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PREPARATION OF Fe_3O_4 NANOPARTICLES USING TRIS (ACETYLACETONATO) *IRON* [III] AS SINGLE SOURCE PRECURSOR (SSP) AND ITS USE IN REMOVAL OF TOXIC CADMIUM

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ABSTRACT

The chemistry of transition metal chalcogenides are good candidates in material science due to their vast applications. Various synthetic strategies can be used to adjust the morphology, shape and size of material. Fe_3O_4 is a wide bandgap (2-3 eV) material and it has a wide range of uses, including those for drug delivery systems, magnetic resonance imaging (MRI), as a catalyst and as data storage material. There are 35 metals that are of concern for us because of residential or occupational exposure, out of which 23 are there. The Cadmium is a heavy metal which has considerable toxicity with impact on most organs. Long-term exposure to cadmium through air, water, soil, and food will leads to cancer and organ system toxicity such as skeletal, urinary, reproductive, cardiovascular, central and peripheral nervous, and respiratory systems. Cadmium levels can be measured in the blood, urine, hair, nail and saliva samples. Cd binds to DNA and impair vitamin D. It is also responsible for oxidative stress. Herein, we demonstrate preparation of Fe_3O_4 nanoparticles using *Tris*(acetylacetonato*)* iron[III]. The complex was characterised by

UV-vis and FTIR. The complex was used as single source precursor for preparation of Fe_3O_4 nanoparticles. The brown colour powder was characterized by powder XRD, SAED and TEM. The powder XRD shows (220) (311) (222) (422) (311) with cubic phase which matches with (JCPDS 19-0629). TEM image shows particles size from 20-30. Further, the prepared Fe_3O_4 nanoparticles shows Adsorption Cd in acidic pH which can subsequently eliminated.

KEYWORDS: Fe_3O_4 nanoparticles, Single Source Precursor, Cadmium toxicity.

Due to the very interesting physical and chemical properties of nanoparticles compared to bulk materials, nanoparticles have recently attracted a lot of attention.[1] Knowledge of coordination Compounds can be employed to design of Single Source Precursor which on decomposition that is pyrolysis in horizontal furnace is an easy way to prepare the nanoparticle.[2] The choice of metal-organic precursor that is single source precursor(cation and anion present in single molecule) over multiple source precursors (cation, anion, combustible compound, capping agent) plays a critical role in determining the final properties of the nanoparticles. The preparation of nanoparticles through the thermal decomposition of coordination complexes is indeed a fascinating area of research, as it allows for the production of materials with control stoichiometry, phase pure material specific characteristics and control over morphology. Co-ordination chemistry provides us new synthetic approach for preparation of complexes which after pyrolysis will subsequently give us materials which are having specific size, shape, morphology and stoichiometry as we have different ratios of cation to anion is possible but very few have application in designing final Divice1.[1,2,3,4] The various parameters such as precursor, annealing time, decomposition temperature are having impact on size, shape and morphology of nanoparticles. The process involves the decomposition of single source precursor at elevated temperatures, which leads to the formation of phase pure high pure nanoparticles with controlled stoichiometry. The rate of temperature increase during pyrolysis is also important parameter, as it can affect particle nucleation and growth. The molar ratio of the cation to anion precursor impacts the final nanoparticle's composition and uniformity. This can also influence the phase purity, stability, and functional properties of the nanoparticles. The shape and surface morphology of nanoparticles often determine their reactivity and interactions with surrounding materials which is key parameter in catalysis For instance, nanoparticles with specific shapes (e.g., spherical, cubic, or rod-like) may have different catalytic properties.[1,2]

The high surface area, larger area to volume ratio and specific morphology of nanoparticles make them excellent catalysts as more number of atoms on surface. Nanoparticles are used in the fabrication of semiconductors, sensors, and optoelectronic devices. Their size allows them to interact with biological systems at the cellular level, making them ideal for drug delivery and imaging transition metal chalcogenides Nanoparticles are employed followings areas advanced battery technologies, such as lithium-ion batteries, due to their improved conductivity and surface reactivity. The creation of microelectronic circuits, sensors for identifying various harmful gases, smart windows, piezoelectric devices, fuel cells, and super capacitors, coatings for passivating surfaces against corrosion, catalysts, and magnetic materials are all technological applications for transition metal oxides.[3] Iron oxides nanoparticles is immerging candidate since it have been investigated for decades from a variety of angles, both to satisfy the quest for fundamental understanding and because of their potential use in emerging technologies. The main attention has switched over time to magnetic and non-magnetic iron oxide nanoparticles because of their high surface-to-volume ratio and different physical and chemical characteristics from bulk systems. Iron oxide nanoparticles are specifically used in drug imaging (MRI), in cancer therapy, as a corrosion protective pigments in paints and coatings, in protein purification biological separation, as a catalyst and in delivery systems, as magnetic resonance storage material and in spintronic based devices. They show superparamagnetism, i.e. their magnetization is zero, in the absence of an external magnetic field and they can be magnetized by an external magnetic source. The band gap of Fe_3 O_4 is 2.00-3.00ev.[3,5] Figue.1shows different stichometry's of Fe to O atom which depends on preparation condition and thermodynamic parameters, hence design strategy single molecule precursors gives as different form of Fe and O atom.

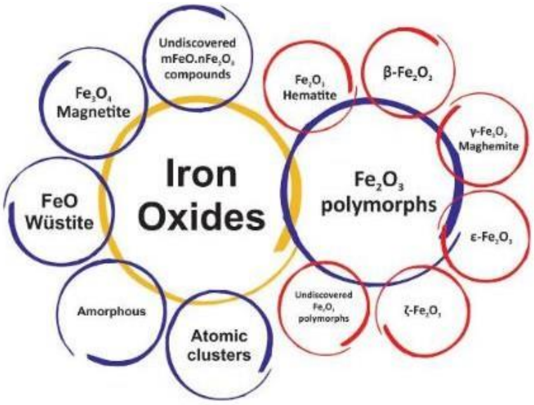

Figure 1: Different Forms of Fe and O atom.

Cadmium (Cd) atomic number forty-eight is an earth's crust natural occurring element and is usually found as a mineral in combination with other elements from p block such as oxygen, chlorine, or sulfur. Cadmium (Cd) toxicity is a serious health concern, with exposure primarily occurring through inhalation, food, and water, leading to oxidative stress and other various health problems, including kidney damage, cancer, and cardiovascular issues. Industries like mining, smelting, and battery manufacturing expose workers to cadmium, Cadmium can contaminate soil and water by all means entered in food chain leading to exposure through food and water consumption. Smoking is a significant source of cadmium exposure, as the lungs absorb cadmium more efficiently than the stomach Cadmium can accumulate in certain foods, such as cereals, vegetables, nuts, and meat.[7,8] Cadmium accumulates in the kidneys and can cause following diseases like renal tubular damage, kidney stones, and emphysema. Cadmium is classified as a human carcinogen and also responsible for oxidative stress and is linked to increased risk of certain cancers, including internal organs like lung, kidney, and bone cancer as it can replace Calcium in bones as both are having oxidation state plus two. Cadmium exposure can contribute to cardiovascular problems, including high blood pressure ionophores can replace Calcium is present in blood having important function as blood clotting along with structure role as bone and teeth's.

Acute exposure to cadmium fumes can cause flu-like symptoms, tracheobronchitis, pneumonitis, and pulmonary edema. Cadmium can also affect the skeletal, reproductive, and nervous systems which leads to oxidative stress. Further Cadmium induces oxidative stress, leading to damage to cells and tissues hence set back to immune system.

Cadmium interferes with various physiological and biochemical processes, including enzyme activities, photosynthesis, and nutrient uptake. Cadmium can induce epigenetic changes in DNA expression that is alter and damage irreversibly genetic information of species is stored, potentially leading to cancer mutation of cell, normal functioning of cell along with other diseases related to various physiological healthy functioning of body. Cadmium is classified as a human carcinogen and is linked to cancers such as lung, kidney, reproductive organs such as prostate, pancreas, breast, and bladder cancer. Chronic exposure leads to renal tubular dysfunction and kidney failure. The kidneys are particularly sensitive to Cd accumulation, which is slow to excrete and can remain in the body for decades Inhalation exposure can cause lung diseases, including emphysema, chronic bronchitis, and lung cancer. Cd exposure is associated with osteoporosis that is bone became brittle leads to factures osteomalacia, and Itai-Itai disease, causing painful bone disorders and fractures Long-term exposure can damage the liver, affect the nervous system (leading to cognitive and behavioral issues), impair immunity, retard growth, and cause hypertension. Acute poisoning can result in severe gastrointestinal symptoms, muscle cramps, dizziness, shock, and convulsions. Cd can cause epigenetic changes, DNA damage, mutations, modifications of DNA and inhibit enzymatic activity critical for cell function and respiration as most of metalloenzymes have bivalent active centers Cd can cross the placenta, potentially causing teratogenic effects and risks to new born. Cd released from industrial processes, mining, fertilizers, and electronic waste accumulates in soil and water contaminate environment. Its high solubility makes it mobile in the environment and persistent due to its long half-life. Cd is taken up by plants and accumulates in animals (especially mollusks and crustaceans), entering the food chains. Cd is highly toxic to aquatic organisms and can impair growth, reproduction, and survival. In soil, it inhibits plant growth and disrupts nutrient cycling. Cd can travel long distances as an air pollutant, depositing far from its original emission source and contaminating remote ecosystems. Measures include minimizing industrial emissions, safe recycling of Cd-containing products (such as Ni Cd rechargeable batteries), Cadmium is a dangerous heavy metal with severe long-term effects on human health and the environment, necessitating robust management, public awareness, and regulatory control we thought that iron oxide nanoparticles are best candidate to monitor Cd toxicity and its removal as itself is biocompatible. Figure.2 indicates the oxidative stress and other toxic hazards to living systems.[7,8,9]

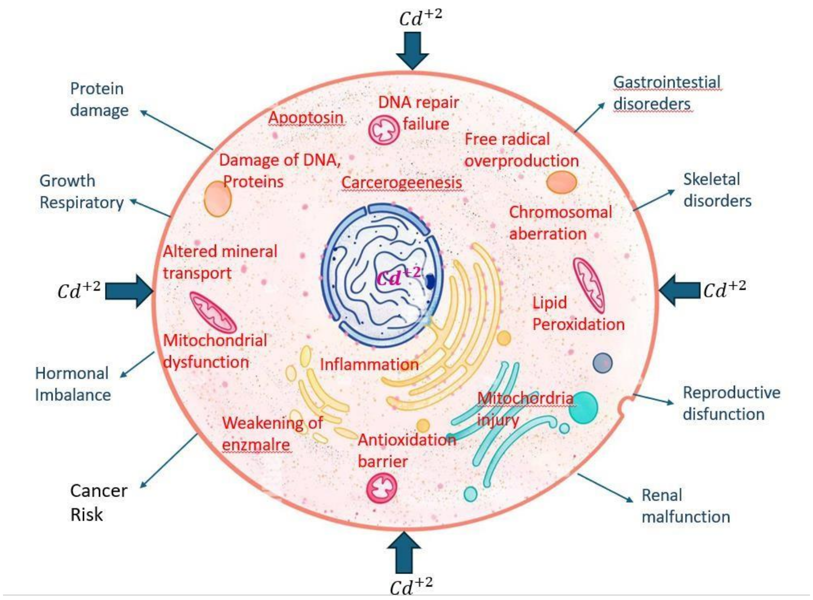

Figure 2: Toxic effect of Cadmium to living cell.

Transition metal complexes with various ligands have been reported namely semicarbazone, curcumin, 2-hydroxy-1-naphthaldehydedato, p-hydroxybenzoate and diketones reported.[6] Here in we report bidented ligand acetylacetonato forms co-ordinate bond through oxygen atom.

Preparation of single source precursor

In typical synthesis of single source precursor 1g of ferric chloride was dissolved in minimum quantity of conc. HCl in a 250 cm^3 beaker and heat it to about 60^{o}C. Add 10 cm^3 of acetylacetone ligand solution drop by drop by continuous stirring to metal salt solution further stir reaction mixture for 20 minutes on ice bath. Add liquor ammonia drop wise till distinct smell of ammonia. Deep red coloured Tris(acetylacetonato) iron[III] complex gradually forms, keep reaction mixture in ice bath for 20 minutes. Filter the complex using Whatman filter paper no.1 using Büchner funnel.(Fig.3). The single source precursor (SSP) was dissolved in ethanol UV spectra was carried out in using Perkin Elmer, model-LAMBDA 25 which showed the λmax at 270 nm in the UV region From(Fig.4) FTIR spectra Fe(acac)$_3$ shows following peaks at 1572 cm $^{-1}$ for ν(C=O), 1524 cm $^{-1}$ for ν(C=C), 1361 cm $^{-1}$ for δ s (C-H), and 1275 cm $^{-1}$ for δ(C=C-H). 29 These peaks, which are characteristic of the acac group indicating co- ordination Via oxygen atom.(Fig.5). from spectral data the proposed structure of SSP(fig. 6)

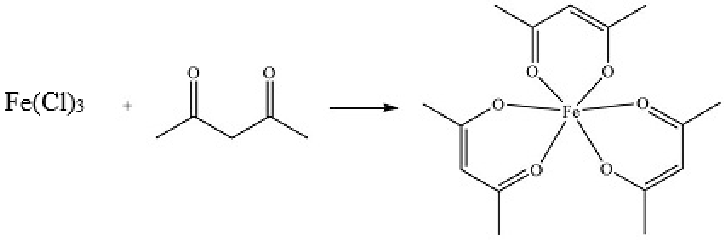

Figure 3: Preparation of Tris(acetylacetonato) iron[III] Single Source Precursor.

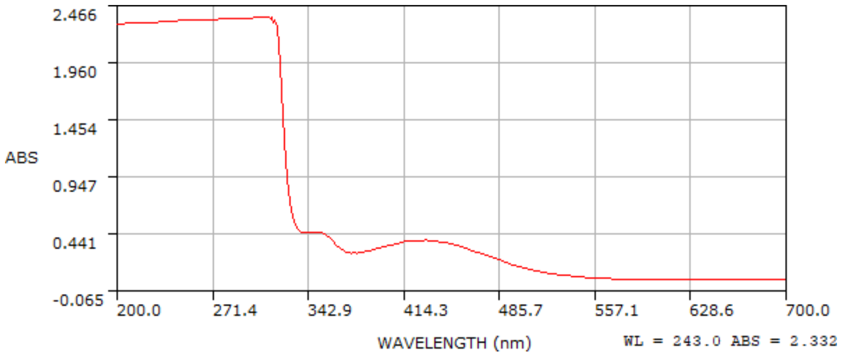

Figure 4: UV-VIS spectra of Tris(acetylacetonato) iron[III] Single Source Precursor.

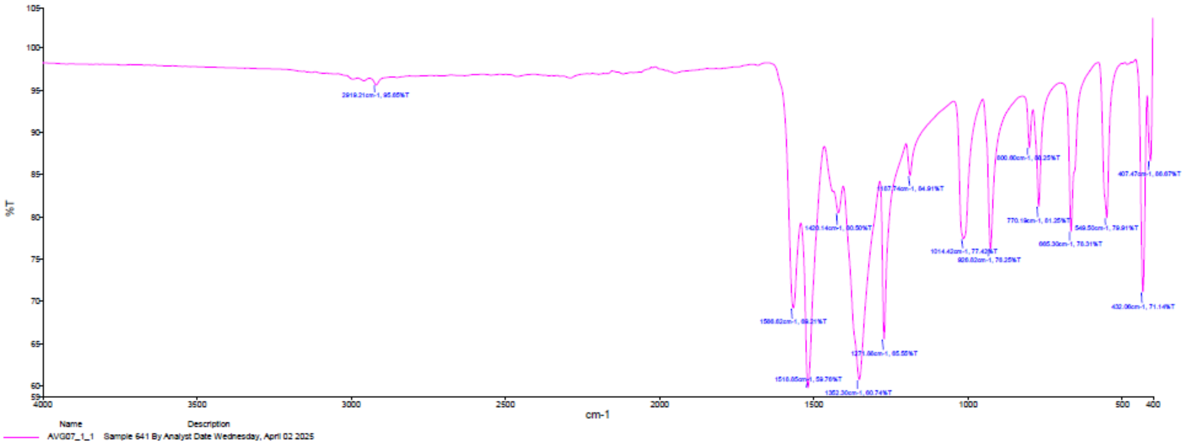

Figure 5: FTIR spectra of Tris (acetylacetonato) iron[III] Single Source Precursor.

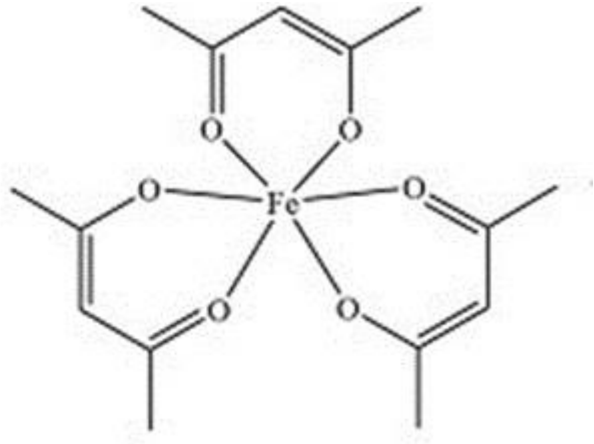

Figure 6: structure Tris(acetylacetonato) iron[III] Single Source Precursor.

Synthesis of Fe_3O_4 nanoparticles

In typical synthesis 0.370 g of Tris(acetylacetonato) iron[III] Single Source Precursor complex was weighed, transferred in silica crucible and placed in horizontal furnace. Temperature was raised to 490^{o}C and it was kept in the furnace at same temperature for annealing process for two hours. After two hours, furnace was allowed to attain room temperature. The brown coloured product which weighed 0.079 g. The practical yield was in agreement with theoretical yield.(Fig.7). The Powder XRD of brown coloured nanoparticles (Fig.7) was carried out by using Panalytical Xpert PRO X Ray Diffractometer, Model: Xpert Pro MPD, Anode: Copper, Wavelength: 1.5405Angstorm, Power: 40KV / 30mA, Detector: Xcelerator Detector with Diffracted Beam Monochromator. It showed the peaks a Peaks matches with (111)(220)(311)(400)(422)(511)(440)(JCPDS 19-0629)Fe_3O_4 Cubic phase.(Fig.8) TEM analysis was carried out by sonicating 5 mg of nanoparticles in 10 cm3 of methanol for half an hour. 2 drops was taken on copper grid by using TEM CM 200, Make:

PHILIPS, Model: CM 200, Operating voltages: 20-200 kv. Grid was air dried and the images were recorded TEM images Fe_3O_4 showing particle size20-30nm(Fig.9) HRTEM images Fe_3O_4 showing Cubic Phase Pure Material(Fig.10)

Sample quantity	370 mg
Atmosphere	air
Decomposition temp.	490°C
Annealing	2h

Figure 7: Decomposition parameters for Tris(acetylacetonato) iron[III] Single Source Precursor.

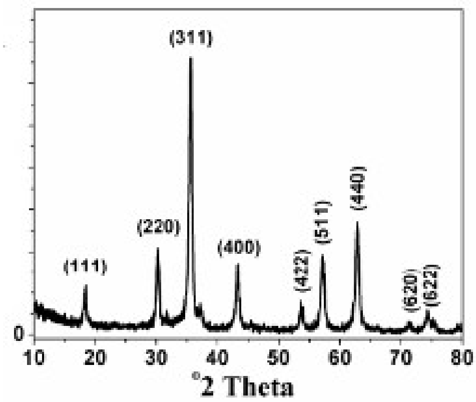

Figure 8: Peaks matches with (111)(220)(311)(400)(422)(511)(440)(JCPDS 19-629)Fe_3O_4 Cubic phase.

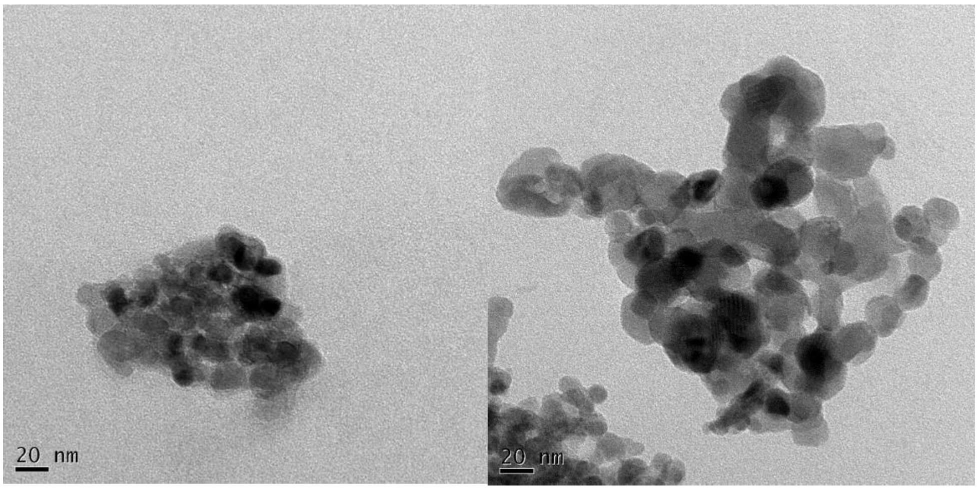

Figure 9: TEM images Fe_3O_4 showing particle size20-30nm.

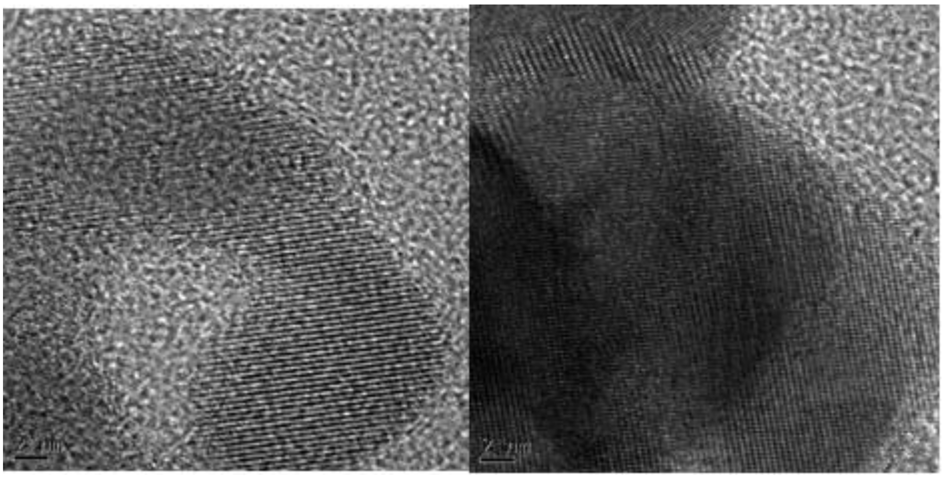

Figure 10: HRTEM images Fe_3O_4 showing Phase Pure Material.

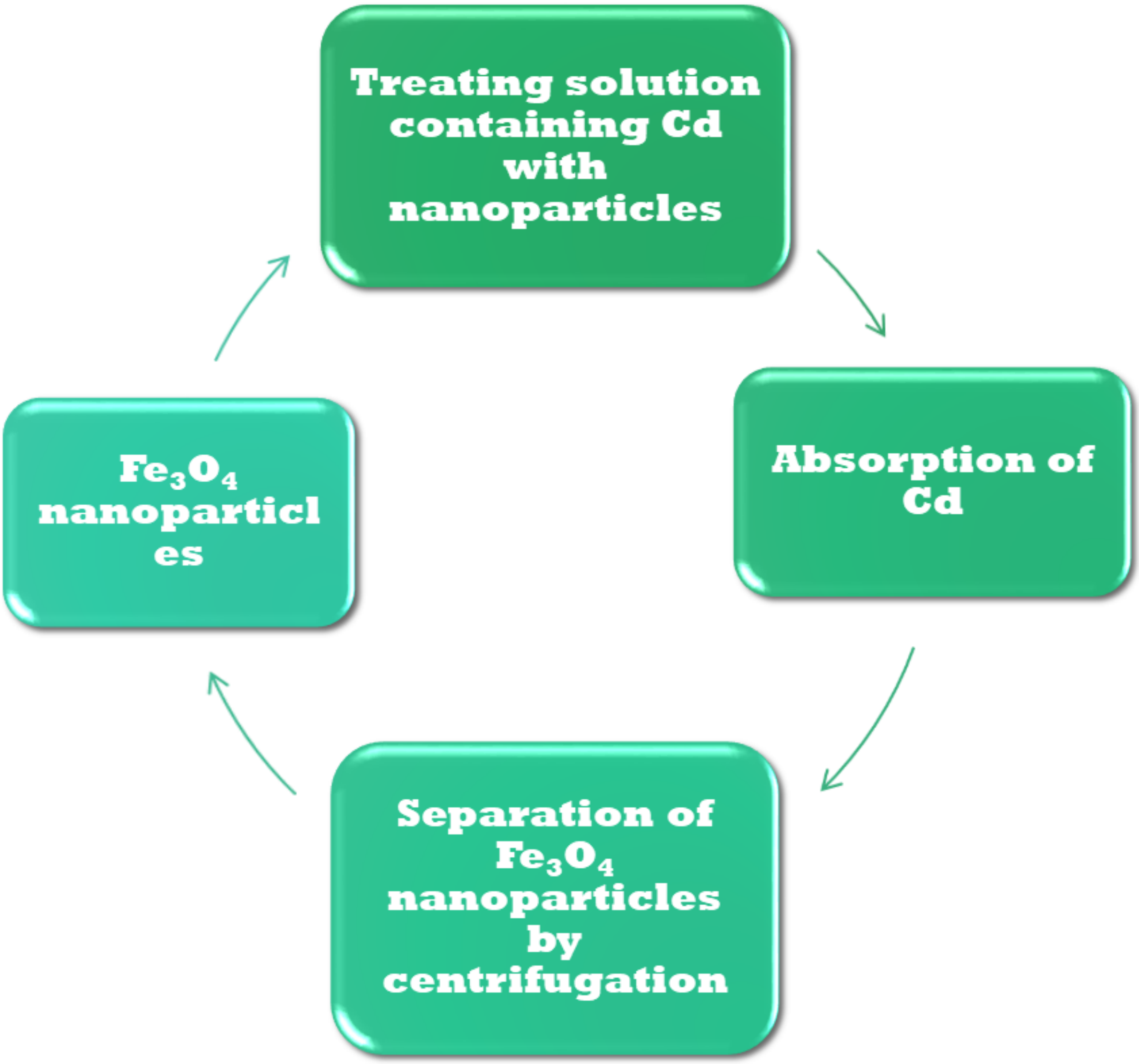

Figure 11a: Use of Fe_3O_4 nanoparticles for removal of toxic Cd.

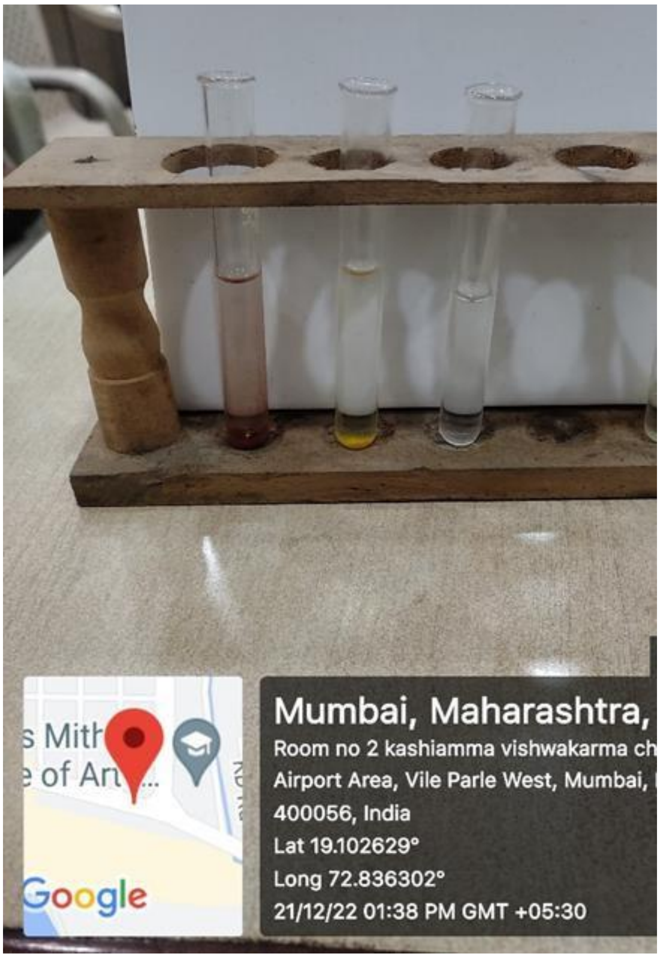

Figure 11b: Use of Fe_3O_4 nanoparticles for removal of toxic Cd precipitated as CdS.

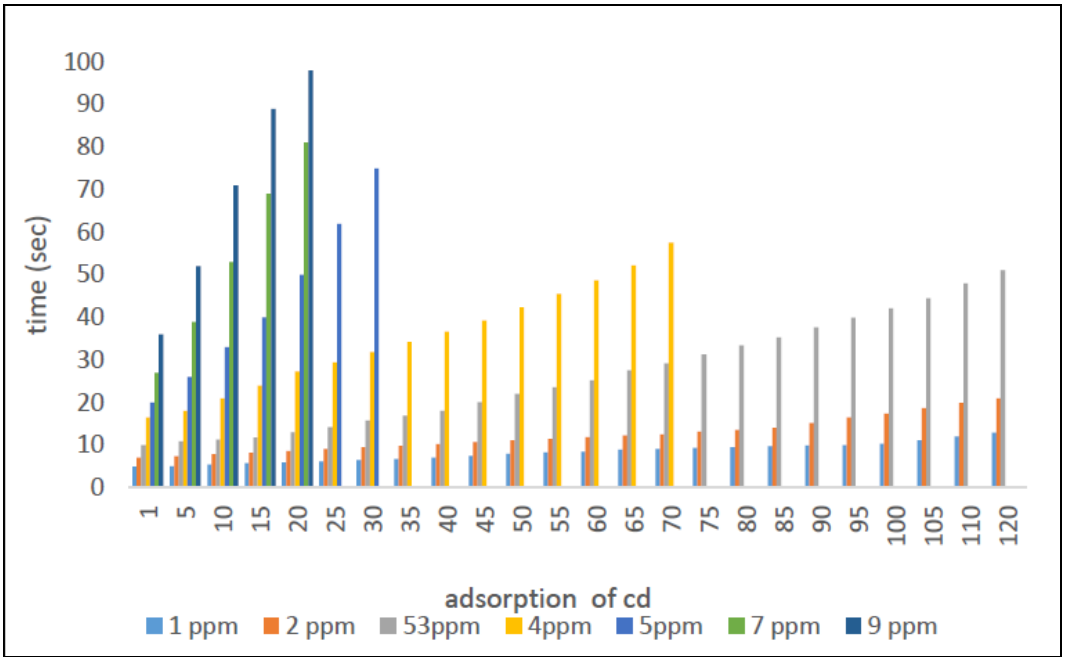

Figure 11c: Use of Fe_3O_4 nanoparticles for removal of toxic Cd precipitated as CdS for 1-9 ppm solutions.

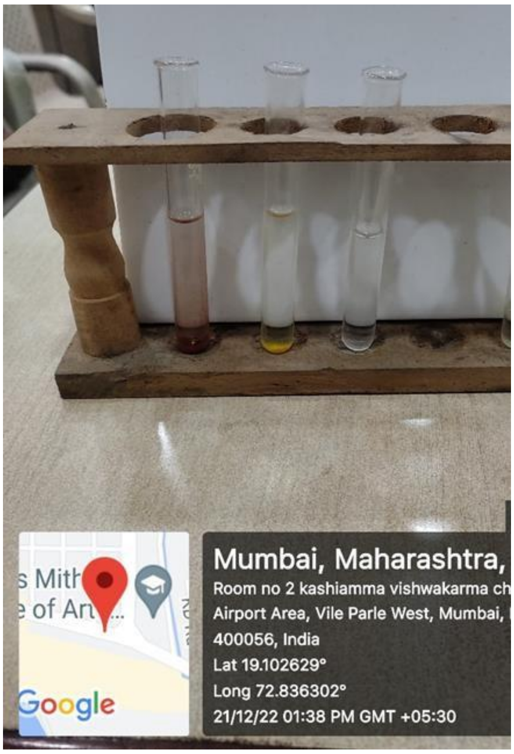

Figure 11 d: Use of Fe_3O_4 nanoparticles for removal of toxic Cd precipitated as CdS.

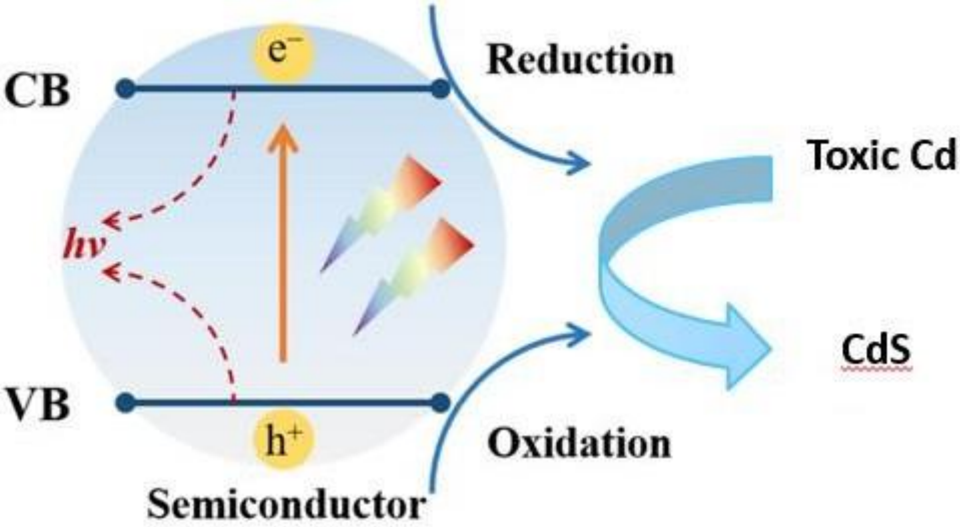

Figure 11e: Proposed mechanism adsorption of Cd at acidic pH on Fe_3O_4 nanoparticles removal of toxic Cd precipitated as CdS.

Further, the one pinch of nanoparticles which were used in the first cycle for adsorption with Cd were separated out with the help of centrifugation method. The residue left behind after centrifugation contained nanoparticles which were collected and used for next cycle of adsorption. Centrifugate pH was raised to pH 2 using dil HCl solution so that Cd get de-adsorbed in acidic condition and followed by Na_2S solution was added to precipitate Cd as CdS (Fig. 11a,b,c,d) The time of adsorption of Cd was mentioned above(Fig. 11b) shows good catalytic efficiency. The adsorption studies were carried out till 100 cycles form concentration one to nine ppm and real samples of Cd.

CONCLUSION

Herein we have demonstrated the preparation of Fe_3O_4 nanoparticles by very easy and sample path. Fe_3O_4 nanoparticles shows removal of Cd According to qualitative and quantitative evaluations, the aim of this research is to develop low-cost, high-efficiency devices with a minimum equipment using transition metal chalcogenides nanoparticles for the degradation of harmful contaminants.

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