Polymeric Composites for Industrial Water Treatment: An Overview

Jordana Bortoluz, Mário César Vebber, Nayrim Brizuela Guerra, Janaina da Silva Crespo, and Marcelo Giovanela

Abstract Several advanced techniques for water treatment depend on materials and chemicals that can pose a secondary pollution risk if not removed, such as nanoparticles, catalysts, and disinfectants. The removal of these compounds requires the use of additional unit operations, making the water treatment process more expensive or even non-scalable. Immobilizing active materials in polymeric composites is an effective way to address these concerns. Such hybrid materials possess a combination of properties that are not normally found in a single constituent, combining the thermal and chemical stabilities of inorganic materials with the processability and flexibility of organic compounds while avoiding dangerous chemicals leech into the treated water. Given that water producers are required to provide high-quality drinking water, polymeric composites have been broadly employed to abate several pollutants. In this context, several nanometric materials have been integrated into polymeric matrices to form state-of-the-art water treatment composites, finding application in microbiological treatment, adsorption and photocatalysis. Due to their chemical flexibility, high surface area, optimal mechanical properties, and cost-effectiveness, such composites have great potential in water purification. The possibilities for tuning polymeric networks are virtually endless, which allows for relatively simple control of functionality (chemical modification, surface modification) and nanomorphology (porosity, structure) of the composites, and fine-tuning of these materials for specific applications and contaminants. In this chapter, we provide an up-to-date review of the importance of polymeric composites in removing several pollutants from water. The main techniques and materials employed in preparing nanocomposites for water treatment, along with their target contaminants, will be addressed, as well as a discussion on their economic feasibility and comparison with well-established techniques.

J. Bortoluz · M. C. Vebber · N. B. Guerra · J. da Silva Crespo · M. Giovanela (✉)
Área do Conhecimento de Ciências Exatas e Engenharias, Universidade de Caxias do Sul, Rua Francisco Getúlio Vargas, 1130, Caxias do Sul, RS 95070-560, Brazil
e-mail: mgiovan1@ucs.br

M. C. Vebber
Department of Chemical and Biological Engineering, University of Ottawa, 161 Louis Pasteur, Ottawa, ON K1N 6N5, Canada

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1 Introduction

The diversification and development of industrial plants and different types of products come to the diversification of pollutants that contaminate the environment, especially through wastewaters (Farré et al. 2008; Pal et al. 2014; Peña-Guzmán et al. 2019). Dyes, heavy metals, pharmaceuticals, pesticides and industrial chemicals in generals are among the micropollutants often detected in the water. Additionally, the inactivation of pathogenic microbes that may be found in the water streams also needs to be carried out for their safe consumption (Geissen et al. 2015; Sauvé and Desrosiers 2014; Dong et al. 2021). While conventional water treatments (filtering, flocculation and chlorination) are suitable to eliminate common, concentrated pollutants; some compounds can go through the process virtually unchanged or as byproducts that are still dangerous, even in trace amounts, and need to be completely removed (Huerta-Fontela et al. 2011; Yang et al. 2017). This has fomented the field of modern treatment technologies, including nanoparticles, adsorption, ultrafiltration, and advanced oxidation processes (AOPs) (Fig. 1). Such treatment techniques can remove pollutants in the parts per billion range and can also be fine-tuned for specific contaminants (Papageorgiou et al. 2014; Hernández-Leal et al. 2011; Restrepo and Villa 2021; Zoschke et al. 2011; Röhricht et al. 2009; Yoon et al. 2013).

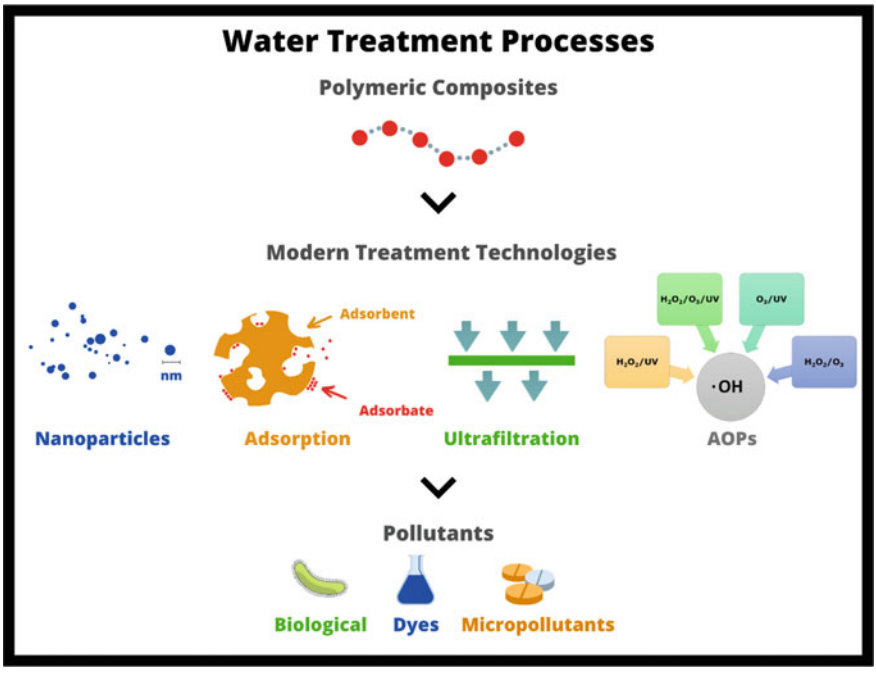

Fig. 1 Water treatment processes using polymeric composites

The main drawback of these techniques is that they often involve introducing materials in the waste stream that may also pose environmental risks. If released into nature, they can affect complex metabolic pathways of microbiomes, which are at the base of any ecosystem, while also increasing oxidative stress, as is the case for peroxides and many catalysts, or even increasing heavy metal concentrations in the case of metallic nanoparticles (Li et al. 2016; Venditti et al. 2013). While additional unit operations can be carried out to remove these materials from water, and this imposes an increase in the cost of water treatment which may not be economically feasible. Moreover, in the case of nanoparticles, complete removal from water is difficult due to their small size.

In light of these facts, the immobilization of active water treatment compounds in polymeric matrices is a promising approach, which prevents or drastically reduces the release of chemical compounds into the treated water while also allowing reutilization of such composites for several cycles (Nunes et al. 2017). A polymeric matrix is especially useful for the versatility of organic compounds, which can be functionalized for specific substrates and have their nanomorphology optimized to increase the efficiency of the final composite. Moreover, polymers have unique chemical resistance and mechanical flexibility, adjusting different types of substrates and water treatment systems (Khan et al. 2021; Yahaya et al. 2021).

In the past 20 years, we have seen a boom in research focused on composites for water treatment as complementary approaches to treatment plants already in place in cities and industries. Most of the efforts in this field involved the optimization of polymer matrixes in achieving high durability, sustainability and increased performance by functionalizing the composites to target specific contaminants. The diversifications of active nanoparticles, either metallic or ceramic, have also gathered large attention from the research community as an approach to increase the efficiency of these materials. More recently, carbon-based composites have arguably been the most extensively explored class of materials, where graphene, activated carbon and carbon nanotubes stand out as both matrix and active components in water-treatment composites (Jaspal and Malviya 2020; Berber 2020). In the following chapter, we will explore a few strategies for producing polymeric composites and their application in different types of water treatment.

2 Polymeric Composites Production Techniques

Techniques employed in the fabrication of polymeric composites should be simple and offer the possibility of tuning the surface properties of the mixture depending on the target pollutant while maintaining the chemical stability of the composite. However, choosing the correct preparation technique is a critical point in pursuing polymer composites with desirable properties (Pandey et al. 2017). Figure 2 illustrates a few of the most common techniques.

Thin films are of great interest for pollutants removal; they have exceptional surface properties and can be supported on a convenient substrate that enables their

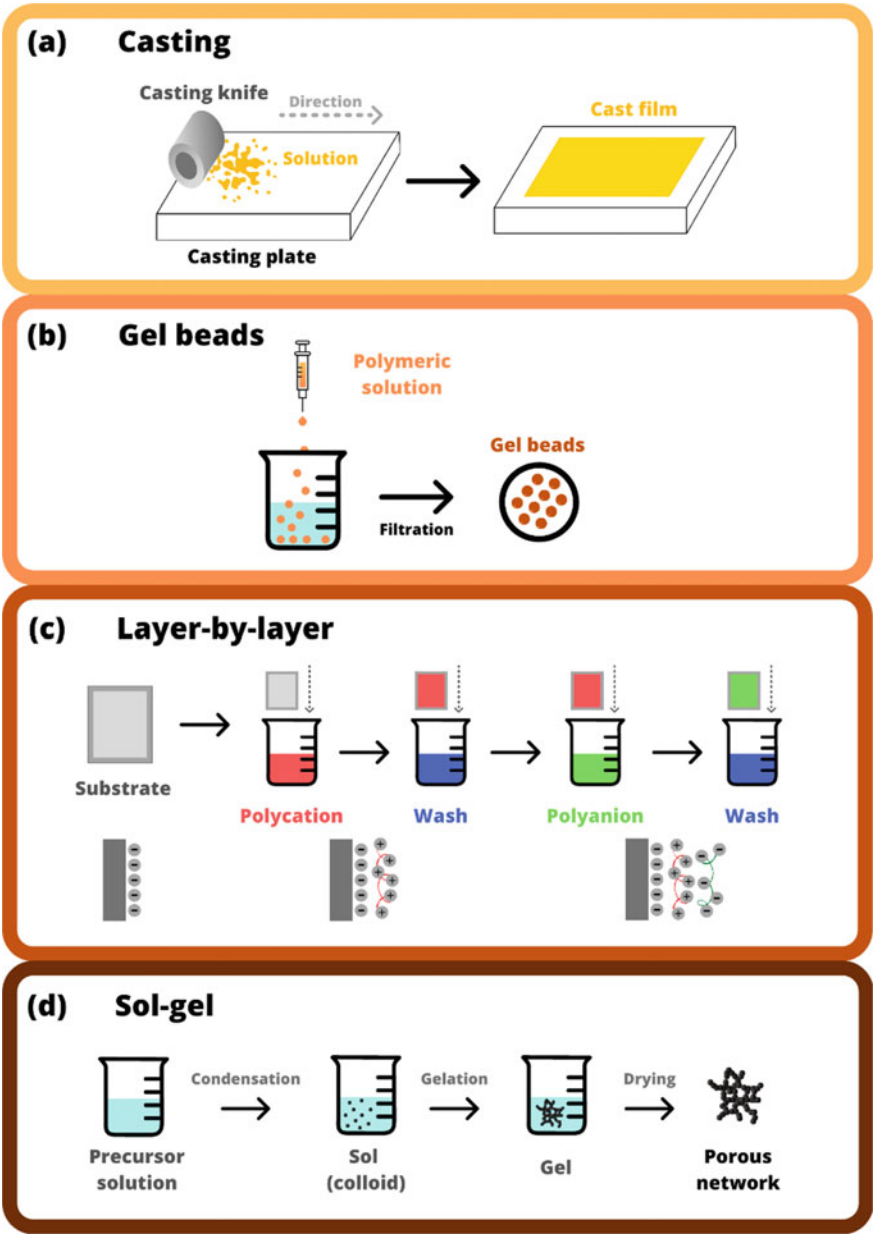

Fig. 2 Illustrative image of the techniques for the preparation of polymeric composites used in the removal of pollutants from wastewater: **a** casting, **b** gel beads, **c** layer-by-layer and **d** sol–gel

reuse. Furthermore, control of the film deposition process enables the formation of organized structures at the nanometer or micrometer level, increasing the efficiency of films (Nunes et al. 2017).

Thin films can be produced using several techniques, such as sublimation, (Liu et al. 2014) castings, (Lefatshe et al. 2017) sol–gel, (Cho et al. 2016) Langmuir–Blodgett (LB) (Dahdal et al. 2016) and layer-by-layer (LbL) (Vebber et al. 2019a). Most of these methods are robust, have experimental simplicity, allow film thickness control, and lead to homogeneous deposition on the substrate (Ferreira 2004). In this section, some techniques for the fabrication of polymer composite films used in water treatment will be discussed in more detail.

2.1 Casting

Casting is arguably the simplest technique employed in producing polymeric films, mainly membranes (Karami et al. 2020). It consists of the mechanical spreading of a forming solution to form polymeric films in a process called phase inversion (Fig. 2a). This technique usually yields thicker films (micrometer range), which rely on porosity to achieve the required properties for water treatment: ultrafiltration, catalysis, or adsorption (Khan et al. 2021; Tan and Rodrigue 2019). Additives and active materials can be incorporated in the forming solution to impart the membrane's active properties, porosity, and increased surface area. Alternatively, the self-assembly of a porous network can be achieved by choice of polymer and film formation conditions (Karami et al. 2020). The phase inversion, which is the phase separation of the components of the forming solution, can be carried out by cooling the solution or adding an anti-solvent to force the precipitation of the polymeric phase. This process is followed by washing the additive or templating phase off the films to yield a porous structure in the film with a high surface area (Tan and Rodrigue 2019). Different porous nanostructures can be achieved by manipulating the forming solution and the phase separation of its components. Traditionally, a mixture of polymers that are known to form domains of the desired pore size is employed (Tan and Rodrigue 2019).

2.2 Gel Beads

The fabrication of polymeric beads is another simple approach for immobilizing and reutilization of active water-treatment materials (Fig. 2b) (Lovatel et al. 2015). These beads are usually of millimetric size and, because of that, do not require any complex process to be recovered from the treated water. This technique is often used to immobilize metallic nanoparticles for microbiological treatment and carriers for clay particles in adsorption (Raota et al. 2019). The relatively large size of the beads tend to reduce the available surface area in contact with water and, with it,

the efficiency of these materials. Similarly to the materials discussed above, porous additives, such as clays can be incorporated to impart porosity to the beads. Nonetheless, the application of hydrophilic polymers with some degree of permeability is necessary for good results (Liu et al. 2012).

Biopolymers are the most often used to fabricate water-treatment beads due to their non-toxicity, biodegradability, permeability and hydrophilicity. In a typical procedure, a concentrated polymer solution is dropped with a syringe into an anti-solvent mixture containing metallic nanoparticles and/or clay particles (Raota et al. 2019). The polymer then immediately precipitates in the format of beads, trapping the active component in the structure. The concentration and pH of the solutions are key parameters that largely affect the final properties of the composites, especially when using the mentioned biopolymers, which contain protic groups that take part in acid–base reactions, changing their solubility, microstructure and porosity (Lovatel et al. 2015; Liu et al. 2012).

2.3 Layer-By-Layer

Broadly, LbL encompasses all techniques consisting of the sequential deposition of nanometric layers onto a substrate (Fig. 2c), whether this is achieved by spin-coating, dip-coating, or vapor deposition, among others (Nunes et al. 2017). In the context of water-treatment composites, the self-assembly of polymeric layers, more specifically polyelectrolytes, stands out as an efficient, high surface area template for catalysis. In this technique, polyelectrolytes of opposite charges are sequentially and alternatively deposited on a chosen substrate to form thin films that can vary from a few nanometers (20–40 nm) until a few micrometers depending on the total number of layers (Decher and Hong 1991). The charged polymers are deposited by adsorption and held together by electrostatic interactions of the second order. The concentration, salinity, and pH control the structure of the resulting film by changing the configuration of the polymer chains, which tend to be linear due to electric repulsion when the chains are mostly ionized and tangled if the ionization rate is low. Tangled chains form thicker, more porous films, suitable for water treatment (Decher 1997). With the same principle at work, other charged species can be incorporated in such films, namely functionalized metallic nanoparticles for microbial treatment and ceramic photocatalysts (Vebber et al. 2019a, b; Faria et al. 2014).

The employment of polyelectrolytes also imparts hydrophilicity and permeability to the films. It has been shown that small species can easily penetrate the film, while large molecules, such as pharmaceutical compounds, do not and tend to interact only on the surface (Eltz et al. 2020). This characteristic also allows for nanoparticles, catalysts, and other active components to be incorporated into the polymeric film in a posterior step, simplifying the manufacture of these films (Liu et al. 2016). Moreover, many environment-friendly polymers are compatible with this technique, further reducing their possible detrimental impact in the treated wastewater (Decher and Hong 1991; Decher 1997).

2.4 Sol–gel

The sol–gel method is a traditional approach to producing inorganic nanostructures of different sizes and properties and is often used to fabricate nanoporous inorganic films (Fig. 2d) (Momina 2021). Crystalline chains of oxides are grown from metal precursors, often an organic compound, utilizing acid or basic hydrolysis. While these sol–gel films generally present high surface activity, they lack important properties for their use as a recoverable material in water treatment, mechanical flexibility and adherence being the main ones (Clarke et al. 2013; Bahuguna et al. 2016). Polymeric composites address these issues, and sol–gel nanoparticles are frequently incorporated in thin films by the techniques mentioned above, although often with some reduction inefficiency (Vebber et al. 2019a, b).

To combine the efficiency of free nanoparticles with the mechanical properties of polymeric thin films, sol–gel and polymerization can be carried out simultaneously to yield a composite with a tunable nanostructure (Liaw and Chen 2007). In this approach, monomers/polymers and nanoparticle precursors react simultaneously. This technique is complex and requires the conditions in terms of solvent, pH, temperature and catalyst to be suitable for two different reactions simultaneously. Nonetheless, it allows for fine control of the nanostructure, such as reducing pore size, the increased mechanical stability of the microstructure, and good dispersion of nanoparticles in the matrix, maximizing the active surface area (Liu et al. 2021).

3 Natural and Synthetic Polymeric Composites for Water Treatment

As mentioned previously, polymer matrices are currently receiving much attention from many researchers due to their ease of surface modification, biodegradability, easy availability and low cost (Pathania and Kumari 2020; Rajeswari et al. 2021). The combination of reinforcing fillers into a polymer matrix can solve the drawbacks of polymers and nanomaterials when used separately. Due to their biphasic nature, these materials are known as polymeric composites and, depending on the origin of the polymer, they can be classified (Khodakarami and Bagheri 2021).

Natural polymers or biopolymers are so-called macromolecules because they are derived from natural sources. Some examples of them are cellulose, lignin, chitin and chitosan, pectin, alginate, gum, gelatin and starch (Fig. 3). Conversely, synthetic polymers are defined as polymers that are artificially produced in laboratories. In wastewater treatment, some of the most commonly used synthetic polymers are polyamide, polysulfone, polyethersulfone, polyvinylidene fluoride, polypropylene and polyacrylonitrile (Ahmed et al. 2014; Siti Aisyah et al. 2014) (Fig. 4).

It is also important to highlight that these materials are diverse in size and chemical structure and that these characteristics make them versatile compounds for stabilization, immobilization and the reduction of nanoparticles (Nasrollahzadeh et al. 2021).

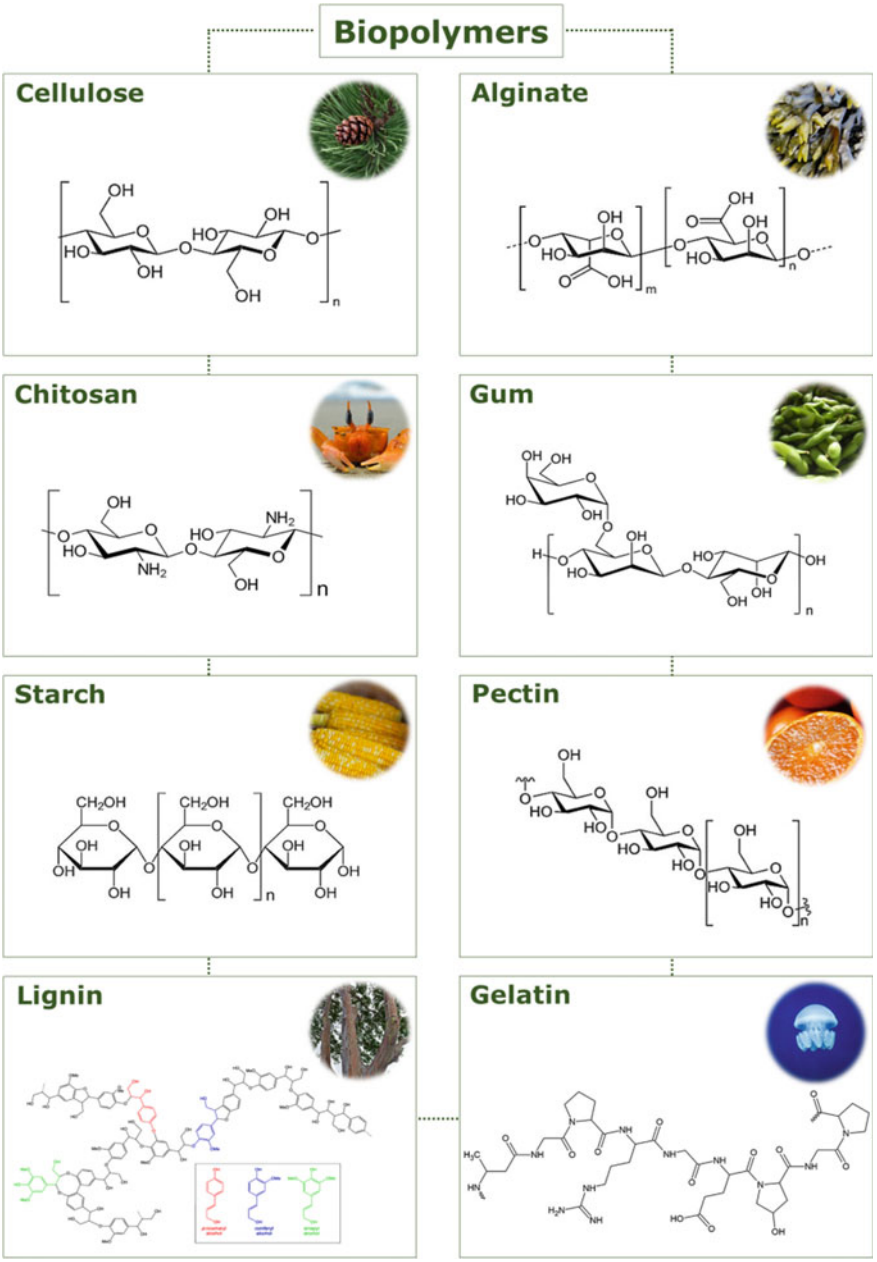

Fig. 3 Examples of natural polymers used in the pollutant's removal

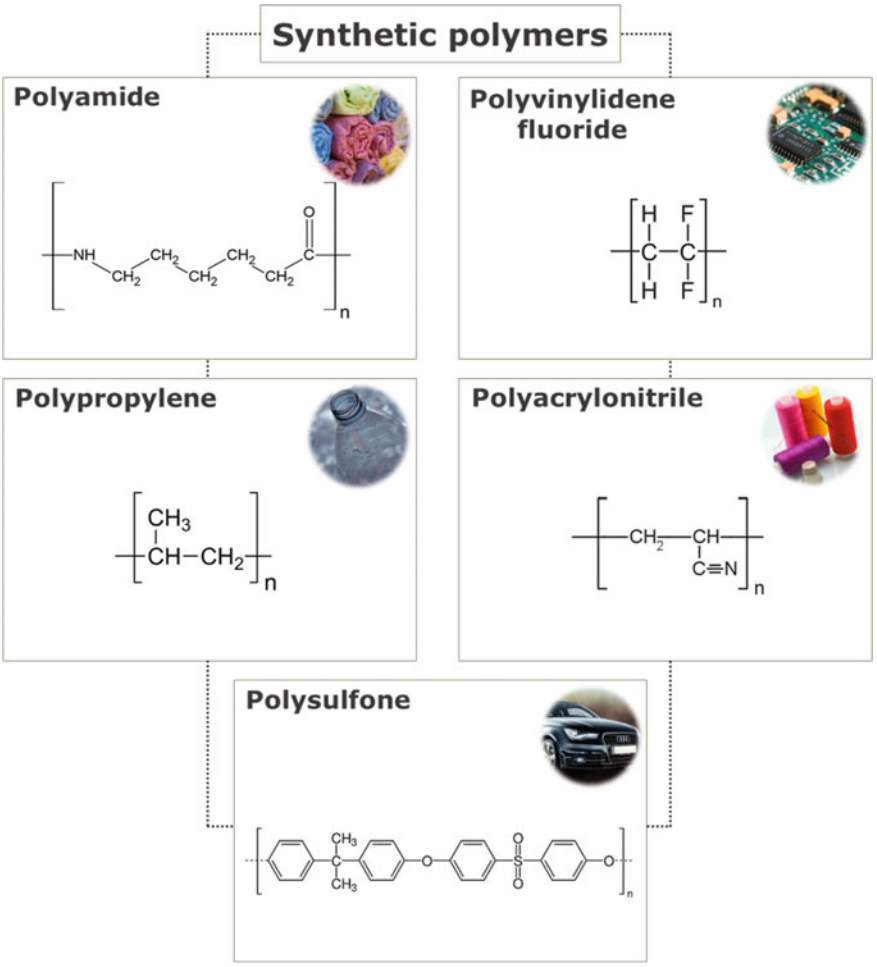

Fig. 4 Examples of synthetic polymers used in the pollutant's removal

In this sense, the investment and advances in the development of polymer composites can bring promising solutions for water treatment (Motshekga et al. 2018).

Different characteristics influence the choice of polymer composite to treat a specific pollutant, among them is the type and concentration of the contaminant present in the wastewater, pollutant removal capacity and the efficiency/cost ratio. It is also important to consider the composite's ability to regenerate and be able to be used several times without modifying or losing the material's physical–chemical properties (Unuabonah and Taubert 2014). In the next sections, we will describe three main categories of pollutants that can be removed from wastewater using natural and synthetic polymeric composites.

3.1 Biological Pollutants

Water purification is motivated by one main reason: all living beings need uncontaminated water to survive. Pure water refers to the nonexistence of heavy impurities and bacteria and microorganisms disease-causing (Chopparapu et al. 2020). It is well known that in the water, there are several microorganisms responsible for many diseases, for example, dysentery and diarrhea are caused by amoeba and *Shigella dysenteriae*, typhoid fever occurs by microorganisms such as *Salmonella paratyphi* and *Salmonella typhi*. In water, there is also *Escherichia coli*, *Giardia lamblia*, *Campylobacter* and *Vibrio cholerae*. Chlorination of water has been used for many years to eliminate biological pollutants; however, due to the formation of harmful waste (*i.e.,* chlorophenols), this technique is rapidly losing ground (Nikolaou and Lekkas 2001). Currently, filtration is usually the most common process of getting pure water as it removes all bacteria, microorganisms and little particles based on size. Chitosan is a natural polysaccharide biopolymer that is mainly used for filtration purposes (Sharma and Kumar 2019). In this sense, attempts are made to purify water through advanced technologies that employ composite polymeric materials.

For example, Charpentier et al. (2012), prepared nanometric films of polyurethane and titanium dioxide (TiO_2). The removal of biological pollutants such as *Escherichia coli* was evaluated using solar irradiation (photocatalysis). According to these authors, in almost 1 h of treatment, they eliminated more than 99.5% of the microorganisms, or which is related to the photocatalytic effect of the TiO_2 in the polyurethane matrix. In another study, Undabeytia et al. (2014) developed Na-bentonite composites with three commercial polymers for *Escherichia coli* removal. The antimicrobial effect was influenced by the charges of the monomers adsorbed on the clay, providing a high cationic density on the surface. Using sodium hypochlorite and hydrochloric acid, the clay-polymer system demonstrated total regeneration of filters loaded with microorganisms.

Zarpelon et al. (2016) fabricated thin films obtained from poly(allylamine hydrochloride) (PAH) and poly(acrylic acid) (PAA) polyelectrolytes with silver nanoparticles (AgNPs) and crosslinked with glutaraldehyde to be used as a biological agent in wastewater treatment. The total *Escherichia coli* count was reduced by 93% after 6 h. Four years later, the same authors (Eltz et al. 2020) evaluated the elimination of *Escherichia coli* from other industrial wastewater using thin films of PAH/PAA/AgNPs and copper nanoparticles (CuNPs). All films studied showed bactericidal activity against this microorganism. However, polymeric thin films with only AgNPs showed better biological results than films containing CuNPs. After 2 h of treatment, it was found that minimal amounts of copper and silver were released (0.027 and 0.0083 mg L^{-1}, respectively). Thin films with smaller nanoparticles and lower concentrations promoted a more effective interaction with microbial membranes, favoring the destruction of bacteria.

Some biopolymer nanocomposites have also been investigated for use in the removal of organic pollutants. Cellulose-based polymers have often been used for

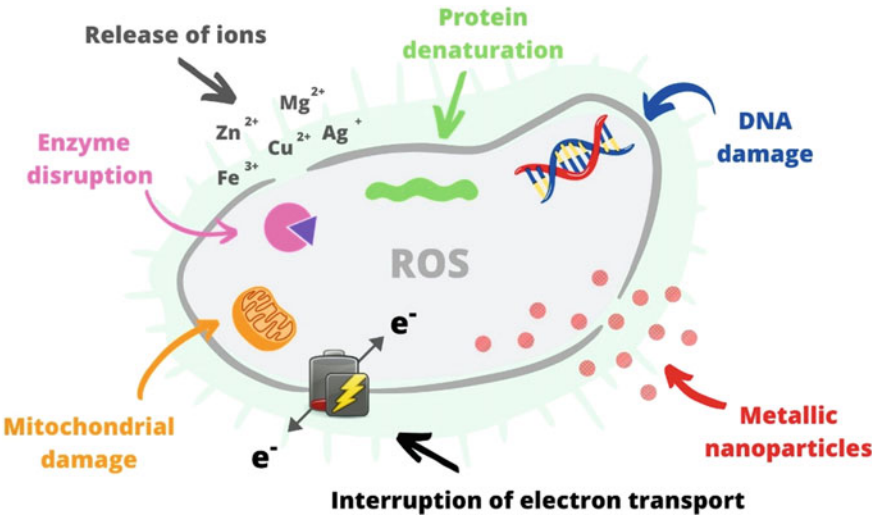

Fig. 5 Illustrative image of the antimicrobial effects of metallic nanoparticles

this purpose since they are non-toxic, biodegradable, and highly efficient in water treatment. Yoosefi Booshehri et al. (2015), for example, performed the deposition of CuNPs onto a cellulose matrix to evaluate its antibacterial activity using *Escherichia coli* and *Staphylococcus aureus*. The tests showed that the disinfection occurred after a contact time between 150 and 180 min. They explained that this result is mainly due to the CuNPs present in the cellulose matrix. The mechanisms of leaching and penetration cells were responsible for interrupting the cellular function of these microorganisms (Fig. 5). In other words, the mechanism of antibacterial activity of Cu(II) ions occurred predominantly through the formation of the copper-peptide complex that catalyzes reactive oxygen species (ROS) formation and drastically increases its production, which results in cell death. The authors also found that the composite can be reused, maintaining the same antibacterial capacity at least three times after the first use.

Lefatshe et al. (2017) used another nanoparticle with the same biopolymer-matrix. They prepared zinc oxide (ZnO)/cellulose nanocomposite with photocatalytic and antibacterial activities against *Staphylococcus aureus* and *Escherichia coli*. This study found that when the ZnO nanoparticles (ZnONPs) were introduced in the biopolymer-matrix, a good activity against both bacteria was verified, as evidenced by a large zone of inhibition (ZOI) around the material. The authors explain that the ZnONPs produces ROS, such as superoxides (O^{2-}), hydroxyl radicals (•OH) and hydrogen peroxide (H_2O_2) when coming into contact with bacteria cells. The ROS generated may promote the peroxidation of the polyunsaturated phospholipid component of the lipids in the microorganism.

Alginate-based nanocomposites have also been shown to be an effective material for water disinfection. For example, Motshekga et al. (2018) fabricated alginate beads

encapsulated with ZnONPs for bacteria disinfection from water. The antimicrobial tests were performed using surface water and synthetic water with *Staphylococcus aureus*. In the surface water samples, it was found that all bacterial activity was inactivated in less than one minute. On the other hand, when synthetic water was used, the nanocomposites showed good results after 120 min, when a low concentration of bacteria was used. Thus, these authors concluded that a small amount of zinc was leached into the aqueous medium. This was a satisfactory result since the environmental limits allowed for water treatment were not exceeded.

Baek et al. (2019) also evaluated a nanocomposite that encapsulated ZnONPs in an alginate biopolymer (ZnONPs-alginate beads). The antibacterial effects were verified with two antibiotic-resistant bacteria (*Escherichia coli* DH5-α and *Pseudomonas aeruginosa*). This study showed that the ZnONPs-alginate beads are promising for disinfection water, once presented 98 and 88% removal, respectively. The generation of ROS is the responsible mechanism of disinfection activity. Finally, the reusability tests showed that this material could be reused, even when it is used for a resistant contaminant such as *Pseudomonas aeruginosa.* Lovatel et al. (2015), in another study, prepared a nanocomposite with AgNPs onto montmorillonite and fixed it in a polymer matrix of sodium alginate. The bactericidal potential was tested using the agar diffusion method with two microorganisms (*Escherichia coli* and *Staphylococcus aureus*). From these tests, the authors were able to verify that there was a significant reduction (~98%) in the total number of coliforms on the disinfection of industrial wastewater. The authors also emphasized that this nanocomposite does not present environmental risks, as the concentration of AgNPs leached into the aqueous medium is almost insignificant (~3.0 $\mu g\ L^{-1}$). In addition, they considered that due to the stability of the material during the 90 min of evaluated treatment, there is a possibility of reusing this material.

Chitosan, a natural polymer that comes from crustaceans, has also been widely used to immobilize metallic nanoparticles for the microbiological treatment of water. Chatterjee et al. (2017) prepared and used chitosan beads with silver and ZnO (Ag-ZnO) to disinfect secondary treated sewage. The antimicrobial action was evaluated with and without sunlight incident on the samples to verify the influence of this external factor on water disinfection. The study showed that the nanocomposite presents good results for the inactivation of coliforms in treated sewage water, mainly under visible light irradiation. The authors explained that bacterial inactivation happens due to the associated effects of H_3O^+, •OH, O_2• and silver species. Finally, the authors also found that the chitosan/Ag-ZnO beads can be reused for at least five operation cycles.

In the same way, Raota et al. (2019) synthesized AgNPs through a green route with an extract of Ives cultivar (*Vitis labrusca*) pomace for subsequent preparation of a chitosan-based nanocomposite. The bactericidal activity of this material was evaluated using two Gram-negative (*Escherichia coli* and *Pseudomonas aeruginosa*) and two Gram-positive (*Staphylococcus aureus* and *Enterococcus faecalis*) bacteria. In this study, the authors verified that three compounds could be responsible for the bactericidal activity of the studied composite: AgNPs, chitosan and phenolic compounds from the pomace grape. AgNPs contribute by penetrating

the cell wall of microorganisms. Chitosan, in turn, acts as a binding agent to the surface of the bacteria. Phenolic compounds also contribute to damage to the cell membranes of bacteria. The authors verified that after 1 *h* of wastewater treatment, the nanocomposite promoted a 47% reduction in the initial counting of *Escherichia coli.*

3.2 Dyes

Nowadays, the existence of dyes in wastewater is a worrying factor for the population, as this contamination causes major problems to human health. If the dyes remain for long periods in running water, this can cause a decrease in dissolved oxygen, which slows down the expansion of aquatic biota (Gao et al. 2019). In this sense, the scientific community has dedicated a large effort to producing materials that are used to eliminate these pollutants from wastewater. Several methods have been used, such as precipitation, solvent extraction, neutralization, reverse osmosis, ion exchange, and adsorption. Among these methods, the cheapest, most efficient and easiest to implement a process for removing pollutants is adsorption, or which works even to eliminate pollutants at relatively low levels (Chung et al. 2014).

Cho et al. (2016) using a sol–gel method, synthesized inorganic–organic particles using an amphiphilic polymer functionalized with alkoxysilane bonded to silica particles (M-APAS-SiO_2) as a precursor. The adsorption essay was used to verify the possibility of M-APAS-SiO_2 particles to adsorb water-soluble dye compounds and water-insoluble dye (organge-16 and solvent blue-35; respectively). The results showed that the tested particles can work as an adsorbent in removing hydrophobic and hydrophilic pollutants. The authors investigated that the hybrid particles studied can be reused by simple pH adjustments.

Magnetite ionic polymer nanocomposites were developed by Atta et al. (2016) at room temperature and tested as methylene blue (MB) adsorbents. For this, the copolymerization of acrylonitrile (AN) and 2-acrylamido-2-methylpropane sulfonic acid (AMPS) monomers was performed and subsequently swollen in ferric chloride ($FeCl_3$) and potassium iodide (KI) solutions to produce magnetite nanocomposites (Fe(AN-*co*-AMPS)). It is known that the electrostatic attraction, chemical interactions, and ionic characteristics of dyes and ionic polymeric nanocomposites influence the adsorption process. In this study, the authors concluded that the presence of negative charges on the surface of Fe(AN-*co*-AMPS) was responsible for the fast diffusion of a large amount of MB dye. MB recoveries were 93.2 and 88.4% after two adsorption–desorption cycles, without loss of adsorption capacity.

El-Shamy (2020) developed inexpensive nanocomposites of polyvinyl alcohol/zinc peroxide (PVA/ZnO_2) and polyvinyl alcohol/carbon dots decorated with zinc peroxide (PVA/CZnO_2) using the casting technique. For the same filler concentration, the dye adsorption was 1972 ± 40 and 1831 ± 20 mg g^{-1} in PVA/CZnO_2 and PVA/ZnO_2, respectively, showing the positive influence of carbon points in the adsorption process. After 1 h of study, a 98% removal of MB was obtained.

According to the recycling experiments, the PVA/CZnO$_2$ nanocomposites were used for five cycles maintaining high efficiency.

Malachite green (MG) is a water-soluble dye used in various industries (textile, wool, cotton, paper, leather, silk and jute). It can also be used as a fungicide and ectoparasiticide in aquaculture and fisheries (Rajabi et al. 2017). In this context, Rajabi et al. (2019) prepared poly(methylmethacrylate)/graphene oxide-Fe_3O_4 (PMMA/GOFe_3O_4) and poly(methylmethacrylate)/graphene oxide (PMMA/GO) nanocomposites to be used as adsorbents for the removal of MG dye. The highest adsorption rate was obtained for nanocomposites containing Fe_3O_4 nanoparticles. This fact was explained by the high interaction between nanoparticles and MG. The adsorption capacity was maintained for up to 35 min under experimental conditions. Different polymer composites have been used as adsorbents for MG removal, such as tetraethylenepentamine-functionalized *Rosa canina*-L fruits activated carbon, (Ghasemi et al. 2016) multi-walled carbon nanotubes (MWCNs), (Rajabi et al. 2016) melamine/maleic anhydride composites, (Rong et al. 2014) and amylopectin-poly(acrylic acid) copolymer, (Sarkar et al. 2014) among others.

Hir et al. (2017) evaluated the photodegradation of methyl orange (MO) dye using polyethersulfone films prepared with different TiO_2 levels. The greatest removal of MO was achieved under acidic conditions since the pH of the zero-point charge (pH_{ZPC}) of TiO_2 is 6.8, therefore, its surface becomes positively charged when the pH is below 6.8. The best photocatalytic activity was exhibited in acidic environments since MO is an anionic dye and easily adsorbs on TiO_2 positive surfaces. The MO photodegradation followed pseudo-first-order kinetics. The authors found that the 13% TiO_2 film can be reused for five cycles without losing efficiency.

Clays are natural materials with intercalated cationic and anionic layers and are widely used as adsorbents for various micropollutants. Montmorillonite was used as filler of a hydrogel polymer to study the removal of three dyes: methyl red (MR), crystal violet (CV) and MB from wastewater (Nakhjiri et al. 2018). Two pH were evaluated, and in the neutral environment, the removal of MR, CV and MB was 51, 80, and 89%, respectively. However, for pH ~12.0, the adsorption values obtained were 23, 86 and 93%, reporting a maximum capacity of 113, 176, and 155 mg g^{-1} for MR, CV, and MB, respectively.

Biopolymers have also been applied as a matrix material to prepare nanocomposites to remove dyes from contaminated water. In this sense, Saber-Samandari et al. (2016) reported the direct red 80 (DR) and MB dyes adsorption process using magnetic gelatin nanocomposite beads comprising carboxylic acid-functionalized MWCNTs. The results showed that the nanocomposite can remove about 96% of the DR dye and 76% of the MB dye, respectively. The charges between the adsorbent and the adsorbate are opposite, resulting in an electrostatic interaction between them. The same did not happen for the cationic dye (MB). In this case, the MB dye ended up being partially repelled due to both material and pollutant having a positive charge. On the other hand, the carboxyl groups present in the CNTs interacted electrostatically with the cationic dye. This made the percentage of removal of this dye still quite significant.

A nanocomposite of gelatin hydrogel with copper oxide nanoparticles (GL-CuO) was developed by Ahmad et al. (2020) for the catalytic reduction of the MO and Congo red (CR) dyes. This study verified that the introduction of this nanocomposite accelerated the reaction of sodium borohydride ($NaBH_4$) reducing agent with the tested dyes (5 min for MO dye and 7 min for CR dye, respectively). The recyclability and activity of the GL-CuO were monitored for three successive MO reduction reactions. A chitosan–gelatin hydrogel with Zr(IV) selenophosphate nanoparticles (CH-GEL/ZSPNC) was prepared by Kaur and Jindal (2019) for the adsorption and photodegradation of MB dye. The Zr(IV) selenophosphate nanoparticles (ZSPNPs) were incorporated to make this material selective for cationic dyes. The tests showed that the higher percentage removal (~99% of MB dye) and photodegradation was observed under sunlight. The maximum adsorption capacity was equal to 10.46 mg g^{-1}, and the degradation efficiency was satisfactory during the first four cycles (82% dye removal). Even so, CH-GEL/ZSPNC can be reused.

The CuO/chitosan nanocomposite thin film was investigated by Kumar et al. (2015) in the photocatalytic degradation of rhodamine B (RhB) dye. This study revealed that this nanocomposite could degrade about 99% of the dye. The authors explained that this occurs due to the slow electron–hole pair recombination rate of nanosized CuO in the chitosan matrix, the large surface area of the nanoparticles, and the high absorption capacity associated with the biopolymer. The reusability tests showed that the decolorized percentages of RhB dye solution during five cycles of use were 98.8, 78.3, 72.8, 70.6, and 69.2%, respectively.

The utilization of di-aldehyde alginate crosslinking gelatin hydrogel decorated with AgNPs was investigated in removing MB dye from water by Abou-zeid et al. (2019). The authors performed a comparative study using a composite without AgNPs. This experiment showed that the best result was achieved when AgNPs were present on the composite surface, as they provide additional sites for electrostatic interaction with the cationic molecules of the MB dye. The maximum adsorption capacity found was equal to 625 mg g^{-1}. In the same way, a sodium alginate hydrogel with silver nanoparticles was tested by Karthiga et al. (2016) as a biosorbent for the removal of MB dye from water. The adsorption tests showed that the nanocomposite exhibited excellent adsorption property toward removing MB dye from an aqueous solution. The adsorption kinetic data follows the pseudo-first-order model, and the Elovich model confirmed the chemisorption mechanism.

Mohammed et al. (2015) produced cellulose nanocrystal-alginate hydrogel (CNC-ALG) beads to be applied to remove MB dye from water. This study showed that the adsorption process followed the Langmuir isotherm, and the maximum adsorption capacity of CNC-ALG was equal to 256.4 mg g^{-1} for MB dye. The kinetics exhibited a better correlation with the pseudo-second-order model, and the nanocomposite showed that it was reusable as it presented more than 97% dye removal during five adsorption–desorption tests.

Masilompane et al. (2018) studied the removal of brilliant black (BB) dye using a nanocomposite of lignin-chitosan-based with titania nanoparticles (TiO_2NPs). The functional oxygen groups present in all nanocomposite components, the amine groups in chitosan, and the π-electron system in the benzene structure of the lignin

matrix make this material a potential adsorbent to be applied in the dye removal processes. The value found for k_f (8.98×10^{-5} mg g^{-1}) indicated that the adsorbent had a low adsorption capacity, and the magnitude of n (5.5) suggested that the adsorption process was easily achieved. The kinetics was better correlated with the pseudo-second-order model.

The gum tragacanth, the polysaccharide extracted from plants of the genus *Astragalus*, was used by Sharma et al. (2021) The authors prepared a gum tragacanth-based hydrogel nanocomposite modified with TiO_2NPs for the adsorption of MG dye from an aqueous solution. The TiO_2NPs were incorporated in this material to improve the adsorption character of the matrix. In fact, an enhancement in dye removal was observed with the increase in the amount of TiO_2NPs in the material (from 88.57 to 103.09 mg g^{-1}). The adsorption study also showed that the material presented about 99% of the dye removal under optimized conditions. The study also showed that the nanocomposite can be reused up to three times without losing significant efficiency.

Vanaamudan et al. (2018) developed a chitosan/guar gum blend with green AgNPs (from palm shell extract) to evaluate the catalytic activity degradation of an individual and binary mixture of dyes (reactive blue-21 (RB), reactive red-141 (RR), and rhodamine-6G (Rh)). The results revealed that this nanocomposite is efficient on heterogeneous catalysis. The complete degradation of the individual dyes occurred between 2 and 15 min. In the case of a mixture of dyes, a degradation of 95% to RB-Rh, 100% to RR-Rh and 90% to RB-RR mixtures was observed. The reusability test showed that after three cycles of the catalytic process, the efficiency decreased by only 2–3%, proving that this material can be reused.

Starch, a natural carbohydrate biopolymer that has low cost, biodegradability and versatility, was used as a matrix material by Gomes et al. (2015), who prepared starch/cellulose nanowhiskers hydrogel composite to remove MB dye from water. Incorporating 5 wt.% of nanoparticles in the hydrogel matrix increased the adsorption capacity from 1872.90 to 1918.81 mg g^{-1}. However, the authors highlight that the adsorption capacity decreased with the additional increase in the amount of cellulose nanowhiskers (more than 5 wt.%). It was related that in optimized experimental conditions, the maximum adsorption capacity of the nanocomposite was 2050 mg g^{-1} and about 90% removal for all tested concentrations of MB dye.

3.3 Micropollutants Removal

The production and consumption of pharmaceutical products have increased considerably in recent years. This fact has become a concern for water resources because the pharmaceutical industry produces many toxic residues for biological life. These contaminants could have an antagonistic impact on the marine ecosystem and human health through endocrine disturbance and the development of antibiotic resistant bacteria (superbugs) (Yang et al. 2017; Patel et al. 2019). The drugs most commonly found in industrial wastewater are antibiotics, anti-depressants, anti-inflammatories, lipid regulators, tranquillizers, (Gros et al. 2010; Yuan et al. 2014), and hormones

including natural estrogens and 17 β-estradiol, as well as the contraceptive 17 α-ethinylestradiol (Amouzgar et al. 2016).

In this sense, Tang et al. (2019), using a simple method, manufactured a multifunctional adsorbent composed of Zr(IV)/carboxymethyl-β-cyclodextrin (Zr/CM-β-CD) to remove female hormones from the liquid medium. The oligosaccharide CD cavities allowed encapsulating the hormone micropollutants through physical interactions between host–guest. The maximum adsorption capacity was 210.53 mg g^{-1}, achieving the best fitting with the Langmuir isothermal model. The Zr/CM-β-CD composite continued to be highly efficient after five cycles.

Bisphenol A (BPA) can cause several endocrine disorders, including female and male infertility, risk of breast and prostate cancer, etc. Here, cyclodextrin again appears as an option to degrade BPA in the aqueous medium. Zhang et al. (2012) evaluated the photocatalytic capacity of TiO_2 with cyclodextrin (TiO_2/β-CD). TiO_2/β-CD is an efficient catalyst of BPA. However, the evident degradation of BPA was mainly caused by O_2•, which was generated through the charge transfer complex between cyclodextrin and TiO_2.

Two adsorbent materials, the polyaniline and MWCNT, were mixed until obtaining the composite PANI/MWCNTs, to be used to eliminate meloxicam from the liquid medium (Dutra et al. 2018). The adsorption capacity of the mixture was 221.2 mg g^{-1}, and the adsorption process presented a better fit with the theoretical pseudo-second order and Elovich models. The kinetic study indicated that adsorption was favored by the surface chemistry and the porous structure of the energetically heterogeneous composite.

The biopolymer nanocomposites have shown that they can also remove micropollutants from water. Photocatalysis was used by Sarkar et al. (2015) who immobilized TiO_2NPs with calcium alginate beads (TIAB) to degrade chlorhexidine digluconate (CHD), ibuprofen (IBP), atenolol (ATL) and carbamazepine (CBZ). The performance of the nanocomposite beads was compared with the efficiency of free TiO_2. This study showed that about 55% removal of CHD was possible using TIAB in a packed bed photoreactor (PBPR), while almost 70% removal was achieved using free TiO_2. The percentage of steady-state removal was reached 58, 85, and 80% for ATL, IBP and CBZ, respectively. These three results were lower when are compared with the free TiO_2. However, the authors emphasized that although the TIAB has lower performance, many medications can be removed with this system. The TIAB was reused without becoming ineffectiveness for five cycles in PBPR.

Using the LbL technique, Vebber et al. (2019a) prepared a nanostructured thin film of PAA, PAH and TiO_2, intending to degrade IBP in aqueous media by photocatalysis using the film and solar energy. The film achieved to degrade, after 150 min, up to 95% of IBF, as well as 50% of its aromatic centers using neutral pH. In another study carried out by these same authors, (Vebber et al. 2019b) the self-assembled thin films of PAA, PAH, TiO_2, and copper were evaluated in the photodegradation of IBP. All films showed high stability in water, releasing only 0.4 wt.% TiO_2 and the photocatalytic efficiency was demonstrated during three cycles of 150 min each. The films with copper showed greater photocatalytic degradation when compared to those without copper, indicating an IBF degradation rate of 76%. Through a

composite experimental design, Kerwald et al. (2020) evaluated the influence of different factors on the photocatalytic activity of PAA/PAH/TiO_2 thin films on the drug bezafibrate (BZF). The results exposed that the film degraded more than 80% of BZF, and the incorporation of AgNPs provided a 64% increase in the photocatalytic efficiency compared to films without AgNPs. Film reuse has been demonstrated for up to four cycles without loss of efficiency.

Attallah and Rabee (2020) investigated a nanocomposite with pectin, chitosan and zinc oxide (Pec/CS/ZnO) to remove CBZ in aqueous solutions under direct sunlight. The results showed that under optimum conditions, the degradation efficiency of CBZ was found to be 69.5%. Two main phenomena were observed: firstly, there was the adsorption of CBZ on Pec/CS/ZnO nanocomposite, and then the photodegradation of this molecule happened. When direct sunlight illuminated ZnO in Pec/CS/ZnO nanocomposite, photons were absorbed, and electron–hole (e^-–h^+) pairs and free radicals were created. Thus, the solution's adsorbed and free CBZ molecules were degraded due to those free radicals that disrupted their conjugation. The authors verified that the Pec/CS/ZnO nanocomposite did not lose significant efficiency in the degradation of the drug after three consecutive uses.

An ecofriendly and sustainable nanocomposite was fabricated by Mohamed and Mahmound (2020) with biochar from *Pisum sativum* pods and starch hydrogel (N-PSPB/SHGL) for naproxen (NAP) removal. The results showed that this material presented an excellent adsorption capacity value (309.82 mg g^{-1}) and was confirmed to be an advantageous nano sorbent for pharmaceutical removal from aqueous solutions. The N-PSPB/SHGL showed high removal percentage value of NAP (90.07%) even after five cycles.

Hu et al. (2019) developed a bilayer amino-functionalized cellulose nanocrystals/chitosan beads nanocomposite (CNC-ED@CS-ED) for diclofenac sodium (DS) removal. The authors explain that the functionalization of adsorbent materials with amino groups become capable of forming an ionic bond with the carboxyl group of DS at suitable experimental conditions. As expected, the adsorption experiments presented good results for DS removal with an adsorption capacity equal to 444.44 mg g^{-1}. The adsorption process can be described by pseudo-second-order kinetic model and Langmuir adsorption isotherm. The reusability tests showed that even after five adsorption/desorption process cycles, the adsorption capacity of CNC-ED@CS-ED for DS was only reduced by about 10%.

Pesticides used in agriculture are another example of micropollutants that can cause serious health and environmental problem. Thus, several studies have investigated ways to remove them from the aquatic ecosystem. In this sense, TiO_2/chitosan beads (TCB) were tested by Balakrishnan et al. (2020) for photocatalytic degradation of broadleaf pesticide2,4-dichlorophenoxyacetic acid. The TCB showed a maximum photocatalytic degradation under UV light of 92% compared to TiO_2 (53%), P25 (64%) and chitosan (39%). According to the authors, the altered bandgap of catalyst, better adsorption due to incorporating chitosan, and retarded fast e^-–h + pair recombination's affected by aeration were the responsible factors to the improved results beads. The reusability studies showed the nanocomposite can be reused since the degradation efficiency only decreased from 92 to 80% after 11 consecutive cycles.

In another study, Dehaghi et al. (2014) used batch adsorption experiments and fabricated chitosan-ZnO nanoparticles (CS-ZnONPs) composite beads to remove Permethrin pesticide, a neurotoxin widely used in agriculture. The experiments showed satisfactory results, removing 99% of the pesticide from an aqueous solution. The desorption experiment showed that this nanocomposite could be regenerated about 56% after three cycles. The removal of the pesticide from liquid media using a polymeric composite of mesoporous silica and polyaniline monomer (MSNPs/PANI) was studied by El-Said et al. (2018) The exclusion efficiency of the pesticide chloridazon MSNPs/PANI mesosorbent was around 96%, and the Langmuir and Freundlich models were the ones with the best fit. The ability to reuse composites over several cycles without losing or modifying their physical integrity and adsorption activity was investigated. The authors identified that MSNPs/PANI can be used up to seven cycles, performing simple washes with acetonitrile solution.

4 Applications and Comparison with Traditional Techniques

Although the use of polymeric composites in water treatment is mainly a topic of research, there are a few examples of real pilot-plant scale treatment processes that employ the materials described in this chapter. In this sense, to employ lab-scale adsorption technology in a large-scale purification system, Aziz et al. (2020) conducted experiments in a steady-flow reactor and modelled the adsorption kinetics. Activated charcoal was immobilized in alginate beads for easy recovery and employed to eliminate heavy metals from wastewater in a fixed bed column. Nearly 100% of dissolved cadmium was removed in large-scale tests, and the composite became saturated only after 25 h of treatment. Additionally, activated charcoal composites have the distinction of being used not only for targeted micropollutants but are also widely used in domestic tap water treatment due to their large absorbance capacitance and non-selectivity (Jaspal and Malviya 2020). These composites remove fine suspended particulate and microorganisms, odors, tastes and regulate the pH, which are the main contaminants of domestic waters (Siong and Atabaki 2014).

Silver nanoparticles incorporated in membranes have been employed for simultaneous filtration and disinfection of drinking water, (Ursino et al. 2018), as well as being incorporated in inorganic ceramic filters, reaching a bacteria inactivation of 97–100% (Ren and Smith 2013; Oyanedel-Craver and Smith 2008). Photocatalytic TiO_2-polyvinylidene fluoride/PMMA nanocomposites flat sheet membranes were produced by phase inversion and a modelled crossflow filtration system, aiming the removal of pollutants with contrasting chemical properties. In this study, filtration and photocatalysis processes using ultraviolet light were used simultaneously. The combination of these treatments removed nearly 100% of CR dye and over 80% of Tartrazine, with reduced fouling (Errahmani et al. 2021). In fact, when moving composites from laboratory to pilot-plant scale, especially in the form of films and

membranes, the fouling and general contamination of these selective materials can be major drawbacks. Therefore, the transfer of technologies needs to be closely controlled to ensure the composites functionality (Berber 2020; Bouziane Errahmani et al. 2021).

As the efficiency of these advanced materials improves and the regulations for wastewaters become stricter, the use of composites in water treatment is expected to keep increasing, especially to target specific pollutants than conventional treatment plants do not remove. That is supported by the numbers shown above in the pilot plant scale for adsorption, photocatalysis, and microbiological treatments using polymeric composites. While the composites could remove between 80 and 100% of the targeted micropollutants, traditional water treatment generally removes less than 50% of these emerging chemicals, often less than 20% (Huerta-Fontela et al. 2011; Huang et al. 2003). Given that health effects and ecosystem disruption can happen at concentrations of parts per billion in the case of pesticides and pharmaceuticals, less than 50% abatement is not nearly enough for safe drinking water or even wastewater (Huerta-Fontela et al. 2011; Huang et al. 2003). Alternatively, many of these advanced techniques cannot be optimally employed in the treatment of macropollutants, as they are sensitive to the aqueous environment around them, which is the case of photocatalysis that can become poisoned by excess organic matter occupying active sites, (Vebber et al. 2019a; Pettibone et al. 2008) as well as adsorbents (organic or clay), whose performance is highly sensitive to pH, which can vary greatly in a highly contaminated source (Berber 2020). Therefore, it should be clear that the techniques reviewed here function as complementary treatments that, nonetheless, are necessary due to the increasing complexity and variety of chemicals that end up in water streams (Yang et al. 2017).

5 Challenges, Perspectives and Environmental Sustainability

The mission of offering adequate treatment for different water contaminants sometimes becomes difficult and expensive. For this reason, there is currently an abundant request for cutting-edge and cheap technologies. However, as described in this chapter, several pollutants have been eliminated using polymer compounds of natural and synthetic origin, demonstrating their potential in wastewater treatment. Arguably, the use of nanostructured materials as a filling for polymeric matrices has emerged as an emerging area to increase the potential for removing aqueous contaminants. Substantial research has been devoted to achieving high adsorption performance, recyclability, selectivity of different pollutants and durability. However, despite all the efforts made, the market does not have a polymer composite that can eliminate 100% of the contaminants, which can be produced on a large scale.

Another challenging issue in selecting polymeric compounds for water treatment is preserving the environmental sustainability of the ecosystem. It is known that

the use of some chemical processes in wastewater treatment can become a possible source of environmental pollution in the long term. In this sense, the development of green composites through green approaches has gained prominence in recent years as alternatives for ecosystem security. Green compounds are eco-friendly, efficient, reliable and alternative to traditional compounds for environmental sustainability. Furthermore, using materials from natural sources can help reduce costs and facilitate large-scale applications (Mukhopadhyay et al. 2020).

In this sense, the authors identify as a possible future challenge the synthesis of compounds that present specific functional groups that increase the performance of the final composite, ensuring environmental safety. Continuing studies on possible pollutant elimination mechanisms is still a very important challenge, as we need to understand these mechanisms in greater depth, which helps to define a better choice of material. Despite the challenges to be overcome, polymer composite research opens a new route in science for a pollution-free environment.

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