

Review

# Diversity of Iron Oxides: Mechanisms of Formation, Physical Properties and Applications

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**Abstract:** Iron oxide compounds have naturally formed during the whole of Earth’s history. Synthetic compositions with iron oxides are produced with the use of various techniques and widely used for scientific and applied purposes. This review considers an attempt to classify all the information on different iron oxide compound formation mechanisms and intended applications in biomedicine, catalysis, waste remediation, geochemistry, etc. All the literature references analyzed were divided into several groups by their number of included iron oxide compounds: compositions containing only one compound (e.g., magnetite or wüstite), including various polymorphs of iron(III) oxide ( $\alpha$ -,  $\beta$ -,  $\gamma$ -,  $\epsilon$ -,  $\zeta$ -,  $\delta$ -Fe<sub>2</sub>O<sub>3</sub>); compositions with two different distinguishable iron oxide phases (e.g., maghemite and hematite); compositions containing non-crystalline phases (amorphous iron oxide or atomic clusters); and compositions with mixed iron oxide phases (indistinguishable separate iron oxide phases). Diagrams on the distribution of the literature references between various iron oxide compounds and between various applications were built. Finally, the outlook on the perspectives of further iron oxide studies is provided.

**Keywords:** iron oxides; phase diagrams; mechanisms of formation; synthetic materials; biomimetic materials; biogenic materials; iron oxide application



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## 1. Introduction

The chemistry of iron is of great interest because iron is an abundant element present in various fields [1]. The elemental abundance of oxygen, hydrogen and iron at the surface of and within the Earth’s crust has fostered widespread occurrences of iron oxides and oxyhydroxides in a diverse range of aquatic and terrestrial environments; most of the known iron oxides and oxyhydroxides are known to occur in nature [2]. Iron oxides are formed naturally through the weathering of Fe-containing rocks both on land and in the oceans and play an important role in geochemistry [3]. Iron-rich sedimentary rocks have had important implications in the evolution of Earth’s atmosphere and hydrosphere [4]. Iron oxide copper gold, apatite-magnetite and other ore deposits have very important heavy industrial applications [5,6]. There are iron oxides on the surface of Mars, in the depths of Earth, in old rusting factories, in pigeon brains and magnetotactic bacteria [7]. Despite the precise mechanism of biogenic magnetite mineralization on early Earth still being unknown, the understanding of this mechanism includes the origin of banded iron formations [8]. Iron oxides are also linked to pathological states of the human body, such as iron dysregulation in the brain and neurological disorders [9,10].

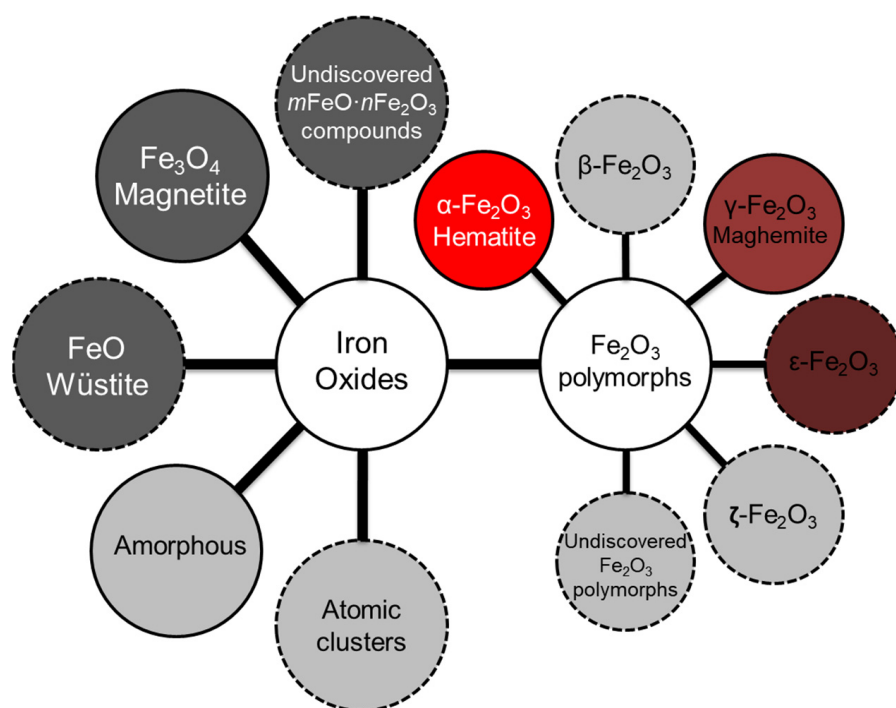
Metallurgy has been developed relating to iron and iron oxides and used for various applications including colored pigments, magnetic materials, catalysts, water oxidation, biomedical uses including therapy and diagnostics, etc. [1,11–14]. The stability of the structural incorporation of uranium into the hematite crystal structure suggests the feasibility of iron oxides for inhibiting the mobility of aqueous uranium (VI) [15]. The use of special

stainless steels (i.e., Eurofer steel) for some portions of the main wall of a nuclear fusion experimental reactor may come into consideration in industrial applications of nuclear fusion; therefore, the detailed knowledge and quantification of their interactions between atoms, molecules and plasma, including electron impact ionization cross sections of iron oxide molecules, is of considerable interest [16]. Iron oxides are, furthermore, of great interest with regard to the corrosion and oxidation processes of iron metal and steel, which are mediated by the surface whose structure depends greatly on environmental variables such as temperature, oxygen or water partial pressures [17,18]. Additionally to the various Fe oxidation states, ferric oxide ( $\text{Fe}_2\text{O}_3$ ) may be stable or metastable in the known  $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\epsilon$ ,  $\zeta$  and  $\delta$  polymorphs [19–22]. Polymorphism in this case means a possibility for a compound to exist in two or more solid phases that are isochemical but have distinct crystal structures and thus, different physical properties. Due to their different physical properties, which arise from the differences in their crystal structures, all of the polymorphs have found applications in nanotechnology [20]. Moreover, there are also not simply polymorphs of known iron oxides, but distinct compounds with the formula  $\text{Fe}_4\text{O}_5$  [23]. Other iron oxides with unconventional stoichiometry, such as  $\text{Fe}_5\text{O}_6$ ,  $\text{Fe}_5\text{O}_7$ ,  $\text{Fe}_7\text{O}_9$  and more complicated compounds have been predicted in theory, some of which have been successfully synthesized at pressures of 10 ~ 80 GPa and annealed from high temperatures [24]. Magnetite ( $\text{Fe}_3\text{O}_4$ ), maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) and hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ), including in the form of the oxidized zerovalent iron core-shell structure, are widely used in heterogeneous catalysis processes and have been used as attractive alternatives for the treatment of wastewaters and soils contaminated with organic compounds [25–27].

Structures containing the various iron oxide crystalline or amorphous phases can either be macro- (ceramics [18]), micro- (microbial-induced precipitates [28]) or nanometer-sized (nanoparticles and nanoclusters [29]). The nucleation and growth of inorganic crystals, including iron oxides, from solutions occurs throughout geochemical, biological and synthetic systems [30]. Iron oxide biominerals are formed under “green” conditions without a loss of functionality such that they have the potential for numerous scientific as well as industrial applications [31]. Global iron cycling is driven by both abiotic and biotic reactions, and in the presence of oxygen and under circumneutral pH conditions, ferrous iron is quickly oxidized to Fe(III) and precipitates as iron oxides [32,33].

The physical properties of the various iron oxide compounds can be extremely different, e.g., magnetite and maghemite have been commonly used in biomedicine because of their saturation magnetization being the highest [34]. The  $\epsilon$ -polymorph of  $\text{Fe}_2\text{O}_3$  possesses the highest coercive force (up to 2 T at room temperature [20,35–37]) among the other ferrimagnetic oxides, while maghemite-magnetite nanoparticles (NPs) can have almost zero coercivity [38] with a very low difference in their average size. In some applications, e.g., in catalysts, amorphous iron oxide NPs can be more active than crystalline polymorphs of the same diameter thanks to their “dangling bonds” and higher surface–bulk ratio in their amorphous phase [39]. Based on the literature data, a generalized scheme illustrating the diversity of the known iron oxide compounds, including stable, metastable, atomic clusters and amorphous, is given in Figure 1.

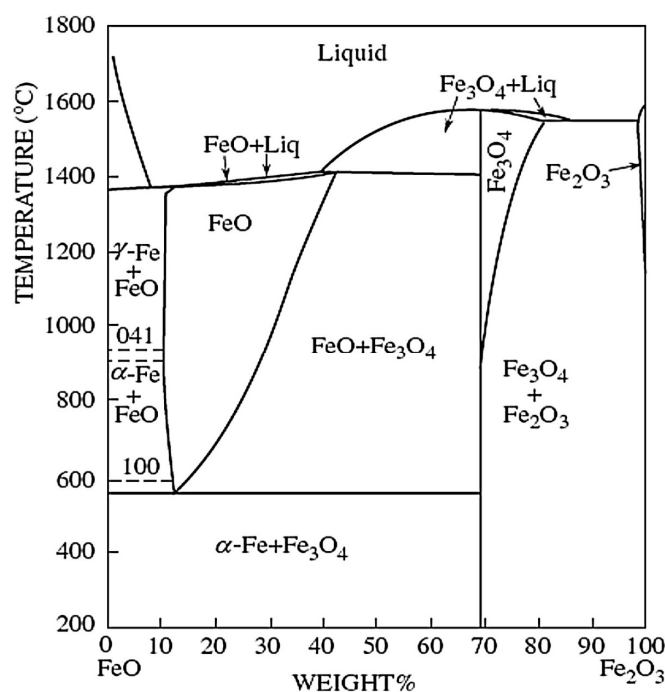
The current review attempts to summarize the main information on the various iron oxide compounds to date. The next section is devoted to a comparison of some of the main physical properties of the various iron oxide compounds.



**Figure 1.** Diversity of stable (solid line) and metastable (dashed line) iron oxides.

## 2. Physical Properties of Various Iron Oxide Compounds

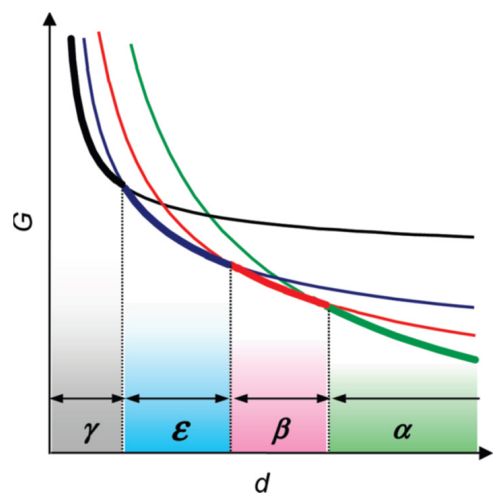
An Fe–O phase diagram is given in Figure 2. According to the ratio of  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$ , the phase diagram of Fe–O can be divided into six phase zones from right to left [40].



**Figure 2.** Equilibrium phase diagram for Fe–O system. Reprinted from [40], with permission from Elsevier.

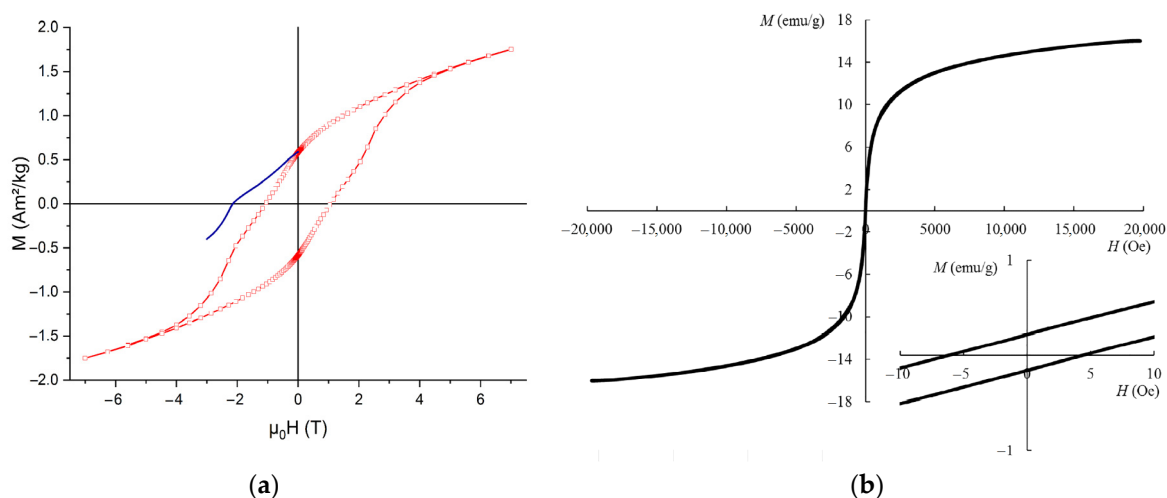
The shown phase diagram does not take into account the polymorphisms of ferric oxides. Sakurai et al. [41] discuss the crystal structures and magnetic properties of the four phases are the following: ferrimagnetic  $\gamma\text{-Fe}_2\text{O}_3$  with a spinel structure; ferrimagnetic

$\varepsilon$ -Fe<sub>2</sub>O<sub>3</sub> with an orthorhombic structure; antiferromagnetic  $\beta$ -Fe<sub>2</sub>O<sub>3</sub> with a bixbyite structure; and weak ferromagnetic  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with a corundum structure. The observed phase transformations for Fe<sub>2</sub>O<sub>3</sub> phases inside of the mesoporous silica matrix are due to the surface (or interface) energy  $G_S$  contribution to the total free energy  $G = G_B + (6V_m/d)G_S$ , where  $G_B$  is the free energy in the bulk,  $V_m$  is the molar volume and  $d$  is the diameter of the NP. The  $G$  vs.  $d$  curves should appear as shown in Figure 3.

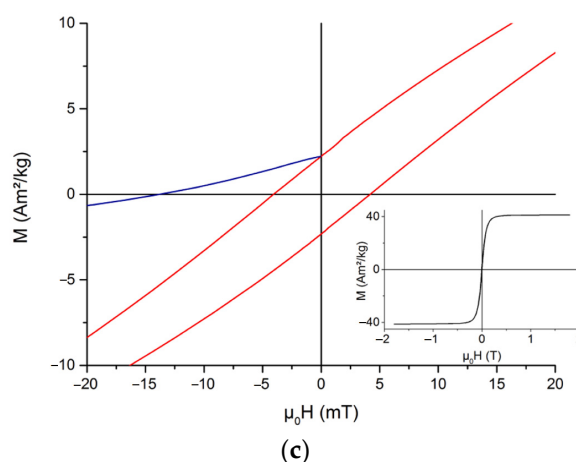


**Figure 3.** Representation of free energy  $G$  vs. particle diameter  $d$  curves for the four Fe<sub>2</sub>O<sub>3</sub> phases. Gray, blue, green and red lines represent the  $G$  values of  $\gamma$ -,  $\varepsilon$ -,  $\beta$ - and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, respectively. Thick solid lines indicate the most stable Fe<sub>2</sub>O<sub>3</sub> phases over the corresponding size ranges. Copyright 2009 by the American Chemical Society. Reprinted with permission from [41].

Such a strong correlation between the most stable iron oxide crystal phase and the matrix makes it possible to create nanomaterials with diametrically different magnetic properties. Our team has provided the studies on the natural and synthetic silica-based systems containing inclusions of iron oxide NPs. Figure 4 illustrates the static magnetic characteristics of the silica-based systems with NPs of  $\varepsilon$ -Fe<sub>2</sub>O<sub>3</sub> with a coercive force  $H_c = 1.07$  T [37],  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>-Fe<sub>3</sub>O<sub>4</sub> with  $H_c = 0.5$  mT [38] and natural magnetite from the Kovdor deposit with  $H_c = 4$  mT [6]. Thus, despite their almost identical chemical composition, the similar structures with iron oxides can possess different  $H_c$  values by more than three orders of magnitude.



**Figure 4.** Cont.



**Figure 4.** Static magnetic properties of various samples of silica–iron oxide systems: (a)—Hysteresis loop and backfield demagnetization curve of a sample containing  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub>. Reprinted from [37], license CC BY 4.0; (b)—Magnetic hysteresis curve of Fe<sub>m</sub>O<sub>n</sub>-SiO<sub>2</sub> synthetic colloidal nanoparticles. Reprinted from [38], with permission from Elsevier; (c)—Backfield curve and central part of the hysteresis loop of magnetite ore powder; full loop in the 1.8 T maximum field is shown in the inset. Reprinted from [6], with permission from Elsevier.

Iron oxide compounds can differ not only by their coercive force, as shown above, but also by their other physical characteristics. Some of them are shown in Table 1.

**Table 1.** Comparison of the physical characteristics of various iron oxide compounds [42–73].

| Parameter                                                  | Iron Oxide Compound |                                |                                          |                                         |                                          |                                            |
|------------------------------------------------------------|---------------------|--------------------------------|------------------------------------------|-----------------------------------------|------------------------------------------|--------------------------------------------|
|                                                            | FeO                 | Fe <sub>3</sub> O <sub>4</sub> | $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> | $\beta$ -Fe <sub>2</sub> O <sub>3</sub> | $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> | $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> |
| Mineral name                                               | Wüstite             | Magnetite                      | Hematite                                 | –                                       | Maghemite                                | –                                          |
| Crystal structure                                          | Cubic [42]          | Cubic spinel [43]              | Rhombohedral [44]                        | Cubic [62]                              | Cubic spinel [45]                        | Orthorhombic [46]                          |
| Static dielectric permittivity                             | 22.6 [47]           | 20–40 [48,49]                  | 12–26 [48–50]                            | n/a                                     | 20 [48]                                  | n/a                                        |
| High-frequency dielectric permittivity                     | 10.8 [47]           | 7–16 [51]                      | 7.6–7.9 [50]                             | n/a                                     | 14.2 [52]                                | 4–10 [53,54]                               |
| Saturation mass magnetization <sup>1</sup> at 300 K, emu/g | 11–18 [55,56]       | 92–94 [57]                     | 0.3–1.9 [58,59]                          | 0.02–0.05 [63,64]                       | 74–80 [60,61]                            | 15 [46]                                    |
| Curie/Neel point, K                                        | 196 [47,55]         | 838–856 [66]                   | 948–963 [41,58]                          | 110–119 [62,64]                         | 618–928 [41,67]                          | 480–495 [41,46]                            |
| Optical band gap at 300 K, eV                              | 1.0 [47]            | 0.2 [68,69]                    | 1.9–2.2 [59,70–72]                       | 1.7–1.9 [64,65]                         | 2.0 [60]                                 | 2.0–2.4 [73]                               |

<sup>1</sup> The values of saturation mass magnetization are given for the bulk materials.

The crystal structure of an iron oxide compound can be attributed to various symmetry groups: cubic for FeO [42], Fe<sub>3</sub>O<sub>4</sub> [43] and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> [45], rhombohedral for  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> [44], and orthorhombic for  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> [46]. Crystal structure variation influences the electron zone structure, which can be semi-metallic with an optical band gap (at 300 K) of 0.2 eV for Fe<sub>3</sub>O<sub>4</sub> [68,69] and semiconductive for FeO with an optical band gap of 1.0 eV [46], as well as for  $\alpha$ -,  $\gamma$ - and  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> with an optical band gap of 1.9–2.4 eV [58,59,70–73]. There is no clear correlation between the crystal structure of an iron oxide compound and its dielectric permittivity, for which values are in the range of 12–40 for static permittivity [46–49] and

4–16 for high-frequency permittivity [46,49–53]. Since the various iron oxides can possess antiferromagnetic or ferrimagnetic properties, their value of saturation mass magnetization can differ by a few orders of magnitude: from 0.3 emu/g (at 300 K) for pure hematite [57,58] to 92–94 emu/g for stoichiometric magnetite [56]. Such variation in electrical and magnetic characteristics lies in the versatility of the structures containing iron oxides for their wide spectrum of possible applications. The next section is devoted to the main mechanisms of iron oxide formation disclosed in the scientific literature.

### 3. Mechanisms of Iron Oxide Formation

The literature data on the iron oxide formation mechanisms were divided into several groups according to the iron oxide compounds in the studied structures: the structures containing pure crystal iron oxide phases, the ones containing iron oxide atomic clusters and amorphous iron oxides, the ones containing two co-existing iron oxide crystal phases, and the ones containing three or more co-existing iron oxide phases. Their formation mechanism, either natural or synthetic, is briefly described based on the data in the cited reference. Furthermore, their existing and potential (declared) applications are also given.

#### 3.1. The Structures Containing Pure Phases of FeO, Fe<sub>4</sub>O<sub>5</sub>, Fe<sub>3</sub>O<sub>4</sub>, and $\alpha$ -, $\beta$ -, $\gamma$ -, $\delta$ -, $\epsilon$ - and $\zeta$ -Polymorphs of Fe<sub>2</sub>O<sub>3</sub>

The information on the structures with a pure iron oxide crystal phase is given separately for each iron oxide compound in Tables 2–6.

Based on the gathered information, it was possible to determine the most frequently obtained form of the structures containing FeO (Table 2). This form is FeO NPs produced for biomedical applications via various chemical or physical routes, but not naturally originated, since this iron oxide compound is metastable under normal conditions [42,74–80].

As one of the stable iron oxide compounds, magnetite can be obtained from the various structures, including natural abiotic or biogenic and synthetic ones (Table 3). Fe<sub>3</sub>O<sub>4</sub> NPs can be considered as one of the most frequently used iron oxide magnetic materials and their production techniques are mostly chemical since this approach is most suitable for biomedical applications [81–89]. Additionally, “green” techniques, involving the use of natural plant extracts [90,91], biomimetic formation [92] or microbial mineralization [93–98], are widely used. Other commonly described forms of magnetite are its inclusion within ore samples [6,99–104] widely used in geosciences and the external magnetite layer of metal surfaces [105–109], which is an important object of iron corrosion studies.

The most stable (under normal conditions) iron oxide compound, hematite, attracts the highest attention of scientists, according to the overall amount of published papers (Table 4). Similarly to magnetite, the most frequently obtained form of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is a chemically synthesized NP, predominantly used for photocatalysis, biogeochemistry and the bioremediation of toxic compounds [105–119]. The second important form of hematite is the natural ore with inclusions of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, which is used in Earth sciences [117–124]. The use of the pure  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> is quite rare. The most frequently produced form of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> is as NPs (Table 5), including chemically synthesized [125–129] or biogenic [93].

Structures with  $\beta$ -Fe<sub>2</sub>O<sub>3</sub>,  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub>,  $\zeta$ -Fe<sub>2</sub>O<sub>3</sub>,  $\delta$ -Fe<sub>2</sub>O<sub>3</sub> or Fe<sub>4</sub>O<sub>5</sub> metastable phases (Table 6) are much less described compared to the main stable compounds of iron oxides.  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub> is the only one seriously applicable in biomedicine [130,131], electronics [132,133] or geosciences [134,135] and can be obtained both synthetically [37,136,137] or by extraction from various archeological objects [138–140]. There have also been some attempts to use  $\beta$ -Fe<sub>2</sub>O<sub>3</sub> NPs in biomedicine [63], sensors and lithium-ion batteries [20,64].



**Table 2.** Data on the structures containing an FeO phase.

| Composition                                      | Main Mechanisms of Iron Oxide Formation                                                                                                                    | Declared Applications                                                                   | Phase Verification Techniques                                                                                                         | Refs.      |
|--------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------|------------|
| FeO NPs                                          | Thermal decomposition of the iron(II) precursor, mechanochemical reduction of magnetite, flame synthesis, laser target interaction in liquid carrier media | Biomedicine, electronics, spintronics, magnetic force microscopy, metastability studies | XRD <sup>1</sup> , UV-Vis <sup>2</sup> , MALDI-TOF MS <sup>3</sup> , EELS <sup>4</sup> , F-AAS <sup>5</sup> HAADF-STEM <sup>6</sup> , | [41,74–80] |
| Ultra-thin FeO film                              | Oxidation of iron monocrystal surface                                                                                                                      | Iron oxidation kinetics study                                                           | RMDS <sup>7</sup>                                                                                                                     | [141]      |
|                                                  | Electron-beam deposition on Au(111) surface                                                                                                                | Iron catalysis, electronics, biomedicine                                                | STM <sup>8</sup>                                                                                                                      | [142]      |
| Millimeter-sized iron oxide particles            | Magnetite reduction with iron as reducing agent                                                                                                            | Catalysts for ammonia synthesis                                                         | TG-DSC <sup>9</sup>                                                                                                                   | [40]       |
| FeO layer on the metal alloy surface             | Invar oxidation in a static carbon dioxide atmosphere                                                                                                      | Iron oxidation kinetics study                                                           | XRD, TG-DSC, TEM <sup>10</sup>                                                                                                        | [143]      |
| Wüstite inclusions in titanomagnetite particles  | Titanomagnetite ironsand-fluidized bed reduction by hydrogen                                                                                               | Commercial iron making                                                                  | XRD                                                                                                                                   | [144]      |
| FeO powder                                       | Reduction of hematite in a gas-controlled electric furnace                                                                                                 | Earth's mantle sound velocity studies                                                   | XRD, IXS <sup>11</sup>                                                                                                                | [145]      |
| FeO inclusions in the mold flux                  | Iron oxide formation in molten mold flux                                                                                                                   | Study of the oxidation mechanism of mold flux-covered molten iron                       | XRF <sup>12</sup>                                                                                                                     | [146]      |
| FeO inclusions within the dense iron shell       | Porous hematite gas reduction under isothermal conditions                                                                                                  | Industrial exploitation of low-grade iron ores                                          | TG-DSC, XRD                                                                                                                           | [147]      |
| FeO clusters within the stable iron oxide matrix | The reduction of magnetite/hematite at temperatures of 400–500 °C                                                                                          | Iron catalysis                                                                          | Quantitative theoretical analysis                                                                                                     | [148]      |

<sup>1</sup> X-ray powder diffraction. <sup>2</sup> UV-visible(-NIR) spectroscopy. <sup>3</sup> Matrix-assisted laser desorption ionization time-of-flight mass spectrometry. <sup>4</sup> Electron energy loss spectroscopy. <sup>5</sup> Flame atomic absorption spectroscopy. <sup>6</sup> High-angle annular dark-field scanning transmission electron microscopy. <sup>7</sup> Reactive molecular dynamics simulations. <sup>8</sup> Scanning tunneling microscopy. <sup>9</sup> Thermogravimetry/differential scanning calorimetry. <sup>10</sup> Transmission electron microscopy. <sup>11</sup> Inelastic X-ray scattering. <sup>12</sup> X-ray fluorescence.

**Table 3.** Data on the structures containing an Fe<sub>3</sub>O<sub>4</sub> phase.

| Composition                                                     | Main Mechanisms of Iron Oxide Formation                                                                                                                                                                                                                                                                                                                          | Declared Applications                                                                                                                    | Phase Verification Techniques                                                                                                                                                   | Refs.           |
|-----------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------|
| Fe <sub>3</sub> O <sub>4</sub> NPs                              | Co-precipitation from iron salt solution, co-precipitation from iron oxyhydroxide solution, solvothermal synthesis, electrochemical formation from a pure iron, thermal decomposition of the iron oleate complex, biomimetic process with use of a leaf extract, nucleation mediated by iron-binding protein Mms6, biogenesis with a use of amyloid peptide Aβ42 | Biomedicine, magnetic separation, antimicrobial and antioxidant applications, contaminant removal, black pigment production, ferrofluids | TEM, XRD, SAXS <sup>1</sup> , RS <sup>2</sup> , FTIR <sup>3</sup> , XPS <sup>4</sup> , HAADF-STEM, EELS, TG-DSC, UV-Vis, PL <sup>5</sup> , MSP <sup>6</sup> , SAED <sup>7</sup> | [81–91,149–162] |
| Bacterial magnetosomes                                          | Bacterial biomineralization, transient phosphate-rich ferric hydroxide reduction to magnetite, formation by dissimilatory iron-reducing bacteria                                                                                                                                                                                                                 | Biomedicine, paleomagnetism, microbial iron cycle studies, bioremediation of toxic compounds                                             | HAADF-STEM, TEM, XAS <sup>8</sup> , SAED, XMCD <sup>9</sup>                                                                                                                     | [93–98,163]     |
| Inclusions of Fe <sub>3</sub> O <sub>4</sub> within ore samples | Abiotic hydrothermal mineralization, iron oxide formation derived from continental weathering, extrusive magmatic formation from iron oxide-melt liquid                                                                                                                                                                                                          | Geochemistry, environmental magnetism studies, early Earth iron cycle studies, origin and evolution of iron oxides studies               | XRD, RS, XPS, EDS <sup>10</sup> , ICP-AES <sup>11</sup>                                                                                                                         | [99–102]        |
| External magnetite layer on a metal surface                     | Oxidation of a steel surface, slow oxidation of green rusts at room temperature, high-temperature corrosion                                                                                                                                                                                                                                                      | Corrosion studies                                                                                                                        | XRD, EDS, XRF, RS, XPS, AES <sup>12</sup>                                                                                                                                       | [105–110]       |
| Fe <sub>3</sub> O <sub>4</sub> microparticles                   | Microbial-induced precipitation with the use of <i>Sporosarcina pasteurii</i>                                                                                                                                                                                                                                                                                    | Green synthesis of magnetite                                                                                                             | EDS                                                                                                                                                                             | [28]            |
|                                                                 | Aging of ferrous hydroxide gels at elevated temperatures                                                                                                                                                                                                                                                                                                         | Colloidal crud formation studies                                                                                                         | XRD, TEM                                                                                                                                                                        | [162]           |



Table 3. Cont.

| Composition                                                             | Main Mechanisms of Iron Oxide Formation                                                   | Declared Applications                   | Phase Verification Techniques  | Refs. |
|-------------------------------------------------------------------------|-------------------------------------------------------------------------------------------|-----------------------------------------|--------------------------------|-------|
| Self-assembled Fe <sub>3</sub> O <sub>4</sub> mesocrystalline films     | Heat-up method with the use of iron(III) chloride and sodium oleate                       | Biomedicine and industrial applications | TEM, SAED, XAS, SAXS           | [163] |
| Fe <sup>0</sup> /Fe <sub>3</sub> O <sub>4</sub> composite               | Controlled reduction of the starting Fe <sub>3</sub> O <sub>4</sub> with H <sub>2</sub>   | Treatment of wastewater                 | MSP, XRD                       | [164] |
| Magnetite nanowires                                                     | Supercritical fluid inclusion within a mesoporous silica matrix                           | Soft magnetic materials                 | TEM, SAED, XRD, FTIR           | [165] |
| Inclusions of Fe <sub>3</sub> O <sub>4</sub> NPs                        | Bacterial reduction of amorphous hydrous ferric oxide                                     | Biogeochemistry                         | TEM, SAED, XRD, EDS            | [166] |
| Fe <sub>3</sub> O <sub>4</sub> layer on the zerovalent iron surface     | Surface oxidation of iron by oxygen in an aqueous medium                                  | Organic pollutant removal               | EDS, XRD                       | [167] |
| Epoxy/magnetite nanocomposites                                          | Reduction of anhydrous ferric chloride by ammonium hydroxide                              | Marine coatings of steel                | FTIR, XRD, TEM                 | [168] |
| Iron oxide nanocomposite hydrogel                                       | Co-precipitation process by ammonium hydroxide                                            | Biomedicine                             | XRD, TEM, TG-DSC, EDS          | [169] |
| Surface film containing Fe <sub>3</sub> O <sub>4</sub> NPs              | Bacterial mineralization in the air–water interface in Arctic tundra waters               | Anaerobic microbial carbon cycle        | TEM, EDS, STEM, EELS, FTIR, RS | [170] |
| Nanocomposite hydrogel with Fe <sub>3</sub> O <sub>4</sub> NPs          | Reduction with ammonia from a remixed solution of FeCl <sub>3</sub> and FeCl <sub>2</sub> | Biomedicine                             | TEM, XRF, EDS, TG-DSC, FTIR    | [171] |
| Biochar composite with Fe <sub>3</sub> O <sub>4</sub> NPs               | One-pot solvothermal method using phoenix tree leaf-derived biochar                       | Treatment of wastewater                 | TEM, XRD, FTIR, XPS, ICP-AES   | [172] |
| Fe <sub>3</sub> O <sub>4</sub> NP inclusions in the surface layer       | Formation of NPs along with cracks and pores during pre-oxidation                         | Plasma nitriding of steel               | XRD                            | [173] |
| Chitosan/graphene oxide composite with Fe <sub>3</sub> O <sub>4</sub>   | Co-precipitation of Fe <sub>3</sub> O <sub>4</sub> and chitosan/graphene oxide            | Organic pollutant removal               | XRD, XPS, RS, FTIR             | [174] |
| Fe <sub>3</sub> O <sub>4</sub> layer on carbon fibers of a carbon paper | Deposition on the carbon paper gas diffusion layer at the cathode                         | Corrosion studies                       | XRD, EDS                       | [175] |

Table 3. Cont.

| Composition                                                                | Main Mechanisms of Iron Oxide Formation                                                           | Declared Applications                      | Phase Verification Techniques   | Refs. |
|----------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------|--------------------------------------------|---------------------------------|-------|
| Mesocrystals assembled from Fe <sub>3</sub> O <sub>4</sub> nanocubes       | Heat-up method with the use of iron(III) chloride and sodium oleate                               | Mesocrystal applications                   | TEM                             | [176] |
| Fe <sub>3</sub> O <sub>4</sub> nanorods                                    | Formation in electron-beam-induced deposition from iron pentacarbonyl                             | Electronics                                | TEM, EELS                       | [177] |
| Lipase immobilized on coated Fe <sub>3</sub> O <sub>4</sub> NPs            | Solvothermal method with the use of FeCl <sub>3</sub> ·6H <sub>2</sub> O and ethylene glycol      | Biodiesel production                       | TEM, XRD, FTIR                  | [178] |
| Spherical mesoporous magnetite aggregates                                  | Precipitation from iron(III) ethoxide with ethanol in the surfactant solution                     | Catalysis, sustainability                  | FTIR, XPS, EDS, TEM, MSP        | [179] |
| Perfluorocarbon-loaded hydrogel microcapsules                              | Coaxial interface shearing double emulsion method                                                 | Biomedicine                                | –                               | [180] |
| Mesoporous magnetite                                                       | Ball milling of Fe <sub>3</sub> O <sub>4</sub> and SiO <sub>2</sub> followed by partial reduction | Recyclable absorbent for toxic Cr(VI) ions | TEM, XRD, XPS, ICP-AES          | [181] |
| Magnetite crystal model                                                    | Local spin-density approximation density-functional calculation                                   | Magnetite electron structure studies       | Density-functional calculations | [43]  |
| Spherulite nanostructure with inclusions of Fe <sub>3</sub> O <sub>4</sub> | Electron-beam irradiation of the precursor solution with iron nitrate                             | Crystal growth dynamics studies            | TEM, STEM, EDS                  | [182] |

<sup>1</sup> Small-angle X-ray scattering. <sup>2</sup> Raman spectroscopy. <sup>3</sup> Fourier-transform infrared spectroscopy. <sup>4</sup> X-ray photoelectron spectroscopy. <sup>5</sup> Photoluminescence spectroscopy. <sup>6</sup> Mössbauer spectroscopy. <sup>7</sup> Selected area electron diffraction. <sup>8</sup> X-ray absorption spectroscopy. <sup>9</sup> X-ray magnetic circular dichroism. <sup>10</sup> Energy-dispersive X-ray spectroscopy. <sup>11</sup> Inductively coupled plasma atomic (optical) emission spectroscopy. <sup>12</sup> Auger electron spectroscopy.

**Table 4.** Data on the structures containing an  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> phase.

| Composition                                                           | Main Mechanisms of Iron Oxide Formation                                                                                                                                                                                              | Declared Applications                                                                                                                   | Phase Verification Techniques                                                                        | Refs.             |
|-----------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------|-------------------|
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> NPs                          | Hydrothermal synthesis, precipitation from a ferric salt solution using a natural leaf extract, precipitation and aging of ferrihydrite in an oxidized system, direct transformation of $\alpha$ -FeOOH via high-energy ball milling | Biomedicine, bioremediation of toxic compounds, photocatalysis, geochemistry, electronics, antibacterial activity studies, geochemistry | XRD, FTIR, UV-Vis, EDS, TEM, RS, XPS, XAS, ICP-AES, EPR <sup>1</sup> , HAADF-STEM, WAXS <sup>2</sup> | [105–111,180–192] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in ore samples | Precipitation from oxygenated iron-rich water or biomineralization, dissolution of Fe(III) hydroxides by Fe(III)-reducing bacteria, terrestrial subglacial oxidation of glacial iron fluvial deposition                              | Terrestrial iron oxide concretion studies, Precambrian iron formation studies, Antarctic glacier studies, biogeochemistry               | EDS, RS, TEM, HAADF-STEM, XRD, SAED, FTIR, UV-Vis                                                    | [112–119]         |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> layer on a metal surface     | Anodic potentiostatic oxidation of stainless steel sheet                                                                                                                                                                             | Anodic passivation of stainless steel                                                                                                   | AES                                                                                                  | [193]             |
|                                                                       | Oxidation of steel in an O <sub>2</sub> -N <sub>2</sub> atmosphere at high temperature                                                                                                                                               | Improvement of steel coating quality                                                                                                    | TEM, EDS, GD-OES <sup>3</sup>                                                                        | [194]             |
|                                                                       | Corrosion of chromia-forming alloys in simplified combustion atmosphere                                                                                                                                                              | Fireside corrosion studies                                                                                                              | EDS, XRD                                                                                             | [195]             |
| Porous $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanostructures        | Hydrothermal synthesis from FeCl <sub>3</sub> ·6H <sub>2</sub> O in a microwave reactor                                                                                                                                              | Lithium-ion batteries                                                                                                                   | XRD, TEM, SAED, XPS, TG-DTG                                                                          | [196]             |
|                                                                       | Sol-gel transformations of precursors in self-organized nanocellulose                                                                                                                                                                | Energy conversion and storage                                                                                                           | XRD, TEM, SAED, XPS, TG-DSC                                                                          | [197]             |
| Martian hematite deposits                                             | Precipitation from oxygenated iron-rich water or biomineralization                                                                                                                                                                   | Search for evidence of life on Mars                                                                                                     | EDS, TEM                                                                                             | [198]             |
| Hematite layers on sandstone grains                                   | Precipitation from oxidizing iron-saturated fluid                                                                                                                                                                                    | Geochemistry                                                                                                                            | XRD, ICP-MS <sup>4</sup>                                                                             | [199]             |
| Double-walled hematite nanotubes                                      | Growth of Fe nanowires inside porous templates and oxidation                                                                                                                                                                         | Photocatalysis, biomedicine                                                                                                             | XRD, EELS, HAADF-STEM, RS                                                                            | [200]             |

Table 4. Cont.

| Composition                                                                 | Main Mechanisms of Iron Oxide Formation                                                           | Declared Applications                   | Phase Verification Techniques  | Refs. |
|-----------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------|-----------------------------------------|--------------------------------|-------|
| Coral-like and nanowire $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>            | Thermal oxidation of iron foils in air- and water vapor-assisted conditions                       | Removal of Cr ions from aqueous systems | XRD, RS, TEM, XPS              | [201] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> NPs on mineral surfaces            | Weathering of Fe-bearing silicate minerals or partial oxidation of Fe <sub>3</sub> O <sub>4</sub> | Paleoclimate studies                    | XRD, TEM, SAED                 | [202] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanorods                           | Controlled aqueous growth from FeCl <sub>3</sub> ·6H <sub>2</sub> O and NaNO <sub>3</sub>         | Photoelectrochemical water splitting    | XRD                            | [203] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in regolith simulant | Ball milling of commercial $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> samples in isopropyl alcohol  | Combustion studies                      | XRD, TG-DSC                    | [204] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in stone matrix      | Bacterial mineralization                                                                          | Heritage sciences                       | XRD, EDS, RS                   | [205] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in auriferous quartz | Terrigenous abiotic mineralization                                                                | Geochemistry                            | EDS                            | [206] |
| Hematite layers on sandstone grains                                         | Terrigenous co-precipitation with sandstone and uranium                                           | Geochemistry of radionuclides           | Gamma-ray spectrometry, ICP-MS | [207] |
| Hematite inclusions encapsulated in chert                                   | Dehydration of the interstitial goethite to hematite microplates                                  | Geochemistry                            | TEM, XRD, EDS                  | [208] |
| Hollow $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanofibers                  | Electrospinning with a use of iron chloride and poly(vinylpyrrolidone)                            | Photoelectrochemical water splitting    | EDS, TEM, SAED, TG-DSC, UV-Vis | [209] |
| Fossilized bacteria with $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>           | Biomining by anoxygenic photoferrotrophy                                                          | Biogeochemistry                         | RS                             | [210] |
| Porous $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> xerogel and aerogel         | Sol-gel synthesis from Fe(III) salts with addition of propylene oxide                             | Catalysis, sensors, biology             | TEM                            | [211] |
| Iron oxide nanostructures                                                   | Microbial Fe(II) oxidation of carbonate green rust by Fe(II)-oxidizing bacteria                   | Precambrian iron formation studies      | MSP                            | [212] |
| Iron oxide biogenic precipitates                                            | Bacterial mineralization                                                                          | Biogenic iron oxide formation studies   | XAS                            | [213] |

Table 4. Cont.

| Composition                                                                | Main Mechanisms of Iron Oxide Formation                                                                                         | Declared Applications                        | Phase Verification Techniques          | Refs. |
|----------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------|----------------------------------------|-------|
| Steel-wearing ejected debris with $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> | Steel fretting wear controlled by oxygen ingress to the contact                                                                 | Steel fretting wear studies                  | XRD                                    | [214] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> NPs on a steel surface            | Oxidation of iron-bonded diamond precision-polishing wheel                                                                      | Grinding of hard and brittle materials       | XRD, XPS, TEM                          | [215] |
| Nanostructured $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> films              | Electrochemical anodization of steel in an alkaline solution                                                                    | Photocatalysis, anti-bioadhesion             | RS, UV-Vis                             | [216] |
| Monodispersed micaceous $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>           | Hydrothermal synthesis from iron chromium hydroxide precursors                                                                  | Iron chromium grinding waste recycling       | ICP-AES, XRD, XPS                      | [217] |
| Nanoporous $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> layer on an iron foil  | Anodization of iron in an ethylene glycol and NH <sub>4</sub> F aqueous solution                                                | Photocatalysis                               | TEM, RS, XRD, UV-Vis, EDS, EELS        | [218] |
| Natural $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> from the iron deposits    | Terrigenous abiotic mineralization                                                                                              | Photocatalytic recycling of toxic wastewater | RS, EDS, UV-Vis                        | [219] |
| Nanocomposite containing $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>          | Wet impregnation of Co <sub>3</sub> O <sub>4</sub> powder with an Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O solution | Catalysis                                    | XPS, XRD, TG-DSC, EDS, TEM             | [220] |
| Stepped $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> (0001) surfaces           | First principles spin-polarized density-functional theory simulation                                                            | Chloride-induced iron depassivation studies  | Density-functional theory calculations | [221] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> powder                            | In situ generation of iron oxide via decomposition of Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O                      | Catalysis                                    | XRD                                    | [222] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanorods                          | Hydrothermal precipitation and air calcination of goethite nanorods                                                             | Catalysis, lithium-ion batteries, sensors    | XRD, MSP, UV-Vis, EDS, TG-DSC          | [223] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nano- and microparticles          | Chemically synthesized commercial $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> samples                                              | Mechanisms of oxide toxicity toward bacteria | FTIR, XAS                              | [224] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> nanowires                         | Heating of iron wires suspended between two electric contacts                                                                   | Vacuum electronic devices                    | TEM, EDS, XPS, RS                      | [225] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> layer on zerovalent iron NPs      | Iron oxide film formation under aerobic conditions                                                                              | Remediation of water pollutants              | TEM, FTIR, XPS, XRD                    | [226] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in rock varnish     | Terrigenous abiotic mineralization or biotic processes                                                                          | Geomicrobiology                              | XRD, RS, EDS                           | [227] |

Table 4. Cont.

| Composition                                                                 | Main Mechanisms of Iron Oxide Formation                                              | Declared Applications                          | Phase Verification Techniques | Refs. |
|-----------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------------------------------------|-------------------------------|-------|
| Nanolayers of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in polymer composite | Iron pentacarbonyl transformation with diamond anvil cells in Ar gas                 | High-energy density solid studies              | RS, TEM, XRD                  | [228] |
| Jian ware blue-colored glaze with $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>  | Calcination of a milled mix at a high temperature in oxidizing atmosphere            | Ancient ceramics studies                       | XRD, UV–Vis, TEM, XPS         | [229] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in sediment samples  | Microbial reduction of surface Fe(III) by iron-reducing bacteria                     | Microbial iron reduction studies               | XRD                           | [230] |
| Core-shell iron/iron oxide NPs                                              | Zerovalent Fe core-controlled oxidation during deposition                            | Oxide formation under e-beam radiation studies | TEM, EELS                     | [231] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> film on a dielectric substrate     | Liquid-phase atomic layer deposition of crystalline hematite                         | Catalysis, sensors, lithium-ion batteries      | XRD, UV–Vis                   | [232] |
| Cube-shaped $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> microstructures        | Facile hydrothermal method using hydrated ferric nitrate and NaOH                    | Ethanol gas sensing                            | XRD, FTIR, EDS, RS            | [233] |
| Iron oxide/Ti composites                                                    | Plasma electrolytic oxidation, impregnation and annealing                            | Phenol photodegradation                        | XRD, EDS, FTIR, XPS           | [234] |
| Microporous $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> NPs                    | Precipitation from iron(II) sulfate using a natural leaf extract                     | Sustainability                                 | XRD, UV–Vis, XPS, FTIR        | [235] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in artificial clay   | Fe(OH) <sub>3</sub> colloid mixing into chemically pure kaolin                       | Laterite engineering                           | XRD                           | [236] |
| Iron oxide nanotubes                                                        | Potentiostatic anodization of iron foil in electrolytes containing NH <sub>4</sub> F | Catalysis, sensors, supercapacitors            | XRD, TEM, SAED                | [237] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> thin film                          | Spray pyrolysis from FeCl <sub>3</sub> and methanol solution                         | Electrochemical supercapacitors                | XRD, UV–Vis                   | [44]  |
| Corroded steel tube samples with $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>   | Steel corrosion in an aqueous medium with oxygen and chlorine                        | Pipeline corrosion assessment                  | XRD, EDS, TEM, SAED           | [238] |
| Inclusions of $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> in stone samples     | Formation by washing and leaching of a stone object by rainwater                     | Limestone artifact studies                     | RS, FTIR, EDS, XRF            | [239] |
| Iron oxide-loaded slag                                                      | Precipitation from FeCl <sub>3</sub> solution with NaOH into melted slag             | Arsenic removal from water                     | ICP-AES, XRD                  | [240] |

Table 4. Cont.

| Composition                                                                | Main Mechanisms of Iron Oxide Formation                                                            | Declared Applications                                  | Phase Verification Techniques        | Refs. |
|----------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------|--------------------------------------------------------|--------------------------------------|-------|
| 3D-ordered macroporous $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>            | Impregnation of polymer matrices and high-temperature calcination                                  | Catalysis                                              | XRD, TG-DSC, FTIR, SAED, UV-Vis, XPS | [241] |
| $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> /mesoporous silica core-shell NPs | Solvothermal synthesis from ferric nitrate with sol-gel silica coating                             | Catalysis, biomedicine                                 | XRD, TEM, FTIR, UV-Vis               | [242] |
| Spindle-shaped $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> mesocrystal        | Interface-driven nucleation by ferrihydrate oxidation and attachment                               | Thermoelectronics, photonics, catalysis, photovoltaics | TEM, SAED, FTIR, EDS                 | [243] |
| Hematite nanopillars                                                       | Electron-beam evaporation using anodized aluminum oxide templates with well-defined pore diameters | Photoelectrochemical water splitting                   | XRD, XPS, UV-Vis                     | [244] |

<sup>1</sup> Electron paramagnetic resonance spectroscopy. <sup>2</sup> Wide-angle X-ray scattering. <sup>3</sup> Glow-discharge optical emission spectrometry. <sup>4</sup> Inductively coupled plasma mass spectrometry.



**Table 5.** Data on the structures containing a  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> phase.

| Composition                                                          | Main Mechanisms of Iron Oxide Formation                                                                                                                                       | Declared Applications                                                                                                                           | Phase Verification Techniques                                                                                  | Refs.        |
|----------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------|--------------|
| $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> NPs                         | Solvothermal synthesis from iron salts, bacterial mineralization, lepidocrocite calcination in an air atmosphere, hydrothermal and solvothermal synthesis from salt solutions | Catalysis, biomedicine, nucleation and formation of biogenic iron oxide studies, electronics, maghemite to hematite transition studies, sensors | TEM, EDS, SAED, XRD, XPS, EPR, FTIR, UV-Vis, ICP-AES, HAADF-STEM, MSP, in situ total scattering, XAS, SAXS, RS | [93,125–129] |
| $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> NPs in silica matrix        | Gas-phase synthesis in a furnace aerosol reactor from iron pentacarbonyl                                                                                                      | Biomedicine                                                                                                                                     | XRD, TEM, EDS, FTIR, UV-Vis                                                                                    | [245]        |
|                                                                      | Dehydration of iron(III) hydroxide to magnetite followed by oxidation                                                                                                         | Catalysis                                                                                                                                       | XRD, FTIR                                                                                                      | [246]        |
| $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> powder                      | Chemically synthesized commercial $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> samples                                                                                            | Catalytic oxidation of S(IV)                                                                                                                    | ICP-MS, FTIR                                                                                                   | [247]        |
| 26-faceted maghemite polyhedrons                                     | Direct burning of ferrocene in different solvents in an alcohol lamp                                                                                                          | Lithium-ion batteries                                                                                                                           | XRD, TEM                                                                                                       | [248]        |
| Magnetic polymeric NPs with $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> | Co-precipitation of FeCl <sub>3</sub> /FeCl <sub>2</sub> ·4H <sub>2</sub> O with NH <sub>4</sub> OH solution                                                                  | Biomedicine                                                                                                                                     | TEM, TG-DSC, FTIR                                                                                              | [249]        |
| $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> NP superlattice thin films  | Chemically synthesized commercial $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> samples                                                                                            | Electronics, optical coatings                                                                                                                   | Grazing incidence small angle X-ray scattering                                                                 | [250]        |
| Maghemite-decorated graphene nanoscrolls                             | Hydrolysis of FeCl <sub>3</sub> ·6H <sub>2</sub> O and W(CO) <sub>6</sub> , promoted with hydrazine                                                                           | Energy storage                                                                                                                                  | TEM, XPS, TG-DSC, RS                                                                                           | [251]        |
| Hollow iron oxide NPs                                                | Gas-phase vaporization synthesis of Fe NPs and oxidation to $\gamma$ -Fe <sub>2</sub> O <sub>3</sub>                                                                          | Optics, nanoelectronics                                                                                                                         | TEM, HAADF-STEM, EDS                                                                                           | [252]        |
| Mesoporous iron oxide                                                | Inverse micelle synthesis from Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O butanol solution                                                                          | Arsenic removal from water                                                                                                                      | XRD, FTIR, RS, XPS                                                                                             | [253]        |

**Table 6.** Data on the structures containing a  $\beta$ -Fe<sub>2</sub>O<sub>3</sub>,  $\epsilon$ -Fe<sub>2</sub>O<sub>3</sub>,  $\zeta$ -Fe<sub>2</sub>O<sub>3</sub>,  $\delta$ -Fe<sub>2</sub>O<sub>3</sub> or Fe<sub>4</sub>O<sub>5</sub> metastable phase.

| Composition                                                                                                | Main Mechanisms of Iron Oxide Formation                                                                                                       | Declared Applications                       | Phase Verification Techniques          | Refs. |
|------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------|----------------------------------------|-------|
| Pristine and co-substituted $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub>                                     | Simulated crystal structure with a use of density-functional calculations                                                                     | Magnetoelectric material development        | Density-functional theory calculations | [254] |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> embedded in biomimetic graphene                                 | Precipitation from ferric and ferrous chloride with a biocompatible polymer                                                                   | Biomedicine                                 | XRD, TEM, SAED, RS, XPS, TG-DSC, FTIR  | [130] |
| Epitaxially stabilized thin-film $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub>                                | Epitaxy on (100)-oriented yttrium-stabilized zirconia substrates                                                                              | Electronics, permanent magnets, biomedicine | XRD, HAADF-STEM                        | [136] |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> in ancient black glazed wares                                   | Surface iron enrichment and a firing of wares under reducing conditions                                                                       | Electronics, spintronics                    | XRF, XAS, XRD, RS, TEM, EDS            | [132] |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> NPs                                                             | Hydrolysis of tetraethoxysilane in a solution of ferric nitrate and annealing                                                                 | Wireless technologies, electronics          | XRD, TEM, THz-TDS <sup>1</sup>         | [255] |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> inclusions in fired clay samples                                | Stabilization of $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> NPs in a matrix of silicates during firing of clays                               | Paleomagnetism                              | XRD, EDS                               | [134] |
| Y <sub>3</sub> Fe <sub>5</sub> O <sub>12</sub> matrix including $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> | Formation of $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> in the Y <sub>3</sub> Fe <sub>5</sub> O <sub>12</sub> matrix using the sol–gel method | Magnetoelectric material development        | XRD, XPS, TG-DSC, FTIR                 | [256] |
| $\delta$ -Fe <sub>2</sub> O <sub>3</sub> in layered double hydroxyl                                        | Dry impregnation of layered double hydroxyl structure with ferric nitrate                                                                     | Photocatalysis                              | XRD, FTIR, XRF, TG-DSC, UV–Vis         | [22]  |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> -SiO <sub>2</sub>                                               | Reverse micelle method with the use of ferric nitrate                                                                                         | Oxidative dehydrogenation of n-butene       | XRD                                    | [136] |
| $\beta$ -Fe <sub>2</sub> O <sub>3</sub>                                                                    | Milling of Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> and NaCl and calcination at 550 °C in air                                          |                                             |                                        |       |
| Ga-substituted $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> NPs                                              | Calcination of a mesoporous silica impregnated with metal nitrates                                                                            | Biomedicine                                 | XRD, XRF, TEM, ICP-MS                  | [131] |

Table 6. Cont.

| Composition                                                                                                         | Main Mechanisms of Iron Oxide Formation                                                                                                      | Declared Applications                               | Phase Verification Techniques            | Refs.   |
|---------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------|------------------------------------------|---------|
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> in archeological brick and baked clay                                    | High-temperature firing of bricks and clays in air                                                                                           | Archaeomagnetism, paleomagnetism                    | RS                                       | [138]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> in archeological samples, $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> NPs | Sol-gel synthesis from ferric and barium nitrate with tetraethyl orthosilicate                                                               |                                                     | XRD, RS                                  | [135]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> coatings on Si(100) substrates                                           | One-pot sol-gel recipe assisted by glycerol in an acid medium                                                                                | Paleomagnetism, biomedicine, electronics            | RS, XAS, EELS, HAADF-STEM                | [257]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> /SiO <sub>2</sub> composite powder                                       | Sol-gel synthesis from ferric and barium nitrate with tetraethyl orthosilicate                                                               | Electronics                                         | XRD, TEM                                 | [133]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> nanorods                                                                 | Chemical vapor deposition from the Fe organic liquid source                                                                                  | Photocatalysis, electronics                         | XPS                                      | [258]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> /SiO <sub>2</sub> composite                                              | Sol-gel synthesis from nitrate with tetraethyl orthosilicate and nitric acid                                                                 | Electronics, spintronics, magnetizable printing     | TG-DSC, XRD, TEM                         | [259]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> NPs                                                                      | Immersion of mesoporous silica with an FeSO <sub>4</sub> or Fe(C <sub>10</sub> H <sub>9</sub> CHO) solution and high-temperature calcination | High-coercivity material development                | TEM, XRD, MSP, TEM, SAED                 | [20,41] |
| $\beta$ -Fe <sub>2</sub> O <sub>3</sub> NPs                                                                         |                                                                                                                                              | Sensors, lithium-ion batteries                      |                                          |         |
| Epitaxial $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> films on GaN substrate                                         | Pulsed laser deposition on the Ga-terminated surface of the GaN (0001)                                                                       | Electronics                                         | XRD, RHEED <sup>2</sup> , TEM, XAS, XMCD | [260]   |
| Silica-coated $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> NPs                                                        | Sol-gel treatment of $\beta$ -FeOOH nanorods with tetraethoxysilane and calcination                                                          | Electronics                                         | XRD, TEM, EDS, MSP                       | [261]   |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> in a Hare's Fur Jian ware                                                | High-temperature firing of local iron-rich area on the ceramic glaze                                                                         | Magnetoresistance materials                         | XRF, XAS, EDS, XRD, RS                   | [140]   |
| Metal-substituted $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub>                                                        | Impregnation of mesoporous silica NPs with rhodium-substituted $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub>                                    | Electronics, magnetic force microscopy, biomedicine | XRD                                      | [262]   |

Table 6. Cont.

| Composition                                                         | Main Mechanisms of Iron Oxide Formation                                                                     | Declared Applications                           | Phase Verification Techniques          | Refs. |
|---------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------|-------------------------------------------------|----------------------------------------|-------|
| $\beta$ -Fe <sub>2</sub> O <sub>3</sub> NPs                         | Thermally-induced solid-state reaction of NaCl with Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> in air  | Sensors, lithium-ion batteries                  | XRD, MSP, TEM, SAED                    | [20]  |
| $\zeta$ -Fe <sub>2</sub> O <sub>3</sub>                             | Pressure treatment of $\beta$ -Fe <sub>2</sub> O <sub>3</sub> NPs at pressures above 30 GPa                 | n/a                                             |                                        |       |
| $\epsilon$ -Fe <sub>2</sub> O <sub>3</sub> in a thin MgO(111) layer | Pulsed laser deposition from MgO and Fe <sub>2</sub> O <sub>3</sub> targets ablated using a KrF laser       | Electronics                                     | RHEED, XRD, neutron reflectometry      | [263] |
| Single crystal of Fe <sub>4</sub> O <sub>5</sub>                    | Synthesis in the diamond anvil cell at high pressure after laser heating                                    | Solid Earth studies                             | Density-functional theory calculations | [23]  |
| Nanometer-scale lamellae of Fe <sub>4</sub> O <sub>5</sub>          | High-pressure and high-temperature multi-anvil synthesis                                                    | Deep Earth studies                              | XRD, TEM, SAED, EDS, STEM              | [264] |
| Powder of Fe <sub>4</sub> O <sub>5</sub>                            | High-pressure and high-temperature direct synthesis from a mixture of Fe <sub>3</sub> O <sub>4</sub> and Fe | Electronics                                     | XRD, neutron diffraction               | [265] |
| $\beta$ -Fe <sub>2</sub> O <sub>3</sub> NPs                         | Thermally-induced solid-state reaction of NaCl with Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> in air  | Optoelectronics, sensors, lithium-ion batteries | XRD, MSP, TEM                          | [64]  |
|                                                                     | Hydrolysis of 2M FeCl <sub>3</sub> in boiling water and cooling down slowly at room temperature             | Biomedicine                                     | UV-Vis, TEM, XRD, FTIR, EDS, SAED      | [63]  |

<sup>1</sup> Terahertz time-domain spectroscopy. <sup>2</sup> Reflection high-energy electron diffraction.

### 3.2. The Structures Containing Iron Oxide Atomic Clusters and an Amorphous Iron Oxide Phase

In their thermodynamic equilibrium state under normal conditions, iron oxides possess a crystal structure; therefore, there are only a few papers describing compositions with amorphous or poorly crystalline iron oxides, which are obtained via chemical [266–269] or biomineralization routes [270] and intended for various potential applications (Table 7). Iron oxide atomic clusters are a more frequently studied object and can be obtained either by synthetic chemical [271–274], physical [275] or biomimetic [276,277] techniques. Potential applications for iron oxide clusters include biomedicine [271,273], electronics [271,276], catalysis [273,274], and natural iron storage process studies [276].

The next type of iron oxide structure is ultra-thin, including two-dimensional films on metal surfaces [142,278–280], which are obtained using various chemical and physical techniques and can be applied to the production of molecular hydrogen [278], removal of contaminants [279], catalysis [142] and electronics [280]. Simulated iron oxide atomic clusters [281] and ultra-thin layers on a metal surface [282] are also described and can be useful for the prediction of the magnetic properties of  $\text{FeO}_x$  NPs [281] and chemical water treatment technique development [282].

### 3.3. The Structures Containing Two Co-Existing Iron Oxide Crystal Phases

Real structures containing iron oxides in various cases are inhomogeneous, for example, due to the partial oxidation of magnetite to maghemite for synthetic [38,283–293], natural abiotic [6] and biogenic [294] origins. In this section, compositions with iron oxides containing two co-existing crystal phases are described (Tables 8–11).

Crystal phases of  $\alpha\text{-Fe}_2\text{O}_3$  and  $\gamma\text{-Fe}_2\text{O}_3$  quite rarely co-exist (Table 8), in comparison, for example, with the phases of magnetite and maghemite, according to the analyzed research articles. Nevertheless, such a combination can be found in natural and synthetic objects, including oxidized iron items [295],  $\alpha/\gamma\text{-Fe}_2\text{O}_3$  isoelement synthetic heterostructures with different crystal content [296], loess and paleosol [297] and saprolite soils [298] samples, graphene-iron oxide nanotube composites [299] and polyacrylonitrile/iron oxide composites [300]. The main applications of such structures are in the removal of contaminants [299–301], pedogenic process studies [297,298] and corrosion studies [302].

A more frequently discovered combination is the co-existence of  $\alpha\text{-Fe}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$  crystal phases (Table 9). They can be found in natural ore samples and can be explained by abiotic [303–308] or biogenic [309–311] processes. Besides these natural formations, such combinations of iron oxide phases can be synthetically obtained with the use of chemical [312–314], physical [315] or biomimetic [316] techniques. The main applications of the compositions are geosciences [306,307,310,311], biomedicine [312,314,317] and catalysis [313,316,317].

Cubic iron oxides, wüstite and magnetite can be co-existing (Table 10), despite these cases being rare, in comparison to the structures containing co-existing  $\alpha\text{-Fe}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$  phases [303–328], and can be attributed to applications of iron oxides in Earth's mantle studies [329], porous iron growth mechanism studies [330], environmental remediation, electronics, catalysis, biomedicine and energy storage [331].

Structures containing iron oxides with a spinel structure, magnetite and maghemite, are widely used in various applications with a predominance of NPs intended for biomedicine [45,332–337] (Table 11). It is possible to propose the partial oxidation of magnetite NPs to maghemite in the vast majority of cases (except those with inert atmospheric preservation), but this effect can be distinguished only by the use of some additional instruments, including Mössbauer spectroscopy [6], zero-field and field cooling measurements to reveal a Verwey transition and high-resolution transmission electron microscopy [295] to show the crystal structure, while more widely used X-ray powder diffraction cannot resolve magnetite and maghemite [38]. The most frequently used synthetic routes used to obtain  $\gamma\text{-Fe}_2\text{O}_3\text{-Fe}_3\text{O}_4$  NPs are by thermal decomposition [337–340] and chemical precipitation [332,336,341,342].

The iron oxide layer on metal surfaces is also a possible structure containing co-existing magnetite and maghemite phases. Such structures can originate from both natural [343,344] and synthetic [345–349] routes, generally implying chemical or electrochemical oxidation in a liquid medium. Overall, such compositions play an important role in iron corrosion studies [344,346,347,350,351]. Finally, compositions with magnetite and maghemite, presumably due to the presence of iron(II) and iron(III) cations, are actively used for the remediation of waste [348,352–357].

**Table 7.** Data on the structures containing iron oxide atomic clusters and an amorphous iron oxide phase.

| Composition                                                            | Main Mechanisms of Iron Oxide Formation                                                                                                                                                                                                                                                                                            | Declared Applications                                                                                                                                          | Phase Verification Techniques                                                                                                                                           | Refs.         |
|------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|
| Iron oxide atomic clusters                                             | Combustion synthesis from $\text{Fe}(\text{CO})_5$ mixed with hydrogen and oxygen, high irradiance laser ionization from pressed $\text{Fe}_2\text{O}_3$ and $\text{Fe}_3\text{O}_4$ tablets, biomineralization inside the ferritin shell, reaction of laser ablated iron foil with 5% $\text{O}_2$ seeded in a helium carrier gas | Catalysis, biomedicine, electronics, sensors, prediction of the magnetic properties of $\text{FeO}_x$ NPs, natural iron storage process studies, photovoltaics | MBMS <sup>1</sup> , PMS <sup>2</sup> , RMDS <sup>3</sup> , TEM, LI-TOFMS <sup>4</sup> , density-functional theory calculations, European Synchrotron Radiation Facility | [271–277,281] |
| Surface iron oxide layer on metal                                      | Multicycling of an iron foil electrode between the switching potentials, formation of iron oxide species after reaction with $\text{Cr}(\text{VI})$ and $\text{Cu}(\text{II})$                                                                                                                                                     | Chemical water treatment, production of molecular hydrogen, removal of contaminants                                                                            | RMDS, XRD, XPS, FTIR, EDS                                                                                                                                               | [278,279,282] |
| Amorphous ferric oxides                                                | Adding $\text{Fe}(\text{II})$ or $\text{Fe}(\text{III})$ to seawater                                                                                                                                                                                                                                                               | Bioavailable iron studies                                                                                                                                      | XAS, XRD                                                                                                                                                                | [266]         |
|                                                                        | Addition of $\text{Fe}(\text{III})$ to synthetic buffered solution or soluble microbial systems                                                                                                                                                                                                                                    | Chemical water treatment                                                                                                                                       | UV–Vis                                                                                                                                                                  | [267]         |
| Amorphous $\text{Fe}_2\text{O}_3$ in a silica matrix                   | Impregnation of mesoporous silica with ferric nitrate and calcination                                                                                                                                                                                                                                                              | Antibiotic adsorption                                                                                                                                          | TEM, XRD, FTIR, UV–Vis                                                                                                                                                  | [268]         |
| Poorly crystalline iron oxides                                         | Iron oxide biomineralization by iron-reducing bacteria                                                                                                                                                                                                                                                                             | Geochemistry                                                                                                                                                   | ICP-MS                                                                                                                                                                  | [270]         |
| Amorphous iron oxide nanostructures                                    | Photothermal reaction inside a droplet of iron(III) acetylacetonate solution                                                                                                                                                                                                                                                       | Electronics, sensors                                                                                                                                           | TEM, SAED, EDS, RS                                                                                                                                                      | [269]         |
| Two-dimensional iron oxide on Au(111)                                  | Evaporating iron atoms, annealing and cooling down to 300 K in $\text{O}_2$                                                                                                                                                                                                                                                        | Catalysis                                                                                                                                                      | STM, density-functional theory calculations                                                                                                                             | [142]         |
| Iron oxide layer on zerovalent iron NPs                                | Zerovalent iron corrosion in an electrolyte solution                                                                                                                                                                                                                                                                               | Treatment of contaminated aquifers                                                                                                                             | UV–Vis, XAS                                                                                                                                                             | [283]         |
| Ferric oxide NPs                                                       | Protein-promoted conversion of $\text{Fe}(\text{II})$ into insoluble ferric iron oxides                                                                                                                                                                                                                                            | Mitochondrial iron mishandling studies                                                                                                                         | UV–Vis                                                                                                                                                                  | [284]         |
| Ultra-thin iron oxide nanowhiskers                                     | Iron oleate complex followed by selective decomposition at 150 °C                                                                                                                                                                                                                                                                  | Biomedicine                                                                                                                                                    | TG-DSC, TEM, SAED, RS, XPS, FTIR                                                                                                                                        | [285]         |
| High valent iron oxo complexes                                         | Fluorine-substituted Fe–tetra-amidomacrocyclic ligand oxidation                                                                                                                                                                                                                                                                    | Photocatalysis                                                                                                                                                 | UV–Vis, EPR, high-resolution mass spectrometry                                                                                                                          | [286]         |
| $\text{FeO}(\text{111})$ -like film on $\text{Fe}(\text{110})$ surface | Initial oxidation of $\text{Fe}(\text{110})$ in oxygen via Frank–Van der Merwe mechanism                                                                                                                                                                                                                                           | Catalysis, pigments, electronics                                                                                                                               | XPS, XAS, STM, AES, LEED <sup>5</sup> , STS <sup>6</sup>                                                                                                                | [280]         |
| Colloidal $\text{Fe}-\text{Fe}_x\text{O}_y$ composite NPs              | Oxidation of metal NPs via a nanoscale Kirkendall process                                                                                                                                                                                                                                                                          | Clean fuels, catalysis, electrochemical energy                                                                                                                 | TEM, SAXS, WAXS, RMDS                                                                                                                                                   | [287]         |
| Biogenic microtubular iron oxides                                      | Biotic formation of organic sheaths and subsequent abiotic deposition of Fe                                                                                                                                                                                                                                                        | Catalysis, pigments                                                                                                                                            | EDS, RS, TEM, XRD, STEM                                                                                                                                                 | [288]         |
| Iron oxide model thin-film electrodes                                  | Thermal oxidation of pure metal iron substrates at $300 \pm 5$ °C in air                                                                                                                                                                                                                                                           | Lithium-ion batteries                                                                                                                                          | RS, XPS, SIMS <sup>7</sup>                                                                                                                                              | [289]         |
| Iron(III) oxide/hydroxide nanonetworks                                 | Synthesis of iron(III) oxide/hydroxide xerogels from a hydrated ferric nitrate                                                                                                                                                                                                                                                     | Electronics, catalysis, sensors                                                                                                                                | XPS, FTIR, XRD, TEM                                                                                                                                                     | [290]         |
| $\text{Fe}^0$ -iron oxide core-shell NPs                               | Precipitation from ferrous sulfate with leaf extracts                                                                                                                                                                                                                                                                              | Removal of nitrate in aqueous solution                                                                                                                         | EDS, XRD, FTIR                                                                                                                                                          | [291]         |
| Soil samples with amorphous iron oxides                                | Abiotic mineralization in soil pore structures                                                                                                                                                                                                                                                                                     | Soil weathering studies                                                                                                                                        | XRD, ICP-AES                                                                                                                                                            | [292]         |
| Reticular pipeline cracks filled with iron oxide                       | Decarburization and diffusive oxidation of steel matrix                                                                                                                                                                                                                                                                            | Corrosion resistance studies                                                                                                                                   | EDS                                                                                                                                                                     | [293]         |

<sup>1</sup> Molecular beam mass spectrometry. <sup>2</sup> Particle mass spectrometry. <sup>3</sup> Reactive molecular dynamics simulations. <sup>4</sup> Laser ionization orthogonal time-of-flight mass spectrometry. <sup>5</sup> Low-energy electron diffraction. <sup>6</sup> Scanning tunneling spectroscopy. <sup>7</sup> Secondary ion mass spectrometry.

**Table 8.** Data on the structures containing co-existing  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> phases.

| Composition                                               | Main Mechanisms of Iron Oxide Formation                                        | Declared Applications                   | Phase Verification Techniques | Refs. |
|-----------------------------------------------------------|--------------------------------------------------------------------------------|-----------------------------------------|-------------------------------|-------|
| Saprolitic soil samples                                   | Aerobic weathering of Fe-bearing minerals                                      | Pedogenic process studies               | XRF, UV-Vis, XRD              | [298] |
| Loess and paleosol samples with iron oxides               | Aerobic weathering of Fe-bearing silicate minerals                             |                                         | XRD, UV-Vis                   | [297] |
| Oxidized iron items                                       | Soil iron corrosion limited by the diffusion of dissolved oxygen               | Heritage science                        | EDS, XRD, RS                  | [295] |
| Surface iron oxide layer on metal                         | Anodic film formation on steel immersed in sour acid media                     | Corrosion resistance studies            | XRD, EDS                      | [302] |
| Graphene-iron oxide nanotube composite                    | An adept template-free hydrothermal route from ferrous sulfate                 | Removal of the toxic heavy metal Cr(VI) | EDS, XRD, FTIR, UV-Vis, TEM   | [299] |
| Polyacrylonitrile/iron oxide composite                    | Hydrothermal method for in situ growth of iron oxide; iron alkoxide hydrolysis | Removal of Congo red dye from water     | FTIR, XRD, EDS, ICP-AES       | [300] |
| Carbon/Fe <sub>x</sub> O <sub>y</sub> magnetic composites | Mechanical mixing and thermal treatment under N <sub>2</sub> atmosphere        | Wastewater treatment                    | XRD, TG-DSC, EDS, FTIR        | [301] |
| Isoelement synthetic heterostructures                     | Hydrothermal method combined with controlled partial annealing process         | Visible-light photocatalysis            | XRD, TEM, XPS, UV-Vis, EPR    | [296] |

**Table 9.** Data on the structures containing co-existing  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub> phases.

| Composition                               | Main Mechanisms of Iron Oxide Formation                                                                                                                                                                                                   | Declared Applications                                                                                                               | Phase Verification Techniques                     | Refs.     |
|-------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------|-----------|
| Inclusions of iron oxides in ore samples  | Precipitation during protracted hydrothermal fluid/rock interaction, biological oxidation of Fe(II) by photoautotrophs, microbial sedimentary ferric iron flux, infiltration by hypogene and supergene fluids during or after deformation | Banded iron formation studies, geochemistry, late Archean and early Paleoproterozoic studies, iron oxide copper gold system studies | ICP-MS, EDS, XRF, SAED, TEM, XRD, ICP-AES, TG-DSC | [303–311] |
| Surface iron oxide layer on metal         | Tribo-oxidation wear of the cast iron disc                                                                                                                                                                                                | Brake system wear studies                                                                                                           | EDS, XRD, TEM, SAED                               | [315]     |
| Iron oxide NPs                            | Anodization of Fe sheet in ethylene glycol electrolyte and calcination                                                                                                                                                                    | Biomedicine, catalysis, photovoltaics, electronics                                                                                  | XRD, EDS, XPS, RS, FTIR                           | [312]     |
| Iron oxide inclusions in concrete samples | Corrosion of a steel-reinforcing bar in air-entrained concrete with chlorides                                                                                                                                                             | Corrosion resistance studies                                                                                                        | EDS                                               | [318]     |



Table 9. Cont.

| Composition                                     | Main Mechanisms of Iron Oxide Formation                                      | Declared Applications                            | Phase Verification Techniques           | Refs. |
|-------------------------------------------------|------------------------------------------------------------------------------|--------------------------------------------------|-----------------------------------------|-------|
| Iron oxide nanosheets and nanowires             | Thermal oxidation of iron foils in the presence of water vapor               | Cr(VI) removal                                   | XRD, TEM, RS, UV-Vis                    | [319] |
| Iron oxide hollow spheres                       | Microwave-hydrothermal ionic liquid method, calcination and autocatalysis    | Photocatalysis                                   | XRD, TEM, UV-Vis                        | [314] |
| Inclusions of iron oxides in mineralized rocks  | Abiotic formation of a mineral deposit                                       | Geochemistry                                     | XRF                                     | [320] |
| Theoretically calculated iron oxide phases      | Radiation-chemical oxidation of Fe depending on pH and oxygen content        | Precambrian studies                              | Kinetics of iron oxidation calculations | [321] |
| Iron oxide NPs supported on biogenic silica     | Iron oxide NP impregnation under hydrothermal conditions and calcination     | Rhodamine B photocatalytic degradation           | EDS, XRD, UV-Vis, TEM                   | [316] |
| Sediment samples with inclusions of iron oxides | Mineralization by variable diagenetic processes                              | Rock magnetism studies                           | XRD, EDS                                | [322] |
| Iron oxide nanorods                             | Sols of ferric hydroxide radiolysis in water under gamma irradiation         | Electronics, biomedicine                         | XRD, TEM                                | [314] |
| Spinel-bearing peridotite                       | Oxidation of ferrous iron in olivine and pyroxene into ferric iron           | Serpentinization studies                         | FTIR, EDS                               | [323] |
| Iron oxide inclusions in kaolin clay samples    | Abiotic chemical precipitation                                               | Clay chemistry and morphology studies            | ICP-AES, XRD, XRF, TG-DSC               | [324] |
| Precipitates containing iron oxide inclusions   | Biomining by photosynthetic Fe(II)-oxidizing bacteria                        | Banded iron formation studies                    | XRD, EDS                                | [325] |
| Iron-mineralized biofilms                       | Dissolution and re-precipitation of iron oxide minerals                      | Bioremediation of iron ore mines                 | –                                       | [326] |
| Iron oxide nanotubes                            | Template-based electrodeposition and calcination under oxidizing atmospheres | Biomedicine, electronics, gas sensors, catalysis | TEM, XRD, SAED                          | [317] |
| Iron oxide powder                               | Hydrothermal process with a use of pyrite cinder lixivium                    | Pyrite cinder reutilization                      | FTIR, XRD, TEM, SAED                    | [327] |
| Growth model for submarine deposits             | Transformation of primary (hydr)oxides via reduction by organic matter       | Banded iron formation studies                    | –                                       | [328] |

Table 10. Data on the structures containing co-existing FeO and Fe<sub>3</sub>O<sub>4</sub> phases.

| Composition                                                           | Main Mechanisms of Iron Oxide Formation                                                                                                                             | Declared Applications                                                          | Phase Verification Techniques         | Refs. |
|-----------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------|---------------------------------------|-------|
| Fe-rich carbonates with inclusions of iron oxides                     | Laser heating of natural goethite in a diamond anvil cell in CO <sub>2</sub>                                                                                        | Earth's mantle studies                                                         | XRD, XAS, TEM, EELS, HAADF-STEM, SAED | [329] |
| Samples with partially reduced FeO and Fe <sub>3</sub> O <sub>4</sub> | Porous iron growth from wüstite in CO/CO <sub>2</sub> and H <sub>2</sub> /H <sub>2</sub> O systems                                                                  | Porous iron growth mechanism studies                                           | –                                     | [330] |
| Fe/oxide core-shell NPs                                               | Formation of Fe <sub>3</sub> O <sub>4</sub> during the oxidation of Fe NPs; high-temperature reduction of Fe <sub>3</sub> O <sub>4</sub> to FeO by an electron-beam | Environmental remediation, electronics, catalysis, biomedicine, energy storage | TEM, SAED, EELS, HAADF-STEM, EDS      | [331] |



**Table 11.** Data on the structures containing co-existing  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub> phases.

| Composition                                          | Main Mechanisms of Iron Oxide Formation                                                                                                                                                                                                                           | Declared Applications                                                                                                               | Phase Verification Techniques                                                                         | Refs.                        |
|------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------|------------------------------|
| Iron oxide NPs                                       | Thermal decomposition of iron oleate, continuous flow synthesis, co-precipitation of Fe <sup>3+</sup> /Fe <sup>2+</sup> ions, aerosol spray pyrolysis with the use of ferric nitrate and ferric chloride, precipitation from iron salts with natural leaf extract | Biomedicine, soil remediation, metal removal, wastewater treatment, electronics, catalysis, energy storage, groundwater remediation | TEM, XRD, FTIR, SAED, TG-DSC, UV-Vis, SAXS, neutron diffraction, EDS, MSP, EELS, EPR, ICP-MS, XAS, RS | [45,332–340,352–355,358–362] |
| Surface iron oxide layer on metal                    | Oxidation of a pure iron surface in oxygen, electrochemical reduction of lepidocrocite and ferrihydrite, in situ formation on an iron surface depending on the applied potential                                                                                  | Iron oxidation studies, atmospheric steel corrosion studies, groundwater remediation, corrosion protection studies                  | XPS, XRD, XAS, RS, AES, ellipsometry                                                                  | [345–349]                    |
| Oxidation layer on archaeological steel              | Combined iron oxidation/iron(III) oxyhydroxide reduction without O <sub>2</sub>                                                                                                                                                                                   | Corrosion studies on ancient metallic objects                                                                                       | EDS, RS                                                                                               | [343]                        |
| Iron oxide-TiO <sub>2</sub> nanorod heterostructures | Precipitation by injection of Fe(CO) <sub>5</sub> into stirred TiO <sub>2</sub> containing mixture                                                                                                                                                                | Optoelectronics, biomedicine, catalysis                                                                                             | XRD, XAS, ICP-AES, TEM, UV-Vis                                                                        | [363]                        |
| Iron oxide in nanoscrolls and nanoribbons            | Precipitation from ferric and ferrous chloride with ammonia solution                                                                                                                                                                                              | Lithium-ion storage, photocatalysis, biosensors                                                                                     | TEM, FTIR                                                                                             | [364]                        |
| Iron oxide hollow core/Shell NPs                     | Solvothermal synthesis from FeCl <sub>3</sub> and urea in ethylene glycol and calcination                                                                                                                                                                         | Biomedicine                                                                                                                         | XRD, TEM, TG-DSC, UV-Vis                                                                              | [365]                        |
| Thin-film nanocomposite membrane with iron oxide     | In situ synthesis from aqueous solutions containing ferric chloride                                                                                                                                                                                               | Biofouling protection                                                                                                               | EDS, TEM, XPS, UV-Vis, XRD, TG-DSC                                                                    | [366]                        |
| Magnetoferritin iron oxide NPs                       | Controlled mineralization from recombinant human H-chain ferritin                                                                                                                                                                                                 | Biomedicine                                                                                                                         | TEM                                                                                                   | [367]                        |
| Iron oxide-based hollow magnetic nanoparticles       | Synthesis from iron pentacarbonyl in 1-octadecene and oleylamine                                                                                                                                                                                                  | Exchange bias studies                                                                                                               | XRD, TEM, FTIR, MSP, F-AAS                                                                            | [368]                        |
| Albumin protein-based magnetic NPs                   | Co-precipitation of FeCl <sub>2</sub> and FeCl <sub>3</sub> by ammonia in the presence of protein                                                                                                                                                                 | Biomedicine                                                                                                                         | TEM, TG-DSC                                                                                           | [369]                        |

Table 11. Cont.

| Composition                                         | Main Mechanisms of Iron Oxide Formation                                                                | Declared Applications                            | Phase Verification Techniques   | Refs. |
|-----------------------------------------------------|--------------------------------------------------------------------------------------------------------|--------------------------------------------------|---------------------------------|-------|
| Composite of organic matrix and iron oxide NPs      | Thermal decomposition of iron(III) oleate complex                                                      | Biomedicine                                      | TEM                             | [370] |
| Iron oxide powder                                   | Photochemical oxidation of siderite (FeCO <sub>3</sub> ) by ultraviolet radiation                      | Banded iron formation studies                    | XRD                             | [371] |
| Interfacial iron oxide layer on iron artifacts      | Iron corrosion in an anoxic environment after a pH increase at the interface                           | Anoxic corrosion of archaeological steel studies | HAADF-STEM, RS, EDS, SAED, SIMS | [344] |
| Iron oxide hydroxyapatite core/shell nanocomposites | Precipitation from ferric and ferrous chloride with ammonia under N <sub>2</sub>                       | Biomedicine                                      | TEM, FTIR, XRD, AAS, EDS        | [372] |
| Chitosan-based beads with iron oxide NPs            | Co-precipitation from ferric and ferrous chloride with NaOH solution                                   | Remediation of water sources                     | XRD, FTIR, TG-DSC, EDS          | [356] |
| Silica–iron oxide nanocomposite                     | Co-precipitation from ferric and ferrous chloride with ammonia solution                                | Toxic species removal                            | XRD, TEM, FTIR, UV-Vis, SAED    | [357] |
| Vertical tube-shaped iron-oxide accumulations       | Deep water corrosion of carbon steel                                                                   | Marine corrosion studies                         | EDS                             | [351] |
| Hydrogels with embedded iron oxide NPs              | In situ mineralization of iron ions in a hydrogel matrix                                               | Dye removal                                      | XRD, FTIR, TG-DSC, TEM          | [373] |
| Corroded reinforced concrete                        | Iron corrosion in a laboratory corrosion chamber                                                       | Steel rebar corrosion studies                    | XRD, EDS                        | [350] |
| Porous hollow iron oxide NPs on carbon nanotubes    | Etching of Fe-Fe <sub>x</sub> O <sub>y</sub> intermediate with nitric acid aqueous solution and drying | Biomedicine, catalysis, separation               | TEM, XRD                        | [374] |
| Iron oxide embedding of bacterial cells             | Biomining by thermophilic iron-reducing bacteria                                                       | Biogenic iron mineral formation studies          | XRD                             | [375] |
| Activated carbon aerogel with iron oxide inclusions | Hydrothermal synthesis from ferrous sulfate with ammonia                                               | Catalytic oxidation of pesticides                | XRD, FTIR, XPS, TEM             | [376] |
| Polyglycerol-grafted iron oxide NPs                 | Thermal decomposition of iron(III) acetylacetonate in triethylene glycol                               | Biomedicine                                      | TEM, TG-DSC, FTIR, ICP-AES      | [377] |

### 3.4. The Structures Containing Three or More Co-Existing Iron Oxide Phases

The co-existence of three or more iron oxide compounds in a single heterogeneous composition makes it useless to try to precisely distinguish every standalone phase. Thus, studies devoted to such a case are considered in this section and listed in Table 12. Iron oxide NPs and the surface oxide layer on metal surfaces are the largest groups of papers on the structures containing mixed iron oxide phases. Synthesis techniques for obtaining mixed iron oxide NPs include physical ones (flame synthesis [378], thermal oxidation [379] and laser ignited combustion [376]) and chemical ones (thermal decomposition [82,380] and precipitation [341], including “green” process [381]). The main application of such NPs is in biomedicine [82,378–380].

**Table 12.** Data on the structures containing mixed iron oxide crystal phases [42,79,380–407].

| Composition                                                                                                                              | Main Mechanisms of Iron Oxide Formation                                                                                                                                                                                            | Declared Applications                                                                       | Phase Verification Techniques                             | Refs.        |
|------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------|-----------------------------------------------------------|--------------|
| Iron oxide NPs                                                                                                                           | Thermal decomposition of an iron oleate in the presence of oleic acid, flame synthesis from $\text{Fe}(\text{CO})_5$ , precipitation from ferric chloride in a natural leaf extract, thermal oxidation of polycrystalline Fe foils | Biomedicine, ferrofluids, electronics, immobilization of Cd in soils, catalysis, metal fuel | PMS, TEM, EDS, TG-DSC, SAED, XRD, XPS, FTIR, UV-Vis, EELS | [79,380–384] |
| Surface iron oxide layer on metal                                                                                                        | Carbon steel corrosion at room temperature, iron carboxylate transformation to iron oxides, electrochemical anodization of metal in simulated acid rain solution                                                                   | Corrosion resistance studies, railway industry, fireside corrosion studies                  | EDS, XRD, XRF, RS, XPS, FTIR, TG-DSC                      | [385–389]    |
| Powder containing iron oxide microparticles                                                                                              | Carbothermal reduction of red mud by heating in a microwave furnace                                                                                                                                                                | Alumina production by-product recycling                                                     | XRD, EDS, XRF, TG-DSC                                     | [400]        |
| Iron oxide microfibers arranged in a complex hierarchical structure                                                                      | Thermal decomposition of $\text{Fe}(\text{CO})_5$ and silicone oil and microwave vaporization                                                                                                                                      | Environmental safety, biomedicine, sensors                                                  | EDS, XRD                                                  | [396]        |
| Fe-based nanocomposite catalysts containing agglomerates of the two types                                                                | Melting of iron, aluminum and copper salts and reduction                                                                                                                                                                           | Low-temperature catalytic oxidation of CO                                                   | XRD, TEM, XAS, EDS                                        | [393]        |
| Iron oxide powders containing hematite, magnetite and maghemite                                                                          | Chemical precipitation from ferric nitrate and ferrous sulfate and heating                                                                                                                                                         | Inositol phosphate selective retention in soil                                              | XRD                                                       | [399]        |
| Silica–iron oxide nanocomposite with hematite, magnetite and wüstite                                                                     | Silica promotion upon the reduction of amorphous iron oxide in hydrogen                                                                                                                                                            | Catalysis                                                                                   | XRD, TEM, EELS, STEM, SAED                                | [394]        |
| Iron(II) and (III) oxides inclusions in char composites                                                                                  | Microwave pyrolysis of Moso bamboo samples with ferric chloride                                                                                                                                                                    | Syngas production                                                                           | XRD                                                       | [401]        |
| Ultra-thin magnetic iron oxide films containing $\text{Fe}_3\text{O}_4$ , $\gamma\text{-Fe}_2\text{O}_3$ and FeO                         | Thermally induced phase transformation of ultra-thin iron oxide films                                                                                                                                                              | All oxide heterostructures                                                                  | XRD, XPS                                                  | [402]        |
| Surface iron(II) and (III) oxide layer on iron granules                                                                                  | Atomization of the molten semi-steel with a rotary cup atomizer                                                                                                                                                                    | Iron powder production                                                                      | TG-DSC, XRD                                               | [403]        |
| Iron oxide ( $\text{Fe}_3\text{O}_4$ , $\gamma\text{-Fe}_2\text{O}_3$ and $\alpha\text{-Fe}_2\text{O}_3$ ) inclusions in fly ash samples | Coal combustion and flue gas cooling at various temperatures                                                                                                                                                                       | Selenium adsorption by iron minerals                                                        | XRF, MSP, XPS                                             | [404]        |
| High-pressure metastable phases $m\text{FeO}-n\text{Fe}_2\text{O}_3$                                                                     | Formation of complex iron oxide crystals under high-pressure conditions                                                                                                                                                            | Earth and planetary deep interior studies                                                   | –                                                         | [24]         |
| Iron(III) oxide submicron inclusions in a biofilm on a basalt surface                                                                    | Microbial direct or non-direct biomineralization                                                                                                                                                                                   | Biovermiculation studies                                                                    | EDS                                                       | [388]        |
| Inclusions of poorly crystalline iron(III) oxides in ore samples                                                                         | Microbial biomineralization                                                                                                                                                                                                        | Mine remediation, waste stabilization                                                       | SIMS                                                      | [390]        |
| Iron oxide ( $\text{Fe}_3\text{O}_4$ , $\gamma\text{-Fe}_2\text{O}_3$ and $\alpha\text{-Fe}_2\text{O}_3$ )/iron composite                | Reactive spark plasma sintering of mechanically activated Fe powders                                                                                                                                                               | Magnetic material development                                                               | XRD, TG-DSC                                               | [405]        |

Table 12. Cont.

| Composition                                                                                                                                                   | Main Mechanisms of Iron Oxide Formation                                  | Declared Applications                              | Phase Verification Techniques | Refs. |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------|----------------------------------------------------|-------------------------------|-------|
| Core–shell Fe@Fe <sub>2</sub> O <sub>3</sub> nanowires containing Fe(II) and Fe (III) oxides                                                                  | Ferric chloride reduction with sodium borohydride and surface oxidation  | Cr(VI) removal                                     | XRD, UV–Vis, TEM, XPS         | [406] |
| Iron oxide submicron particles accumulated in the cytoplasm of cells                                                                                          | Intracellular or extracellular microbial biomineralization               | Hydrothermal vent field studies                    | XRD, TEM, SAED, EDS           | [389] |
| Deposit samples with inclusions of iron(II) and (III) oxides                                                                                                  | Anoxygenic photosynthesis by a photoferrotrophic bacterium               | Banded iron formation studies                      | –                             | [391] |
| Bovine serum albumin–iron oxide suspensions                                                                                                                   | Precipitation from ferric nitrate with NaOH in N <sub>2</sub> atmosphere | Boreal forest studies                              | FTIR                          | [398] |
| Iron oxide (Fe <sub>3</sub> O <sub>4</sub> , $\gamma$ -Fe <sub>2</sub> O <sub>3</sub> and $\alpha$ -Fe <sub>2</sub> O <sub>3</sub> ) magnetic short nanotubes | Anion-assisted hydrothermal route by using phosphate and sulfate ions    | Biomedicine, ferrofluids, electronics, spintronics | TEM, SAED, XRD                | [407] |
| Fe-biochar composites containing iron(II) and (III) oxides                                                                                                    | Pyrolysis of ferric chloride in a biochar matrix at various temperatures | Arsenic removal, environmental remediation         | XRD, XPS, RS, FTIR            | [397] |
| Fe <sub>x</sub> O <sub>y</sub> @C spheres embedded with highly dispersed iron oxide NPs                                                                       | One-pot hydrothermal cohydrolysis-carbonization using iron               | Catalysis                                          | TEM, EDS, XAS, XRD, MSP       | [395] |
| Powder with inclusions of submicron iron oxide particles                                                                                                      | Reduction of solid ferric hydroxide by iron-reducing bacteria            | Microbial iron reduction studies                   | EDS, RS                       | [392] |

An iron oxide layer on the metal surface is the second commonly considered structure, which plays an important role in corrosion studies [383–385] and can be formed both naturally (e.g., iron carboxylate transformation in a leaf contamination on rails [386] or fireside corrosion of steel in the furnace walls in boilers [384]) and synthetically (e.g., in situ oxidation of the surface of a steel sample in a controlled atmosphere [387] or electrochemical anodization of metal in a simulated acid rain solution [385]).

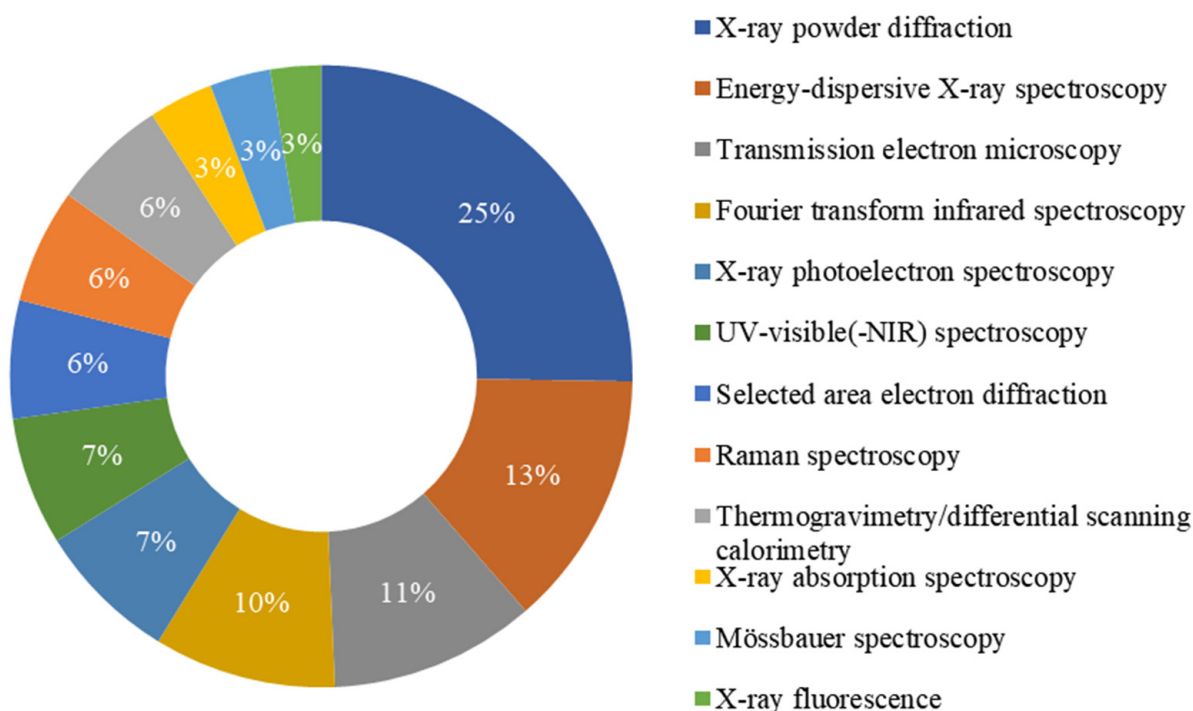
Compositions with mixed iron oxides can originate via a biogenic route, including microbial direct or non-direct biomineralization [388–390], anoxygenic photosynthesis by a photoferrotrophic bacterium [391] and the reduction of solid ferric hydroxide by iron-reducing bacteria [392]. Biogenic iron oxide structures can be used in microbial iron reduction studies [392], banded iron formations [391] or hydrothermal vent field studies [389] and waste remediation [390].

Similarly to the above mentioned iron oxide structures, various compositions containing mixed iron oxide phases can be applied for catalytical purposes. These compositions include iron oxide NPs [379], Fe-based nanocomposites [393], silica–iron oxide nanocomposites [394] and Fe<sub>x</sub>O<sub>y</sub>@C spheres [395] and can be obtained with the use of chemical [394,395] or physical [379,393] synthesis.

Finally, in some cases, such structures are used for various environmental tasks, including environmental safety [396], environmental remediation [397], boreal forest studies [398], inositol phosphate selective retention in soil [399], biovermiculation studies [388] and the Earth and planetary deep interior studies [24].

### 3.5. The Main Characterization Techniques Used to Verify Phase Composition

From analyzing Tables 2–12, the most frequently used characterization techniques can be revealed. These techniques include X-ray powder diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS), transmission electron microscopy (TEM), Fourier-transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), UV-visible(-NIR) spectroscopy (UV–Vis), selected area electron diffraction (SAED), Raman spectroscopy (RS), thermogravimetry/differential scanning calorimetry (TG-DSC), X-ray absorption spectroscopy (XAS), Mössbauer spectroscopy (MSP) and X-ray fluorescence (XRF). The diagram showing the partial distribution between these techniques is presented in Figure 5.



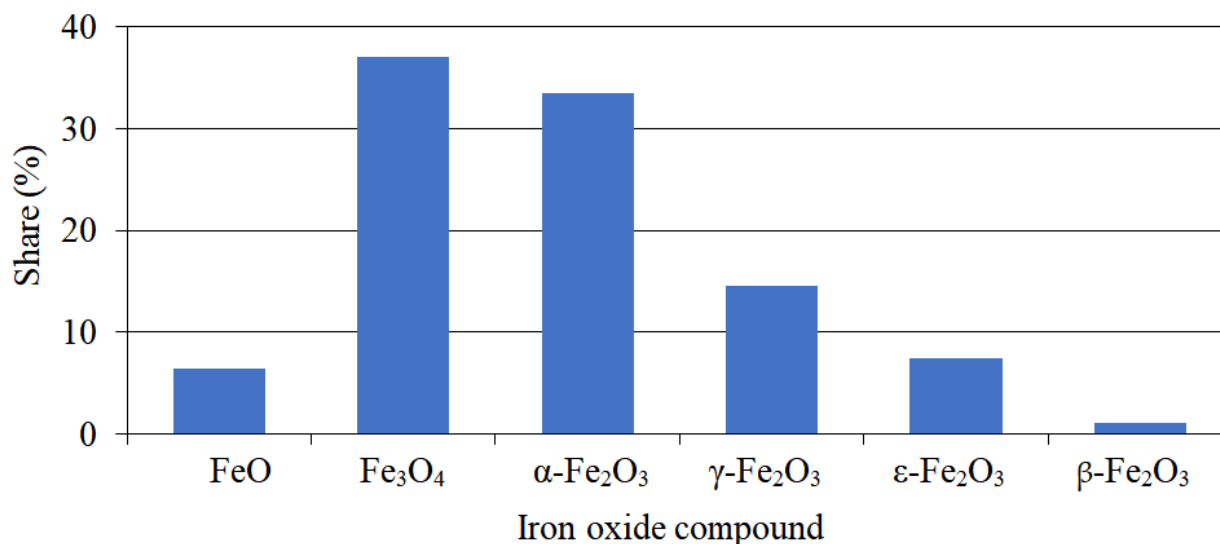
**Figure 5.** Main characterization techniques used to verify phase composition.

In some cases, while the crystal structure of the co-existing iron oxide compounds differs significantly (e.g., for hematite and magnetite), the most common and available techniques are suitable for phase composition verification, including XRD and XRF. For instance, XRF and XRD techniques were used to determine the chemical and crystalline phase composition, accordingly, of clay samples containing iron oxides [324]. Another possible example is  $\epsilon$ - $\text{Fe}_2\text{O}_3$ , a crystal structure that is also quite distant from the other iron(III) oxide polymorphs and can be distinguished with the use of RS and XAS [258], or even with the use of a standalone XRD technique [37]. Contrary to the above, the simultaneous existence of magnetite and maghemite crystal phases cannot be adequately analyzed with XRD, SAED or some other techniques due to the similar crystal structure of these compounds and non-stoichiometry of synthetic [38] or natural [6,295] magnetite. In this case, the MSP technique can give additional information on the crystal structure, including magnetite to maghemite partial transition or the superparamagnetic state of NPs [38]. MSP can be applied to characterize samples composed of homogeneously sized iron oxide NPs above the blocking temperature in a superparamagnetic regime [368]. Mössbauer measurements were performed to investigate detailed iron mineralogy compositions in magnetic fractions of fly ashes [405]. Perecin et al. showed that although Mössbauer spectra with two sextets were expected for pure magnetite, an extra sextet suggested the maghemite phase's presence in the sample, in agreement with the FTIR results [89]. The high oxidation degree of magnetite can be confirmed by low isomer shift values [342]. MSP results can show a shift of the Morin transition in hematite upon increasing  $\text{Ru}^{3+}$ -to- $\text{Fe}^{3+}$  substitution, similar to the shift in the Morin transitions occurring in temperature-dependent magnetization measurements [224].

### 3.6. The Analysis of the Distribution of Iron Oxide Compounds by their Frequency of Mention

Based on the data provided in Tables 2–12 (in total, more than 300 research articles were analyzed), a histogram was built (Figure 6). Compositions containing magnetite are the most frequently considered in scientific articles. This can be explained by taking into account the highest saturation magnetization of this iron oxide compound among others and also because of the wide biomedical application of magnetite NPs. The second most commonly described compound is hematite, presumably due to its use in photocatalysis

and its abundance (the same for magnetite ores) in nature. The third important iron oxide compound is maghemite, since  $\gamma\text{-Fe}_2\text{O}_3$  NPs are often used instead of magnetite NPs due to the high oxidation instability of  $\text{Fe}_3\text{O}_4$  in air atmospheres and, therefore, difficulties in its preservation without an inert atmosphere or a protective shell. Other iron oxide compounds, including  $\text{FeO}$ ,  $\beta\text{-Fe}_2\text{O}_3$  and  $\epsilon\text{-Fe}_2\text{O}_3$ , are metastable and/or their synthesis procedure is too complex, therefore scientific studies on them are relatively rare.



**Figure 6.** Distribution of iron oxide compounds by their frequency of mention (based on the analyzed research articles mentioned in Tables 2–12).

### 3.7. The Main Mechanisms of Iron Oxide Formation

Using the information given in Tables 2–12, the generalized scheme illustrating some widely considered mechanisms of iron oxide formation was drawn (Figure 7). Terrigenous formation implies abiotic mineralization [100,102,206,219,292,324]. Extraterrestrial formation including samples from Mars [113] originated from precipitation from oxygenated iron-rich water [198] and abiotic formation in an aqueous environment of deposition [124].

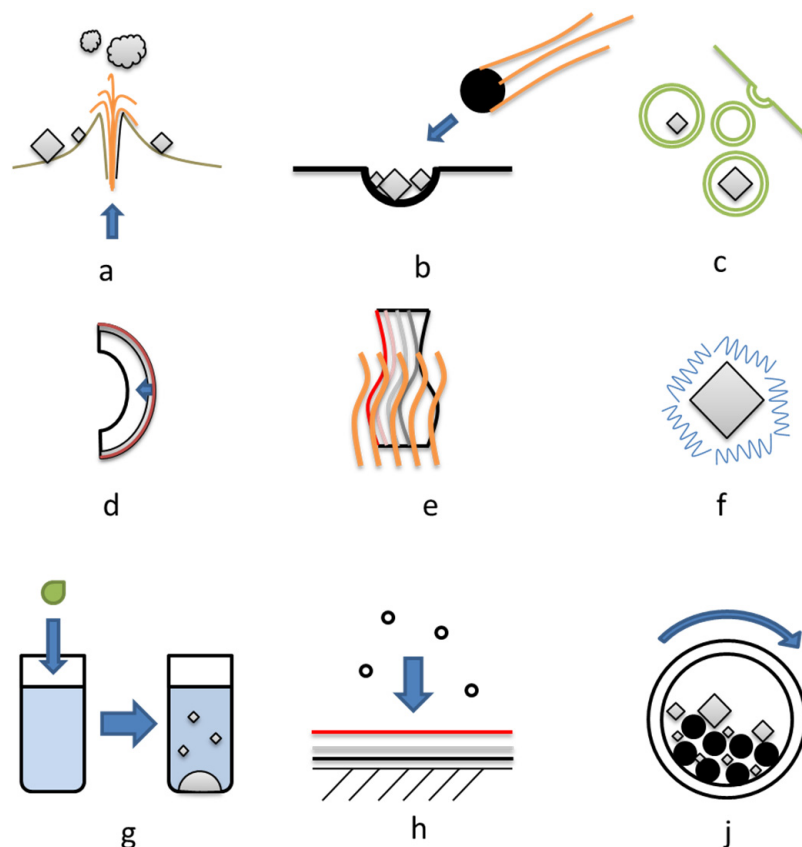
Biomineralization implies bacterial magnetosome formation [94,96–98], formation by dissimilatory iron-reducing bacteria [95], bacterial reduction of iron hydroxide [122,166,392], biomineralization by anoxygenic photoferrotrophy [210], biomineralization inside the ferritin shell [276,277], biogenesis of magnetite with a use of the amyloid peptide  $\text{A}\beta_{42}$  in the case of brain diseases [160], etc.

Iron and iron alloy corrosion include chemical [106,109,238,350] and electrochemical [86,349,385] processes, either natural or intended. Ceramics firing implies the calcination of a milled mix at a high temperature in an oxidizing atmosphere [229], high-temperature firing of local iron-rich area on a ceramic glaze [140] and surface iron enrichment and firing of wares under reducing conditions [132]. Biomimetic synthesis refers to a process using natural plant extracts [91], iron oxide NP formation on biogenic silica [316], nucleation of  $\text{Fe}_3\text{O}_4$  NPs mediated by the iron-binding protein Mms6 [92] and protein-promoted conversion of  $\text{Fe(II)}$  into insoluble ferric iron oxides [284].

Chemical precipitation includes co-precipitation by sodium hydroxide from an iron chloride solution [82], precipitation from  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  ions by urea with chitosan [149], precipitation from iron(II) sulfate heptahydrate with  $\text{NaOH}$  [158] and precipitation from iron(III) ethoxide with ethanol in the surfactant solution [179]. Physical deposition implies electron-beam deposition [142], liquid-phase atomic layer deposition [232], pulsed laser deposition on the Ga-terminated surface of a  $\text{GaN}$  (0001) [260] and chemical vapor deposition from an Fe organic liquid source [258]. Finally, mechanochemical synthesis includes mechanical mixing and thermal treatment under a  $\text{N}_2$  atmosphere [301], reactive spark plasma sintering of mechanically activated Fe powders [405] and milled zerovalent



iron corrosion in anaerobic synthetic groundwater [348]. All the listed routes of iron oxide formation can be either intended (controlled) or natural (uncontrolled).



**Figure 7.** Mechanisms of iron oxide formation: (a)—Terrigenous formation; (b)—Extraterrestrial formation; (c)—Biomineralization; (d)—Iron and iron alloy corrosion; (e)—Ceramics firing; (f)—Biomimetic synthesis; (g)—Chemical precipitation; (h)—Physical deposition; (j)—Mechanochemical synthesis.

#### 4. The Main Applications of the Structures Containing Iron Oxides

The largest amount of scientific papers being analyzed describes various biomedical applications of iron oxides, mainly magnetite/maghemite NPs. Photocatalytic oxidation and other applications in catalysis are the second major practical use of iron oxides, with a predominance of compositions with hematite. The third important field of use is electronics, including spintronics, data storage development and optoelectronics, mainly for thin ferrimagnetic or antiferromagnetic iron oxide films. Corrosion science is a quite obvious, but still very important area of application, since iron oxide passivation can provide a better reliability of steel pipelines, safety of ancient artifacts for heritage science, etc. Waste remediation is generally based on the possibility of iron oxidation from iron(II) to iron(III) and to absorb or bind inorganic and organic pollutants. Finally, serious attempts in geosciences with the use of various iron oxide compositions are still being made, despite a long history of research. Studies of natural iron ores can give much information about Earth's evolution, including Precambrian research.

Biomedical applications of iron oxides include  $T_2$  magnetic resonance imaging (MRI). MRI contrast agents are based on superparamagnetic NPs; their nanocluster formation increases the magnetic signal and subsequently enhances imaging sensitivity or cell labeling efficiency [29]. Magnetic hyperthermia with alternating magnetic fields requires magnetic NPs having an effective heating rate to enable therapeutic applications [11]. There are diverse bioinspired approaches for the synthesis of magnetic nanochains with optimal properties for biomedical applications, including magnetically guided drug delivery [13].

The integration of magnetic NPs and organic dyes into single platforms demonstrated their use as bimodal imaging agents for both in vitro and in vivo imaging and in multifunctional platforms that perform several tasks in parallel (e.g., dual-mode imaging and photodynamic therapy or drug delivery) [14].

Iron oxide formation mechanisms are important for understanding formation processes of iron-relevant minerals in Precambrian banded iron formations, granular iron formations and associated iron-poor strata [4,33]. This understanding also includes the origin of probably the first protosensory system evolved on Earth, i.e., magnetotaxis, while the precise mechanism of biogenic magnetite mineralization on early Earth is still unknown [8]. Well-known processes of the biomineralization of iron can help in better understanding human body iron metabolism and in curing diseases linked with iron-damaged regulation [10]. Today, by far the single most important use (by volume) of iron oxides is as a source of Fe, which is subsequently processed to make steel [3]. Another important iron oxide industrial application is photoelectrochemical water splitting. It is a leading strategy for producing a promising renewable store of energy—hydrogen [12]. Iron oxides, including magnetite, maghemite and hematite, are widely used in heterogeneous catalysis processes and have been attractive alternatives for the remediation of polluted soil, groundwater and wastewater based on a heterogeneous Fenton reaction (a combination of a solid Fe-based catalyst and  $\text{H}_2\text{O}_2$ ) [25,26]. Partially oxidized zerovalent iron NPs with a core-shell structure can be used to remediate groundwater and wastewater contaminated by chlorinated organic compounds, heavy metals, dyes and phenols [27].

Rare iron(III) oxide polymorphs can also be practically used. Thus,  $\beta\text{-Fe}_2\text{O}_3$  has found a few applications in an electro-catalyst for the reduction of hydrogen peroxide, in optoelectronics and in red ferric pigments [19]. A very high room-temperature coercive field makes  $\epsilon\text{-Fe}_2\text{O}_3$  suitable for use in recording media; its magnetoelectric coupling and millimeter-wave ferromagnetic resonance are useful in electric/magnetic field tunable devices and for millimeter wave absorption on the walls of an interior room or on the body of a car, train or airplane [36]. In some cases, e.g., in catalysts, amorphous  $\text{Fe}_2\text{O}_3$  NPs can be more active than nanocrystalline polymorphs or particles of metallic iron of the same diameter [39]. The structures of iron oxides are common to many binary systems and complex solid solutions; therefore, a rich set of isostructural compounds and solid solutions with tunable properties may be synthesized [23].

Ceramics, including composites containing inclusions of amorphous iron oxides, are suitable for various industrial applications. The process of transforming iron oxides from a glass network into a crystal nucleus was studied for the novel field of glass ceramics based on waste glass [408]. Prim et al. showed that iron oxide from a metal sheet treatment process may be used as a ceramic pigment by encapsulation in a crystalline and amorphous silica matrix [409]. Intended for hazardous waste incineration, glass ceramics containing hematite exhibited a superior compressive strength, volume density and water absorption [410]. Alumina–zirconia–titania ceramic membranes coated with a nanosized hematite layer can be applied in a combined ozonation–membrane filtration process [411]. The formation of solid solutions between mullite and transition metal cations, including iron, affects the thermal expansion of mullite ceramics through the distortion of the Al–O octahedral [412].

Less than 20 a wt% addition of iron oxide significantly lowered the softening and melting temperatures of  $\text{CaO-Al}_2\text{O}_3\text{-MgO-SiO}_2$ -based glass ceramics [413]. The lower melting temperature leads to a significant decrease in the price of the vitrification procedure and to the suppression of heavy metal evaporation during glass melting [414]. Such a class of glass ceramics possessing excellent mechanical characteristics (bending strength of 120 MPa, hardness of 9 GPa and fracture toughness of  $1.6 \text{ MPa}\cdot\text{m}^{1/2}$ ) was discussed, together with the remarkable effect of their vitrification on heavy metal immobilization [415]. One of the most low-temperature techniques, the sol–gel method, which involves the hydrolysis of the precursors of constituent oxides followed by their gelation, has the potential to yield magnetic ceramics, including bioceramics, with a more flexible composition range, better homogeneity, better bioactivity and controllable porous structure [416]. Nanostructured



catalyst-modified composite cathodes can be obtained by infiltrating a metal ion solution into a ceramic scaffold, followed by heating at a high temperature. A 3D heterostructured electrode decorated by amorphous iron oxide that works stably at 650 °C with an oxygen reduction reactivity comparable to that of a Pt-decorated one was obtained [417].

A wide range of metals such as gold, silver, copper, zinc, iron, platinum and palladium are fabricated in the form of NPs using algae and cyanobacteria and can be applied for infection control, diagnosis, drug delivery, biosensing and bioremediation [418]. An approach for the synthesis of highly pure, crystalline and biocompatible hematite NPs through the sole use of *Psidium guajava* leaf extract was proposed. The antibacterial efficacy of the obtained hematite NPs against Gram-positive as well as Gram-negative bacteria was established [419]. Both plants and microbes offer various ways to synthesize magnetite and maghemite NPs for potential dye degradation from industrial effluents from a variety of routes due to their vast genetic diversity and presence of various enzymes, respectively [420]. Co-substituted magnetite NPs were produced during the enzymatic reduction of a synthetic co-ferrihydrite using *Geobacter sulfurreducens* as an analogue to bioreduction processes in the natural environment to understand the natural biogeochemical cycling of cobalt in Fe-rich environments undergoing microbially mediated redox transformations [421]. Natural biogenic iron oxide extracted from banded iron formations showed high removal potential with the maximum sorption efficiency of 88.65% at a 30 g/L adsorbent dose [422].

Amorphous iron oxides are the promising material for various energetic and catalytic applications, including biomedicine. Compared to well crystalline Fe<sub>2</sub>O<sub>3</sub>, amorphous Fe<sub>2</sub>O<sub>3</sub>/graphene composite nanosheets exhibited superior sodium storage properties such as high electrochemical activity, a high initial Coulombic efficiency of 81.2% and a good rate of performance for sodium-ion batteries [423]. Efficient water oxidation catalysts have nominally amorphous mixed-metal oxide phases on their surface which are responsible for catalytic activity [424]. Amorphous iron oxide-packaged oxaliplatin prodrugs can be effective for cancer treatment, since the Fe<sup>2+</sup>/Fe<sup>3+</sup> ions released by the amorphous iron oxide NPs produce a large amount of reactive oxygen species through Fenton's reaction [425]. Nanosized amorphous iron oxide showed higher catalytic activity with lower oxidant consumption in comparison to Fe<sub>3</sub>O<sub>4</sub>- and Fe<sub>2</sub>O<sub>3</sub>-based clay composites [426]. Amorphous Fe<sub>2</sub>O<sub>3</sub> nanoflakes were biosynthesized by a novel sol-gel method using *Aloe vera* leaf extract, and their catalytic effect on the thermal decomposition of ammonium perchlorate was investigated [427]. Amorphous Fe<sub>2</sub>O<sub>3</sub> NPs can act as efficient and robust photocatalysts for solar H<sub>2</sub> evolution without any cocatalysts [428]. Amorphous Fe<sub>2</sub>O<sub>3</sub>/reduced graphene oxide/carbon nanofiber films were tested as flexible and freestanding anodes for lithium-ion batteries [429].

Corrosion resistance studies involving iron oxides are also important. Thus, potassium and chlorine may interplay to accelerate the corrosion of Fe-rich oxide scales, and an understanding of this process may open up new ideas for ways to decrease corrosion in highly corrosive environments [430]. Wheat straw fiber-reinforced polyvinyl chloride composites pigmented with iron oxide pigment have better seawater corrosion resistance, including better fiber/matrix interfacial interaction, lower total discoloration and higher surface hydrophobicity, mechanical properties and thermal stability [431]. Hydroxyapatite-bioglass-Fe<sub>3</sub>O<sub>4</sub>-chitosan coatings showed an effective improvement of the surface properties, hemocompatibility and in vitro corrosion rate of a biodegradable magnesium alloy [432]. The corrosion resistance of the epoxy coating was experimentally improved using micaceous iron oxide and Al pigments [433]. In the case of stainless steel, iron oxide formation corresponds to a low pitting potential and corrosion resistance and leads to the degraded protective property of the oxide film [434].

Based on the data in Tables 2–12, a diagram illustrating the main declared applications of various compositions with iron oxides was built (Figure 8).

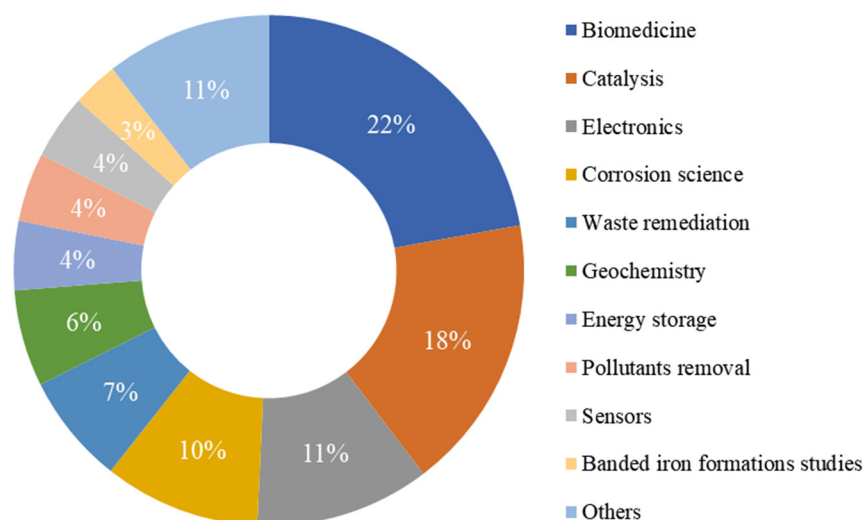


Figure 8. Main applications of iron oxides.

## 5. Summary and Perspectives

Iron oxide compounds are widely presented in various scientific and industrial areas due to their abundance on Earth. The possibility of changing the iron oxidation state between  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  lies in the basement of the biogenic iron cycle, which results in band iron formation accumulations and the deposition of fossilized magnetotactic bacteria, called magnetofossils [435]. The mass production of metallurgy and large iron deposits, e.g., the Kovdor deposit [6], provides a low cost of iron oxides compositions and their applicability for different technical purposes. High biocompatibility and modern synthesis techniques, including continuous-flow, biomimetic and biogenic processes, make it possible to translate academic research to clinical practice [435–437].

Integrating magnetic NPs with polymers allows for the fabrication of multifunctional systems for chemotherapy and magnetic hyperthermia therapy, which can also be simultaneously monitored by utilizing the magnetic resonance imaging capabilities of magnetic nanoparticle–polymer conjugates [14]. Such systems, e.g., iron-loaded crosslinked magnetic chitosan/graphene oxide, can also be widely applied for the practical environmental remediation of wastewater effluents containing organic pollutants [174]. The novel iron oxide-based materials can be used to improve solar fuel production [110]. The use of ferritin protein as a carrier of iron oxide NPs renders it more suitable for cancer diagnosis as an effective  $T_2$  contrast agent with an expected reduced toxicity due to the prevention of NP interaction with the environment [367]. Two-phase iron oxide NPs (e.g., magnetite/maghemite core-shell structures) are promising for applications implying an intrinsic exchange bias effect [368].

As paleoenvironmental proxies, the iron abundance, speciation and isotopic composition recorded for an Archean ocean analogue in the future can assist in understanding the iron biogeochemistry in the water column and explain the information recorded in sedimentary rocks of the Precambrian ocean [104]. More empirical and experimental research is needed to quantify controlling factors of fractionation that occur with iron oxide crystallization in hydrothermal mineral systems [303]. Future work that reconstructs Archean seawater iron and Si concentrations will be crucial in evaluating the extent to which ferrous hydroxide auto-oxidation controlled the Archean iron cycle and the oxidation of the young Earth [103]. The crystal structure of iron oxides synthesized under high pressures, their bonding nature and build-up structural motifs may guide us in discovering novel iron oxide phases and will be useful in revealing the chemistry and physics of Earth and planetary deep interiors [24].

Despite the long history of iron oxide research, they continue to attract the high attention of scientists all over the world; therefore we can suppose the future fundamental and

applied discoveries in this field. The main possible tendencies, which can be predicted from the current state of the research, include the further integration of various scientific analytical approaches, e.g., well-developed in geosciences and nanotechnologies [37,38,294,438], a wider implementation of “green” and biomimetic technologies [439] and a combined use of natural iron oxides and synthetic components in a single structure [6].

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