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REMOVAL OF IRON (II) FROM POLLUTED WATER USING ZNO NANOPARTICLES

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Abstract
For the manufacture of ZnO nanoparticles, sol gel method was used. using ethanol as a solvent and zinc acetate as a primary material. sodium hydroxide was added in order to form zinc hydroxide. the synthesized hydrated zinc oxide nanoparticles were annealed at 600 °C in the air. Using field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy (EDX), the morphology of zinc oxide surface was examined. Fourier transform infrared (FTIR) analysis was used to characterize the functional groups. The Debye-Scherrer equation was used to calculate the size of the particles using X-ray powder diffraction (XRD). ZnO nanoparticles are created as a spherical structure with average (25-90) nm. These nanoparticles were successfully used in the investigation to act as adsorbents, and a high value of adsorption percentage reached to 97.9%.

Keywords: Removal of iron (II), Sol-gel method, Synthesis of ZnO nanoparticles,

Water pollution is one of the consequences of rapid development of industry and expansion of human population. Thus, this leads to exhausting of freshwater resources. Dumping of untreated industrial and domestic waste containing inorganic compounds in water bodies causes water-borne diseases. In addition, long-term excessive ingestion of these pollutants particularly heavy metal ions such as Chromium (Cr), Copper (Cu), Manganese (Mn), Nickel (Ni), Lead (Pb), Silver (Ag) and Cadmium (Cd) could damage kidney, liver, brain function, nervous system or even death[1,2].
Several techniques have been used to remove heavy metals from industrial wastewater including precipitations [3], ion exchange [4], membrane filtration [5,6], and adsorption [7,8]. Recently, increasing attention has been focused on metal oxide sorbents such as iron oxide, aluminum oxide, titanium oxide, manganese oxide, zirconium oxide. The nano sized metal oxides are classified as the promising ones for heavy metals removal from aqueous systems. This is because of their large surface areas and high activities caused by the size quantization effect [9,10]. However, these techniques do not eliminate the contaminants completely. Advanced oxidation process using heterogeneous photocatalysts such as titanium dioxide (TiO_2) and zinc oxide (ZnO) is another alternative technique which is extensively studied by researchers as it can remove both inorganic and organic contaminants simultaneously [11].

ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

In this work, ZnO particles was chosen because it is cheap and has good photocatalytic performance in heavy metal ions removal [12,13]. The high surface area of ZnO particles is responsible for adsorbing positive metal ions in the wastewater effectively. Most research has been focused on the reduction of toxic Cr (VI) by using ZnO photo catalyst [14,15]

RESULTS AND DISCUSSION

Characterization of ZnO Nanoparticles Using (SEM-EDX) Spectra

The prepared oxide was characterized by using (FESEM-EDX) the images show that ZnO oxide in nano size figure (1) shows the morphologies of ZnO nano oxide. ZnO nanoparticles appears at the range of (25-90) nm with distorted spherical shape [17].

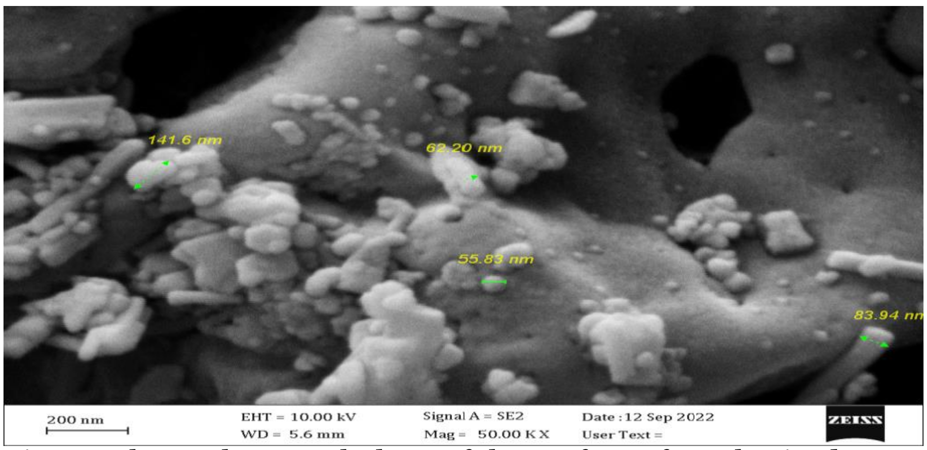

Fig. 1.FESEM image shows the morphology of the surface of synthesized ZnO nanoparticles

The EDX technique was used to examine the elemental analysis, the elemental analysis is performed as can be seen in figure (2). The percentage of ZnO Nano particles is 58.2% oxygen is 32.3 % [18].

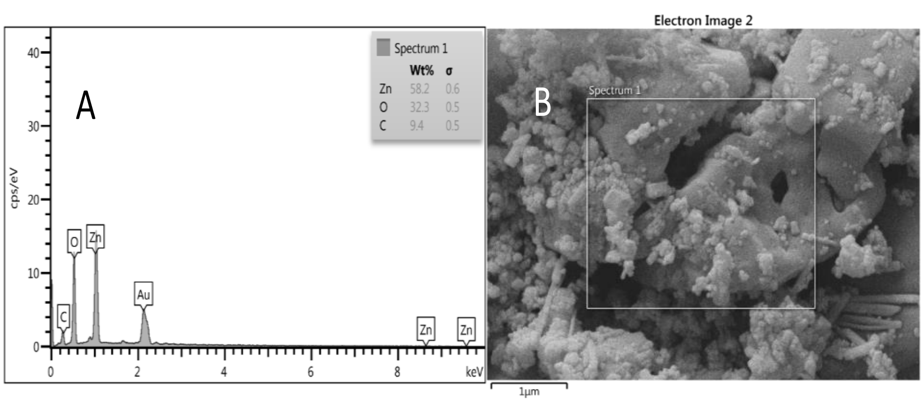

Fig. 2.EDX spectra for ZnO nanoparticles, (A) elemental analysis(B) the scanned area of the surface

Fourier Transform Infrared analysis (FTIR)

The functional groups of the Zinc oxide nanoparticles were diagnosis by using FTIR spectrum in the range from (400-4000) cm-1, where we notice from Figure (3) the appearance of an average peak at

ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

(3467) cm-1 as a result of OH-expansion associated with the surface of the materials of water molecules. It is a characteristic vibration of hydroxyl and the appearance of an average peak at (587) cm-1 and it characterizes the vibrations extending from the Zn-O bonds and the peak at 997-1005 cm-1 [19].

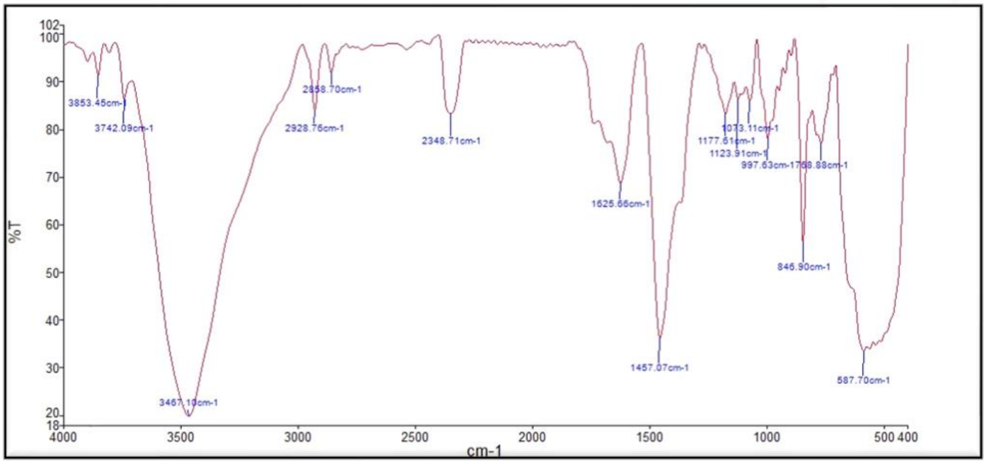

Fig. 2.FTIR analysis of ZnO nanoparticles

Powder Diffraction of X-rays

The X-Ray diffractogram and values of ZnO is determined to be in good agreement when associated to the standard JCPDS data. The synthesized sample is characterized for their structure by X-ray diffraction (Bruker AXS8) with (CuKα) radiation (λ = 1.5406 Å) by using Scherrer equation (1) [20]

D=0.9λ/β COSθ......... (1)

Hence, (D) represents the mean grain size, (λ) represents the wavelength (CuKα), (β) represents the full width at the half-maximum, and θ represents the diffraction angle. The X-ray diffraction peak caused by ZnO nanoparticles are shown in Figure (4). The average particle size of each sample was found to be (55–58) nm correspondingly [21].

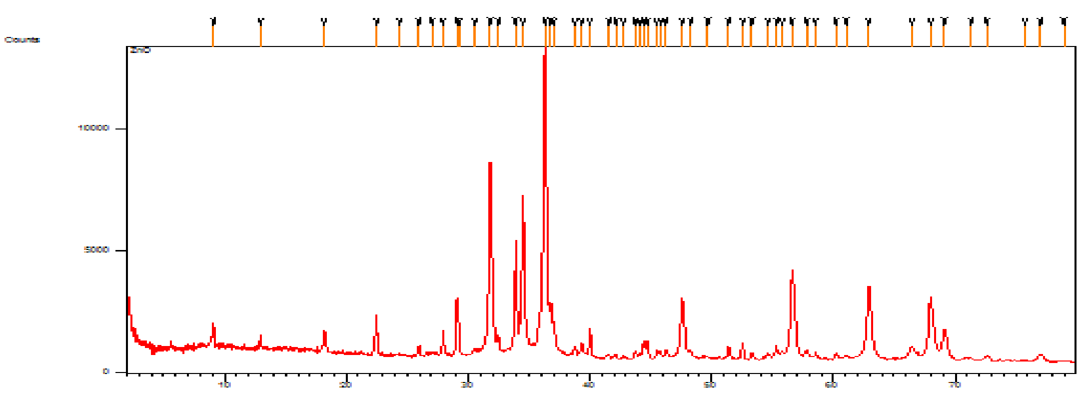

Fig.4. Shows the XRD pattern for ZnO nanoparticles

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ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

Application of ZnO Nanoparticles

The synthesized ZnO nanoparticles are used as an adsorbent material of adsorbed iron (II) from polluted water. Spectrophotometric method was used for determination of iron (II) before and after adsorption. Figure (5) shows the maximum wavelength of 1,10-phenanthroline with iron (II) at (510) nm. For calculation of percentage of adsorption of iron(II) before and after adding of ZnO nanoparticles.

The standard curve of iron-1,10-phenanthroline was determined in the range (5-25) nm

Figure 6 shows this study.

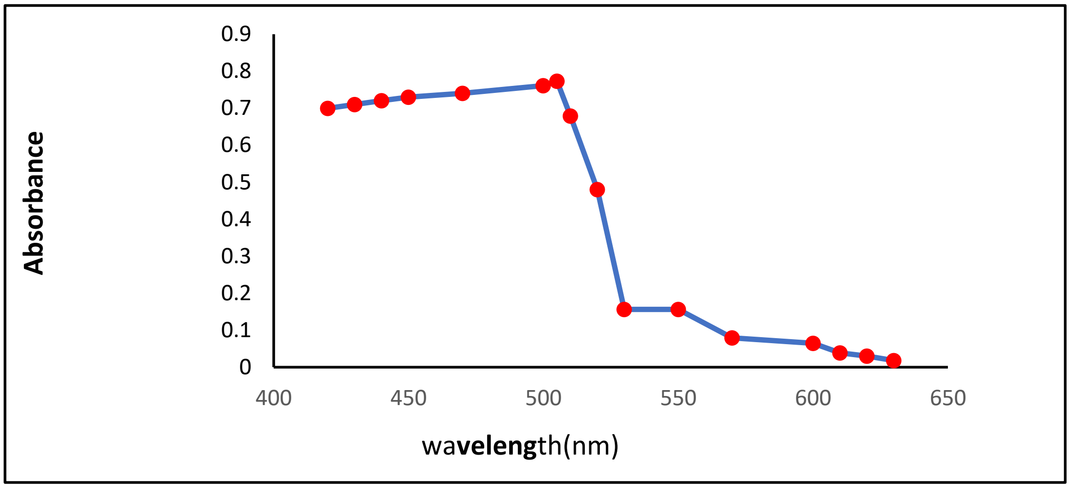

Fig. 5. The maximum wavelength of iron-1,10-phenanthroline using spectrophotometric method

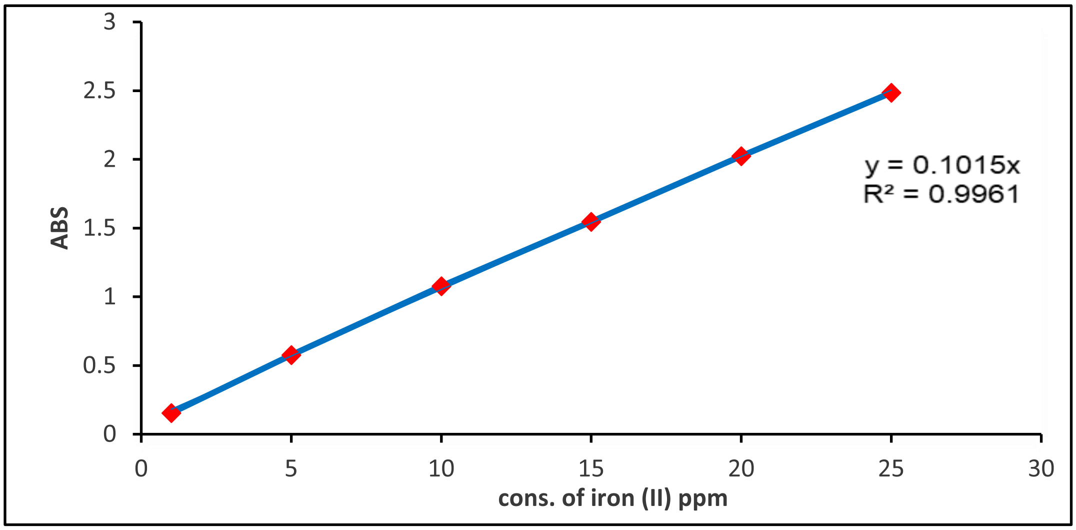

Fig. 6. Calibration curve of iron-1,10-phenanthroline using

ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

From the standard curve (10) ppm concentration from iron(II) was chosen for examine the adsorption percentage. The synthesized ZnO nanoparticles used as an adsorbent. The adsorption percentage of adsorption of iron was calculated using the equation (2). [22]

〖Ads%=(A〗_0-A_t/A_0)*100 .... (2)

The optimum condition of this experiment was studied before adsorption. Many parameters which affect the adsorption were studied such as equilibrium time, temperature, acidity function and initial adsorbent dosage.

The adsorption percentage was 98.7% using ZnO. From our knowledge this is the first time that zinc oxide nanoparticles used for releasing iron(II)from polluted water using spectrophotometric method and high percentage of removing was get.

Adsorption Studies

The degrees of remove heavy metals by ZnO nanoparticles was shown in figure 4. The results showed that ZnO nanoparticles have relatively high adsorption capacity owing to their larger surface area, which contributed to the increase in the number of adsorption sites for the heavy metals. In addition, ZnO have abundant surface functional groups.

Effect of Contact Time

Effect of contact time on the removal of iron ions by adsorption by ZnO nanoparticles was studied to define the equilibrium time for maximum absorption and to ascertain of the adsorption process. The relationship between sorbent and contact time was examined, and the findings seen in Figure 7. ZnO nanoparticles eliminated iron quickly, 15 minutes). In the early stages the effectiveness of ZnO nanoparticles at removing metal ions sorbent increase quickly because of the widespread availability [17,18]

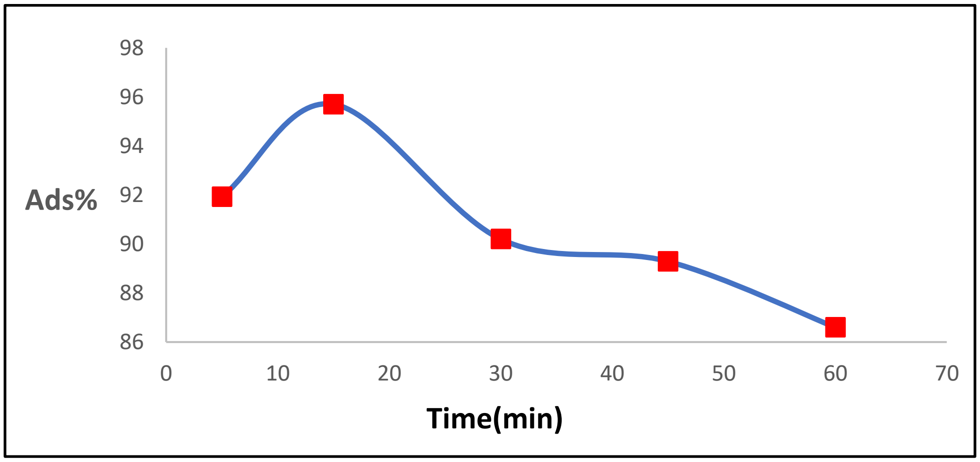

Fig.7.The optimum condition for adsorption of iron by ZnO nanoparticles shows the equilibrium time of adsorption is 15 min.

ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

Effect of the adsorbent dosage on iron ions removal

As shown in Figure 9, with increasing the amount of Nano ZnO sorbent (g), the metal uptake (q) will increase too and the % removal will increase too to a limit of (0.1 g) then it decreases again after the dose increases from (0.05 -1) gm, this behavior is expected since increasing the amount of sorbent will increase the area available for the adsorption and more metal ion is removed. The high adsorption capacity exhibited by ZnO nanoparticles may be explained by its nano-scale particle size giving access to a larger surface area to volume ratio [18,23].

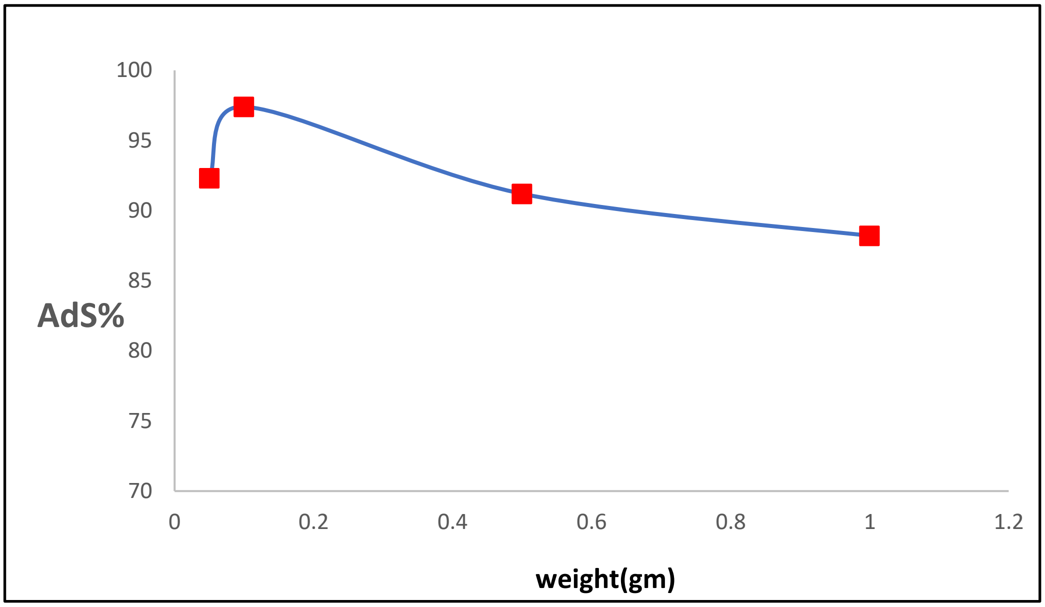

Fig.8. The optimum condition for adsorption of iron by ZnO nanoparticles shows the equilibrium weight of adsorbent is 0.1 gm

Effect of solution pH for iron ions removal

One of the most crucial factors that affect how a metal adsorbs is thought to be the hydrogen ion concentration in the adsorption process. in aqueous solutions of ions. It has an impact on the metal ions' solubility in part of the positive ions at the active sites are replaced by the solution. This influences howmuch the adsorbate is ionized

The pH function is considered one of the important factors in the study of adsorption because the change in the pH value affects the nature of the adsorbents and the adsorbents [24].

The acid functions with a range of (2.0-9.0) were studied for iron at a temperature of 25°C. The effect of the higher pH values of these ranges was not studied due to the deposition of elemental ions in the form of hydroxides. It was noted that the solutions of Fe (II) ions were fogged at the pH function (7.0 - 8.0).

Throughout the process. It affects the solubility of the metal ions in the solution, replaces some of the positive ions found in the active sites and affects the degree of ionization of the adsorbate during the

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ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

reaction [45]. The effect of initial pH on the sorption of Fe (II) ions onto Nano ZnO particles was evaluated within the pH range of 2-9. As after that the metal ions started to precipitate as metal hydroxides, the metal uptake was almost stable from the pH of 7 to 9 [46]. The effect of pH on the adsorption behavior of Nano CuO for Fe (III) is shown in Figures 6 and 7. The initial pH of the solution significantly affected the adsorption capacity of the adsorbent; adsorption capacity was highest when pH was 6 and decreased by either raising or lowering pH under the present range of the experimental condition. At lower values, the metal ion uptake was limited in this acidic medium, this can be attributed to the presence of (H+) ions which compete with the Fe (II) ions for the adsorption sites. On the other hand, the metal ion was prone to Fe (OH)3 deposition through hydrolysis at higher values of pH [47]. That was the reason that the metal uptake became almost constant as there were not extra metal ions to be removed after precipitation. This result is different from the normal pH range for iron to obtain the highest removal degree of iron; this can be contributed to the different of the adsorbent nature [17,18,23].

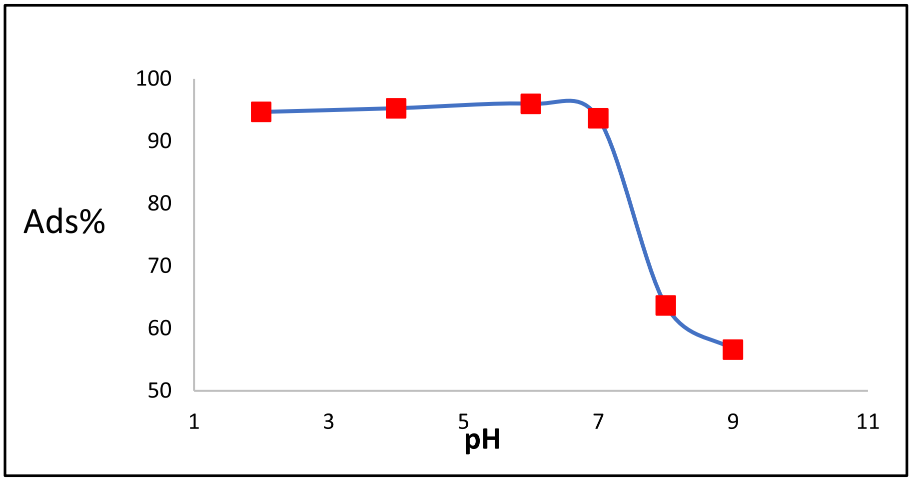

Fig. 9. The optimum condition for adsorption of iron by ZnO nanoparticles shows the equilibrium pH is 6

Effect of initial concentration

Initial concentration provides a crucial energetic force for enhancing contaminant molecule mass transfer resistances between the solid phases and aqueous solution. The adsorption rate is therefore enhanced by higher initial contaminant concentration, which necessitates a longer equilibrium time. The initial numbers of contaminating molecules to the exposed surface area ratio is frequently low at low concentrations. As a result, the initial contaminant concentration has no effect on the adsorption rate. However, at high concentrations, the vacant adsorption sites become fewer, hence the rate of contaminant removal is dependent on the initial concentration, when adsorbent dosages are constant [25,26].

ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

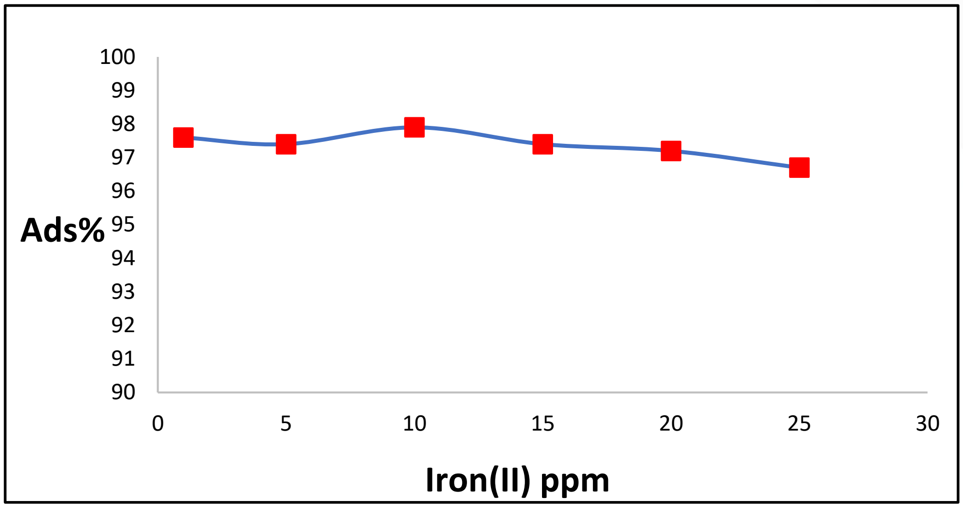

Fig.10.The optimum condition for adsorption of iron by ZnO nanoparticles shows the equilibrium of initial concentration (10ppm)

EXPERIMENTAL

The sol-gel technique is employed in this experiment. Materials used in this study include sodium hydroxide, ethanol, distilled water, and zinc acetate dihydrate. The precursor is zinc acetate dihydrate, while the reagent is methanol. As a solvent, distilled water is employed. All the chemical reagents used in this experiment were purchased from commercial sources as guaranteed-grade materials and employed straight away. In this work, ZnO nanoparticles are manufactured using the sol-gel method. As a zinc precursor in this experiment, zinc acetate dihydrate was used. mixing ethanol and 0.2M zinc acetate dihydrate at ambient temperature. Following a 120-minute ultrasonic mixing process at 25°C with this solution, a clean, transparent sol without any precipitate or turbidity was achieved. The sol was now given a 0.02 M NaOH addition, and it was ultrasonically agitated for 60 minutes. When white precipitation has settled to the bottom of the sol, leave it alone. The precipitate was filtered after precipitation and then washed with more ethanol to eliminate the starting material. On a heated plate, precipitates were dried for 15 minutes at 80°C. Following this, precipitates were annealed for 30 minutes at 400°C [16].

CONCLUSIONS

This bottom – up strategy provides a straightforward, quick, non-toxic, and economically feasible method for the synthesis of ZnO nanoparticles. Sol gel approach is successfully used to create ZnO nanoparticles with a crystallite size less than (54). By using X-ray diffractograms to determine their structure, these nanoparticles are identified as nanostructures. Using Debye Scherer's formula, it is determined that the crystallite size of ZnO nanoparticles is in Nano range. By using SEM-EDS, the elemental analysis is performed, and the morphology of the nanoparticles almost is spherical of oxide. The production of ZnO is confirmed also by Fourier Transform Infrared Spectroscopy. The study of

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using these nanoparticles as adsorbents were done successfully and the high value of adsorption percentage about 98% was get.

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CONFLICT OF INTEREST
No conflict of interest was declared by the authors

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ISSN: 2776-1010 Volume 3, Issue 11, Nov., 2022

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