Review Paper:

Advances in Magnetic Materials: Classification and Synthesis

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Abstract

Nanoscience plays a pivotal role in mitigating lethal pollutants, contributing to environmental rejuvenation. The rising demand for magnetic nanomaterials is driven by their extensive applications in drug delivery, biosensors, environmental remediation, magnetic resonance imaging (MRI), catalysis and cell separation. Various synthesis methods including solvothermal, co-precipitation, thermal decomposition, hydrothermal and microemulsion processes, have been developed to prepare these materials. This study highlights the latest advancements in the preparation of magnetic nanomaterials, showcasing their versatile utility across multiple domains.

By focusing on cutting-edge techniques, we aim to provide insights into the innovative approaches enhancing the efficiency and functionality of magnetic nanomaterials, underscoring their significance in contemporary scientific and technological applications.

Keywords: Magnetic materials, Magnetic properties, Synthesis.

Introduction

Contamination of water is considered to be constant threat to world. The presence of various contaminants such as heavy metals, dyes, oil spills etc. in surroundings negatively affect the nature. The risk of water contaminant can affect the photosynthesis processes of aquatic life by reduction in light penetration and reduction of water oxygen, increase in the water-borne diseases, toxicity of drinking water etc. Therefore, environmental remediation is required to counter such harmful effects. As a result, various techniques can be used and can be tailored in an attempt to decontaminatethe water but on account of low output/input ratio, complex operating mechanism, their employment is pocket-sized. Taking into account, these limitations of conventional techniques, nanotechnology has attracted much attention, which not only provides noxious-free ambience but also is of enormous effectiveness in conservation of resources[20,34].

In the past few decades, magnetic nano-materials have engrossed considerable attention. Magnetic nano-materials show novel physical property at the nanoscale which differentiates them from their bulk corresponding material. This process is called nano-magnetism and it provides distinctive properties like super para magnetism to magnetic materials. It should be eminent that distinct nanostructures in magnetic materials are decisive to attain these distinctive properties. Magnetic materials have been under intense concern due to their advantages in the biomedicine, drug delivery, environment, sensors, magnetic resonance imaging (MRI) and catalysis, etc[8,30,41].

Various magnetic nanocomposites such as bimetallic, spherical, usual core-shell magnetic NPs (MNPs), multinuclear MNPs inlaid in core-shell structures, MNP-silica nanocomposites, MNP encapsulated in mesoporous materials and MNP-decorated silica spheres can be utilized to catalyze several reactions. Specific magnetic properties were possessed by magnetic metal oxides (like$CoFe_2O_4$, Fe_3O_4 and $MnFe_2O_4$) and metal alloys (like FePt and $CoPt_3$) calculated by the magnetization cycle in a hysteresis loop and have been effectively applied in heterogeneous catalysis. Furthermore, hybrid magnetic NPs have a great deal of consideration because of functionalization of materials and can be utilized as appropriate supports, also can be coated by carbon, polymer, metal, metal oxide and silica to form core@shell structure. Large surface area, small size and high reactivity result in the usage of magnetic nanomaterials for the removal of various contaminats[4,25,26].

Evidently, magnetic nanocomposites adsorbents are found to be very significant nanocomposites adsorbent. Here, we focus on the cost effectiveness, eco-friendly and efficient method for the synthesis of magnetic nanomaterials. Also, this study represents a complete overview of distinctive properties of magnetic nano-materials. Also, their advantages as better adsorbents for the adsorption of various pollutants such as inorganic, organic and biological have been discussed.

Types of Magnetic Materials

Magnetic Metallic Nanomaterials: General metallic nanomaterials comprise of metals and metal oxides like Mg, Cu, Ag, Pd, Au, Al_2O_3, V_2O_5, CuO, TiO_2, MnO_2, ZnO, CeO_2, CoO and ZrO_2[43]. Large specific surface area, high adsorption capacity, good chemical stability, surface reactivity, easy modification at low temperature and fast equilibrium are the significant properties. They have tendency to agglomerate due to van der Waals interactions and other forces[1]. The small particle size of metal loaded adsorbents results in difficult separation. Several studies

were performed by incorporating metallic materials on magnetic supports to conquer these problems. Physical or chemical change via chelating reagents or other organic compounds results in the advancement of efficiency and selectivity of these magnetic metallic materials. The extraction efficiency is affected by various factors such as surface area, morphology, surface modification and crystal structure of the metallic materials[15].

Magnetic Molecularly Imprinted Polymers (MIPs): MIPs have modified binding sites consequent to the size, shape and functional groups of template molecules. MIPs have attracted extreme consideration due to high selectivity of the target analyte, predictability of structure and the specificity of recognition.

Generally, copolymerization of cross linkers and functional monomers was used to synthesize MIPs in the presence of template molecules. Explicit customized binding sites are absent as quickly as the templates are eluted to permit MIP to recognize target molecules according to their stereo structure, unique size and chemical function. Magnetic molecularly imprinted polymers (MMIPs) were synthesized by the combination of MNPs with MIPs and have core-shell structure. MMIPs supported by porous-materials, MIPs supported by magnetic-nano sheet and MIPs supported magnetic-nanotube composite structures have surfaced in modern years[5,32].

Siliceous Magnetic Nanomaterials: Since the discovery of ordered mesoporous siliceous nanomaterials such as silica microspheres, M41S, SBA-1, SBA-15, MCM-48, MCM-41 and FSM-16, nanomaterials based on silicone have opened up a wide advantage vision in the study of adsorption. Siliceous MNPs are synthesized by salinization which is a general process for the surface modification of the magnetic core and the silicon material can provide protective outer coating of the magnetic core. Easy surface modification, large specific surface area, mesoporous channels, low cost, easy control of the inter particle interaction, good biocompatibility, high porosity and mechanical stability are the characteristic properties of siliceous MNPs.

MNPs have limiting advantages in the field of adsorption that involves sufficient binding sites as the majority of siliceous MNPs do not have specific surface properties. The usage of inorganic adsorption species including metal phosphates, molybdophosphate and hydrotalcite, in addition to titanate-based materials has been reported whereas organic loads like phenyl, porphyrin and graphene oxide have been applied.

Nanocarbons: Nanocarbons (NCs) are considered to be carbon materials with minimum 1-D of dispersed phase less than 100 nm. Lately, the study of carbon nanotechnology has been found to be quite vigorous and several nanocarbons such as barrel like, needle-likeand rod-like have appeared in a continuous stream. NCs are found to be very potential materials because they have large surface area, affinity to multiple molecules and a short diffusion path resulting in the fast adsorption process in comparison to traditional SPE materials. Various types of NCs are shown in figure 1.

Covalent Organic Frameworks: Covalent organic frameworks (COFs) are porous materials which constitute light elements (H, O, C, N, B, Si) and are considered to be a two- or three dimensional crystals porous structure composed of strong covalent bonds between organic monomers.

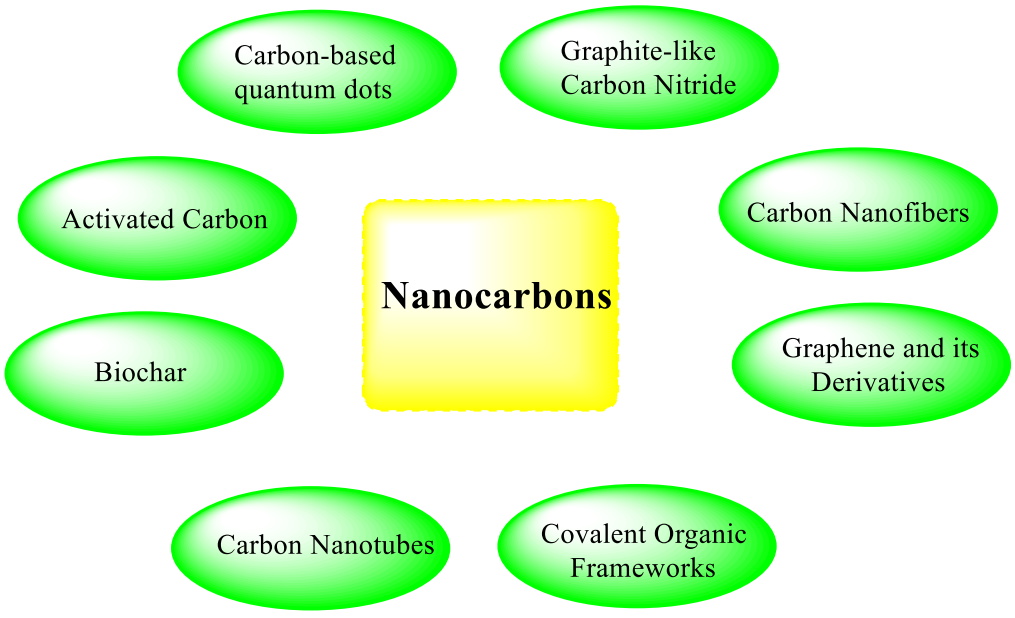

Figure 1: Various types of Nano Carbons

Using core-shell magnetic carboxylate-functionalized covalent organic framework composites (Fe_3O_4@COF–COOH), Hu et al[16] developed a novel multifunctional sorbent that allows for the simultaneous adsorption of these target analytes through mixed-mode solid phase extraction. The Freundlich and Langmuir isotherm models were used to evaluate the synthetic composite's adsorption behaviours for PAHs, TCs and TDs. Quantum chemistry calculations revealed that COF-COOH and guest molecules interact through π-π stacking, hydrogen bonding and electrostatic attraction.

Wang et al[42,43] developed $NiFe_2O_4$-based magnetic covalent organic framework nanocomposites ($NiFe_2O_4$@COFs) using a simple synthesis method at room temperature. $NiFe_2O_4$@COFs had a higher adsorption capacity for brominated flame retardants than carbon nanotubes due to hydrophobic interactions, π-π stacking interaction and van der Waals forces. Ultimately, the produced magnetic material was effectively employed to determine five BFRs from actual water samples using magnetic solid-phase extraction adsorbers. Shakeri et al[33] prepared functionalized Fe_3O_4-based melamine-rich covalent organic polymer (COP) and identified it using various physicochemical techniques including Fourier-transform infrared, X-ray diffraction, field emission scanning electron microscopy, Brunauer-Emmett-Teller analysis and vibrating sample magnetometer analysis.

The compound was used to remove auramine O (AO) and rhodamine B (RB), both cationic dyes, from an aqueous solution using electrostatic attraction, H-bonding, Lewis acid-base interaction and π-π stacking. The significant variables such as pH, Fe_3O_4/COP mass, AO and RB concentrations and sonication time were examined and the optimised conditions were found to be 6.5, 12 mg, 10 mg/L and 4 minutes respectively.

Synthesis Methods of Magnetic Materials

Co-precipitation Method: Magnetic NPs are synthesized by this method. This process is generally utilized for the preparation of ferrites and metal oxides. The base is added to the aqueous salt solution of Fe^{2+}/Fe^{3+} for the preparation of Fe_2O_3 under inert atmosphere. The change in temperature of the reaction used salt, pH, Fe^{2+}/Fe^{3+} ratio and ionic strength of the media results in the control of the composition, size and shape of the MNPs.

$$Fe^{2+} + 2OH^- \longrightarrow Fe(OH)_2$$

$$Fe^{3+} + 3OH^- \longrightarrow Fe(OH)_3$$

$$Fe(OH)_2 + Fe(OH)_3 \longrightarrow Fe_3O_4 + 4H_2O$$

The overall reation may be written as

$$Fe^{2+} + Fe^{3+} + 8(OH^-) \longrightarrow Fe_3O_4 + 4H_2O$$

This method is utilized for the preparation of ferrites in an aqueous medium.

Chemical equation involved is written as:

$$M^{2+} + 2\,Fe^{3+} + 8OH^- \longrightarrow MFe_2O_4 + 4H_2O$$

where M may be Fe^{2+}, Mn^{2+}, Co^{2+}, Cu^{2+}, Mg^{2+}, Zn^{2+}, or Ni^{2+}

Different oxidizing agents are utilized for the partial oxidizing of hydroxide suspension which is an alternate method that falls in the category of co-precipitation. For example, Fe (II) salt, mild oxidant (nitrate ions) and a base were used for obtaining the spherical magnetite particles with 30 - 100 nm of mean diameter.

Yadav et al[45,46] used the chemical co-precipitation method to produce Fe_3O_4 nanoparticles and a Fe_3O_4/NiO composite for Cr(VI) removal. Fe_3O_4 and Fe_3O_4/NiO composite had adsorption capacities of 96.15 mg gm^{-1} and 150 mg g^{-1}, respectively determined by the Langmuir type 1 model. Fe_3O_4 and Fe_3O_4/NiO composite had saturation magnetizations of 60.54 emu/g and 11.09 emu/g respectively. Yadav et al[45,46] used Fe_3O_4 nanoparticles and a porous magnetic chitosan composite derived from Kaphal (Myrica esculenta) leaves to remove cadmium ions from wastewater. The porous magnetic chitosan (MCS) composite and Fe_3O_4 nanoparticles (MNPs) showed maximum Cd adsorption capacities of 426 mg/g and 290 mg/g, respectively. The adsorption procedures both adhered to second-order kinetics. This study describes the removal performance of phosphate and nitrate from water using a stable inorganic-organic hybrid composite of porous carbon (porous carbon derived from papaya seeds) that was created through one-pot synthesis.

Cobalt ferrite coated-porous carbon composite ($CoFe_2O_4$@PC) was used to remove phosphate and nitrate from water. The different parameters for this process were optimized. At 303 K, the maximum adsorption capacity for phosphate is 92.34 mg/g and for nitrate, it is 78.98 mg/g. The Freundlich adsorption isotherm model was found to be superior for fit and heterogeneous surface interaction of phosphate and nitrate ions on the $CoFe_2O_4$@PC composite. The prepared $CoFe_2O_4$@PC composite demonstrated good removal capacity and was successfully used to remove phosphate and nitrate ions from real water samples. Furthermore, this study offers helpful information about how magnetically separable $CoFe_2O_4$@PC composite removes phosphate and nitrate from water and wastewater[19].

Nguyen et al[24] developed magnetite (Fe_3O_4) nanoparticles by employing the ultrasound assisted co-precipitation technique. Fe_3O_4 nanoparticles were found to have a cubic structure. The spherical Fe_3O_4 particles have a size range of 10 to 15 nm. As a magnetic adsorbent, Fe_3O_4 magnetic

nanoparticles were used to extract Congo red from an aqueous solution. It is considered that the pseudo-second-order kinetics equation governs the adsorption process. The Langmuir model fits equilibrium data quite well. 169.8 mg/g is the maximum adsorption capacity. This implies that the produced magnetic nanoparticles may prove to be a useful adsorbent for the removal of organic dyes from aqueous solutions.

Karthikeyan et al[19] used the co-precipitation method to produce $CuMn_2O_4$/CuMnO nanocomposites which were then used to remove dye pollution from an aqueous solution. The nanocomposites demonstrated ferromagnetic behaviour at low fields (coercivity ~ 1082.55 Oe) and antiferromagnetic behaviour at high fields. Furthermore, their photocatalytic properties for the degradation of eriochrome black T under visible light irradiation were studied. The tests were repeated with sucrose added as a capping agent. Using a 0.03 g nanocomposite, 76% of the photodegradation of an eriochrome black T solution at 10 ppm was obtained. Compared to other pH levels, the rate of eriochrome black T degradation at acidic pH was higher, taking 90 minutes to reach 80.45%.

In order to produce the magnetic nanosorbent, Ravi and Sundararaman[29] used powdered chicken eggshell waste that had been coated in iron oxide. Co-precipitation was assisted by an ultrasonic bath.The effectiveness of batch adsorption in removing Cr(VI) from an aqueous solution using CM magnetic nano adsorbent was examined. According to the characterizations, the CM adsorbent had a cubic shape, a uniform distribution and minimal agglomeration. The crystalline nature and average particle size of 24 nm were characterised, as well as the magnetization value of 14.46 emu g^{-1}. The highest Cr(VI) removal rate of 92.87% was achieved with an initial concentration of 94.5 mg/L, a contact time of 66.2 minutes, a solution pH of 5.5 and mechanical shaking of 200 rpm.

Foroutan et al[11] used the co-precipitation method to produce a magnetic nanocomposite of clinoptilolite (CLT), starch and $CoFe_2O_4$. Crystal violet dye (CVD), methyl violet dye (MVD) and methylene blue dye (MBD) were all removed from water media using the magnetic composite powder that had been prepared. According to the BET analysis, the specific surface area of CLT, $CoFe_2O_4$ and CLT/Starch/ $CoFe_2O_4$ powder was reported to be 18.82 $m^2.g^{-1}$, 151.4 $m^2.g^{-1}$ and 104.75 $m^2.g^{-1}$ respectively. This improvement was attributed to the use of starch and CoFe2O4 nanoparticles in the modification process. For the desired composite, the maximum adsorption capacities of CVD, MBD and MVD were found to be 32.84 $mg.g^{-1}$, 31.81 $mg.g^{-1}$ and 31.15 $mg.g^{-1}$ respectively.

Solvothermal Method: Iron based NPs are prepared at a pressure and temperature of range of 1-10,000 atm and 100–1000 °C respectively by utilizing an organic solvent like methanol, ethylenediamine and hydrazine[28]. Here agglomeration can be prevented by using capping agents like polyacrylic acid, oleic acid and sodium dodecylbenzene sulfonic. Shaterabadi et al[35] synthesized magnetite nanorods by using solvothermal technique. Lately, Dar et al[10] utilized a solvothermal approach to form pure and iron doped SnS nanoparticles. Pure SnS and Fe doped SnS samples showed a small stretching vibration in Sn-S modes, according to the FT-IR and Raman data. Samples have spherical, ball-like shapes according to SEM analysis, but the average particle size determined from TEM images is between 25 and 30 nm. The binding energy and chemical makeup of both pure and Fe-doped SnS are confirmed by XPS.

Microemulsion: In this method, magnetic NPs are formed by utilizing water-in-oil microemulsion comprising of three components: oil, water and surfactant[18,22]. The aqueous phase is enclosed in a monolayer of surfactant molecules and is dispersed as microdroplets in the continuous non-aqueous phase. In a microemulsion, a water-soluble metal salt will reside within the aqueous droplets surrounded by oil. If two microemulsions are formed with the same reactants, upon mixing, their microdroplets will continuously collide, coalesce and form a precipitate within the aqueous portion of the microemulsion.

Fe_3O_4/SiO_2/P(NIPAM-co-AMPTMA) nanocomposites were developed by Nayeem et al[23] utilising the microemulsion technique. Islam et al[17] used the microemulsion method to prepare cobalt ferrite nanoparticles.VSM measurements showed that the saturation magnetization, remanent magnetization and coercivity were 12.05 emu/g, 0.77 emu/g and 118.912 Oe respectively. Barad et al[6] developed maghemite nanoparticles by employing the microemulsion method to adsorb Cr(VI) from wastewater. A maximum of 99% adsorption efficiency was discovered.

Hydrothermal reactions: Hydrothermal treatments are generally taking place with an aq. media in reactors or autoclaves under a pressure higher than 2000 psi and temperatures of more than 200°C. The advantage of this technique is the exemption from further calcination, low cost and is ecologically friendly. Two different approaches can be used for preparing magnetic NPs, the first requires hydrolysis and oxidation and the second requires neutralization of mixed metal hydroxides. The PEG-mediated pathway is another type of hydrothermal technique which has been utilized for the formation of Magnetic NPs. The reaction temperature and time play vital roles in controlling the size and morphology of MNPs.

Ethylene glycol (EG) mediated hydrothermal synthesis is used by Tang and co-workers[40] for the formation of nanoparticles of strontium M hexaferrite under diverse situations. The effect of the quantity of EG and temperature of the reaction is studied on the particle size, magnetic properties and morphology of strontium M hexaferrite. Ethylene glycol plays a crucial role in the morphology and

phase transformation of strontium M hexaferrite. The growth of the crystal in the reaction medium is retarded by ethylene glycol. The movement of ions in the reaction medium is hindered by a higher amount of ethylene glycol. Metal oxide NPs with specific morphology and sizes are prepared by a continuous hydrothermal process[44]. Slow reaction kinetics at any temperature is the major drawback of the conventional hydrothermal process. The kinetics of crystallization is enhanced by using microwave assistance during the hydrothermal preparation and such a combination is known as a microwave hydrothermal process[31].

Thermal Decomposition: This approach permits control over size distribution, crystallinity and shape and is considered a useful method for the preparation of magnetic NPs. It has great significance for various sensing applications and has increased control over susceptibility and particle saturation magnetization is also probable[13,47].

By using successive ionic layer deposition from reaction solutions, Popkov et al[28] developed 2D nanocrystals of $Zn_2Fe_4(OH)_{12}SO_4$ that exhibit phase and chemical purity, have a lattice parameter of a = b = c = 8.4394 Å and exhibit superparamagnetic nanoparticle behaviour. Their effective magnetic moment is μeff = 24.3 μB per formula unit and their magnetization is 1.85 (298 K) and 12.79 (77 K) emu/g with an applied field of 18 kOe. Phase-pure superparamagnetic $ZnFe_2O_4$ nanoparticles may find application as the building blocks for magnetic fluids, gas sensors, anti-hyperthermia medications and diagnostic magnetic resonance imaging.

Sol-Gel Method: NPs can be prepared for desired properties using a wet chemical method. This process involves hydroxylating metal precursors in a "sol" of microscopic particles. The self-assembly of NPs during the washing process is this technique's main drawback. To address this issue, numerous modified methods for producing monodispersed and non-agglomerated nanoparticles have recently been described. In addition, the variables that affect the gel's formation and properties include pH, solvent type, agitation time, temperature and concentration[2,7,14]. $NiFe_2O_4$ was recently prepared using the sol-gel method, as reported by Majid et al[21] Findings showed that the synthesis process had an impact on $NiFe_2O_4$'s structural, magnetic and dielectric characteristics. The XRD data confirmed the inverse spinel structure of the Ni Fe_2O_4 and the hydrothermal method yielded a crystalline size of 29.39 nm, while the sol-gel route produced a crystalline size of 52.16 nm. Along with the dielectric properties, prepared $NiFe_2O_4$ displayed varying responses in terms of saturation magnetization (MS), retentivity (Mr) and coercivity (HC)[21]. In his research, Silva and colleagues[38] looked at how PVP affected the IONPs' size, crystallinity and other characteristics.

Biological synthesis method: Biological synthesis technique is used for the preparation of metal NPs by investigating yeast, fungi, viruses, actinomycetes and bacteria. Various advantages associated with this technique are: high efficiency, eco-friendly nature and cleanness and limitation is poor dispersion of metal NPs. Electron shuttle quinones, nitrate reductase action and the mixed mechanisms are the probable mechanism recommended for the mycosynthesis of metal NMs. The complete preparation mechanisms are not well known, yet MNPs have been synthesized by using this greener and clean biological synthesis technique[37,39]. This technique is employed for the synthesis of enzyme MNMs[36]. Additionally, Ahmadi et al[3] prepared iron nanoparticles (FeNPs) with this method by utilizing the essential oil of Satureja hortensis. The findings showed that Fe nanoparticles with a cubic morphological structure and a particle size range of 9.3-27 nm were formed.

In order to develop a magnetic nano-sized solid catalyst from bio-waste Citrus sinensis peel ash (CSPA)@Fe_3O_4 for the production of biodiesel from waste cooking oil (WCO), Changmai et al[9] investigated into the biosynthesis approach. The catalyst's core-shell structure improved the surface characteristics and kept the catalyst's size and shape under control, which greatly increased the catalyst's stability. Several analytical methods were used to characterize the produced catalyst. Citrus sinensis peel ash (CSPA) catalyst is very basic due to its high potassium and calcium content and it was a key catalyst in the transesterification of WCO. A maximum biodiesel yield of 98% was achieved by the CSPA@Fe_3O_4 catalyzed transesterification under the ideal reaction conditions, which included a 6:1 methanol/oil molar ratio, a 6 weight percent catalyst loading, a temperature of 65 °C and a 3-hour duration.

Conclusion

The magnetic nanomaterials are attractive and have fertile applications in several fields. Great progress has been achieved for the preparation of controlled shape, size and relevant properties of magnetic materials. Magnetic nanomaterials exhibit unique physicochemical properties such as low toxicity, low cost, high surface area and easy magnetic separation. Hydrothermal, co-precipitation, microemulsion, high-temperature decomposition of organic precursor and solvothermal methods are used for the preparation of magnetic materials. High quantity and quality of magnetic materials are produced by these preparation methods. Magnetic materials can function as MRI contrast enhancement, imaging probes, nanosensors, therapeutic agent, drug/gene delivery, hyperthermia and cell separation/labeling.

On the other hand, magnetic materials are utilized for the removal of environmental pollutants from the wastewater by the usage of an external magnetic field. Their non-toxic and economic nature make them promising as adsorbents. Hence, these are good candidates for the treatment of industrial effluents. Finding a economic, more proficient means of purifying polluted water to meet safety standards is increasingly occupying the attention of environmental scientists and Governments around the world. This study

mainly focuses on the current advances in the preparation and modification of magnetic materials.

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