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Comprehensive Review of Magnetic Polymeric Nanocomposites with Superparamagnetic Iron Oxide Nanoparticles for Oil Removal from Water

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***Abstract**: Several oil spills in aquatic environments have been reported over the last few years, and a great effort has been made to develop new techniques for collecting and removing oil from water on a large scale to prevent environmental pollution by this contaminant. In view of the various problems involving traditional methods, such as the generation of secondary pollution, high costs and complexity of synthesizing material and expenses to transport equipment, among others, new technologies have been developed for removal of oil from water. Among these, the use of magnetic polymeric nanocomposites has presented promising results, since they have high oil adsorption efficiency, ease of material removal through an external magnetic field, low cost of synthesis and possibility of reusing the material for several cycles, among others. However, a lack of studies about these promising systems exists regarding this technology and its procedures. Therefore, here we present a brief bibliographic review of the synthesis routes to obtain magnetic polymeric nanocomposites containing superparamagnetic iron oxide nanoparticles developed for oil removal from water and report future trends and perspectives for progress of this technology.*

***Keywords**: magnetic polymeric nanocomposites, superparamagnetic nanoparticles, water treatment*

Introduction

Oil spillage in the ocean is a recurring problem in many parts of the world and has been causing serious environmental damage. The extent of devastation caused by accidents, such as in Dalian, China (2010), Gulf of Mexico (2010), Campos, Brazil (2011), and many others show that new remediation strategies should be continuously studied [1]. In the Gulf of Mexico oil spill, an estimated 750 million liters of oil was released into the sea, making it the most serious in history. In 2019, there was an oil spill in Brazil that affected more than 2 thousand kilometers of the coastline of the Northeast and Southeast regions of the country. The recurrence of these accidents means that new studies are carried out every year to develop more effective remediation techniques for oily wastewater treatment.

Many traditional techniques are used to treat oily water, such as solvent extraction [2], catalytic cracking [3], mechanical centrifugation [4-6], membrane separation [7-9], gravity [10,11], biodegradation [12-15], mechanical recovery by oil skimmers [16], and many others. However, many reports can be found in the literature about the low separation efficiency, secondary pollution generation, high costs to synthesize and transport materials, large equipment needed, and many other drawbacks that restrict the use of these methods [15]. Among various methods, physical sorption by high porosity, superhydrophobic and superoleophobic sorbents is simple, fast, and cheaper than the others mentioned. Many materials, such as natural and synthetic adsorbents [15,17-21], have been developed over the years.

A new approach using nanoparticles with magnetic properties has been widely studied for oily water treatment, where the oily phase can be recovered by magnetic separation [21-25].

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Through the incorporation of these magnetic nanoparticles in macroporous polymers, it is possible to produce magnetic polymeric nanocomposites with new properties in relation to conventional systems [26], since the material can be removed from the medium in which it is inserted through an external magnetic field.

In general, magnetic nanocomposites can be divided into three main groups: core-shell inorganic nanocomposites [27]; self-assembled colloidal nanocomposites [28]; and organic-inorganic nanocomposites [29]. Each type has particular morphology for the desired applications. Here we present a summary of these materials, including their morphologies, with focus on organic-inorganic magnetic nanocomposites and their in situ [30] and ex-situ [31] synthesis routes. The most used nanoparticles with magnetic properties are described to demonstrate the phenomenon of superparamagnetism [32-35] required to remove the nanocomposite from oily water.

Finally, we present a bibliographic review to evaluate the use of magnetic polymeric nanocomposites containing Fe_3O_4 and γ-Fe_2O_3 for oil removal from water, demonstrating the importance of these magnetic nanocomposites. Several advantages, such as simplicity, low environmental impact, high oil adsorption efficiency, convenient extraction procedure, and cheap synthesis have been reported by various authors.

Magnetic nanocomposites

Magnetic nanocomposites are considered recent materials because the publication of scientific articles and filing of patents involving nanotechnology only began to grow in 1989. The first application of magnetic nanocomposites dates to 1993, in magnetic resonance contrast agents, where the use of magnetic nanocomposites improved the accuracy of the diagnosis of lymphatic metastasis from 60% to 94% [36].

The current applications are diverse, including controlled release of drugs [37], immunoassays [38,39], molecular biology [40,41], DNA purification [42], cell separation and purification, solid-phase separation as scavenger resins [43], magnetic inks, magnetic sensors, and other applications. The VOSviewer© software was used to check the correlation strength between the keywords: Magnetic + Nanocomposites + Applications during the period between 2011 and 2021 in the Web of Science Database. Only the keywords that appeared in at least 20 articles were evaluated. The bibliometric map can be observed in Figure 1.

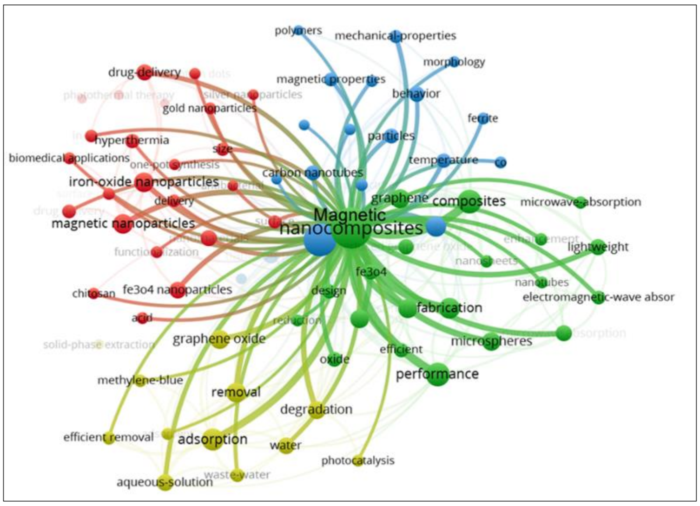

Figure 1. Bibliometric map of magnetic nanocomposites' applications using VOSviewer© software

These materials can be developed in different ways and can be found in the form of powders, suspensions, fibers, films, and three-dimensional solids, each of which has a wide number of applications according to its properties. Several advantages regarding the use of nanocomposites are described in the literature. These materials have improved properties compared to conventional systems due to the presence of magnetic nanoparticles that have a high specific surface area, which contributes to the bonding and entanglement of these nanoparticles in the respective matrices [44]. The mechanical properties of nanocomposites usually show better values of specific strength, stiffness, and toughness than conventional systems [45]. They can also have better resistance, gas barrier and flame retardant properties, among other characteristics.

The evolution of published papers with the keywords ‘’magnetic nanocomposite’’ over the last decade in the Science Direct database can be seen in Figure 2. Only research articles and reviews were taken into account. It is possible to observe through a second-order quadratic polynomial fit that the development of these materials has been arousing increasing interest in the scientific community over the years.

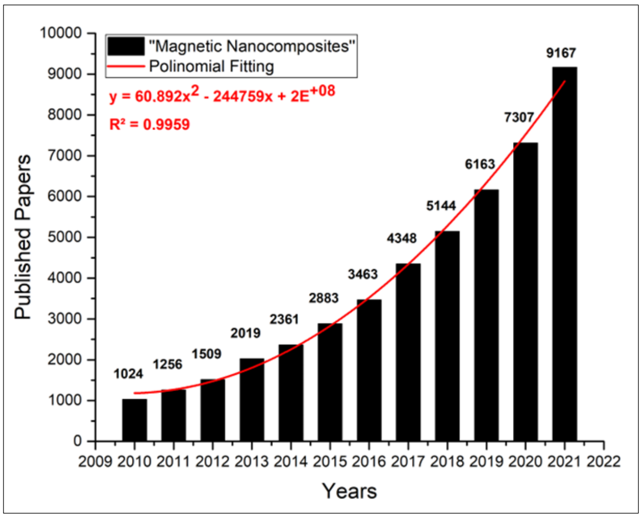

Figure 2. Evolution of published papers using the ‘’magnetic nanocomposites’’ eyword phrase over the years. The data were obtained from the Science Direct database

However, the main advantage of using magnetic nanocomposites over others is the fact that the material can be collected and separated through the induction of a magnetic field. These properties depend directly on the size distribution of the magnetic nanoparticles and the presence or not of interactions between the surface of the polymeric matrix and the contaminants [46-48]. In general, three main types of magnetic nanocomposites are described in the literature: core-shell inorganic nanocomposites [26]; self-assembled nanocomposites [27]; and organic-inorganic nanocomposites [28].

Magnetic core-shell inorganic nanocomposites are formed by combining two materials on a nanoscale in a single hybrid particle that ends up having dual properties from the materials that compose it [49,50]. Silica (SiO_2) has been widely used for the production of nanocomposites due to its coating action and ability to encapsulate magnetic nanoparticles such as magnetite (Fe_3O_4) and other iron oxides.

The presence of silanol groups on its surface, susceptible to various types of functionalization, as well as the presence of a high surface area containing well-defined pores, favors the use of this material for separation processes, catalysts, and various biomedical and environmental applications, also due to its biocompatibility, degradation stability and the hydrophilic character of both materials [26,30]. Studies have also been conducted using metals such as TiO_2 [51], Palladium (Pd), Platinum (Pt), and Gold (Au) to form magnetic nanocomposites with magnetite [52].

Magnetic self-assembled colloidal nanocomposites are synthesized through the formation of small building blocks containing magnetic nanoparticles arranged in macroscopic structures, forming two-dimensional or three-dimensional networks with a high translational degree and crystallographic alignment [49].

The organization of these blocks can generate crystals or almost colloidal crystals. In this regime, the electronic structure and the magnetic and optical properties of the material can be optimized by modifying the size of the crystallites and the building blocks, which can lead to several phenomena, in particular superparamagnetism, meaning that by modifying the electronic structure, several properties can be obtained without changing the chemical composition of the material [28]. These materials are also known as nanocrystal (NC) solids and are used in optoelectronic applications such as light-emitting devices [53], lasers [54], as well as in biomedical applications aimed at magnetic separation of biological targets [55] and drug delivery [56], among other applications.

Magnetic organic-inorganic nanocomposites are the most common materials, and by definition have in their composition a polymeric matrix containing some type of inorganic magnetic or superparamagnetic material [29] dispersed on the surface and/or inside a porous crosslinked structure [57,58]. The use of copolymers is a good alternative, mainly because they keep the magnetic material isolated, protected, and separated from the medium in which it will be applied, mechanically reinforcing the material and improving its processability, in addition to introducing unique optical, electrical and chemical properties, thus expanding the applicability of these materials [59].

The representations of the main morphologies found in the literature on magnetic nanocomposites are shown in Figure 3.

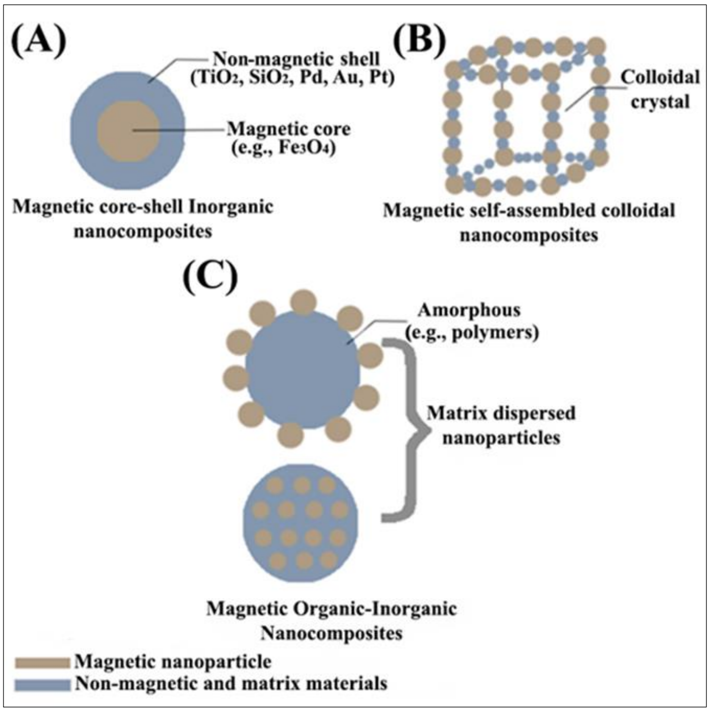

Figure 3. Main morphologies of magnetic nanocomposites.
(A) Magnetic core-shell inorganic (B) self-assembled colloidal
(C) organic-inorganic

The focus of this literature review is to evaluate studies containing polymeric magnetic nanocomposites for oil removal from water. Therefore, the main ways of obtaining them are briefly described below.

Synthesis of magnetic polymeric nanocomposites

One of the main obstacles to the synthesis of magnetic polymeric nanocomposites is the aggregation that occurs between the nanoparticles during the process. This aggregation can alter the magnetic nanoparticles' average size and consequently cause the loss or reduction of the desired properties of the magnetic nanocomposite obtained through the nanometric range [60].

To overcome this problem, several methods are described in the literature to obtain nanocomposites with greater homogeneity, i.e., to improve the dispersion of the nanoparticles in the polymer. These methods can be classified as in situ, ex situ, and other more specific techniques (Figure 4).

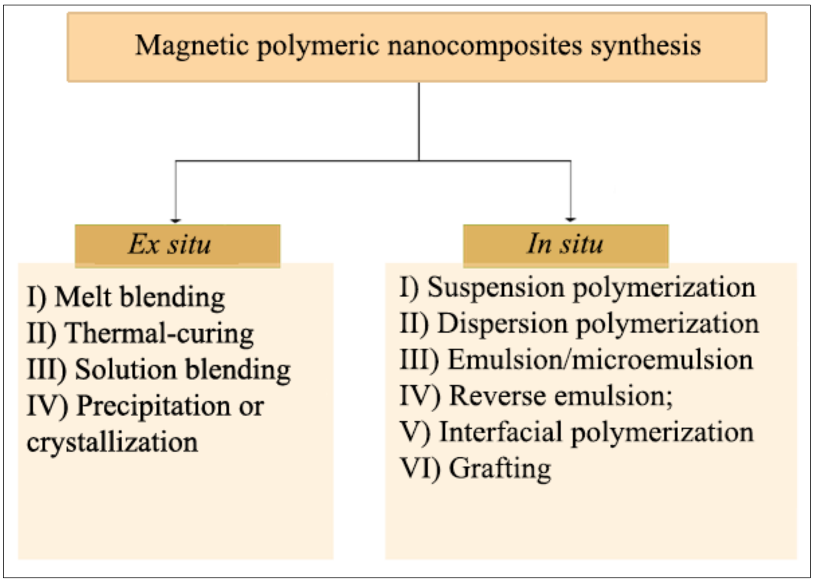

Figure 4 - Main routes to obtain magnetic polymeric nanocomposites

Ex-situ methods

Traditionally, *ex situ* methods are considered the simplest for the preparation of magnetic nanocomposites, considering that they occur through the direct incorporation of nanoparticles in polymers through various types of processing, such as extrusion, where blends and polymeric films are produced. Inclusion of nanoparticles can generally be done by melt-blending processes of the polymer [61]. On the other hand, no matter how much the processes are optimized to facilitate the uniform dispersion of the nanoparticles in the extruded material, characterization analyses of the material surface show the aggregation of nanoparticles [50].

Several other ex situ strategies and methods are described in the literature: (I) coupling of polymer chains to nanoparticles; (II) simultaneous combination of polymeric chains and nanoparticles; (III) formation of ex situ nanoparticles followed by polymerization of the organic component [48, 62-64], and (IV) ex situ formation of polymeric nanoparticles followed by precipitation or crystallization, among other methods [30].

In-situ methods

During the *in situ* methods, the inorganic precursors of the nanoparticles are introduced in a solvent containing monomers, and under polymerization conditions the nanoparticles are formed through chemical reactions within the pores of the polymer matrix [30], preventing their aggregation and consequently favoring good distribution of the material throughout the matrix. The main drawback of this technique is the fact that the remaining molecules of both the monomer and inorganic material can influence the final properties of the nanocomposite [65]. Among the *in situ* methods, the standouts are suspension [66,67], dispersion [68], emulsion/microemulsion, reverse emulsion [69-71], interfacial polymerization [72], polycondensation [73], and grafting.

Given the various applications and routes to synthesize nanocomposites, the choice of the components is extremely important to obtain the desired final material properties. In this respect, the magnetic nanoparticles commonly used to obtain magnetic polymeric nanocomposites are discussed below.

Magnetic nanoparticles (MNPs)

To facilitate removal of the material from the medium, the nanoparticles introduced into the polymer matrix must have magnetic properties enabling their removal by an external magnetic field [74]. The magnetic nanoparticles (MNPs) in association with polymers can improve oil absorption and generate a new product capable of treating oily water (Figure 5).

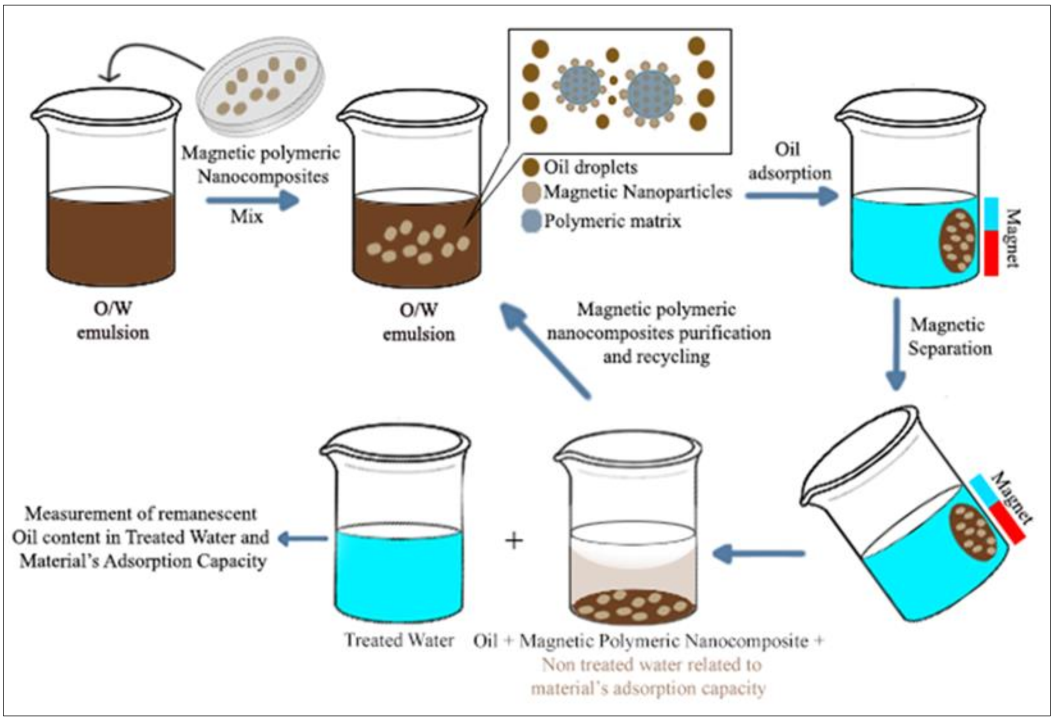

Figure 5. Oil/Water emulsion treatment with magnetic polymeric nanocomposites mechanism

The MNPs used to form polymeric nanocomposites have been synthesized through several methods. The best -known are sol-gel deposition [75-77], chemical coprecipitation [33,78], microemulsion [79], hydrothermal [80-84], and solvothermal synthesis [85-87], among others. Many synthesis parameters, such as temperature, *p*H, and ion concentration, as well as changes in the average particle size, distribution, geometry, and crystalline ordering, among other variables, are widely discussed in the literature [88-90].

Magnetic nanoparticles have been widely studied for the preparation of nanocomposites, such as Fe_3O_4, γ-Fe_2O_3, g-Fe_2O_3 [33,91-93], metallic Co, Fe, Ni [91,92,94], spinel-type ferrite ($MgFe_2O_4$), $MnFe_2O_4$ [58,94,95], $CoFe_2O_4$, FeCo, $CoPt_3$, and FeCoPt nanoparticles [45,57,96]. However, the largest number of studies for oil removal involve magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3). When magnetite and maghemite particles in general have diameter smaller than 20 nm, they present the superparamagnetism phenomena. This occurs because with this average size, the nanoparticles can be considered individually as single magnetic monodomains, although an ensemble can be considered non-magnetized [33-35], which means that these nanoparticles can be temporarily magnetized in the presence of an external magnetic field, followed by disappearance of the magnetization after the removal of the field, i.e., there is no remanent magnetization [97-100].

These magnetic nanoparticles have been incorporated into various organic structures, such as polymers, graphenes, molecularly printed polymers (MIPs), surfactants, and inorganic materials such as metal oxides and silica for various applications [101]. The evolution of studies over the years using keywords (Magnetic polymeric nanocomposites + magnetite) and (Magnetic polymeric nanocomposites + maghemite) in the Science Direct database, where only peer-reviewed papers were counted, is reported in Figure 6. The total papers published according to the database using magnetite as MNPs were 5,334, in contrast with maghemite with a total of 1,609 for production of magnetic polymeric nanocomposites for different applications.

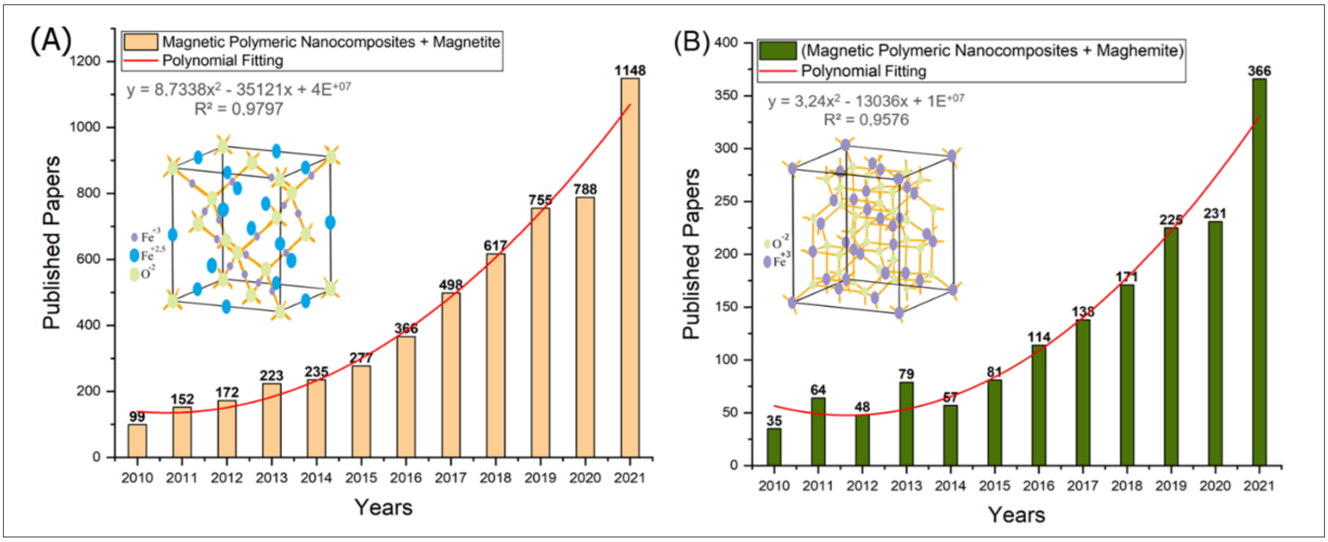

Figure 6. Published papers from 2010 to 2021 of (A) “magnetic polymeric nanocomposites + magnetite”, and (B) “magnetic polymeric nanocomposites + maghemite”, and their respective polynomial fitting and inorganic MNP structure

The importance of using these materials for the formation of nanocomposites is evidenced by the growing number of papers published in the last ten years. Nanocomposites containing magnetite and maghemite MNPs are commonly used in separation techniques, among which are chromatographic separation [77], solid-phase extraction [102], micro-extraction in packaged sorbents [103], solid-phase microextraction [104], and dispersive solid-phase extraction [105]. The separation using MNPs depends on the type of molecule, sorbent material used, and the interaction between the analyte molecules and the functional groups present on the nanocomposite surface [106].

Magnetic polymeric nanocomposites for removal of oil from water

Due to environmental concerns, there is rapid growth in the field of water treatment for oil removal. The development of new materials is fundamental in the process of water treatment.

The use of sorbent materials with superoleophilic properties is a promising strategy for oil removal from water surfaces. Several synthetic polymers, such as polyurethane (PU), polyethylene (PE), polypropylene (PP), polyvinylidene fluoride (PVDF), polystyrene (PS) and polyacrylonitrile have been reported for oil removal from aqueous systems due to their good hydrophobic properties [107-111].

However, since synthetic fibers are not biodegradable and can sink, the full recovery of the material after the oil adsorption is fundamental to prevent secondary pollution. Therefore, a good strategy to overcome this problem is to add iron oxides to the material, making the recovery easier through an external magnetic field. The surface of these nanoparticles can be functionalized by different organic groups combining the advantages of high surface area with multiple functionalities, improving the supereoleophilic properties.

A huge number of environmental clean-up technologies have been established using iron oxide nanomaterials for water treatment, mainly for the enhanced removal of heavy metals. However, these nanomaterials can also be used for the removal of other pollutants such as hydrocarbons and organic dyes, but to date just a few reports are available in the literature using these magnetic polymeric nanocomposites with iron oxide nanoparticles (Table 1).

Table 1. Magnetic polymeric nanocomposites with magnetite/maghemite for oil removal from water

Magnetic polymer nanocomposite	**Technique**	**Oil removal**		**Year**	**Ref**
		%	**$g.g^{-1}$**		
γ-Fe_2O_3/biopolymer	Alkyd cured resin with impregnated magnetic powder	-	7.16 ± 0.18 - 8.33 ± 0.19	2010	[112]
Fe_3O/lignin-CNSL-formol	Polycondensation	-	11.2 ± 0.5	2012	[113]
Fe_3O_4/epoxidized natural rubber	Epoxidation	-	7	2012	[114]
Fe_3O_4/PS	Emulsion polymerization	-	3	2013	[115]
Fe_3O_4/P(S-DVB)	Swelling polymerization	-	8.42	2014	[116]
Fe_3O_4/P(MMA/S/ DVB)	Two-step emulsion polymerization	-	4.26 – 9.41	2014	[117]
Fe_3O_4/PVP	Modified polyol method	93.3 - 100	-	2014	[21]
Fe_3O_4/PS	Microemulsion polymerization	82	-	2014	[118]
Fe_3O/PS/PVDF		-	35-46	2015	[108]
Fe_3O_4/PS	Emulsion polymerization	-	2.492	2015	[109]
Fe_3O_4/ PAni	Emulsion polymerization	99.7	60.3	2016	[119]
Fe_3O_4-nanozeolites /chitosan/PAN	Electrospinning	-	93.4	2017	[107]
Fe_3O_4/PS-palygorskite	Heterogeneous phase polymerization	98	-	2017	[120]
γ-Fe_2O_3/PU	Emulsion polymerization	-	10.0 ± 0.2	2017	[121]
Fe_3O_4/MSp	-	-	3.24 $mg.mg^{-1}$	2018	[122]
γ-Fe_2O_3/alkyd resin/lignin	-	-	10.3 ± 0.8 – 11.7 ± 0.9	2018	[123]
γ-Fe_2O_3/PBS	Fusion	-	9.63 ± 0.46 - 11.06 ± 0.48	2019	[124]
Fe_3O_4/POP-mag	ion-exchange and co-precipitated method	-	5-8	2019	[125]
Fe_3O_4 / bacterial celulose nanofiber aerogel	-		37-87	2020	[126]
Fe_3O_4-SA /PU-PDA	PDA self-polymerization, followed by a covalent deposition of magnetic content and further stearic acid modification	-	28-42	2021	[20]
Fe_3O_4-SiO_2/geopolymer	pore-forming agent in the presence of magnetic content and geopolymer matrix.	-	67 ± 1	2021	[127]

According to several authors, the synthesis of magnetic polymeric nanocomposites has been arousing huge attention due to their distinctive properties, such as high surface area-to-volume ratio, surface modifiability, high chemical, thermal and mechanical stabilities, excellent magnetic properties, easy recovery, recyclability, regeneration, biocompatibility, and considerable amounts of oil removal from aqueous environments [128-131]. The nanocomposite wettability is another feature considered important to determine the hydrophobicity and oleophilicity of the material. However, just a few reports were observed in the literature investigating these promising systems, showing a huge gap in the possible use of these systems for water treatment.

Among the reports, Silveira-Maranhão et al. (2021) reported geopolymers are promising low-cost materials for oil removal from water. The geopolymers are inorganic polymers with repeated units of aluminosilicates, which in the presence of an alkaline solution can form a three-dimensional structure with covalent bonds. The authors also reported that it is possible to confer porosity to the materials by the insertion of H_2O_2, to improve the geopolymer's sorption capability. The presence of ions in the geopolymer matrix attracts the different chemical compounds in oil, allowing the sorption. The authors obtained an oil removal capability of 67 ± 1 $g.g^{-1}$, which is one of the highest values obtained as reported in Table 1. Therefore, it is necessary to encourage the development of new biomaterials for this application.

The crude oil removal measurement used by several authors consists of an analytical gravimetric procedure according to equation 1, where m1 is the mass of the magnetic nanocomposite before the oil sorption, m2 is the oil mass and after the oil sorption procedure the oil residue is weighted and its value is considered as m3.

$$Oil\ Removal = \frac{(m2 - m3)}{m1} \tag{1}$$

Palchoudhury et al. (2014) evaluated the adsorption/absorption capacity of the material through two methods, quantification by UV-vis and subsequent validation by GC-MS. The results obtained through spectroscopy were 93.13% to 100% adsorption, which was validated through the chromatography technique, where it was possible to observe an absorption peak of 100% for the oily content.

Lu et al. (2021) described a new absorption test. The authors used gravity separation or a vacuum pump system. To measure the oil absorption efficiency, the water content in the organic solvent was measured after separation, indicating that the foam had a high absorption capacity and could collect 28 to 42 times more oil than its weight.

Marinho et al. (2018) proposed a new analytical method to evaluate the use of magnetic nanocomposites as good oil spill cleanup tool. The authors proposed the evaluation of the material's oil removal capability (ORC) and intrinsic oil removal capability (IORC). The ORC is an exponential function of the sorption capability ($g.g^{-1}$) depending on the amount of material used according to equation 2:

$$ORC = ORC_0 + Aexp\frac{-[MNC]}{t} \tag{2}$$

where ORC_0 is the function's offset, A is the amplitude, [MNC] is the amount in grams of the magnetic nanocomposites, and *t* is the e-folding time. Through the results obtained with this function, the IORC values of the samples were evaluated by extrapolation of the magnetic nanocomposites amount to zero according to equation 3:

$$\lim_{MNC \to 0} ORC(MNC) = IORC \tag{3}$$

The ORC and IORC measurement was a promising method to evaluate the materials' efficiency in removing oil from water, since it was possible to observe the materials' behavior according to the amount of the nanocomposite used in the experiment and its intrinsic ability to remove oil. Silveira- Maranhão et al. (2021) observed through ORC/IORC measurements that a greatest oil sorption was achieved using smaller contents of geopolymer. From the economic point of view, we believe this analytical approach should be used by other researchers since it can give a wider range of results in function of the amount of materials used and the oil removal from water.

Future trends and perspectives

Magnetic polymeric nanocomposites are indeed promising materials for oil removal and water treatment. However, their use has only been developed on a laboratory scale and some considerations should be evaluated for further research. Below are recommendations for new technologies based on these systems:

- the synthesis of these systems on a laboratory scale is expensive and therefore a better explanation about the nanocomposites' regeneration mechanism using organic solvents should be explored and optimized, since the material can be reused;
- there is a strong need to develop new biomaterials to replace synthetic polymers, such as development of magnetic biopolymer nanocomposites, as reported by Samadi et al. (2017), Ieamviteevanich et al. (2020), and Silveira-Maranhão et al. (2021). Further research should be encouraged, since it is a promising material with higher oil removal capacity compared to synthetic systems;
- it is necessary to develop pilot-scale tests for these materials and evaluate their applicability by simulation, since the environmental conditions, such as wind and rain, can affect the nanocomposite removal from the media,
- another technology should be explored to be used together with the magnetic polymeric nanocomposites to treat the remaining oil.

Conclusions

This review presents the use of magnetic polymeric nanocomposites as an alternative method to traditional techniques used to remediate oil spills, starting from the historical background and including all the methods described in the literature to synthesize and develop these materials. The first section presented the necessary magnetic nanocomposites' chemistry and applicability followed by a brief bibliometric study showing the rising interest in these materials through the years. The second section highlighted the in situ and ex situ routes for synthesis of magnetic polymeric nanocomposites, and the magnetic nanoparticles found in the literature to compose these nanocomposites. The last section focused on the desired properties of the synthesized nanoabsorbents and their oil removal capacity. The magnetic polymeric nanocomposites are considered promising materials with high oil adsorption capacity, easy regeneration for reuse employing inexpensive solvents, leading to a decrease in the secondary pollution produced by conventional synthetic systems, and fast oil removal from the environment through an external magnetic field. However, a lack of studies exists about these promising materials and their technology and procedures. Therefore, it is necessary to intensify studies in this field to understand more about these systems.

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