of zinc, oxygen, silica and arsenic.
Key Words : Adsorbent; Isotherm; Kinetics; ZnO-NPs-AS; Ocimum sanctum; Azadirachta indica.

Introduction[1-7]

Hasty industrialization has led to remarkable bloat in the use of heavy metals. Over the precedent predictably resulted in an increased flux of metallic substances in the aquatic environment. The decisive environmental confront for industry is the safe disposal of wastewater. Among the assorted industries, electroplating units are the most imperative because of their lucrative applications. In contrast to other industries, the electroplating units use less water, thus produce small volume of wastewater which is highly toxic in nature because of the presence of hazardous metals such as Ni(II), As(III), Zn(II), Pb(II), As(V), Cr(VI) and Cd(II). Assorted treatment technologies have been developed for the exclusion of these metals from wastewater, such as precipitation, oxidation/reduction, coagulation flocculation, electro coagulation,

cementation, membrane separation, membrane filtration, solvent extraction, ion exchange and adsorption depending on the concentration of these metals. In regards of its simplicity and high-efficiency characteristics, the adsorption process are one of the few alternatives accessible for the removal of heavy metals at low concentrations from industrial effluents, it is an effectual inexpensive and proficient method. Adsorption technique is quite trendy due its simplicity and high efficiency, as well as the ease of use of a wide range of adsorbents. Various adsorbents have been tested and used for the removal of heavy metals from polluted water. Among the kinds of adsorbents, nano Zinc oxide NPs have been attracted interesting recently because it exhibit high surface area to volume ratio. However, the other methods have momentous disadvantages such as incomplete metal removal, particularly at low concentrations and high capital investment as well as creating sludge disposal problem. These heavy metals reach tissues through the food chain and accumulate in the human body. If the metals are ingested beyond the permitted concentration, they can cause serious health disorders. Therefore it is necessary to treat metal contaminated waste water prior to its discharge to the environment. The World Health Organisation (WHO1993) recommends a maximum acceptable concentration of Cu(II), Ni(II) and Zn(II) in drinking water of 2, 0.02 and 3 mg/ L respectively. Symptoms of arsenic poisoning begin with headaches, confusion, severe nausea and drowsiness. The poisoning develops convulsions and changes in fingernail pigmentation called leukonychia. When the poisoning becomes acute symptoms may include vomiting, blood in the urine, cramping muscles, hair loss, stomach pain and more convulsions. The organs of the body that are usually affected by arsenic poisoning are the lungs, skin, kidneys and liver. The final result of arsenic poisoning is coma and death. It is therefore essential to search benevolent product and to transform such materials to adsorbents. Increasing awareness towards green chemistry and biological processes has led to the efficacy and feasibility of an eco-friendly approach for the synthesis of ZnO nanoparticle ingrained on activated silica as proficient adsorbent for removal of As(III). The present work investigates the potential use of two different adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZI*) for the removal of As(III) from aqueous solution using plant extracts like *Ocimum sanctum* and *Azadirachta indica.*

Materials and Methods[1-7]

Preparation of Activated Silica (AS)

Activated silica (AS) is prepared by treating silica at a temperature of 500ºc for 6 hrs. The thermal pretreatment always increases the available active sites and increases adsorption capacities. The properties of coated silica (ZnO-NPs-AS) with plant extract had a change in specific surface area, pore size and pore volume.

Synthesis of Green Adsorbent

Green synthesis method was used to prime down adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi)* from plants like *Ocimum sanctum* and *Azadirachta indica*. 0.25ml, 0.5ml and 1ml aqueous leaf extracts of the respective plant was introduced into 250ml beaker with 50ml of distilled water and was vigorously stirred for 30 minutes in different sets. Then 1g of Zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$) was added and stirred. After 1 hr stirring 10g of activated silica was introduced into the above solution. Addition of drops of aqueous NaOH resulted in a white aqueous solution at pH 10-12. This was then positioned in a magnetic stirrer for 2 hrs. ZnO nanoparticle ingrained on activated silica were then filtered and washed with double distilled water. The synthesized ZnO-NPs-AS with plant extract was maintained at 60ºC for 12 hrs. A mortor was used to homogeneously ground the Zinc oxide nanoparticle ingrained on activated Silica with plant extract (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*). The proposed sorbent were stored in air at room temperature[8-9].

Characterization of Adsorbent

The external morphology and percentage elemental composition of the sample were characterized by scanning electron microscope (SEM) (LEO 1530FEGSEM) with Energy Dispersive X-ray Analysis (EDAX) [10].

Batch Experiments

Sequence of batch adsorption experiments was conducted to resolve the effect of adsorbent (ZnO-NPs-AS) in the removal of As(III). Therefore various adsorbent dosages of 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, 5.0, 5.5, 6.0, 6.5, 7.0, 7.5 and 8.0g were introduced into 250mL flasks with 20mL solution containing 0.005, 0.0075, 0.01, 0.02, 0.03, 0.04, 0.05, 0.06, 0.07, 0.08, 0.09 and 0.10 N of As(III) ions. The flasks were then

placed in an orbital shaker at an agitation speed of 50, 100, 150, 200, 250, 300, 350, 400, 450 and 500 rpm. Samples were taken at predetermined time intervals of (10, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110 and 120 minutes) in a pH range of 1, 2, 3, 4, 5, 6, 7 and 8 and then separated by centrifugation. Concentration of As(III) ions in aqueous phases were analyzed volumetrically in duplication to observe the reproducibility. The effect of each parameter is studied by changing one parameter progressively by keeping the others constant. The parameter obtained from different models of isotherms like Freundlich, Langmuir, Temkin and Brunauer Emmet Teller (BET) provides important information on the surface properties of the adsorbent and its affinity. The physisorption and chemisorptions kinetic rate equation is determined using Pseudo first and second order rate equation. The quantity of As(III) adsorbed by adsorbent was calculated using the following formula:

$$\% \text{ Removal} = (C_0\text{-}C_e) \times 100 / C_0$$

$$q_e = (C_0\text{-}C_e) \times V / W$$

Where C_o and C_e are initial and equilibrium concentration of As(III), q_e the amount of arsenic(III) adsorbed under given condition, V the volume of the solution(ml) and W the weight (g) of the adsorbent used[11].

Results and Discussion

Characterization of Adsorbent (ZnO-NPs-AS-Os)

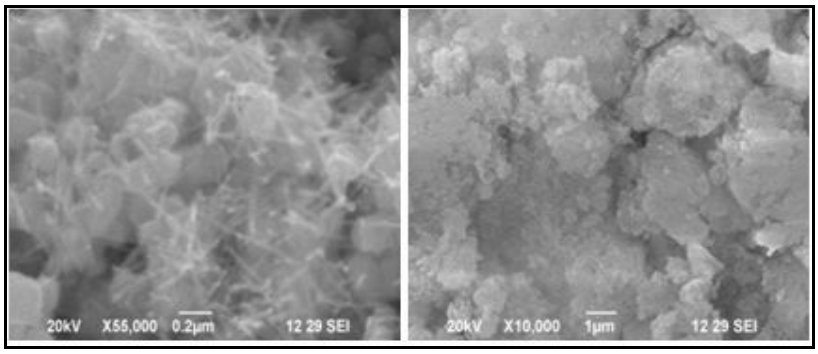

Figure 1 SEM image of ZnO-NPs-AS-*Os* before and after adsorption of As(III)

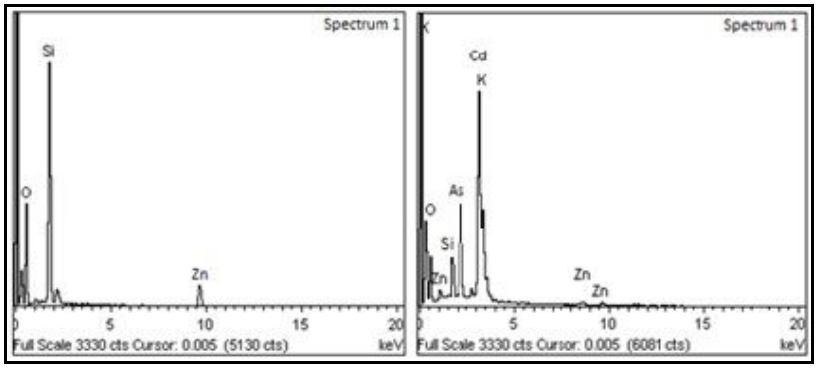

Figure 2 EDAX image of ZnO-NPs-AS-*Os* before and after adsorption of As(III)

Figure 1 and 2 shows SEM and EDAX image of Zinc oxide nanoparticle from aqueous leaf extract of *Ocimum sanctum* ingrained on activated silica (ZnO-NPs-AS-*Os*) from Zinc acetate and sodium hydroxide with water as beningn solvent before and after adsorption of As(III). The observations indicated that the particle size of the adsorbent (ZnO-NPs-AS-*Os*) is in the range of 0.2µm with fine needle shapes before adsorption and 1µm after adsorption of As(III). Figure 1 confirms the surface of the adsorbent (ZnO-NPs-AS-*Os*) is not smooth and uniform which enhances more adsorption of As(III). The composition of elements are in consistent with EDAX with zinc (Zn), oxygen (O) and silica (Si) elements before adsorption and zinc (Zn), oxygen (O), silica (Si) and arsenic (III) elements after adsorption is authenticated from figure 2[12].

Characterization of Adsorbent (ZnO-NPs-AS-AZI)

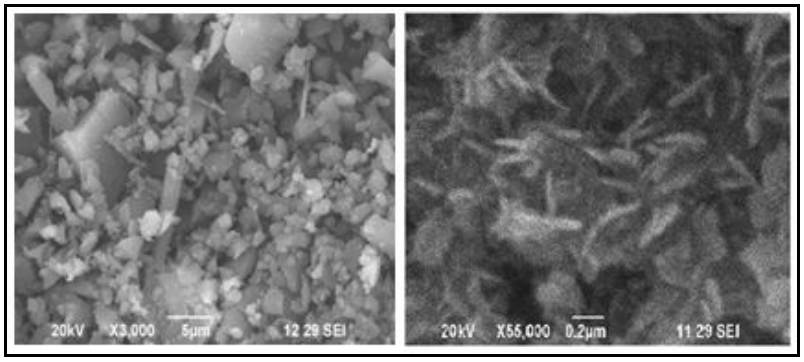

Figure 3 SEM image of ZnO-NPs-AS-*AZI* before and after adsorption of As(III)

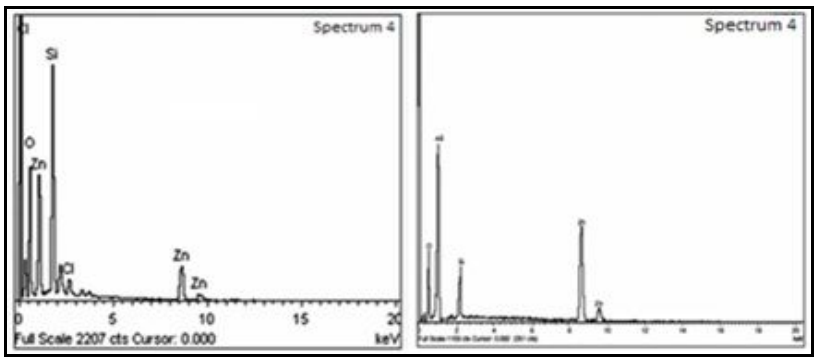

Figure 4 EDAX image of ZnO-NPs-AS-*AZI* before and after adsorption of As(III)

SEM and EDAX patterns of Zinc oxide nanoparticle obtained from *Azadirachta indica* and activated silica (ZnO-NPs-AS-*AZI*) shows preferential orientation for zinc, oxygen and silica before and after adsorption is shown in Figure 4. The observations in Figure 3 indicated that the particle size of the adsorbent (ZnO-NPs-AS- *AZI*) is in the range of 5µm with cube shapes before adsorption and 0.2µm with flower shape after adsorption of As(III). The strong intense and narrow width of the peak indicates that the adsorbent is of high purity. This confirms that ZnO-NPs-AS-*AZI* can be used as an adsorbent to adsorb As(III) from aqueous solution. After adsorption shows preferential orientation for zinc, oxygen, silica and arsenic (Figure 4). This authenticates that ZnO-NPs-AS-*AZI* can be used as an appropriate adsorbent to adsorb As(III) from aqueous solution[13-14].

Effect of Concentration

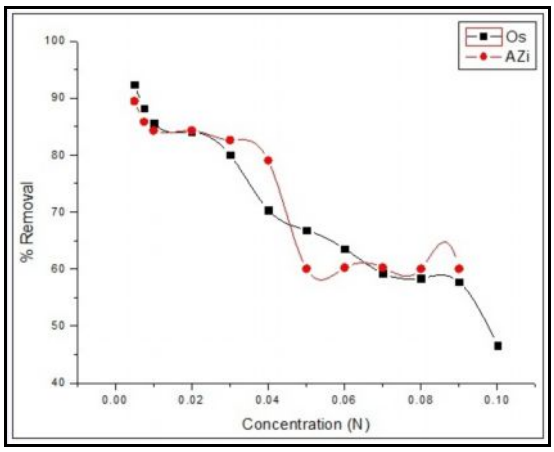

Figure 5 Comparative Effect of Concentration on the Adsorption of As(III) by Adsorbents

The adsorption capacities of two different adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZI*) per initial concentration are represented in figure 5. Different adsorbent capacities might be due to various factors

such as solubility, size and affinity of adsorbent, surface area, pore volume and the phytoconstituents of the respective plant. At lower initial concentration the adsorption ability was significantly higher for ZnO-NPs-AS-*Os* than ZnO-NPs-AS-*AZi*. The percentage removal of As(III) for ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi* and are 92.38% and 89.47%. The percentage removal decreases with increase in initial As(III) concentration and the percentage removal of As(III) was almost complete nearly at 0.07 N, and 0.05 for the adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi)*. It reduces to 59.28% and 60.05% at 0.07 N and 0.05 for adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*). Marginally higher adsorption at lower concentration may be due to the high collision efficiency between the metal ions and the adsorbent. At higher concentration there may be lack of available sites for adsorbing As(III) ions on the adsorbent surfaces and may prevent further adsorption of As (III) ions. The hierarchy of adsorption of the adsorbent in terms of concentration is as follows: ZnO-NPs-AS-*Os* > ZnO-NPs-AS-*AZi*[15].

Effect of Adsorbent Dosage

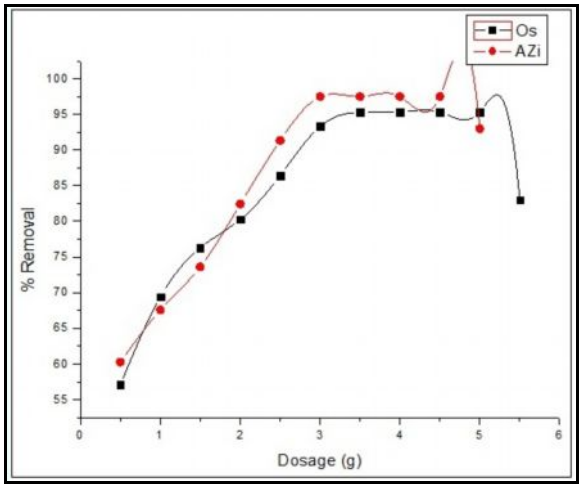

Figure 6 Comparative Effect of Adsorbent Dosage on the Adsorption of As(III) by Adsorbents

Figure 6 indicates the amount of adsorbent dosage on As(III) uptake from 2 different adsorbent (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*). Increase in adsorbent dosage from 0.5g to 6g increases the removal of As(III). At 0.5g the removal of As(III) is 57.11% for ZnO-NPs-AS-*Os* and 60.26% for ZnO-NPs-AS-*AZi*. The maximum removal reached upto 95.3% for ZnO-NPs-AS-*Os* and 97.5% for ZnO-NPs-AS-*AZi*. The hierarchy of adsorbent for As(III) removal is as follows: ZnO-NPs-AS-*AZi* > ZnO-NPs-AS-*Os* at 3g dosage. This means that at greater than 3.5g adsorbent dosage, adsorption of the adsorbent got lowered. Hence optimal adsorbent dosage for all adsorbent is selected to be between 3 to 3.5g[16-17].

Effect of pH

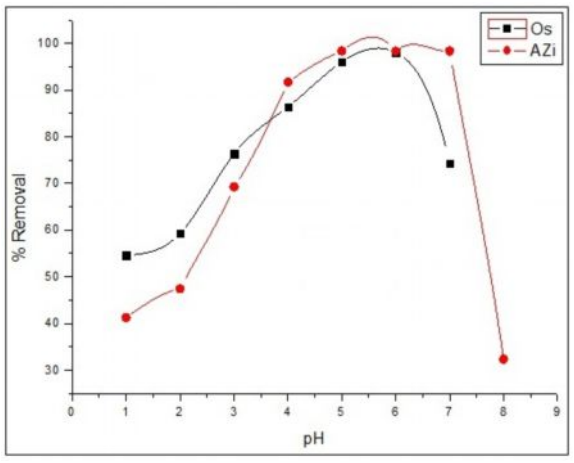

Figure 7 Comparative Effect of pH on the Adsorption of As(III) by Adsorbents

It is well known that adsorption of As(III) ions depend on pH of the aqueous solution. Figure 7 indicated the effect of pH on As(III) adsorption by two different adsorbent ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*. The pH of the solution was varied from 1 to 8 while As(III) removal was 54.58 % and 41.21% for ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi* at pH 1 respectively. This situation indicates that the adsorbent ZnO-NPs-AS-*Os* is not very different from ZnO-NPs-AS-*AZi*. The removal of As (III) increased rapidly at pH values from 5 to 6 and reached maximum upto 98.35% for ZnO-NPs-AS-*AZi*. The sequence is as follows: ZnO-NPs-AS-*AZi* >ZnO-NPs-AS-*Os* At pH 6 As(III) ions compete with each other for the surface of the adsorbent , which would hinder As(III) ions from reaching the binding sites of the sorbent caused by the respective forces. So the metal removal is minimum presumably due to the enhanced competition of proton with As(III) for ligand binding sites and complex formation. Hence at pH > 6 the As(III) ions get precipitated and the removal of As(III) gets decreased the optimal pH was selected to be 5-6 for all adsorbents[18].

Effect of Contact Time Six Adsorbents

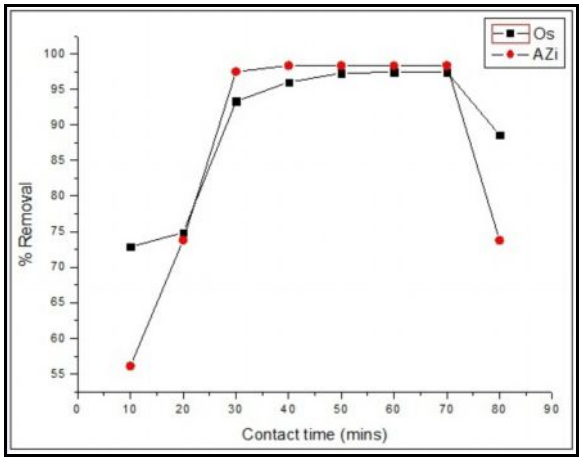

Figure 8 Comparative Effect of Contact Time on the Adsorption of As(III) by Adsorbents

The effect of contact time (Figure 8) on the adsorption of As(III) was investigated at different time intervals on the range of 10-90 minutes. The equilibrium adsorption was established rapidly within 90 min indicating that the initial adsorption was very fast and maximum uptake was reached within 50 minutes for ZnO-NPs-AS-*Os* with the maximum As(III) removal of 96.11% and 40 minutes for ZnO-NPs-AS-*AZI* with 98.35%. Maximum uptake of As(III) was reached within 40-60 minutes, thereafter the amount of adsorption remained almost constant. This was due to the decrease of adsorption sites on adsorbents which gradually interacted with the As(III) ions, but the adsorption rate depends on the As(III) ions, which transported from the bulk liquid phase to the actual adsorption site. In this study As(III) adsorbed onto ZnO-NPs-AS-*Os* is 97.29% and ZnO-NPs-AS-*AZi* is 98.35% .On the basis of these results, it can be observed that, ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi* can be used to remove metal ions .The removal capacity for different adsorbent was in the order of ZnO-NPs-AS-*AZi* > ZnO-NPs-AS-*Os*. It is indicated that ZnO-NPs-AS-*AZi* showed stronger preference for As(III) uptake than other adsorbents with a contact time of 40 minutes[19].

Effect of Agitation

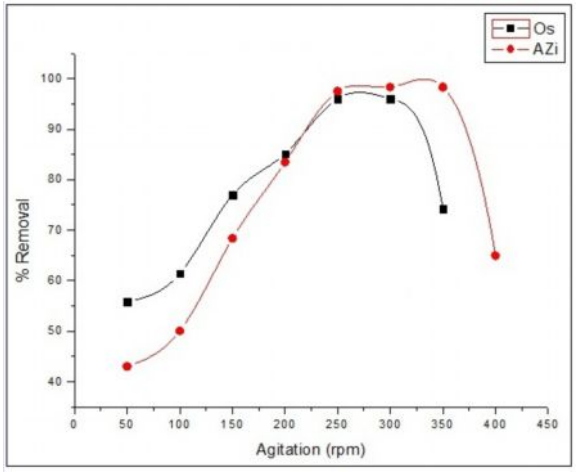

Figure 9 Comparative Effect of Agitation on the Adsorption of As(III) by Adsorbents

It was found from figure 9 that the rate of removal of As(III) increases with increase in agitation time to some extent. The initial rapid adsorption subsequently gives equilibrium within a period of 250 rpm to 300 rpm for both adsorbents. The variation in percentage removal of As(III) with agitation speed at different rpm ranging from 50 rpm to 500 rpm is shown in figure 9. It was observed that the maximum amount of As(III) adsorption takes place within 250 rpm to 300 rpm and it reaches equilibrium at 300 rpm for both adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*). After that no significant change was observed in the extent of adsorption. The percentage of As(III) removal increased from 55.85% and 43.03% for ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi* to 96.02% and 98.31%. This is due to the fact that with increase in agitation speed increases the mass film boundary of the adsorbents (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*) which inhibits adsorption .The hierarchy of adsorption of As(III) for adsorbents is as follows: ZnO-NPs-AS-*AZi* > ZnO-NPs-AS-*Os*[20] .

Equilibrium Study

Table 1 Comparison of Adsorption Isotherm Parameters

Equilibrium Isotherm	Equilibrium Parameters	Adsorbent ZnO-NPs-AS-*Os*	Adsorbent ZnO-NPs-AS-*AZI*
Freundlich	n	9.09	1.35
	K_F (L/g)	6.237	1.06
	R^2	**0.902**	**0.897**
Langmuir	K_L (L/mg)	66.66	45.45
	R_L	0.176	0.3055
	R^2	**0.992**	**0.974**
Tempkin	A	1.396	1.238
	B	0.025	0.025
	R^2	**0.906**	**0.943**
BET	R^2	**0.990**	**0.956**
	Surface Area (m^2/g)	300 m²/g	205.4 m²/g
	Pore size (nm)	29.048 Å	32.48 Å

In order to know As(III) adsorption behavior, the experimental data were applied to Freundlich, Langmuir, Tempkin Isotherm and BET. Table 1 shows the parameters obtained from different models which afford important information on the surface properties of the adsorbent and its affinity for adsorbate. Several conventional isotherm equation fitted to Freundlich, Langmuir, Tempkin Isotherm and BET. Linear correlation coefficients for these equations determined by linear regression for Freundlich, Langmuir, Tempkin Isotherm and BET showing that data correctly fits Langmuir isotherm than Freundlich isotherm ,Tempkin isotherm and

BET proving monolayer and homogenous surface of adsorbent with R^2 nearly 0.992 and 0.974 for (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*) adsorbents respectively. Table 1 shows the high fit of the Langmuir model among Freundlich, Tempkin Isotherm and BET. The choice of Langmuir model is confirmed by high R^2 value and R_L value (0.992, 0.974 and 0.176, 0.305) which is between 0 and 1. Hence it confirms the adsorption is favorable. Hence (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*) can be used as adsorbent because of its high adsorption capacity. The sequence of adsorption of adsorbent is as follows ZnO-NPs-AS-*Os* > ZnO-NPs-AS-*AZi* on the basis of high R^2 value proving Langmuir monolayer adsorption which is in consistent with SEM and EDAX[21-22].

Kinetic Study

Table 2 Comparison of Adsorption kinetics

Adsorption Kinetics	**Adsorbent ZnO-NPs-AS-*Os***	**Adsorbent ZnO-NPs-AS-*AZi***
Pseudo first order equation	0.711	0.132
Pseudo second order equation	0.982	0.906

Correlation coefficients for these equations determined by linear regression for all kinetic models, pseudo-first and pseudo-second order were worked out. It can be inferred from table 2 that adsorption did not obey well with the pseudo first order model because of the absence of linearity between $\ln(q_e-q_t)$ and t (R^2=0.711 and 0.132). In this study pseudo second model fitted better (R^2=0.982 and 0.906) when compared with first order kinetic model. Therefore the adsorption data in the present study supported chemisorptions. The linearity of the plots also showed the validity of pseudo-second order model. The adsorption rate (t) decreased with increasing solute concentration. The sequence of adsorbent is as follows ZnO-NPs-AS-*Os*> ZnO-NPs-AS-*AZi* [40-41].

Conclusion

This new-fangled green synthesis progress shows that the environmentally benign potent adsorbent (ZnO-NPs-AS-*Os* and ZnO-NPs-AS-*AZi*) for the removal of As(III) primed from leaf extracts of *Ocimum sanctum and Azadirachta indica.* Competently wrecked Zinc oxide nanoparticle ingrained on activated silica (ZnO-NPs-AS) using Zinc acetate dihydrate, sodium hydroxide and activated silica synthesised had assorted roles. The Adsorption parameters for the Langmuir, Freundlich, Temkin and BET isotherms were dogged and the constancy data were best described by Langmuir isotherm and fits pretty well with the experimental records with good correlation coefficient of 0.992 and 0.974. The data were analyzed using kinetics models analogous to Pseudo first and second order. All the findings accessible in this study suggested following Pseudo second order equation for the adsorption of As(III) on to ZnO-NPs ingrained on activated silica. From the standards it is consummate that the utmost adsorption corresponds to a saturated monolayer of As(III) molecules on the adsorbent surface persistently. The as synthesized particle was characterized with BET for surface pore, pore size and specific pore volume. The morphology of ZnO-NPs embedded on activated silica was confirmed by scanning electron microscopy (SEM). The sharp peaks by Energy Dispersive X-Ray Analysis (EDAX) pattern show the percentage purity and elemental composition of the sample. Green methods are being good competent for the chemical procedures, which are environment friendly and convenient. The results confirmed that adsorbent synthesized using aqueous leaf extract is a suitable green stencil to prepare ZNO-NPs embedded on activated silica to remove As (III) ions.

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