Nanocomposites of Ferrites with TiO_2 , SiO_2

Subjects: Chemistry, Physical

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Ferrites are important magnetic materials used in electronic devices. Nanocomposites of ferrites with TiO_2 has gained interest due to its unique advantages, such as high chemical stability, surface-active sites, high specific surface area, non-toxicity, excellent optical properties, and tunable porosity.

ferrites carbon quantum dots photocatalysis TiO_2 SiO_2 Nanocomposites

1. Introduction

TiO_2 has four polymorphs: brookite (orthorhombic), rutile (tetragonal), anatase (tetragonal) and TiO_2-B. Anatase, rutile, and brookite phases have bandgaps of 3.2, 3.0, and 3.4 eV, respectively. Brookite is less thermodynamically stable, but rutile is stable and anatase is metastable. At temperatures exceeding 600 °C, irreversible conversion of brookite and anatase TiO_2 to rutile occurs [1].

It has been observed that bandgaps of TiO_2 nanoparticles with 5–10 nm particles narrow up to 0.2 eV. Anatase is the most photocatalytically effective phase of TiO_2 [2] and is widely used in semiconducting materials for environmental applications [3]. It has higher mobility of electron–hole pairs and higher surface hydroxyl density. Brookite-phase activity has not been thoroughly examined [4][5]. Rutile TiO_2 is usually investigated in fundamental investigations. Due to the existence of oxygen vacancies, TiO_2 is classed as an n-type semiconductor [6]. Because crystalline TiO_2 has a less photoexcited electron–hole recombination than amorphous TiO_2, it has higher photocatalytic activity [7][8]. Recombination of photogenerated charge carriers is a disadvantage of TiO_2 photocatalysts, as it decreases overall reaction quantum efficiency [9]. During the recombination processes, the photoexcited electrons return to the valence band non-radiatively or radiatively [10][11][12]. An attractive feature of the TiO_2 photocatalyst is its potential to be activated by visible light. To increase photocatalytic activity and decrease recombination of photoexcited charge carriers, several methods have been followed, such as heterojunction formation and doping with non-metals, metals, and nanosized crystals, which can alter the electronic and optical properties of TiO_2 [13][14][15][16][17]. By using dopants in TiO_2, its bandgap reduces [18]. Metal doping has long been known to improve visible light absorbance of TiO_2 [19][20] and increase its photocatalytic activity under UV irradiation [20][21][22][23], but the introduction of metal ion results in thermal instability, which reduces the reuse of TiO_2 photocatalyst and recombination of electron–hole pairs occur at a fast rate. Non-metal doping is more efficient than metal ion doping due to more thermal stability and less formation of recombination centers [24][25][26][27][28].

Nanocomposites of ferrites with TiO_2 have been synthesized using different chemical methods. A summary of methods used to synthesize magnetic nanocomposites has been discussed in this section.

2. Sol–Gel Method

In typical sol–gel synthesis, formation of gel materials with three-dimensional structure occurs by condensation and hydrolysis reaction of metal ions. It is an irreversible process: once gel is formed, it cannot break down. There is covalent interaction between gel particles. In this method, huge precursors are used during conversions and can be performed at or near room temperature. For chemical catalysts, porous ceramic xerogels with large surface area act as support and thin-film deposition is important for material possessing desired optical and magnetic properties [29]. This method uses simple equipment, produces highly homogeneous and pure products at low temperatures, and allows modification of the surface.

Tatarchuk et al. [30] developed magnetic nanocomposites of $CoFe_2O_4$@TiO_2 using the Pechini sol–gel method with ethylene glycol and citric acid as chelating agents and titanium (IV) polymeric precursor solution. XRD study of $CoFe_2O_4$@TiO_2 indicated the presence of 46% and 54% of anatase and rutile crystal phase, respectively, in the titanium dioxide (TiO_2) component, and the average size of cobalt ferrite ($CoFe_2O_4$) and TiO_2 nanoparticles was about 30 and 8 nm, respectively. SEM revealed that because of deposition of TiO_2 on the $CoFe_2O_4$ surface, particles became large. The IR spectrum of the nanocomposite illustrated the shift of bands, which was the result of isomorphic heterovalent substitution in the octahedral position. Dadfar et al. [31] and Xu et al. [32] fabricated $SrFe_{12}O_{19}/TiO_2$ and $TiO_2/ZnFe_2O_4$ nanostructures by the sol–gel method. XRD indicated the presence of impurity phases (�-Fe_2O_3) at pH 2.5 and 4.5, while pure $SrFe_{12}O_{19}$ and TiO_2 phases were formed at pH 3.5. The production of the rutile phase was prohibited to some extent by highly dispersed $ZnFe_2O_4$ nanoparticles in $TiO_2/ZnFe_2O_4$ nanocomposites. TEM confirmed average particle sizes of 15–75 nm of $SrFe_{12}O_{19}/TiO_2$ and uniform attachment of $ZnFe_2O_4$ nanoparticles to TiO_2 nanoparticles to produce a coupled semiconductor. The electron diffraction pattern displayed very weak intensity of the electron diffraction pattern for the $ZnFe_2O_4$ phase, which was attributed to the dispersion of $ZnFe_2O_4$ nanoparticles amid the TiO_2 nanoparticles. The value of saturation magnetization, remnant magnetization, and coercivity decreased with increasing amounts of TiO_2 in $TiO_2/ZnFe_2O_4$, due to the contribution of the non-magnetic TiO_2 component to total sample volume.

Lahijani et al. [33] fabricated a $PbFe_{12}O_{19}$-TiO_2 nanocomposite using the sol–gel method. From XRD analysis, the average size of crystals was found to be 88 nm. FTIR study indicated that absorption bands at 544, 716, 935 and 1401 nm^{-1} corresponded to stretching modes of Fe-O, Ti-O, Pb-O and C-C bonds, respectively. Heating the compound in the presence of a polyhydroxy alcohol, such as ethylene glycol, promotes polymerization. The metal ions are equally dispersed inside the organic matrix, resulting in a homogeneous resin—the polymeric precursor [34]. The immobilization of metal complexes in such rigid organic polymeric networks can decrease segregation of specific metals, ensuring molecular compositional uniformity. Polymeric precursors can be used to make ferrite–TiO_2 nanocomposites. Mouro et al. [35] used a polymeric precursor technique to make nanometric $TiO_2/CoFe_2O_4$ composites. X-ray diffraction, Raman spectroscopy, surface area through N_2 physisorption, zeta potential, scanning and high-resolution transmission electron microscopy were used to characterize the as-prepared sample. The lack of a rutile phase in the nanocomposites was confirmed by Raman spectroscopy. XRD patterns revealed that thermal treatment did not cause phase segregation. The synthesized nanocomposites showed an increase in surface area, a change in surface charge in relation to pure TiO_2, and selectivity in the photodegradation. The materials had photocatalytic activity due to the presence of TiO_2 on their surfaces, and $CoFe_2O_4$ cores were beneficial for separating and recovering photocatalysts after use in an oxidative process.

3. Ultrasonic Method

The ultrasonic technique involves using sound energy to agitate the particles in a solution, resulting in physical and chemical changes. This method is found to be the most promising method for manufacturing nanocomposites such as oxides, carbides, alloys and colloids with almost uniform distribution of nanoparticles [36]. Ultrasound irradiation causes unstable bubbles in liquids to form, grow, and collapse rapidly at temperatures as high as 5000 K, pressures as high as 20 MPa, and cooling speeds of 10^{10} Ks^{-1} [37]. Ultrasonication was used for the synthesis of titania-coated γ-Fe_2O_3 magnetic activated carbon and Fe_3O_4-TiO_2 photocatalyst with saturation magnetization value of 2.21 and 14 $emug^{-1}$, respectively [38][39]. XRD revealed that the titania coated on the magnetic activated carbon maintained the anatase phase, with the same set of characterization peaks for both Fe_3O_4 and Fe_3O_4-TiO_2, indicating that the ultrasound irradiation had no effect on the crystal structure of the Fe_3O_4 nanoparticles. The tiny crystallite size was shown by the broad nature peak of the deposited TiO_2 on Fe_3O_4-TiO_2. SEM revealed the homogeneous distribution of titania. Superparamagnetic characteristics of Fe_3O_4-TiO_2 photocatalyst at room temperature were confirmed by a magnetization hysteresis loop. HRTEM analysis showed the occurrence of a heterojunction in the Fe_3O_4-TiO_2 nanocomposite. XPS showed that the peaks at 710.5 eV and 458.8 eV originated from Fe 2p and Ti 2p energy levels, respectively, implying that Fe_3O_4 and TiO_2 were largely present as separated phases in Fe_3O_4-TiO_2 composites.

4. Coprecipitation Method

This approach requires the preparation of a mixed aqueous solution of starting chemicals such as chlorides, nitrates, or sulfites of Fe^{3+}, as well as of divalent metal ions in the requisite stoichiometric ratio. The precipitates are then generated by treating the solution with NaOH, filtration, washing twice, and drying. This approach entails four steps: nucleation, growth, coarsening, and agglomeration, all occurring at the same time [40]. Nanocomposites of ferrites of good quality and phase purity can be obtained by this method. This process offers various advantages such as high yield, high product purity, lack of necessity to use organic solvent, good reproducibility and low cost. It is a quick and easy way to make ultrafine particles that are disseminated in a variety of media. By carefully monitoring the preparation parameters, it is possible to achieve control over the surface morphology, structure, and chemical composition with this procedure.

Coprecipitation was employed for fabrication of $CoFe_2O_4/TiO_2$ and $ZnFe_2O_4$-TiO_2 nanocatalysts [41][42]. The average particle size of the coprecipitated nanocatalysts was 50 nm for $CoFe_2O_4$ and 150 nm for $CoFe_2O_4/TiO_2$ according to SEM images, which was consistent with the crystallite size predicted from XRD data. The spherical geometry (9 ± 2 nm) of $ZnFe_2O_4$ nanoparticles and spherical-like morphology (11 ± 3 nm) of $ZnFe_2O_4$-TiO_2 nanocomposites and fine dispersion of black particles ($CoFe_2O_4$) on the gray surface (TiO_2) of $CoFe_2O_4/TiO_2$ were depicted in the TEM images. Bandgaps of $CoFe_2O_4/TiO_2$, $CoFe_2O_4$, $ZnFe_2O_4$-TiO_2, and $ZnFe_2O_4$ were found to be

2.8, 1.1, 2.3, and 2.1 eV, respectively, which suggested the formation of a visible light-active photocatalyst. The large bandgap of the composite may be due to the mixing effect of the bandgap and the interfacial coupling effect between $ZnFe_2O_4$ and TiO_2, $CoFe_2O_4$ and TiO_2. XRD pattern analysis revealed that in $CoFe_2O_4/TiO_2$, the conversion of anatase to the rutile phase of TiO_2 happened at a TiO_2 annealing temperature of ≤600 °C, confirming the purity of $ZnFe_2O_4$ and TiO_2 phase in $ZnFe_2O_4$-TiO_2, which was in agreement with HRTEM and SAED investigations. Haw et al. [43] found that in comparison to conventional rutile-phase TiO_2 and pure urchin-like TiO_2 (3D TiO_2) microparticles, nanocomposites of $CoFe_2O_4$-3D TiO_2 showed an increase in photodegradation of methylene blue, and this nanocomposite demonstrated a generally constant photocatalytic efficacy with low degradation. Two sets of lattice fringes were visible at the TiO_2-$CoFe_2O_4$ interface, with d_{110} = 0.322 nm of rutile phase titania and d_{331} = 0.193 nm of $CoFe_2O_4$. The $CoFe_2O_4$ nanoparticles were clearly visible on the 3D urchin-like TiO_2 structure, and each element was uniformly distributed over the surface of the TiO_2 microsphere on scanning transmission electron microscopy. XRD peaks were designated to the rutile phase of 3D urchin-like TiO_2 and broader peaks confirmed the presence of nanosized $CoFe_2O_4$ in the sample.

5. Hydrothermal Method

The hydrothermal method is a way of crystallizing a chemical, utilizing an aqueous solution at a high vapor pressure as well as temperature [44]. At standard temperatures (100 °C) and pressures (<1 atm), it is generally depicted as crystal formation or crystal synthesis from insoluble chemicals. Autoclaves are used to carry out the process, which is performed at a controlled pressure and temperature. This enables the utilization of temperatures that are higher than the boiling point of water or an organic solution. The concoction reaction that occurs beyond the dissolvable breaking point and at pressures over bar is known as hydrothermal synthesis. The hydrothermal technique has the following advantages: it is simple to acquire nanotube morphology, variations in the synthesis process may be used to improve TiO_2 nanotube characteristics, and it is a method that can be used for a variety of applications.

Hydrothermal deposition of a hard ferromagnetic strontium ferrite ($SrFe_{12}O_{19}$) over TiO_2 can result in a photocatalyst that is both ferromagnetic and visible light-active. The TiO_2-supported $SrFe_{12}O_{19}$ nanocomposite has a strong ferromagnetic property and is quite stable when it comes to losing its magnetic property. XRD images of TiO_2-supported $SrFe_{12}O_{19}$ nanocomposite correspond to mixture of anatase and rutile crystal phases. EDS results confirmed that TiO_2-supported $SrFe_{12}O_{19}$ consists of Fe, Ti, Sr, C, O and Si. The BET-specific surface area of TiO_2 was 49.5 m^2g^{-1} with a total pore volume of 0.1553 cm^3g^{-1}, which compares with 38.3 m^2g^{-1} and 0.0155 cm^3g^{-1} for TiO_2-supported $SrFe_{12}O_{19}$. Due to the deposition of $SrFe_{12}O_{19}$ nanoparticles onto the TiO_2, the surface area and pore volume of the TiO_2 nanocomposite reduces. The paramagnetic impact of TiO_2 lowered the coercivity (H_C) and saturation magnetization (M_S) of TiO_2-supported $SrFe_{12}O_{19}$. The photocatalyst TiO_2-supported $SrFe_{12}O_{19}$ had H_C of 2125.5 G. The coercivity of TiO_2-supported $SrFe_{12}O_{19}$ nanocomposite showed that it was a good ferromagnetic material [45]. Nguyen and Doong [46] and Pongwan et al. [47] fabricated $ZnFe_2O_4$-TiO_2 and $CoFe_2O_4/TiO_2$ nanostructure photocatalysts by the hydrothermal method. The $ZnFe_2O_4$-TiO_2 nanocomposite was prepared by coupling 0.2–2 wt % narrow-bandgap material of p-type $ZnFe_2O_4$ with n-type anatase TiO_2. Transmission electron microscopy (TEM) and high-resolution TEM confirmed average particle sizes of 8–9 nm and 5–35 nm for $ZnFe_2O_4$-TiO_2 and $CoFe_2O_4/TiO_2$, respectively. It was observed that $ZnFe_2O_4$ and TiO_2 were intimately linked, which led to a decrease in electron–hole recombination rate as well as enhanced photocatalytic activity of $ZnFe_2O_4$-TiO_2 heterostructures under visible light irradiation. When the loading amount of $ZnFe_2O_4$ increased from 0.5 to 2 wt %, SEM revealed a slight increase in particle size of $ZnFe_2O_4$-TiO_2 nanocomposites. The N_2 adsorption–desorption isotherms showed that the combination of TiO_2 with $ZnFe_2O_4$ can increase the specific surface area. XRD analysis indicated that $ZnFe_2O_4$-TiO_2 can retain the crystallinity of both nanoparticles. Electron diffraction pattern of $CoFe_2O_4/TiO_2$ displayed the brightness of polymorphic discrete rings of the crystalline particles, suggesting quite a high degree of crystallinity in polycrystals. In this research, the obtained maximum saturation magnetization and coercivity of $CoFe_2O_4$ and $CoFe_2O_4/TiO_2$ nanocomposites were found to be 32.58, 29.64 emug^{-1} and 0.15, 0.05 kOe, respectively.

6. Solvothermal Method

The solvothermal technique employs a non-aqueous solvent and a considerably higher temperature, allowing high boiling point solvents to be utilized. The solvothermal method outperforms the hydrothermal method in terms of shape, size control, distribution, and crystallinity of nanoparticles [48]. Organic solvents, which have a low relative permittivity and are free of ionic species, are used in the solvothermal process to produce a product devoid of foreign anions and ionic species. The benefits of both the hydrothermal and sol–gel methods are combined in this technique. This approach may be utilized in the ceramics sector to regulate the particle size of synthesized materials and to fabricate tiny particles, such as magnetic titania photocatalyst [49]. Atacan et al. [50] developed $ZnFe_2O_4/Ag$-TiO_2 by the solvothermal method. The formation, structure and morphology of prepared samples were characterized by X-ray diffraction, scanning electron microscopy, Fourier-transform infrared spectroscopy and vibrating sample magnetometry. In this research, the obtained value of saturation magnetization of $ZnFe_2O_4/Ag$-TiO_2 was 5.5 emug^{-1}. XRD patterns indicated that no chemical reaction occurred between $ZnFe_2O_4$ and Ag-TiO_2, because no peaks related to other impurities were observed. Nguyen and Doong [51] synthesized $ZnFe_2O_4/TiO_2$ heterostructure by this method.

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