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Environment

Application of magnetic nanocomposites containing carbon derivatives and biopolymer for aquatic contaminants removal

Aplicação de nanocompósitos magnéticos contendo derivados de carbono e biopolímero para a remoção de contaminantes aquáticos

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ABSTRACT

Anthropogenic activities and industrial development are consequences of the increase in the world population that occurred in the last century. The result is more generation of waste through textile and metal-mechanic industries mainly by carrying out disposal, without proper treatment directly in the environment. This practice is criminal and causes irreversible damages to humans and other animals affected by the contaminants. As a result, increase the research, and development of new materials with "green" characteristics as well as new compounds, which is the case of magnetic nanocomposites containing graphene oxide and chitosan in their structure. The present review approaches these materials and their application in process for adsorption of heavy metals contaminants and dyes. The combination of these nanomaterials presents a potential and promising characteristic in the adsorption processes, demonstrating high efficiency, easy handling, and reusable capacity due their magnetic characteristic and combined properties.

Keywords: Adsorption; Dyes; Graphene oxide; Magnetic nanoparticles; Heavy meta

RESUMO

Atividades antropogênicas e o desenvolvimento industrial são consequências do aumento da população mundial ocorrido no último século. O resultado é a maior geração de resíduos por parte de indústrias têxteis e metalmecânicas, principalmente ao realizar o descarte, ou seja, sem o tratamento adequado, diretamente no meio ambiente. Essa prática, além de criminosa, causa danos irreversíveis aos seres humanos e outros animais atingidos pelos contaminantes. Com isso, aumenta a pesquisa e desenvolvimento de novos materiais com características “verdes” e novos compostos, como os nanocompósitos magnéticos, contendo óxido de grafeno e quitosana em sua estrutura. A presente revisão, contempla esses materiais visando a sua aplicação em processos para adsorção de contaminantes metálicos e corantes. A combinação apresenta características potenciais e promissoras nos processos de adsorção analisados, demonstrando elevada eficiência, fácil manuseio e capacidade de reaproveitamento pela característica magnética e propriedades combinadas.

Palavras-chave: Adsorção; corantes; Íons metálicos; Nanopartículas magnéticas; Óxido de grafeno

1 INTRODUCTION

The last century’s population growth is one of the main factors for the increasing demand of water for use in essential life-maintaining activities to human beings, as well as its use in an industrial scale (Bruckmann et al 2022(a)). Therefore, anthropogenic activities and industrial development result in larger waste generation, which are often improperly discarded into the environment (Jimenez; Dal Bosco; Carvalho, 2004; Bruckmann et al., 2022(a)).

Textile industries use great quantities of water in their dyeing processes, causing an effluent with a high rate of dyes, salts, and other organic components (Rodrigues; Madeira; Boaventura, 2014; Oviedo et al., 2022). Dyes are elements that represent large environmental threats, because of their high toxicity and low biodegradability, as well as mutagenic properties (Zhang et al, 2022).

Another origin of contamination of bodies of water is given by metal mechanic industries, sources of release of heavy metals into the environment (Pourbeyram, 2016). The lead, cadmium, copper, chromium, zinc, and nickel ions awaken special worry given its characteristic such as biopersistence, bioaccumulation, and high toxicity (Bruckmann et al., 2022 (c)).

Thus, the industrial effluents need to receive treatment for the removal of the contaminants, methods. Filtration, precipitation, or adsorption can be used to, at last, give it the proper disposal (Burakov et al., 2018; Rhoden et al., 2021; Salles et al., 2023; Vargas et al., 2023). Among these methods, adsorption has been gaining more spotlights for having easy and safe operation, with good indexes of efficiency and lesser economic and energetic cost (Liu et al., 2018; Bruckmann et al., 2022 (b); Bruckmann et al., 2022 (d)).

According to Geankoplis (2003), adsorption is a unit operation of mass transfer where the species (adsorbate) contained in a fluid are transferred to a solid surface (adsorbent). This operation suffers the strong influence of various factors that directly affect the choice of the adsorbent material, such as temperature, surface area, reactional pH, adsorbent and adsorbate concentrations, specific velocity of the reaction, and intermolecular relations (Nunes et al., 2022).

Thus, the nanoadsorbents are very promising materials for this applicability, since they present extremely high specific surface area, offering small intraparticle diffusion distance (Qu et al., 2013). They can also be functionalized to aim to hit specific contaminants, granting them great selectivity (Qu et al., 2013).

In this context, graphene oxide (GO) is a material in growing exploration for its attractive characteristics, such as high surface, stability, and hydrophilicity (Salles et al., 2022; de Oliveira et al., 2023). Its three-dimensional, porous structure permits it to be functionalized, with iron nanoparticles, for example, while also presenting electrostatic interactions directly linked to its affinity with cationic pollutants and aromatic soluble, facilitating adsorption processes (Yu *et al.*, 2017; Harres et al., 2023; Bruckmann et al., 2022 (e)). In addition, GO nanocomposites are also highly attractive for application in adsorption studies due its low toxicity profile (de Oliveira et al., 2022; Viana et al., 2019; Stefanello et al., 2024).

The biopolymer chitosan (CS) possesses potential to sustain the graphene oxide, as its matrix, potentializing its properties together with its singular characteristics, like biocompatibility, great availability, and low cost (Satapathi, 2017; Barbieri et al., 2024; Bruckmann et al., 2022 (f)). The combination of the two components is originated from the reaction between carboxylic groups in GO with the amine present in CS, originating amide, forming a GO-CS composite (Zhang et al., 2016).

Therefore, the following literature revision has as an objective the data collection about the use of magnetic nanocomposites containing graphene oxide and chitosan in its composition, in adsorption processes of heavy metals e dyes present in aqueous solutions. And, to gather information about the efficiency of the processes, as well as evaluate how and how much these materials are being used in wastewater treatment, to expand the theoretical basis of its properties and applications.

2 METHODOLOGICAL PROCEDURES

The research was done based on the methodology described by Gil (2019), defining, in the first moment, the revision steps: plan elaboration, search for bibliographic sources, exploratory and selective reading of the material. The search was done in April 2020 in the Science Direct and Pubs database, between the years of 2012 to 2020.

The following descriptors were used: "graphene oxide; chitosan; magnetic particles; adsorption". Only the papers published in journals were considered, excluding publications in events, bibliographic revisions, book chapters, theses, and dissertations. 397 references were found in total, in which the exclusion criteria were applied. Many of the papers were deleted for not following the demands of this methodology, like literature revision, or, mainly, for presenting themes not relevant to the study. So, 14 papers were left after the exploratory reading, all from the Science Direct database.

3 RESULTS

The material was cataloged and classified according to its purpose: adsorption of dyes and adsorption of heavy metals. Table 1 shows the list of the selected papers as well as its framework according to the theme.

Table 1 - List of articles used in this study

Fabrication of novel magnetic chitosan grafted with graphene oxide to enhace adsorption properties for methyl blue	Fan et al. (2012)
A novel environmental-friendly nanobiocomposite synthesis by EDTA and chitosan functionalized magnetic graphene oxide for high removal of Rhodamine B: Adsorption mechanism and separation property	Marnani, Shahbazi (2018)
Synthesis of magnetic β-cyclodextrin-chitosan/graphene oxide as nanoadsorbent and its application in dye adsorption and removal.	Fan et al. (2013)
Treatment of wastewater from cationic dye using eco-friendly nanocomposite: characterization, adsorption and kinetic studies	Al-Gorair (2019)
Nano-hybrid based on polypyrrole/chitosan/graphene oxide magnetite decoration for dual function in water remediation and its application to form fashionable colored product	Salahuddin et al. (2020)
Novel magnetic chitosan/quaternary ammonium salt graphene oxide composite applied to dye removal	Neves et al. (2020)
Magnetic chitosan-graphene oxide composite for anti-microbial and dye removal applications	Jiang et al. (2016)
Functionalization of magnetic chitosan with graphene oxide for removal of cationic and anionic dyes from aqueous solution	Gul et al. (2016)
Highly selective adsorption of lead ions by water-dispersible magnetic chitosan/graphene oxide composites	Fan et al. (2013)
Adsorption of Pb (II) from aqueous solution using a magnetic chitosan/ graphene oxide composite and its toxicity studies	Samuel et al. (2018)
Magnetic chitosan-GO nanocomposite: Synthesis, characterization and batch adsorber design for Cr (VI) removal.	Debnath, Maity, Pillay (2014)
Adsorption of arsenic using chitosan magnetic graphene oxide nanocomposite.	Sherlala et al. (2019)
Synergistic and antagonistic effect in simultaneous adsorption of Pb (II) and Cd (II) from aqueous solutions onto chitosan functionalized EDTA-silane/mGO.	Shahbazi, Marnani, Salahshoor (2019)
A comparative study of magnetic chitosan (Chi@Fe_3O_4) and graphene oxide modified magnetic chitosan (Chi@Fe_3O_4GO) nanocomposites for eficiente removal of Cr (VI) from water.	Subedi et al. (2019).

Source: author’s construction

3.1 ADSORPTION OF DYES

A group of researchers, Fan et al. (2012), used a nanocomposite magnetic chitosan graphene oxide (MCGO) composite as an adsorbent for analysis of essential parameters to the adsorption: pH, adsorption time, temperature, and starting concentration of the dye. The maximum adsorption capacity ($q_{max)}$ occurred at pH 5.3 and it was 95.31 mg g^{-1}, and the material can be reused for up to four cycles with qmax of 90%. According to the results, it can be observed that MCGO is a very promising composite, as it has high efficiency, easy separation, and extraction.

The same group of researchers developed a similar study the year before. Fan *et al.* (2013) produced a new magnetic composite, containing β-cycledextrines, chitosan, and graphene oxide for methylene blue (MB) adsorption. The nanomaterial developed showed great removal capacity to MB, revealing q_{max} of 84.32 mg g^{-1}. This experiment did not consider the effect of temperature in the adsorption process, however, analyzing the effect of pH in the range of 2.0 to 11.0, it was concluded that as it increases, the q_{max} also does. Thus, the material produced proved itself effective for methylene blue dye adsorption, being able to be used up to five cycles after its recycling.

In a publication of Jiang et al. (2016), the author describes the use of a magnetic chitosan and graphene oxide composite for the removal of methyl orange dye present in an aqueous solution. The maximum adsorption capacity was 398.08 mg g^{-1}, in pH 4.0, and the study also related the increase of this capacity with the increase of adsorbent concentration. Beyond that, it was observed that the adsorption rate grew rapidly in the first 60 minutes of contact, which can be explained by the elevated number of active sites available for the adsorption, since with the passing of time, the material ends up saturating.

Gul et al. (2016) produced a composite in which chitosan was used as a support for Fe_3O_4 particles, after that being decorated with graphene oxide, and applying it in the removal of cationic and anionic dyes from industrial residuary waters. The targets of research were the extraction of methyl violet (MV) and alizarine yellow (AY), in which the maximum adsorption capacity was found in pH 10.0 (9.68 mg g^{-1}) and 6 (9.55 mg g^{-1}), respectively. The study also shows higher removal efficiency through the developed composite when compared to Fe_3O_4, GO, and CS separately.

The adsorption of Rhodamine B through a material containing EDTA, chitosan, and magnetic graphene oxide was studied by Marnani, Shahbazi (2019). After the evaluation of the parameters, it was concluded that removal of 92% (1085.3 mg g^{-1}) was reached in great conditions of pH = 7.5 and temperature of 33 ºC, from a dye concentration of 114 mg L^{-1}. In a lower pH the adsorbent surface turns positively charged, which can be explained as a decrease in adsorption capacity.

Research of Al-Gorair's (2019), shows the production of a material composed by the retention of GO in an arginine/CS hydrogel for the removal of methylene blue. The considered parameters were the pH, contact time and starting dye concentration. After the tests, the biggest adsorption capacity with temperature increase was obtained, with a pH of 8.0 in 75 minutes.

A material based in magnetite-polypyrrole/CS/GO was used for the removal of the ponceau 4R (P4R) dye, by the group Salahuddin et al. (2020). The biggest adsorption capacity of 85% in pH = 2.0 was obtained, decreasing as the pH increased. It was also observed that the removal efficiency decreases with the increase in the starting concentration due to the saturation of the adsorption sites.

Neves *et al.* (2020) developed a composite containing graphene oxide functionalized with magnetic chitosan and ammonium salt, to extract the dye basic brown 4 (BB4) present in aqueous solution. The developed composite had good results for the adsorption of the dye in question, having the maximum adsorption capacity (650 mg g^{-1}) after 30 minutes of contact, with adsorbent concentration starting from 0.4 g L^{-1}, in temperature of 298 K and pH 6.0. The material proved itself to be promising for the removal of BB4 from aqueous solutions, and after recycling of the adsorbent, it can be reused for three more consecutive cycles with an efficiency of approximately

64%. Table 2 shows the different conditions and results obtained for dye adsorption using magnetic nanocomposites containing graphene oxide and chitosan.

Table 2 - Experimental conditions and maximum adsorption capacity of dye adsorption studies

To be continued...

Research	Dye	Operational conditions*	q_{max}	Reference
Fabrication of novel magnetic chitosan grafted with graphene oxide to enhance adsorption properties for methyl blue	methyl blue	200 mg L^{-1}; 303 K, 60 min, pH 5.3	95.31 mg g^{-1}	Fan et al. (2012)
Synthesis of magnetic β-cyclodextrin–chitosan/graphene oxide as nanoadsorbent and its application in dye adsorption and removal	methylene blue	100 mg L^{-1}, 298 K, pH 11.0	84.32 mg g^{-1}	Fan et al. (2013)
Magnetic chitosan–graphene oxide composite for anti-microbial and dye removal applications	methyl orange	50 mg L^{-1}, 298.15 K, 24 h, pH 4.0	398.08 mg g^{-1}	Jiang et al. (2016)
Functionalization of magnetic chitosan with graphene oxide for removal of cationic and anionic dyes from aqueous solution	Methyl violet	10 mg L^{-1}, at ambient temperature, 80 min, pH 10.0	9.68 mg g^{-1}	Gul et al. (2016)

Table 2 - Experimental conditions and maximum adsorption capacity of dye adsorption studies

				Conclusion
A novel environmental-friendly nanobiocomposite synthesis by EDTA and chitosan functionalized magnetic graphene oxide for high removal of Rhodamine B: Adsorption mechanism and separation property	Rhodamine B	114 mg L^{-1}, 306. 15 K, pH 7.5	1085.3 mg g^{-1}	Marnani, Shahbazi (2019)
Treatment of wastewater from cationic dye using eco-friendly nanocomposite: Characterization, adsorption and kinetic studies	Methylthioninium chloride	40 mg L^{-1}, at ambient temperature, pH 8.0	5.2947 mg g^{-1}	Al-Gorair's (2019)
Nano-hybrid based on polypyrrole/chitosan/ graphene oxide magnetite decoration for dual function in water remediation and its application to form fashionable colored product	Ponceau 4R	2.88 mg L^{-1}, 303 K, pH 2.0	6.799 mg g^{-1}	Salahuddin et al. (2020)
Novel magnetic chitosan/ quaternary ammonium salt graphene oxide composite applied to dye removal	Basic brown 4	100 mg L^{-1}, at room temperature, 150 min, pH 6.0	650 mg g^{-1}	Neves et al. (2020)

* initial concentration, temperature, contact time, respectively

Source: author's construction

3.2 ADSORPTION OF HEAVY METALS

Fan et al. (2013) developed magnetic nanoparticles of graphene oxide and chitosan (MCGO), using them for adsorption processes of Pb (II) found in great volume in aqueous solutions. The process was highly influenced by the pH, considerably increasing from pH 1.0 to 5.0, which can be explained by the easiness of protonation of amine groupings in low pH, inducing electrostatic repulsion of the lead ions. The maximum adsorption capacity (76.94 mg g^{-1}) was at pH 5.0 after 60 minutes in the process.

The study of Samuel et al. (2018) also made use of MCGO to adsorb lead ions and revealed high removal potential. After the experimental tests, around 92% of the Pb (II) ions could be removed in an interval of 420 minutes, in pH 5.0 at 300.15 K. With the results obtained by the author, it was possible to observe the maximum adsorption capacity of the magnetic chitosan (31.6 mg g^{-1}) with the developed composite (112.35 mg g^{-1}), showing it to be more effective and promising.

A composite containing GO and chitosan was magnetically modified using $FeCl_2$ and $FeCl_3$, and Debnath, Maity, and Pillay (2014) did an adsorption experiment of hexavalent chromium. After the analysis of some parameters, it was verified that the ideal pH for the adsorption would be 3.0 (lower values had less adsorption for the loss of solubility of the adsorbent and higher values as well because of the decrease of the ionic exchange property), strongly depending on temperature. The maximum adsorption capacity was reached within 30 minutes of the experiment, removing around 94% of Cr (VI). Also, the adsorbent could be used for 5 cycles with no efficiency loss.

The arsenic adsorption was the target of the study of Sherlala et al. (2019), through the adsorbent containing magnetic chitosan and graphene oxide. The process evaluated the influence of pH, observing that the highest efficiency obtained was from acid pH to neuter, being reduced in alkaline pH. The maximum adsorption capacity obtained here was at pH 7.3 with 45 mg g^{-1}, with 67% efficiency. The effect of temperature was also evaluated through the thermodynamic reactions, where it was proved that the arsenic adsorption was more favorable in colder temperatures.

Shahbazi, Marnani, Salahshoor (2019) synthesized a material in which the chitosan is functionalized with EDTA-silane and magnetic graphene oxide to remove Pb (II) and Cd (II) from aqueous solutions. The resulting composite (CS-EDTA/mGO) proved to be efficient in pH 5.0, because in a lower pH, there is a tendency of protonation of the amine, favoring the decrease of qmax, and in values higher than 5.0 what precipitated from the metals tends to dissolve again. As a result, the qmax of 2.33 mmol g^{-1} for lead ions, and 1.05 mmol g^{-1} for cadmium ions, which can be explained by the smaller ionic radius of Pb (II), benefiting the adsorption.

Research done by Subedi et al. (2019) compares the effect of magnetic chitosan (mCS) in the removal of hexavalent chromium from water, with the efficiency of the composite containing mCS modified with GO in its structure. The first option presented a maximum adsorption capacity of 142.32 mg g^{-1} while the structure modifies 100.51 mg g^{-1}, in pH 2.0, because there is more protonation of the adsorbent surface due to the presence of H+ ions, increasing the electrostatic attraction between adsorbent and adsorbate. However, even with the lesser efficiency of one of the composites analyzed, the authors still define that both end up being good and promising choices for the removal of Cr (VI) present in water. Table 3 shows the different conditions and results obtained for heavy metals adsorption using magnetic nanocomposites containing graphene oxide and chitosan.

Table 3 – Experimental conditions and maximum adsorption capacity of heavy metals adsorption studies

Research	Heavy metal	Operational conditions*	q_{max}	Reference
Highly selective adsorption of lead ions by water-dispersible magnetic chitosan/graphene oxide composites	Pb (II)	303 K, pH 5.0 60 minutes	76.94 mg g^{-1}	Fan et al. (2013)
Adsorption of Pb (II) from aqueous solution using a magnetic chitosan/graphene oxide composite and its toxicity studies	Pb (II)	50 mg L^{-1}, 300.15 K, 420 minutes, pH 5.0	112.35 mg g^{-1}	Samuel et al. (2018)
Magnetic chitosan-GO nanocomposite: Synthesis, characterization and batch adsorber design for Cr (VI) removal	Cr (VI)	50 mg L^{-1}, 298.15 K, 24 h, pH 3.0	5.409 mg g^{-1}	Debnath, Maity e Pillay (2014)
Adsorption of arsenic using chitosan magnetic graphene oxide nanocomposite	As (III)	10 mg L^{-1}, 298.15 K, 240 min, pH 7.3	45 mg g^{-1}	Sherlala et al. (2019)
A comparative study of magnetic chitosan (Chi@Fe_3O_4) and graphene oxide modified magnetic chitosan (Chi@ Fe_3O_4GO) nanocomposites for eficiente removal of Cr (VI)	Cr (VI)	40 mg L^{-1}, at room temperature, 240 min, pH 2.0	100.51 mg g^{-1}	Subedi et al. (2019)

* initial concentration, temperature, contact time, respectively

Source: author's construction

3 FINAL CONSIDERATIONS

In this study, it was possible to observe that the magnetic nanomaterials based in graphene oxide and chitosan show promising characteristics and potential for dyes and heavy metals adsorption. The efficiency of the material suffers high influence from the adsorption parameters such as reactional temperature, contact time, and adsorbent concentration, and, mainly, the pH value. All of these parameters depend as well on the material to be adsorbed.

The graphene oxide functionalization with chitosan and magnetite allows the combination of individual properties of these materials increasing the number of active sites available for adsorption. Thus, this work enhances the importance of the MCGO nanocomposites and their application in wastewater remediation furnishing efficient alternatives in dyes and heavy metal removal through efficient and environmental friendly protocols.

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