

UNIVERSIDAD SAN FRANCISCO DE QUITO USFQ

Colegio de Ciencias e Ingeniería

**Iron Oxide Nanoparticles general approach and catalytic agents
for the Environment: A Systematic Literature Review of
Research and Applications.**

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Ingeniería Química

Trabajo de fin de carrera presentado como requisito
para la obtención del título de
Ingeniero Químico

Quito, 23 de diciembre de 2020

UNIVERSIDAD SAN FRANCISCO DE QUITO USFQ
Colegio de Ciencias e Ingeniería

HOJA DE CALIFICACIÓN
DE TRABAJO DE FIN DE CARRERA

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RESUMEN

Esta revisión literaria trata sobre las nanopartículas de óxido de hierro que se utilizan en la remediación catalítica del agua. La revisión se realizó bajo las pautas de una revisión sistemática de literatura. Las nanopartículas de óxido de hierro tienen unas propiedades físicas y magnéticas destacadas, por ello las aplicaciones de las mismas son amplias tan variadas como en biomedicina, energía, remediación, catálisis entre otras. A continuación se presenta un enfoque general de los estudios publicados y seleccionados por año estableciendo una tendencia de crecimiento a lo largo del período de estudio. También se investigó el tipo de óxido de hierro teniendo como resultado que el más utilizado es la magnetita con más de la mitad de los estudios seleccionados. Los métodos de síntesis son muchos, siendo el más común la coprecipitación debido a su facilidad de producción. La remediación catalítica y la biomedicina son las aplicaciones con más publicaciones en los últimos cinco años con un desarrollo prometedor. En cuanto a la remediación catalítica, se encontró que las propiedades físicas y magnéticas más importantes para la caracterización de estas nanopartículas, siendo la forma esférica la más común con más de la mitad de los estudios. El tamaño y el material de recubrimiento son parámetros importantes para estos procesos, teniendo como preferencia pequeñas nanopartículas sin recubrimiento. Finalmente, la mayoría de los estudios reportaron valores bajos de coercitividad que indican un comportamiento superparamagnético con una observación que debe ser confirmada por una medición de baja temperatura así como la mayoría de los nanocatalizadores pueden separarse en el proceso catalítico debido a su alto nivel magnético.

Palabras clave: Nanopartículas de óxido de hierro; técnicas de síntesis; catalizadores magnéticos; degradación del colorante; oxidación avanzada; tratamiento de aguas; calentamiento por inducción magnética.

ABSTRACT

This systematic literature review studied the state of the art of iron oxide nanoparticles applied on catalytic remediation of water. The review was conducted under the PRISMA guidelines for systematic literature reviews. The iron oxide nanoparticles have outstanding physical and magnetic properties, for this reason their application fields are wide, for example for biomedical, energy, remediation or as catalyst. The number of studies published on iron oxide nanoparticles have increased establishing a linear growing trend through the period of study. The type of iron oxide was also investigated having as a result that the most used is magnetite with more than half of the studies. Various synthesis methods are applied, being the most common the co-precipitation due to its easiness of production. Also, applications of the iron oxides nanoparticles were reported to establish the most promising ones. Catalytic remediation and biomedicine are the applications with most publications in the last five years with a promising development. About catalytic remediation the most important physical and magnetic properties were reported being the spherical shape the most commonly used with more than half of the studies. The size and coating material are significant parameters for these processes, obtaining small nanoparticles with no-coating as the preferred material. Finally, most of the studies reported low values of coercivity indicating a superparamagnetic behavior. However, this property should be confirmed by low temperature measurement as well as the separation of the nanocatalysts during the catalytic processes due to their high magnetic saturation values.

Keywords: Iron oxide nanoparticles; synthesis techniques; magnetic catalysts; dye degradation; advanced oxidation; water treatment; magnetic induction heating.

TABLE OF CONTENTS

Introduction	11
Methodology	13
Selection of journals	13
Inclusion and exclusion criteria	14
Categories for the analysis and data coding.....	14
Results and Discussions	16
General Approach	16
Catalytic Remediation.....	20
Physical Properties of the nanoparticles.	21
Magnetic Properties of the nanoparticles.....	24
Conclusions	27
References	28

TABLES INDEX

Table 1. Categories studied in each research questions	15
Table 2. Types of iron oxide reported on the selected studies (Z number=68).	18
Table 3. Synthesis methods of iron oxide nanoparticles.....	19
Table 4. Size of iron oxide nanoparticles for catalytic remediation.	21
Table 5. Comparison of different complexes degraded.	24
Table 6. Coercivity of iron oxide nanoparticles for catalytic remediation.	25

TABLE OF FIGURES

Figure 1. Description of the Systematic Literature Review Guidelines.	13
Figure 2. Number of total and selected studies per year of publication (Z=total number).....	17
Figure 3. Applications of Iron Oxide Nanoparticles.....	20
Figure 4. Different shapes of Iron Oxide Nanoparticles for catalytic remediation.....	22
Figure 5. Coating materials for iron oxide nanoparticles.....	23

INTRODUCTION

Global contamination has been getting worse day by day due to anthropogenic activities. For this reason, researchers have been trying to develop new methods and materials to mitigate this contamination. One of the most popular and abundant pollutants are dyes which are produced by many industrial activities such as textiles colouring, manufacturing of paints, among others [1]. The uncontrolled release of dyes to the environment and its difficult removal is a well-known issue. To solve this problem a promising technique is the use of engineered inorganic nanomaterials like iron oxide nanoparticles (IONPs) which have been of great interest over the last two decades [2]–[4]. The use of IONPs in its different phases is of high importance and beneficial for many applications, being the most common in biomedicine. However their unique properties makes it really interesting for a wide range of applications, including catalytic remediation processes like dye degradation [5], [6].

IONPs present many advantages over conventional catalysts due to the easiness of surface modification which can allow the development of new nanostructured materials designed for specific catalytic processes. On the other hand, their magnetic properties such as coercivity, high magnetic saturation and superparamagnetism provide additional benefits to this material as it can be easily separated with an external magnet and present selective heating under the presence of an alternating magnetic field. These properties are promising for an improvement of environmental catalytic processes[7]–[9]. The different phases of iron oxides such as magnetite (Fe_3O_4) and maghemite ($\alpha\text{-Fe}_2\text{O}_3$) influence the properties mentioned above [10]. In environmental catalytic applications it is advantageous to work with maghemite as magnetite is usually unstable and can be oxidized easily.

The preparation methods of the IONPs plays also an important role in the properties of the material. A wide variety of synthesis methods are applied, being the most common co-precipitation, solvothermal or hydrothermal methods, while the newest trends are in the investigation of green synthesis, sonoelectrochemical or electrochemical methods [5], [11]. Also, the use of eco-friendly solvents extracted from natural raw materials are being explored [12], [8]. The correct choice of the method are fundamental for controlling of the physical as well as enhancing the magnetic properties [13].

The aim of this review is to provide a well-structured guide for students and researchers involved with the synthesis, characterization, and application of IONPs. The aspects that are going to be analyzed in this review are the preparation methods of different size and shape IONPs and their application as water remediation agents. Moreover, key parameters of catalytic remediation processes using IONPs, like composition of the polluted effluents, and catalyst specifications will be analysed.

METHODOLOGY

This systematic literature review will be conducted under the guidelines proposed by [14]–[16] with some adaptations for chemical engineering.

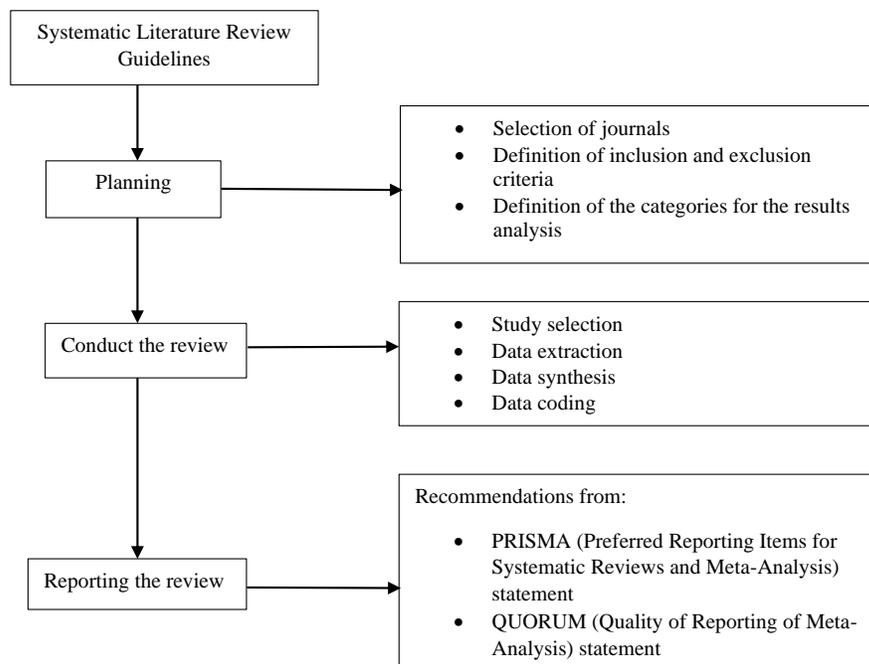


Figure 1. Description of the Systematic Literature Review Guidelines.

Selection of journals

This step consists on the selection of relevant publications for the systematic review in a consistent way. To keep the process methodologically strong and scientifically consistent, a method has been defined in this research for selecting journals. A mental map was developed for a better understanding of the information about the structure, restrictions, databases, and search terms defined. The selection of the journals was made in two of the most important and recognized databases for engineering: Science Direct and Scopus. The definition of the research terms was defined in order to answer the research questions. The search terms chosen were (nanocatalyst OR

nanoparticles) AND (“iron oxide” OR Fe₃O₄ OR magnetite OR maghemite OR magnetic) AND (“preparation methods” OR “synthesis methods”) these terms were searched in the title, abstract and keywords of the publications. The search terms seem to be too general, but the aim of this review is to give a general overview of the iron oxide nanoparticles with a focusing on catalytic remediation hence the publications found with the above terms about dye degradation were chosen too.

Inclusion and exclusion criteria

The general criteria for the SLR are studies published between 2015 and 2020. Also, studies that describe preparation methods and applications of iron oxide nanoparticles.

The specific criteria include studies that report the reactants, temperature of reaction, type of iron oxide formed. Moreover, studies that describe applications of iron oxide nanoparticles especially catalytic bioremediation and studies describing modifications, physical and magnetic properties of iron oxide nanoparticles.

The exclusion criteria were determined and accordingly studies that reached these criteria were excluded studies not identified by “Articles” or “Reviews” in the databases selected and studies not classified as open access in the databases.

Categories for the analysis and data coding

The categories for the analysis of the retrieved articles were defined based on answering the three specific research questions. The use of these categories was used to group all the publications according to specific characteristics. The data was organized in a scientific datasheet for better organization and easier classification.

Table 1. Categories studied in each research questions

	What are the preparation methods, type of iron oxide formed and applications of the iron oxide nanoparticles?	What are the specific parameters of iron oxide nanoparticles that influence in catalytic remediation?	What are the parameters to consider in a degradation process?
Reported preparation methods.	X		
Reported types of iron oxide	X		
Reported applications	X		
Size		X	
Coatings		X	
Shape		X	
Coercivity		X	
Magnetic saturation		X	
Reported complexes degraded			X
Type of catalysts			X
Degradation percentage			X
Time of degradation			X

RESULTS AND DISCUSSIONS

General Approach

In this section the results obtained from conducting the review are reported and discussed with the considerations from the Figure 1. The results are analyzed from the retrieved articles according to the criteria from the methodology section. As a new promising type of material, the analysis of the results from Figure 2 shows how the number of studies published on iron oxide nanoparticles are increasing with time showing a higher percentage of studies in the year 2020.

The final selected studies were included following the methodology reported above. As a result, 85 of all studies were chosen from the selected databases after considering the studies that describe preparation methods, applications, as well as physical and magnetic properties of iron oxide nanoparticles, especially in catalytic remediation. The tendency from the total studies is also maintained for the selected ones (Figure 2), which reinforces the idea that this topic keeps developing and new studies will be published in the following years. Moreover, it is clear that the use of iron oxide in many different applications has been increasing over the past decade as it is a great alternative to commonly used materials for medicine, environment, data recording, sensors, energy storage, catalysis, etc.

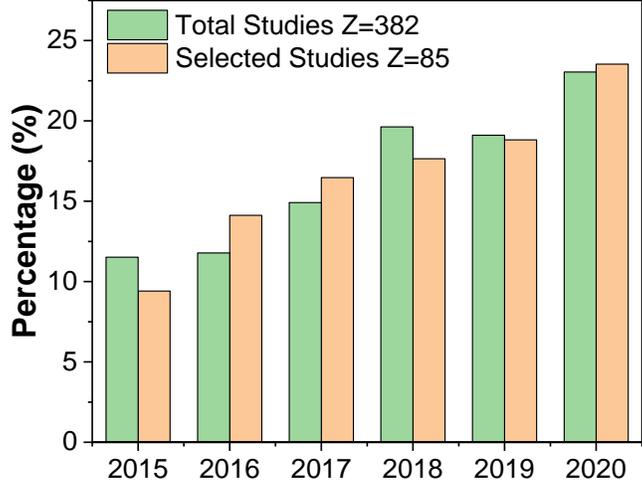


Figure 2. Number of total and selected studies per year of publication (Z=total number).

Regarding the “type of iron oxide”, this category refers to the phases of iron oxide that formed the nanoparticles. These phases can be obtained by different synthesis methods in which parameters like reaction time, temperature, temperature ramp and surfactant play a fundamental role. The majority of nanoparticles are constituted by magnetite (Fe_3O_4) with more than half of the studies. Using magnetite as the main phase in iron oxide nanoparticles can lead to excellent magnetic properties compared with the other phases. Each type of iron oxide provides specific advantages depending on the field of application. In addition, in some studies, a mixture of phases such as magnetite-maghemite are observed in the nanoparticles, providing them with hybrid magnetic characteristics. One thing to consider is that many of these studies are not able to prove with 100 % certainty the production of this magnetite phase and they may be working with materials chemically degraded as magnetite is quite unstable and can be easily oxidized.

Table 2. Types of iron oxide reported on the selected studies (Z number=68).

Sub-Category: Type of iron oxide	Percentage (%)	Studies
Magnetite	57.35	[9], [12], [13], [17]–[51]
Ferrite	16.18	[6], [52]–[61]
Hematite	10.29	[62]–[68]
Maghemite	8.82	[69]–[74]
Magnetite-Maghemite	4.41	[75]–[77]
Magnetite-Ferrite	1.47	[78]
Pyrite	1.47	[79]

With respect to category “Synthesis Methods”, Table 3 summarizes the results obtained in the last 5 years excluding reviews since most of them do not report the synthesis method in a specific manner. It can be seen that the most common synthesis method for iron oxide nanoparticles synthesis is by co-precipitation. On the other hand, thermal methods are the following most used ones in which techniques like hydrothermal, solvothermal, thermal decomposition and combustion method stands out over the recent years. Among the newly methods, microwave assisted synthesis combined with regular methods like thermal decomposition or the polyol procedure, constitute a step forward in the optimization of these processes due to its easy installation and reproducibility. The principal aspect of developing iron oxide nanoparticles by the co-precipitation method lays in the easiness of the procedure with few steps and also the particles that can be obtained are very stable in water unlike the particles using thermal decomposition method. The problem with the co-precipitation synthesis methods is that it is pretty difficult to obtain particles with a narrow size distributions. On the other hand, the high temperature used in thermal methods have the advantage of producing a controlled phase of the iron oxide and better size distributions. New methods have appeared with the need of using green solvents or reducing the byproducts and residues of the

synthesis of iron oxide nanoparticles, for this reason green synthesis methods are being developed and they are gaining more reputation.

Table 3. Synthesis methods of iron oxide nanoparticles.

Sub-category: Synthesis method	Percentage (%)	Years of publication	Studies
Co-precipitation	27.27	2015-2020	[20], [21], [23], [27], [30]–[32], [34], [37], [39], [44], [47], [48], [50], [67], [68], [70], [80]–[82].
Hydrothermal	16.88	2015-2020	[6], [9], [24], [25], [30], [51], [54], [55], [62], [74], [83], [84]
Solvothermal	6.49	2016-2020	[22], [40], [41], [50], [69]
Microwave	7.79	2017-2018	[13], [33], [47], [52], [61], [75]
Thermal Decomposition	6.49	2016-2018	[43], [47], [57], [65], [85], [86]
Combustion Method	6.49	2016-2018	[17], [52], [58], [60], [61]
Precipitation	5.19	2017-2018	[43], [63], [66], [87]
Sol-Gel	5.19	2015-2020	[37], [68], [73], [77]
Green Synthesis	5.19	2016-2020	[12], [29], [36], [71]
Impregnation	3.90	2015-2017	[34], [73], [76]
Polyol	2.60	2015-2016	[53], [78]
Sonochemical	2.60	2018-2020	[42][45]
Ultrasound Probe	1.30	2019	[26]
Electro Synthesis	1.30	2017	[18]
Not specified	1.30	2018	[46]

The different applications of the iron oxide nanoparticles is another category analyzed in this systematic review. Figure 3 shows the results of different applications in the retrieved studies. Almost 50 % of the studies are divided between catalytic remediation and biomedicine. Water treatment, catalysis and electronic devices present around 10 % of the studies each, while 19 % of the studies do not specify their possible applications. And finally, one article studied the application as anticorrosion mechanism. In this review the application of interest is catalytic remediation because as seen in the selected studies, it is a rising subject of application. In the case of biomedicine, iron oxide nanoparticles are especially used for drug delivery and cancer

treatment. Another application that is increasing is water treatment since the properties of the nanoparticles fit for processes like adsorption and degradation of inorganic and organic pollutants. The reactions studied so far in catalysis are transesterification, while catalytic remediation represent the photocatalysis of dyes making them easy to removal.

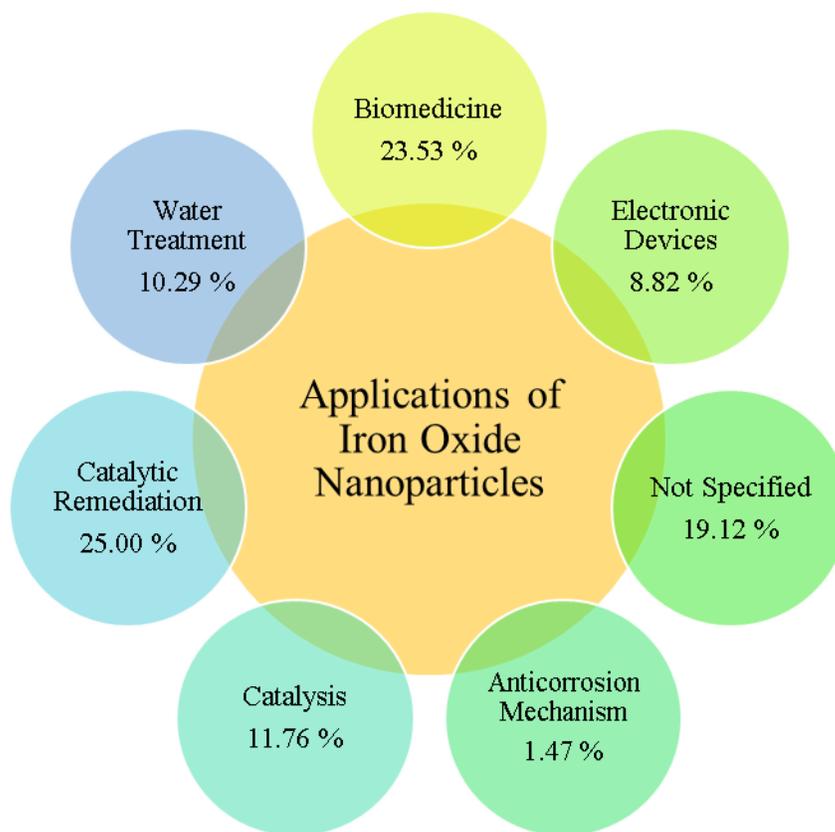


Figure 3. Applications of Iron Oxide Nanoparticles. Catalytic Remediation ([12], [20], [23], [28], [44], [47], [53], [63], [65], [68], [69], [74], [83], [88]–[90][45]). Biomedicine ([6], [13], [22], [31], [43], [46], [48], [51], [52], [60]–[62], [82], [85], [91], [92]). Water Treatment ([29], [30], [32], [36], [40], [59], [71]). Catalysis ([21], [37], [50], [58], [73], [76], [78], [86]), Electronic Devices ([9], [25], [35], [41], [70], [78]). Anticorrosion Mechanism ([24]), Not specified ([17], [27], [38], [42], [55]–[57], [66], [75], [77], [79], [84], [93]).

Catalytic Remediation

The term catalytic remediation can be explained as the use of catalysis processes for mitigation of environmental issues. The main issue is the difficulty of removal of dyes that are present in

water. The difference between water treatment and catalytic remediation is the presence of catalytic reactions and not only the adsorption of pollutants. Photocatalysis is the main mechanism where with the use of solar light the photoreaction is accelerated causing the oxidation of the dyes. The product of this reaction is a new dye much easier to remove with classic methods used in water treatment plants.

Physical Properties of the nanoparticles.

The size of iron oxide nanoparticles applied in catalytic remediation are spread between smaller than 10 nm up to over 100 nm. The category “size of iron oxide nanoparticles” are shown in the Table 4 with the results that the 41.18 % of the studies have a particle size in the range from 1 to 10 nm followed by the 23.53% between 10 to 20 nm. As conclusion, the preferred sizes for catalytic remediation are usually in the very small range due to the high surface areas and the influence of the magnetic properties as well. Even though, the denomination of nanoparticles is given to particles between 1-100 nm, there are some studies that specify iron oxide nanoparticles above this limit. This denomination should be stated more carefully to avoid confusions. One important thing about using catalysts in the nanometric range is that they can provide the benefits of both homogenous and heterogeneous reactions, being very stable and well-dispersed in organic or aqueous media but also, they can be recovered for regeneration and reuse [65], [89].

Table 4. Size of iron oxide nanoparticles for catalytic remediation.

Sub-Category: particle size	Percentage (%)	Studies
Between 1-10 nm	41.18	[44], [50], [53], [54], [69], [83]
Between 10-20 nm	23.53	[12], [20], [63], [94]
Between 20-30 nm	17.65	[65], [68], [90]
Over 100 nm	11.76	[28], [74]
Not specified	5.88	[45]

Regarding the “Shape of iron oxide nanoparticles”, Figure 4 shows that the preferred shape of the iron oxide for catalytic remediation is spherical with more than the half of studies. This can be explained by the surface area that is an essential factor in catalysis as materials with higher values present more active sites for chemical reactions. The shape is influenced by the synthesis method. A correlation between these two aspects would be important to identify.

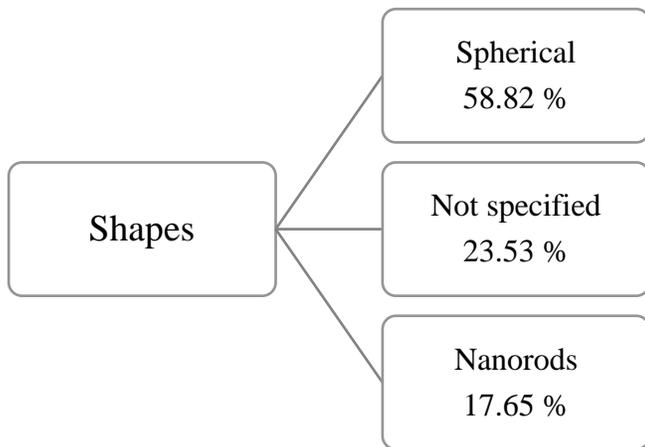


Figure 4. Different shapes of Iron Oxide Nanoparticles for catalytic remediation. Spherical [12], [28], [44], [50], [53], [65], [67], [74], [83], [94]. Nanorods ([63], [68], [69]), Not specified ([20], [45], [88], [89][45])

Focusing on the “Coating Materials” of the review studies, Figure 5 shows that almost half of the studies do not report a coating with the 47.06 % of studies. The coating more used for catalytic remediation is silica followed by titania. All the materials reported in Figure 5 are inorganic, which leads to concluding that the best coatings for catalytic remediation are inorganic and no application of coatings. The coatings are well-combined with metal doping as many catalysts are prepared by metals such as palladium or platinum.

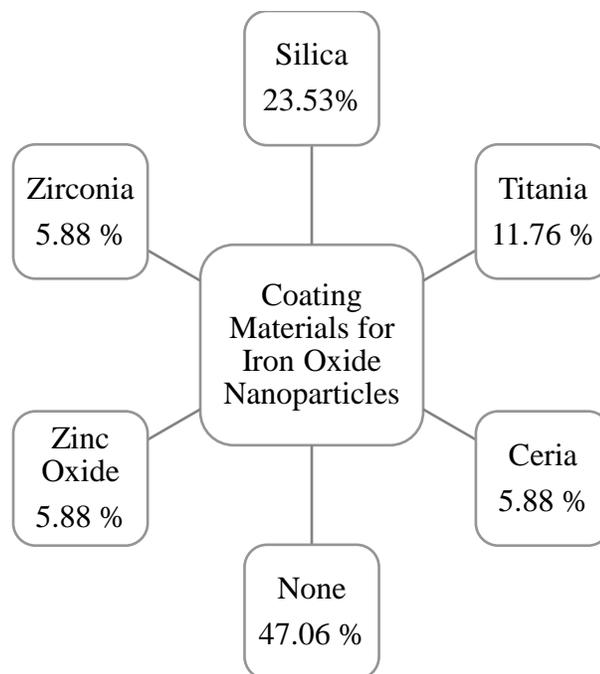


Figure 5. Coating materials for iron oxide nanoparticles. Silica([23], [44], [45], [74]), Titania ([44], [83]), Ceria ([89]), Zinc Oxide ([53]), Zirconia ([69]) None ([12], [50], [54], [63], [65], [67], [68]).

With respect to the type of dyes and complexes degraded, by the nanoparticles in the application of catalytic remediation study can report more than one dye degraded. The main dye degraded is methylene blue with the 26.32 % of the studies [44], [47], [54], [67], [74] followed by rhodamine B and methyl orange with 10.53 % for each one [12], [20], [53], [63], [65], [68]. The important aspects are the degradation percentage and the time that requires to achieve this percentage. Table 5. Comparison of different complexes degraded. shows that dyes are most of the complexes degraded and the percentages of degradation are around 80 % to 100 %. The time needed to reach the reported degradation is above 100 minutes. Most of the catalysts used for catalytic remediation are coated and functionalized by metals. Another industry that contributes to pollute the water are pharmaceutical which are contemplated by the degradation of antibiotics and 4-nitrophenol following the same tendency as dyes degradation but with a reduction of the reaction time. The importance of degradation is that most of the dyes and complexes are difficult to remove as

discharged. After degradation by the remediation reaction the molecules are shortened and the resulting compounds can be removed easier.

Table 5. Comparison of different complexes degraded.

Complex Degraded	Type of Catalyst	Degradation (%)	Time	Studies
Tetracyclines chloride	Fe ₃ O ₄ -chitosan beads	85	120 min	[50]
Methylene Blue	AgPt@γ-Fe ₂ O ₃ /t-ZrO ₂	80	80 min	[69]
	ZnFe ₂ O ₄ @rGO	94.2	60 min	[88]
	TiO ₂ @Fe ₃ O ₄ @C-NF	75-100	10 min	[83]
	Ni@Fe ₃ O ₄	90	60 min	[12]
	α-Fe ₂ O ₃	99	270 min	[90]
	Fe ₃ O ₄ @SiO ₂	97	140 min	[44]
AB92	AgPt@γ-Fe ₂ O ₃ /t-ZrO ₂	90	80 min	[69]
Rhodamine B	ZnFe ₂ O ₄ @rGO	49	60 min	[88]
	Ni@Fe ₃ O ₄	80	60 min	[12]
Antibiotics	TiO ₂ @Fe ₃ O ₄ @C-NF	80-100	125 min	[83]
Congo Red	Ni@Fe ₃ O ₄	80	60 min	[12]
	α-Fe ₂ O ₃	82	20 min	[63]
4 <i>H</i> -drobenzo[<i>b</i>]pyran	Fe ₃ O ₄ @SiO ₂ -guanidine-PAA	94	20-40 min	[23]
dihydropyrano[<i>c</i>]chromene	Fe ₃ O ₄ @SiO ₂ -guanidine-PAA	94	20-40 min	[23]
Rose Bengal	α-Fe ₂ O ₃	86	105 min	[63]
p-nitrophenol	Fe ₃ O ₄ @NH ₂ -mesoporous silica@PPy/Pd	94	180 s	[45]
Methyl Orange	Fe ₃ O ₄ @NH ₂ -mesoporous silica@PPy/Pd	99	600 s	[45]
Orange G	Fe ₃ O ₄ /CeO ₂	98	120 min	[34]
Malachite Green	ZnO@CoFe ₂ O ₄	100	120 min	[53]
Thymol Blue	ZnO@CoFe ₂ O ₄	100	120 min	[53]

Magnetic Properties of the nanoparticles.

One of the main advantage of using iron oxide nanoparticles is that it is possible to enhance catalytic or extraction processes with their magnetic properties. This kind of materials can be easily separated by using an external magnet which presents a cost-efficient process that is less time-consuming common. Moreover, when dealing with superparamagnetic nanoparticles they can behave like normal materials with no remnant magnetization except when they are subjected to a

magnetic field. Then they recover their magnetism and can be dragged towards the magnet [54]. Remarkably, when these particles are subjected to an alternating magnetic field, they present the ability to absorb the magnetic energy and dissipate it as heat which is an advantage for catalytic processes as usually reaction yields increase with the increasing temperature [44], [74].

With respect to the “Coercivity” category, Table 6 shows that almost half of the studies have a low coercivity which means a superparamagnetic behavior since there is no noticeable hysteresis in the magnetization loops [69], [83]. The remnant magnetization of this particles will probably lead to a magnetic aggregation that with time will decrease surface areas and catalytic activity of the material [23], [63]. Therefore, to improve catalytic processes it is better to perform reactions with superparamagnetic nanocatalysts. Also, a high percentage of the studies (47.06 %) did not specify the coercivity of the nanoparticles. It would be interesting to investigate more deeply about this issue due to the influence they have on catalysis.

Table 6. Coercivity of iron oxide nanoparticles for catalytic remediation.

Sub-Category: Coercivity	Percentage (%)	Studies
Between 0-12 Oe	47.06	[20], [23], [28], [34], [44], [74]
Over 12 Oe	5.88	[65], [68]
Not specified	47.06	[12], [45], [47], [54], [63], [67], [69], [83]

Regarding to the magnetic saturation of the studies, the results are wide with almost the half of them not specifying the influence of magnetic saturation [12], [28], [45], [50], [54], [63], [67], [69], [74], [83]. Magnetic saturation shows how magnetic a material can be, superparamagnetic nanoparticles present an interest behavior in which they only act like small magnets when subjected to a magnetic field. For this reason, the magnetic aggregation is probability reduced. Depending on the type of iron oxide the values of magnetic saturation are specific for each bulk

material [95], [96]. Adding coatings to the surface of the nanoparticles reduce the values of magnetic saturation of the nanoparticles. Different coatings can decrease magnetic saturation values, but it is important to maintain a value high enough for magnetic separation. In the selected studies all of them show values enough for magnetic separation [23], [34], [44], [53], [65], [68]. For other applications such as battery production or electronic devices high values of magnetic saturation could be a more valuable property because of the magnetization that can have which derived in more energy storage in addition to control the alternate current through the inductor that some devices can have [41], [70], [97], [98].

CONCLUSIONS

Here, an extensive systematic literature review was performed to analyze, synthesize, and describe the most recent studies performed on iron oxide magnetic nanoparticles. It was possible to develop a well-described guide for future studies and researchers that want to raid in this field of study. With all the information collected, it was possible to understand and describe the most common types of iron oxide nanoparticles, synthesis methods, coatings, magnetic features, and applications. Using iron oxide nanoparticles for different applications has become of great interest in the last decade. For catalytic remediation, processes like degradation with iron oxide nanoparticles are an emerging technology with excellent advantages against common methods. Specifically, and even though some of them fail to prove this affirmation, magnetite is the most common and used material as described in different studies. Moreover, for the preparation of this kind of materials it was possible to summarize an extensive list of techniques and it was possible to identify a new trend using green solvents extracted from plants in order to reduce the residues from the synthesis of the nanoparticles. This trend indicates the interest of making these synthesis processes more appropriate for future needs and applications. Catalytic remediation using IONPs is a promising technique for degradation of dyes and pharmaceutical complexes being the most common studied dye the methylene blue. The percentages of degradation are high for both type of compounds but with a significant shorter reaction time for degradation of pharmaceutical complexes. About magnetic properties, magnetic saturation should be carefully reported since coating diminishes it and can generate some confusion with the reported vales. Many publications talk about superparamagnetic properties of the nanoparticles but this properties should be confirmed by low temperature measurements. In conclusion, iron oxide magnetic nanoparticles are a promising material widely studied during the last five year and with great and growing future prospection.

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