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Original Research

Synthesis, characterization of nano-sized anatase TiO_2 and its adsorption behaviour for environmental contaminant

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Abstract: TiO_2 is one of the most studied material due to its unique properties like chemical stability, non toxicity and cost effectiveness. Nano-sized TiO_2 has been prepared by sol –gel method using titanium butoxide as precursor. X- ray diffraction analysis was used to characterize the phase and crystal size of the synthesized TiO_2 particles It was found that sizes of the particles ranged from 6 to 12 nanometre. FT-IR spectrum was also recorded for the TiO_2 particles to detect the remaining organic residue .Heavy metals like Cr, Cu, Fe, Ni etc are considered major environmental contaminant in water due to their non biodegradable nature and adverse health effect in living beings. In this work the adsorption behavior of heavy metals towards nano-sized TiO_2 was investigated.

Key words: Nano-sized TiO_2; Heavy metals.

Introduction

Heavy metal ion contamination in water is one of the various serious environmental issues since heavy metal ions are non-biodegradable and environmentally persistent (1, 2). Metals having density more than water or specific density 5g/cm^3 are considered as heavy metals including metalloids e.g. arsenic (3,4). Source of heavy metal ions in environment are both natural as well as anthropogenic. Natural sources include volcanic eruption, weathering etc. Anthropogenic sources are mainly mining, metal smelting and various industries involving metal processing (5). Long term exposure to heavy metal ion like Cd^{2+}, Cu^{2+}, Pb^{2+} etc. could cause detrimental effect on health of human beings (6-8). Iron is found both in ground water as well as industrial waste water predominantly as ferrous ions while at higher pH it exists in ferric state to be insoluble in aqueous phase. The maximum acceptable concentration of Iron (II) in drinking water recommended by World Health Organization (WHO) is 0.2 mg/L. The limit of Iron (II) into wastewater is 5 mg/L. The acute toxicity symptom for iron are vomiting, GI haemorrhage, cardiac depression, metabolic acidosis and chronic toxicity include hepatic cirrhosis (9).

Oxide based nanomaterial offer a promising tool for the water purification. Due to their small particle size and large surface area they show remarkable adsorption behavior towards heavy metal ions adsorption from the aqueous solution (10). Nano-crystalline TiO_2 is one of the most explored materials due to some of its unique properties like chemical stability, non-toxicity, chemical structure etc (11, 12). Sorption mechanism of various adsorbents depends upon the pH of the medium, duration of reaction, and concentration. The present work deals with the adsorption behavior of Fe^{2+} ions over nano-sized TiO_2.

Materials and methods

In the present study sol gel method was used for the preparation of nano-TiO_2 .All the used chemicals were of analytical grade. Atmospheric hydrolysis of titanium (IV) butoxide were carried out by dissolving in the toluene. The obtained gel was oven dried and the so obtained white powder was calcined at 500^{0}C for 5 hrs in microprocessor controlled muffle furnace.

Method

The aqueous solution of Fe^{2+} ions was prepared by using Ferrous ammonium sulphate 1.404 g of $Fe(NH_4)_2(SO_4).6H_2O$ taken in 20 ml of conc. H_2SO_4 was slowly added in to 50 ml of water .
0.1 N $KMnO_4$ was then added in to the solution until the faint pink color was maintained. This solution was further diluted to 1000 ml. to be used as stock iron solution.

The concentration of Fe^{2+} was analyzed spectrophotometrically by following the phenanthroline method as described in APHA (American public health association) standard methods for the examination of water and wastewater 22nd edition.

Batch studies/l

In the present study the adsorption experiments were carried out in a series of 100 ml Erlenmeyer flask which contained 50 ml of 0.1 ppm Fe^{2+} solution. Doses of nano TiO_2 were added in each flask ranging from 0.02 g/50ml to 0.1 g/50ml. The suspension was shaken with rotatory shaker for one hour at room temperature. Then the suspension was filtered with whatman (grade 42) filter paper to separate the solution from the undissolved solids. For determination of Fe^{2+}, 25 ml of filtrate was taken

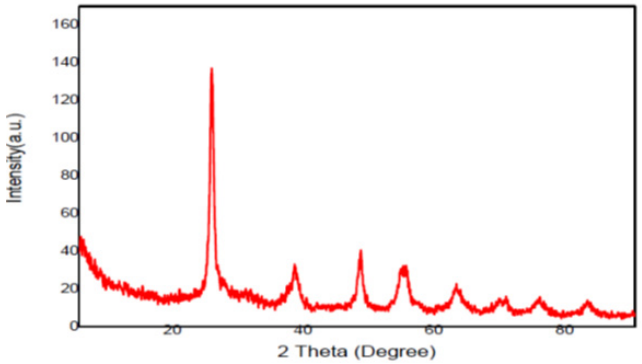

Figure 1. PXRD of TiO_2 Nanoparticles.

in 100 ml of volumetric flask in 4ml of hydroxylamine and 4 ml of phenanthroline were added to raise the volume up to 100 ml and kept for 10 minutes for maximum color development. During the experiment the pH of the solution was maintained within range of 2 to 3. Absorbance of this colored solution was recorded with the help of spectrophotometer and shown in figure 3.

Results and discussion

Characterization of adsorbent

For characterization of the synthesized material, PXRD (powder x-ray diffraction) pattern was carried out on a Rigaku smart lab x-ray diffractometer.The PXRD pattern of prepared material is shown in Figure 1. On analysis of this pattern with JCPDS file reported in literature indicates the formation of antase phase type of TiO_2. Particle size is calculated by Deby-Sherrer formula ranged from 10-15 nm.

FT-IR (Fourier Transform Infrared spectroscopy) spectrum (Figure 2) was obtained from PerkinElmer spectrum2 from the range 5000 to 500cm^{-1}.Peaks around 3400 cm^{-1} and 1630cm^{-1}in the spectrum are due to the stretching and bending vibrations of –OH groups. Absence of any peak at 2900cm^{-1} shows the complete removal of the organic residue remaining at this calcinations temperature.

Effect of adsorbent dose

As shown in plot, (Figure 4) with increase in the dose of nano TiO_2 the extent of absorption at 510 nm in UV-Vis spectrum decreased. The maximum decrease in absorption was found to be 0.08g/50ml indicating that at this dose maximum adsorption of Fe^{2+} occurs. Above this dose there is increase in absorption.

Conclusion

Nano- TiO_2 was successfully synthesized by sol gel method. PXRD analysis confirms anatase phase. The

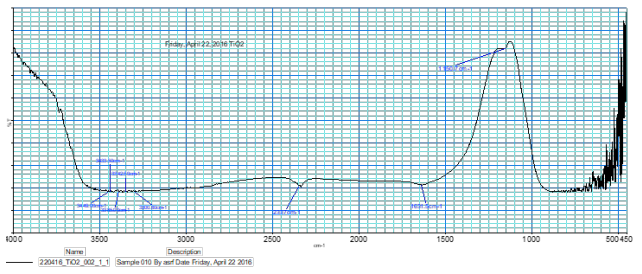

Figure 2. FT-IR of TiO_2 Nano particles.

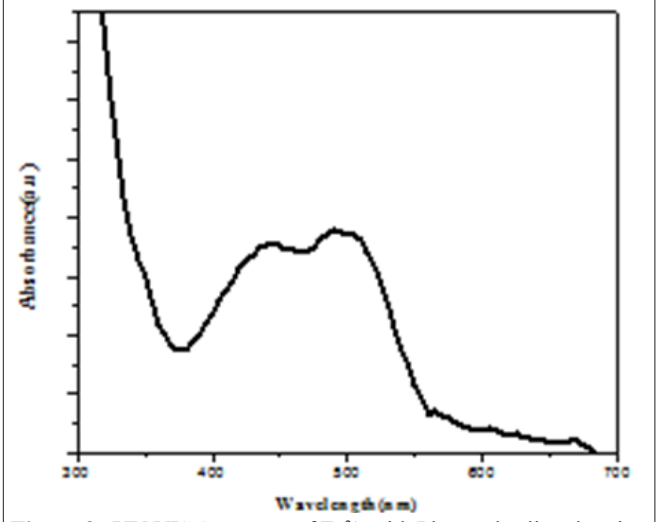

Figure 3. UV-VIS Spectrum of Fe^{2+} with Phenanthroline showing maximum absorbance at extent 510nm.

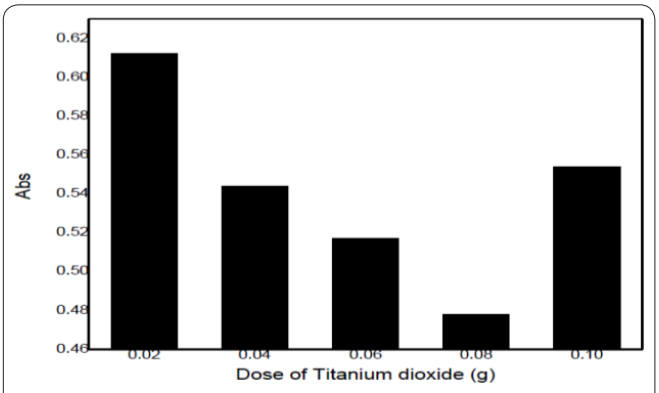

Figure 4. Dose of nano TiO_2 vs absorbance extent at 510nm.

adsorption of Fe^{2+} on the surface of TiO_2 increased as the dose of adsorbent increases at pH 2 to 3 because more surface area are available for adsorption.

X.Xie et al. reported that at lower pH the predominant adsorption process for TiO_2 is chemisorptions in which it exists as Ti-$OH_2^{½+}$. For Anatase, the surface energies of stable crystal plane (110) and (100)/ (010) are higher and higher surface energies make chemisorption's favourable. (13)

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