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Green and chemical method of activated silica as adsorbent for arsenic removal by zinc oxide nanoparticle modification

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ABSTRACT

Presently the progress of green chemistry in the synthesis of Zinc oxide nanoparticle entrenched on activated silica (ZnO-NPs-AS) using environmentally benign leaf extract of *Azadirachta indica,* Zinc acetate, sodium hydroxide and activated silica with diverse roles as adsorbent has fascinated a great attention. In this study, report on the exploit of activated silica (AS) as a proxy for adsorption using Chemical method. The present exploration describes the synthesis and characterization of ZnO-NPs-AS and AS prepared by green and chemical technique. Adsorption parameters for the Langmuir, Freundlich, Tempkin and BET isotherms were dogged and the equilibrium data were best described by Langmuir, Tempkin and BET model and fits quite well with the experimental data with good correlation coefficient of 0.974, 0.9430 and 0.9995 for ZnO-NPs-AS than AS. The results of intraparticle diffusion model suggested that intraparticle diffusion was not the rate controlling process. From the values it is accomplished that the maximum adsorption corresponds to a saturated monolayer of As^{3+} molecules on the adsorbent surface of ZnO-NPs-AS. The data were analysed using kinetics models analogous to Pseudo first and second order. All the findings presented in this study suggested following Pseudo second order equation for the adsorption of As^{3+} on to Zno-NPs-AS. The synthesized ZnO-NPs-AS and AS was characterized for BET surface area, pore size and specific pore volume. The sharp peaks by X-Ray Diffraction Analysis (XRD) pattern show the crystallinity and purity of the sample. Formation of ZNO-NPs-AS and AS was confirmed by scanning electron microscopy (SEM). Particle Size Analyser (PSA) shows the fashioned ZNO-NPs-AS and AS ranged in dimension of about 100 and 1000 nm. The results confirmed that aqueous leaf extract of *Azadirachta indica* is a suitable green template to prepare ZNO-NPs-AS for adsorption. Though chemical and green methods are trendier for adsorption process, the biogenic green fabrication of ZnO-NPs-AS is a better choice of adsorption due to eco-friendliness large surface area and high adsorption of 99% of As^{3+}.

Keywords: Adsorption, Isotherm, Kinetics, Nanoparticle, Activated silica, *Azadirachta indica*.

1. INTRODUCTION

Adsorption and adsorption processes are significant fields of cram in chemistry. Adsorption has gained extensive reception and reputation for taking away of pollutants because it is a well-organized and cost-effectively realistic process for decontamination. The consequence of adsorption in usual and engineering systems is well known. Study of adsorption equilibrium and kinetics are very imperative because their statistics are the origin for the assortment of appropriate adsorbent, intend of separation process, investigation of economical and so on. Even if the equilibrium properties of diverse adsorption systems are well carried out in literature but the kinetics of processes have obtained fewer attentions because of intricacy in carrying out of experiments and also the complexity of its hypothetical depiction in many systems. The ability to distinguish adsorption equilibrium accurately is important in a number of chemical processes, ranging from the study of adsorption at fluid interfaces to the design of heterogeneous chemical reactors. For multi component systems, the description of adsorption equilibrium can be quite difficult, as evidenced by the large body of literature in this field published over many decades. It is often most convenient if adsorption equilibrium are represented by explicit equations. Adsorption is present in many natural physical, biological and chemical systems and is widely used in industrial applications such as activated charcoal, capturing and using waste heat to provide cold water for air conditioning and other process requirements adsorption chillers, synthetic resins, increase storage capacity of carbide-derived carbons and purification. In particular, low cost adsorbents have drawn much attention and a significant amount of research has been carried out [1-3]. Silica gel is an adsorbent widely used in purification and separation of organic mixtures due to its adsorption properties high surface area and porosity [4]. Relatively, research has not been carried out using silica gel as a support for zinc oxide nanoparticles. In this study we are interested in modifying activated silica with zinc oxide nanoparticle for use in metal adsorption. This research demonstrates zinc oxide nanoparticle entrenched activated silica ZnO-NPs-AS found to have more surface area for As^{3+} removal than activated silica AS. To our knowledge activated silica entrenched Zinc oxide nanoparticle has never been used as a support for As^{3+} removal. In this work, activated silica and ZnO-NPs-AS were prepared and characterized and tested in adsorption studies of As^{3+}.The effect of concentration, adsorbent dosage, contact time, pH and agitation speed were studied for their ability to remove As^{3+}.

2. MATERIALS AND METHODS

2.1. Preparation of ZnO-NPs-AS and AS

Zinc acetate dihydrate (99% purity) and sodium hydroxide (pellet 99%) was used as the preparatory material and was supplied by Sigma-Aldrich chemicals, India. ZnO-NPs-AS structure was primed by green synthesis method. Aqueous leaf extract of *Azadirachta indica* was stirred for 30 min to that 1g of Zinc acetate dihydrate was added under vigorous stirring. After 1hr stirring 10 g of activated silica was introduced into the above solution followed by the addition of aqueous NaOH resulted in a white aqueous solution at pH 12. This was then sited in a magnetic stirrer for 2hr. The activated silica supported ZnO nanoparticle were then filtered and washed with double distilled water. The synthesized ZnO-NPs-AS was maintained at 60ºC for 12 hrs. A mortar was used to homogeneously ground the ZnO-NPs entrenched on activated Silica. The proposed sorbent were stored in air at room temperature. Activated silica is prepared by treating silica at a temperature of 500ºc for 6 h. It is believed that the thermal pretreatment increases the available active sites and increases adsorption capacities. The properties of coated silica (ZnO-NPs-AS) are clearly different from that of uncoated silica (AS) leading to a change in specific surface area and pore size and pore volume. The X-Ray powder diffraction pattern of the as-synthesized samples was recorded on an X-ray diffractometer (XRD, PW 3040/60 Philips X'Pert, Holland) using Cu (Kα) radiation (λ =1.5416 A°) operating at 40 kv and 30 mA with 2θ ranging from 10- 90°.The external morphology of the sample were characterized by scanning electron microscope (SEM) (LEO 1530FEGSEM).The surface characteristic and particle size distribution of ZnO-NPs-AS was investigated using Particle Size analyser (PSA, Zetasizer Ver.6.32).

2.2. Experimental details

The experiments were carried out as shown in table 1. The effect of various experimental parameters such as As^{3+} metal ion Concentration, Adsorbent dosage, Contact time, pH and Agitation speed has been investigated. To study the effect of certain parameter, that parameter has been changed progressively keeping the other four constant.

The quantity of As^{3+} adsorbed by ZnO-NPs-AS was calculated using the following formula:

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

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

Where C_o and C_e are initial and equilibrium concentration of As^{3+} respectively, q_e the amount of arsenic adsorbed, V the volume of the solution and W the weight of the adsorbent used.

2.2.1. Adsorption Isotherm

Equilibrium data commonly known as adsorption isotherm describe how the adsorbate interacts with adsorbents and give a comprehensive understanding of the nature of interaction. It is basically important to optimize the design of adsorption system. The parameter obtain from different models provide important information on the surface properties of the adsorbent and its affinity of adsorbent. Several conventional isotherm equations fitted to such as Langmiur, Freundlich,Tempkin and BET isotherm.

2.2.2. Freundlich Isotherm

The Freundlich expression is an empirical equation based on multilayer sorption to a heterogeneous surface and is expressed by the following equation:

$$q_e = K_F C_e^{1/n} \text{ or } \log q_e = \log K_F + 1/n \log C_e$$

Where q_e and C_e are the amount of adsorbed adsorbate per unit weight of adsorbent

Table - 1: Experimental details of ZnO-NPs-AS & AS for adsorption of As^{3+}

Effect of the System		Concentration (N)		Adsorption dosage (g)		Contact time (min)		pH		Agitation (rpm)	
		ZnO-NPs-AS	AS	ZnO-NPs-AS	AS	ZnO-NPs-AS	AS	ZnO-NPs-AS	AS	ZnO-NPs-AS	AS
Concentration (N)	ZnO-NPs-AS	0.005, 0.0075, 0.01, 0.02, 0.03, 0.04, 0.05, 0.06, 0.07, 0.08		0.05		30		4		100	
	AS		0.005, 0.0075, 0.01, 0.02, 0.03, 0.04, 0.05, 0.06		2.5		50		5		300
Adsorption dosage (g)	ZnO-NPs-AS	0.05		0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5		30		5		250	
	AS		0.02		0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4		50		5		300
Contact time (min)	ZnO-NPs-AS	0.05		3		10, 20, 30, 40, 50, 60, 70, 80		5		250	
	AS		0.02		2.5		10, 20, 30, 40, 50, 60, 70, 80		5		300
pH	ZnO-NPs-AS	0.05		3		30		1, 2, 3, 4, 5, 6, 7		250	
	AS		0.02		2.5		50		1, 2, 3, 4, 5, 6, 7, 8		300
Agitation (rpm)	ZnO-NPs-AS	0.05		3		30		5		50, 100, 150, 200, 250, 300, 350, 400	
	AS		0.02		2.5		50		5		50, 100, 150, 200, 250, 300, 350, 400, 450

and unadsorbed adsorbate concentration in solution at equilibrium respectively. And KF and 1/n are Freundlich constant characteristics of the system which are determined from the log qe vs. log Ce.

2.2.3. Langmuir adsorption

Langmuir monolayer adsorption isotherm is very useful for predicting adsorption capacities and also interpreting into mass transfer relationship. The isotherm can be written as follows:

$$C_e/q_e = (1/K_L) + (a_L/K_L)\ C_e$$

The constant K_L (L/g) is the Langmuir equilibrium constant, and the a_L/K_L gives the theoretical monolayer saturation capacity. These Langmuir parameters were obtained from the linear correlations between the values of C_e/q_e and C_e. Generally the Langmuir equation applies to the cases of adsorption on completely homogeneous surfaces.

2.2.4. Tempkin Isotherm

This isotherm describes the behavior of adsorption systems on heterogeneous surfaces and it has generally been applied in the following form:

$q_e = B \ln A + B \ln C_e$

A plot of q_e versus ln C_e enables to determine the constants A and B.

2.2.5. Kinetic Studies

The adsorption kinetics is important as it can predict the rate at which an As^{3+} is removed and provide valuable insights into the mechanism of sorption reactions. To study the rate constant for the adsorption of As^{3+} on ZnO-NPs-AS and AS the following kinetic models were tested to fit experimental data obtained. As^{3+} of initial concentration of 0.005 to 0.08 was treated at different contact durations of 10 to 80 min at pH 1 to 7and adsorbent dosage of 0.5 to 4.5g at an agitation speed of 50 to 400 rpm.

2.2.6. Pseudo first-order equation

The pseudo first-order equation is given as follows:

$\ln(q_e - q_t) = \ln q_e - k_1 t$

Where q_t and q_e are the amounts of As^{3+}adsorbed at time t and equilibrium respectively and k_1 is the pseudo first-order rate constant for the adsorption process. The linear graph of $\ln(q_e - q_t)$ vs. t shows the applicability of first order kinetic.

2.2.7. Pseudo second-order equation

This chemisorption kinetic rate equation is expressed as follows:

$t / q_t = (1/ k_2 q_e^2) + (1/q_e)t$

Where k_2 is the equilibrium rate constant of pseudo second order equation. The linearity of t / q_t vs t suggests the best fitted with pseudo-second order kinetic.

2.2.8 Intra-particle equation

Kinetic data can also be analyzed by an intra-particle diffusion kinetic model formulated as follows:

$q_t = k_p t^{1/2} + C$

Where k_p is the intra-particle diffusion rate constant and C is the intercept of the plot of q_t versus $t^{1/2}$. If this linear plot passes through the origin then intraparticle diffusion is the rate-controlling step.

3. RESULTS AND DISCUSSION

A distinctive XRD pattern of Figure 1 Shows the ZnO-NPs-AS prepared by the green synthesis method at 60˚C for 12 hours for aqueous leaf extract of *Azadirachta indica*.It can be seen that all of these peaks are well matched[5] with that of Zincite phase (JCPDS CARD NO: 36- 1451) equivalent characteristic peak predominantly at about 12˚, 20˚, 32˚, 34˚ and 36˚ are indicative of nano-crystalline nature of ZnO-NPs-AS. At the same time no diffraction peaks from other species could be detected which indicates that all the precursors have been completely decomposed and no other crystal products have been retained after the decomposition process. Figure 2 represents the AS distinctive XRD peaks for silica at 12ºC, 20ºC and 24 ºC. The crystalline sizes of the ZnO-NPs-AS prepared at 60˚c for 12 hours were observed to be 100nm and the size of AS was found to be 200nm which was determined by Debye Scherer's formula according to the equation (d=kλ /β cosθ), Where λ is the wavelength of incident X-ray is the Full Width Half Maximum (FWHM) of diffracted peak and θ is diffracted angle.

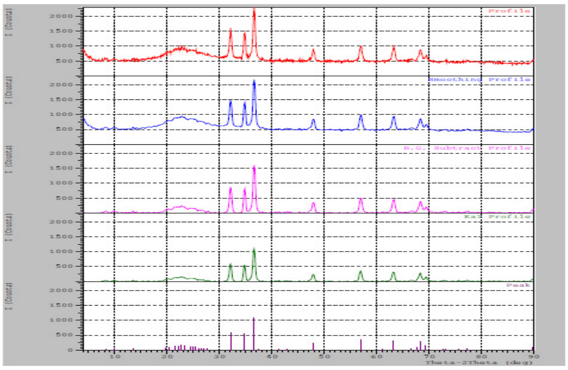


Figure - 1: Distinctive XRD Pattern of ZnO-NPs-AS. Figure 2 Distinctive XRD Pattern of AS.

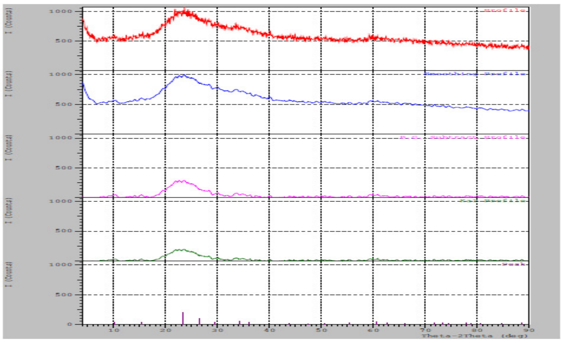
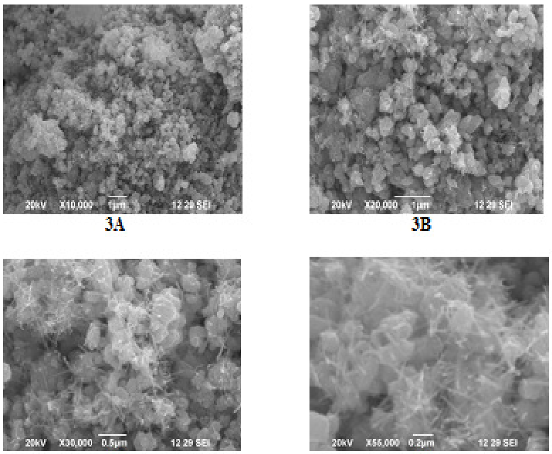


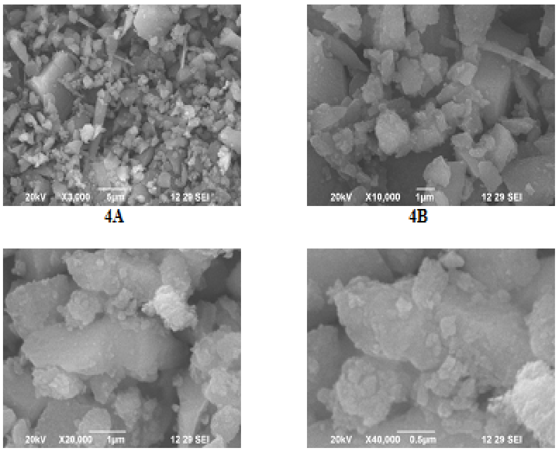
Figure - 2: Distinctive XRD Pattern of AS.

Figure 3 A, B, C, D and 4.A,B,C,D shows the SEM micrograph of homogeneous shape and size for ZnO-NPs-AS and AS at X10,000, X20,000, X30,000, X55,000 and X3000, X10,000, X20,000 and X40,000 which is the image of ZnO-NPs-AS obtained using aqueous leaf extract of *Azadirachta indica* and activated silica. The size and morphology of the obtained ZnO-NPs-AS and AS have been changed by increasing the magnification. The SEM image and Particle size (Figure 3 and Figure 4) distribution of ZnO-NPs-AS and AS is found to have the size ranging 100nm and 1900nm.



3A 3B 3C 3D

Figure - 3(A, B, C, D): SEM Micrograph of Homogeneous Shape and Size of ZnO-NPs-AS.



4A 4B 4C 4D

Figure - 4(A, B, C, D): SEM Micrograph of Homogeneous Shape and Size of AS.

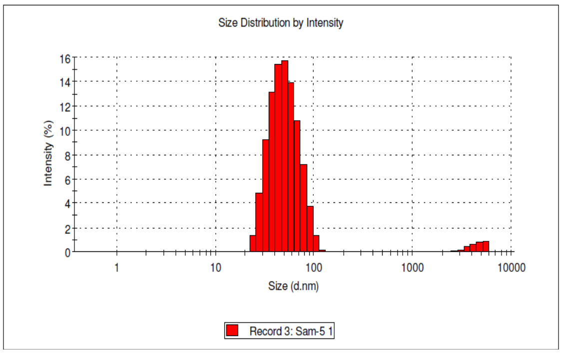


Figure - 5: Particle Size Distribution of ZnO-NPs-AS.

Size distribution by intensity using Particle size analyser reveals that the average particle size was found to be 52.34nm for ZnO-NPs-AS and 1942 nm for AS (Figure 4 and 5)

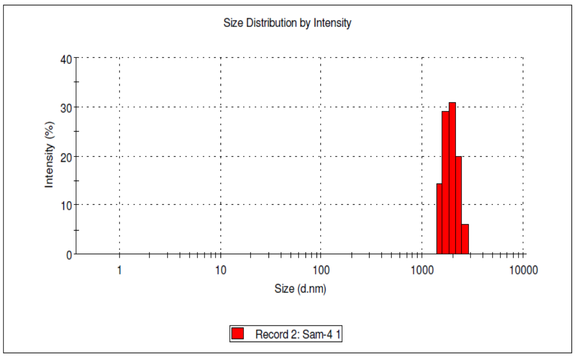


Figure - 6: Particle Size Distribution of AS.

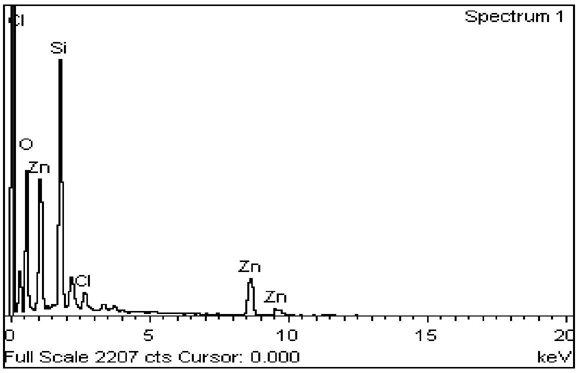


Figure -7: The EDX spectrum of ZnO-NPs-AS

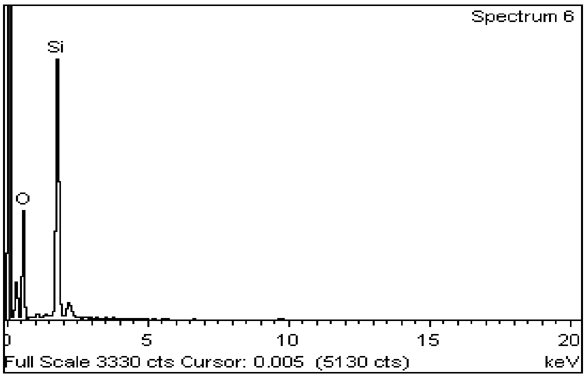


Figure - 8: The EDX spectrum of AS.

The EDX spectrum (Figure7) shows the peak only for the presence of Zinc, Oxygen silicon elements in the as-prepared ZnO-NPs-AS. The percentage of zinc, silica and oxygen is found to be 100% which proves that it is completely free from impurities. The EDX spectrum (Figure 8) confirms the peak only for the subsistence of silicon and Oxygen elements in the prepared AS. This confirms that smaller the particle size, greater the surface area and greater the adsorption. This result from Particle Size Analyzer confirms the higher adsorption of As^{3+} by ZnO-NPs-AS than AS.

The effect of concentration on the sorptive removal of As^{3+} is presented in the Figure

9. The results show gradual decrease in adsorption for ZnO-NPs-AS and AS from 89.47% to 60.05% and 79.6% to 47.40% with increase in concentration from 0.005M to 0.04M and then found to be constant. It was observed that amount of As^{3+} ions concentration adsorbed on ZnO-NPs-AS and AS increases with increase in the initial As^{3+} ions concentration in the solution. The percentage removal of As^{3+} was found to be greater for ZnO-NPs-AS than AS. On the other hand the percentage efficiency of As^{3+} ions removal was found to decrease with rise in the initial As^{3+} ions concentration in the solution depicted in Figure 9 which is in accordance with other researchers [6]. The decrease in percentage of adsorption of AS may be caused by the lack of sufficient surface area to accommodate much more As^{3+} ions available in the solution. Similar trend is also noted by other researchers [7].

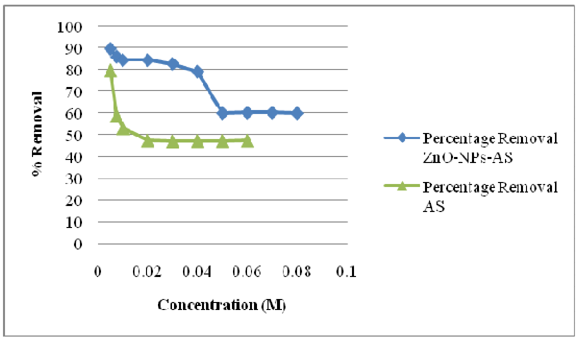


Figure - 9: Effect of Concentration of the Adsorption of As^{3+} by ZnO-NPs-AS vs AS.

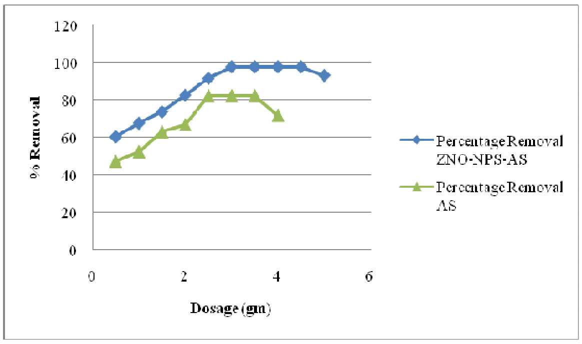


Figure - 10: Effect of Adsorbent Dosage on the Adsorption of As3+ by ZnO-NPs-AS vs AS.

Effect of adsorbent dosage (Figure 10) on the adsorption of As^{3+} is studied by changing the ZnO-NPs-AS dosage from 0.5gm to 5gm and the initial concentration of As^{3+} was fixed as 0.005M. The graph shows an increase in adsorption from 0.5gm to 3gm due to greater availability of the surface area at higher concentration of the adsorbent ZnO-NPs-AS. Any further addition of ZnO-NPs-AS beyond 3gm did not cause any significant change in the adsorption. This may be due to over lapping of adsorption sites as results of overcrowding of adsorbent particle. The effect of AS on the removal of As^{3+} ions was studied by varying the dosage from 0.5gm to 4gm. It was noted that the rate of removal increased from 0.5gm to 2.5gm and it attained the equilibrium. When the adsorbent dosage is higher it is more likely that a significant portion of the available active sites remain uncovered leading to lower specific uptake. The maximum removal of As^{3+} was obtained in the adsorbent dose of 3gm for ZnO-NPs-AS and 2.5gm for AS which is in accordance with other researchers [8, 9].

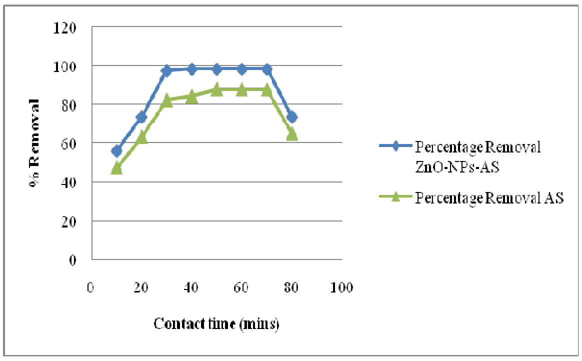


Figure - 11: Effect of Contact Time on the Adsorption of As^{3+} by ZnO-NPs-AS vs AS

The purpose of studying the effect of time on adsorption is to establish the equilibrium reaction time between adsorbent and As^{3+}. The adsorption experiment was carried out using contact time ranging from 10mins to 80mins for ZnO-NPs-AS and the results are depicted in figure 11. It was observed that metal adsorption occurred rapidly. The adsorption efficiency of As^{3+} increased gradually with increasing contact time up to 30mins and reached a plateau afterwards and there was no change in adsorption and the equilibration time is 30mins. It can be observed from the figure that the amount of As^{3+} ions adsorbed on the AS increases linearly up to 50min. But rate of uptake of As^{3+} ions lowered with further increase in the contact time. The equilibrium time of contact required to remove As^{3+} ions from the solution was observed to be 50min for AS in the present work. The data showed that time is a significant factor contributing largely to the adsorption under different sets of condition as time is required for As^{3+} to diffuse in to the ZnO-NPs-AS. Similar results were observed by other reporters [10, 11].

The pH is one of the important parameter controlling the removal of metal. It is clear from the Figure 12 that pH has a significant role on adsorption of metal using the synthesized ZnO-NPs-AS. It was found that increasing the pH of the solution from 1 to 6 increases the percentage removal of metal. This is due to the decline in the competition between proton and metals species for surface sites. Basic pH was also attempted but it could not be investigated due to precipitation.

The effect of pH on the adsorption capacity for AS solution was prepared at different pH values range from 1 to 8. The percentage of adsorption increases with increase in pH from 1 to 5 and remains constant thereafter. In the acidic range the positive surface charge of adsorbent increases and attract the negatively charged functional groups. At higher solution pH the surface charge of the adsorbent may get negatively charged which enhances the electrostatic force of attraction which is in accordance with other reporters [12, 13].

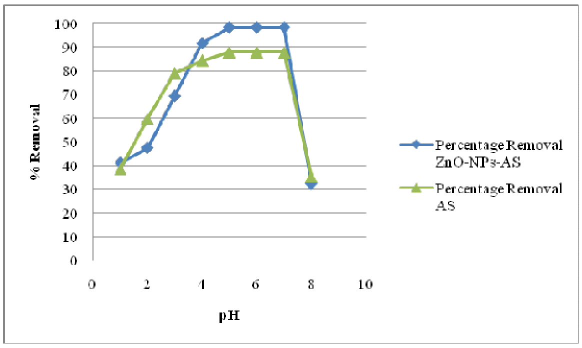


Figure - 12: Effect of pH on the Adsorption Of As^{3+} by ZnO-NPs-AS vs AS.

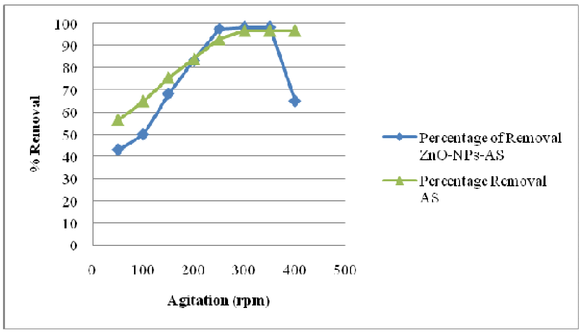


Figure - 13: Effect of Agitation on the Adsorption of As^{3+} by ZnO-NPs-AS vs AS.

The effect of agitation speed on the adsorption of ZnO-NPs-AS rate was investigated by changing the speed in the range 50 to 400 rpm. The rate of As^{3+} removal was very significant from 50rpm to 300 rpm. Increase in agitation makes the particle to collide with each other with a greater speed resulting in detachment of loosely bound ions and also they did not get appropriate time to interact with each other. By increasing the speed there was no further increase in adsorption. This is because all the binding sites have been utilized and no binding sites were available for further adsorption. An increasing agitation rate may reduce the film boundary layer surrounding the ZnO-NPs-AS [14]. The agitation speed varied from 50 to 500 rpm is carried out with a magnetic shaker. As agitating rate of adsorption increased from 50 to 500 rpm adsorption capacity of AS increases up to 300 rpm and then decreases is revealed from the figure 13. The effect of increasing the agitation rate was to decrease the film resistance to mass transfer surrounding the adsorbent particles.

3.1. Adsorption isotherm

The non-linear graph of Freundlich, Langmuir, Tempkin and BET isotherm models using ZnO-NPs-AS and AS was depicted in Table 2 below. It represented that the data were fitted to Langmuir, Tempkin and BET isotherm for ZnO-NPs-As and Langmuir, Freundlich, Tempkin and BET isotherm for AS. The parameters like Freundlich constants (KF and n), Langmuir constants (Qmaxand KL) and their linear correlation coefficient (r2) were mentioned in Table 2. Entire range studied with a good linear correlation coefficient, show that data correctly fit Langmuir isotherm Tempkin and BET isotherm for ZnO-NPs-As than Freundlich isotherm proving monolayer and homogenous surface of adsorbent. The same pattern was not observed for AS.

Table - 2: Adsorption Isotherm Parameters for the Adsorption of As^{3+} onto ZnO-NPs-AS and AS

Equilibrium Isotherm	Equilibrium Parameters	Adsorbent ZnO-NPs-AS	Adsorbent AS
Freundlich	n	0.131	0.374
	K_F (L/g)	0.941	1.856
	R^2	0.897	0.939
Langmuir	Q_{max} (g/g)	7.018	18.69
	K_L (L/mg)	0.022	0.155
	R^2	0.974	0.9658
Tempkin	A	0.025	0.030
	B	0.093	0.024
	R^2	0.943	0.962
BET	R^2	0.9995	0.9994
	Surface Area (m^2/g)	304.3573	164.2008
	Pore size (nm)	30.048	44.688

Table - 3: Adsorption kinetics for the adsorption of As^{3+} onto ZnO-NPs-AS and AS

Adsorption Kinetics	Adsorbent ZnO-NPs-AS	Adsorbent AS
Pseudo first order equation	0.132	0.188
Pseudo second order equation	0.906	0.732
Intra-particle Equation	0.208	0.111

Table 3 described kinetic parameters for the adsorption of As^{3+} onto ZnO-NPs-AS and AS, in which the correlation coefficients (r2) for the linear plots of pseudo-second order plots are better than the pseudo-first order reaction plots for ZnO-NPs-AS. This shows that the pseudo second order kinetic model explains the chemisorption in better way. The pseudo-second order model is based on the assumption that the rate determining step may be a chemical sorption involving valence forces through sharing or exchange of electrons between sorbent and sorbate and not intraparticle diffusion [15].

4. CONCLUSION

In this exertion ZnO-NPs-AS and AS were prepared and characterized and tested in adsorption studies of As^{3+}. The ZnO-NPs-AS has exposed superior adsorption capacities compared to other accessible non-conventional adsorbent AS. The various process parameters like initial concentration, adsorbent dosage, contact duration, pH and agitation speed were investigated in this study. Equilibrium and kinetic studies were carrying out for the adsorption of As^{3+} onto ZnO-NPs-AS and AS. The equilibrium data have been analyzed using Langmuir, Freundlich, Tempkin and BET isotherms. The characteristic parameters for each isotherm and related correlation coefficients have been determined. The Langmuir, Tempkin and BET isotherm for ZnO-NPs-AS shows good linear correlation coefficient than Freundlich isotherm and AS proving monolayer and homogenous surface of adsorbent. At the same way correlation coefficients were obtained for kinetic models like pseudo first- order, second-order model and intra particle diffusion equation. Intraparticle diffusion was not the rate controlling step. The pseudo second-order model is more fitted than first order. This reveals that pseudo-second order model is based on the assumption that the rate determining step may be a chemical sorption involving valence forces through sharing or exchange of electrons between sorbent and sorbate and not intraparticle diffusion. The properties of ZnO-NPs-AS are clearly different from that of AS leading to a change in specific surface area and pore size and pore volume. This research concludes that zinc oxide nanoparticle entrenched on activated silica ZnO-NPs-AS found to have more surface area for metal removal than activated silica AS.

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