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Review

Advances in Chitosan-Based Materials for Application in Catalysis and Adsorption of Emerging Contaminants

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Abstract: The increasing disposal of emerging contaminants in the environment is a worldwide concern due to environmental impacts, such as toxicity, hormonal disorders, and bioaccumulation. The persistence of these pollutants in water bodies makes conventional pollutant removal techniques inefficient or partial, thus requiring the development of new, more effective, sustainable remediation technologies. Therefore, chitosan-based materials have emerged as a promising alternative for application in catalysis and contaminant removal. The biopolymer has functional properties that make it an excellent adsorbent capable of removing more specific pollutants, such as pharmaceuticals, microplastics, agricultural pesticides, and perfluoroalkyl and poly-fluoroalkyl substances, which are increasingly in evidence today. Therefore, this review of recent and advanced research into using chitosan to manufacture catalytic and adsorption materials offers an innovative approach to treating contaminants in aqueous environments, significantly reducing their presence and impact. It discusses the advantages of using chitosan as an adsorbent and catalyst and its role as a support for catalysts and biocatalysts. In addition, the review highlights the diversity of the physical forms of chitosan, such as particles, membranes, and hydrogels, and its possible chemical modifications, highlighting its effectiveness in catalytic applications and the removal of a wide range of emerging contaminants.

Keywords: chitosan; modifications; biocatalysts; adsorption; emerging contaminants

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1. Introduction

Population growth, rapid industrialization, urbanization, and agriculture favor the increase in emerging contaminants in the ecosystem, directly affecting the aquatic environment [1–3]. The excessive use of fertilizers and pesticides in agriculture, industrial effluents with high levels of heavy metals and toxic chemicals, domestic waste, and mining waste are the primary anthropogenic sources that generate this accumulative volume of harmful substances [1–3]. Among these, contaminants are classified as emerging pharmaceutical products [4–6], personal care items [7,8], hormones [1,9], microplastics [10,11], agricultural pesticides [12,13], and perfluoroalkyl and poly-fluoroalkyl substances [14,15], which are increasingly being detected in wastewater and are further exacerbating the problem. In addition, climate change due to global warming, such as heavy rain and strong winds, can favor the dispersion and bioaccumulation of these pollutants in various places, further

aggravating this problem. Therefore, developing and searching for innovative materials to help remove these contaminants is becoming increasingly necessary to preserve fauna and flora [16].

Among the methods for removing contaminants from aqueous solutions, adsorption stands out due to its ease of operation and low cost, especially when combined with sustainable or reusable adsorbents. Chitosan is one of these promising adsorbents, as it is non-toxic, low-cost, biodegradable, and versatile [17–19]. This biopolymer is derived from chitin, which is found in crustacean exoskeletons [4–6] and is presented as an efficient material due to its unique properties, such as the presence of active functional groups in its chemical structure and the possibility of modifications to its structure and morphology, such as increasing the surface area, thus favoring the removal of emerging compounds in effluents [6,20,21]. The various techniques used to obtain chitosan-based adsorbents and catalysts allow their adaptation to different types of pollutants and operational conditions, optimizing the removal of contaminants both on a laboratory scale and in industrial applications [2]. Furthermore, chitosan presents promising advantages when used to support catalysts and biocatalysts, increasing the efficiency of pollutant degradation reactions. The ability to chemically modify chitosan allows for the creation of concrete supports for various types of catalysts, increasing the selectivity and efficiency of these reactions [4].

The biopolymer can support the formation of catalysts where metals are deposited or incorporated, usually forming a composite with the chitosan immobilizing these nanoparticles. Immobilization techniques directly influence biocatalysts' physicochemical properties, including encapsulation, adsorption, and cross-linking [22–24]. In addition, studies have shown that the application of these compounds to the treatment of effluents is favored by the presence of the catalyst, which promotes stability in solutions and improves the mechanical properties of the adsorbent, thus favoring its ability to be reused after the adsorption process of emerging contaminants [25,26]. This review highlights the potential of chitosan as an attractive subclass of functional materials, offering sustainable and high-performance solutions for wastewater treatment. The study also provides new insights into the development of functional catalysts and their application in a wide range of pollutants, indicating their functionality and the selectivity of each pollutant.

2. Properties of Chitosan

Chitosan, a biopolymer that is considered the world's second most abundant organic resource, has attracted significant attention in various research fields due to its unique properties. These include biodegradability, biocompatibility, bioactivity, and favorable physical and mechanical characteristics. Derived from chitin (2-acetamino-2-deoxy-d-glucose connected through β (1 → 4) glycosidic bonds) (Figure 1), which is found mainly in the exoskeletons of crustaceans (shrimps and crabs) and insects (cockroaches and ants), chitosan offers a wide range of applications in areas such as pharmaceuticals, agriculture, biomedical engineering, the food industry, wastewater treatment, and catalysis [4–6,9]. The polysaccharide has a low cost, is non-toxic, is readily available, and is reusable, and its ease of modification/functionalization makes this biopolymer an increasingly attractive material for innovative technological and environmental applications, such as adsorption and catalysis [6,20,21].

Among the forms of chitosan modification/functionalization, we can mention its use in the form of powered gels, flakes, membranes, fibers, nanospheres, beads, and films. Modifications in the chitosan structure aim to increase the surface area and porosity, as well as improving mechanical stability. These characteristics favor the decontamination processes of environmental waters, mainly when the biopolymer is applied as an adsorbent, catalyst, or support for other catalysts [1,9,21,27,28].

The chemical and physical modifications (Figure 1) of chitosan help in research to expand its applications in the field of sustainable catalysts. Chemically, chitosan can be cross-linked with agents such as cyanoguanidine, formaldehyde, glyoxal, tripolyphosphate, isocyanate, ethylene, and glutaraldehyde, creating a more stable three-dimensional network

that improves mechanical and thermal resistance, as well as facilitating the accessibility of adsorbates and consequently increasing adsorption capacity [29–32]. Wang et al. [14] developed chitosan nanofibers cross-linked with glutaraldehyde, which were used as supports for palladium catalysts in the Mizoroki–Heck reaction. The authors observed that the catalysts showed remarkable stability and efficiency, directly influenced by the glutaraldehyde concentration. Cross-linking with glutaraldehyde reduced the energy required to form a chelate complex between the fibers, thus reducing the activation energy of the Mizoroki–Heck reactions.

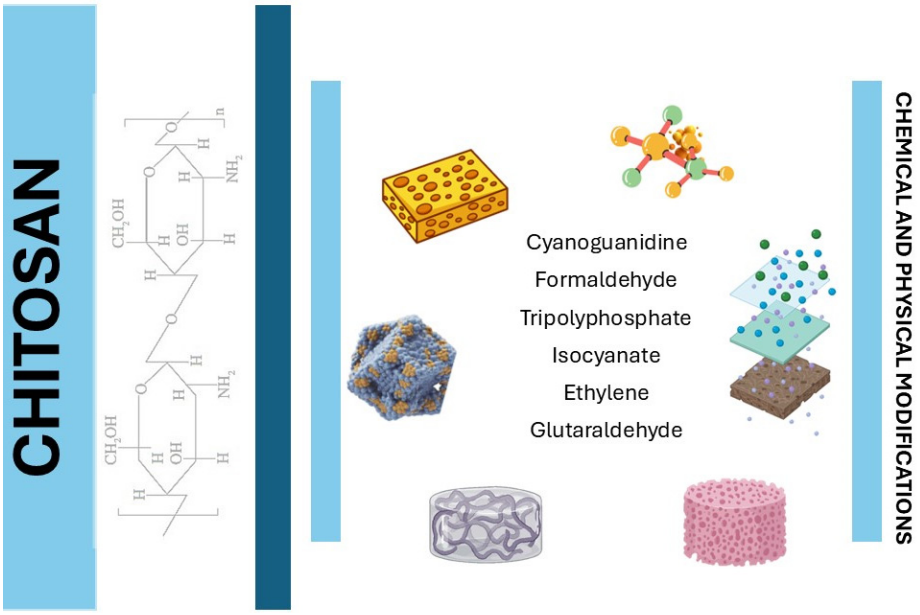


Figure 1. Chitosan modifications for applications.

The chemical properties of chitosan are primarily determined by the presence of primary amine groups, together with N-acetylated groups, and it can catalyze reactions of carbonyl compounds through the enamine/imine mechanism, varying its applicability. In addition, the amine groups allow for its response to be directed precisely, minimizing the formation of by-products. For example, the biopolymer can be used as a basic catalyst for the development of α-diazo carbonyl compounds, representing a significant advance towards environmental sustainability, offering an efficient solution for the synthesis of these important chemical intermediates [33–36].

Physical modifications of chitosan in catalysis: The biopolymer can be applied with various metal ions, enzymes, or nanoparticles, favoring its catalytic activity; it can also be used as a support for other catalysts [28,37]. Cordoba et al. [38] prepared hybrid nanofibers utilizing a blend of chitosan and polycaprolactone, which was used to support titanium dioxide nanoparticles. The chitosan nanofibers improved the adsorption of Rhodamine B and the efficiency of photocatalytic degradation by the nanoparticles. The porous structure of chitosan and its high surface area favor the immobilization of catalytic species and increase selectivity.

Another important variable that should be considered is the degree of deacetylation (DD), which influences chitosan's physical and chemical properties. Chitosan with a high DD has high solubility and crystallinity, which improves the structural stability of a catalyst. A chitosan structure with a higher DD allows for the formation of a more ordered and regular structure, favoring the formation of porous and fibrous structures. On the other hand, a cross-linked chitosan structure will favor its thermal and mechanical resistance, which is advantageous in reactions that take place under harsh conditions, where the stability of the catalyst is crucial [39–41].

3. Chitosan in Catalysis and Adsorption

In wastewater treatment, catalytic methods play a crucial role in efficiently removing persistent contaminants, both organic and inorganic. These methods facilitate the degradation of these pollutants and allow for the strict control of the reaction conditions, thus increasing catalytic activity and maintaining thermodynamic equilibrium without alterations [42,43]. It is, therefore, fundamental to choose the appropriate catalytic method according to the type of emerging contaminant in the solution, concentration, and costs associated with the process. Using catalysts allows for treatment optimization to achieve high removal rates, with a minimal availability of emerging contaminants and their bioaccumulation in water bodies [44].

Catalysis is divided into homogeneous and heterogeneous types, each with distinct characteristics influencing their application. In homogeneous catalysis, the catalyst and reactants are in the same phase, forming intermediate complexes, which are then converted into the desired product. Homogeneous catalysis offers a significant advantage by allowing for the control of reaction variables, such as temperature and pressure, leading to more selective and efficient reactions, optimizing the process [45]. However, the recovery of the catalyst after the reaction can be more challenging, which is why more advanced research includes the development of homogeneous catalysts that are immobilized or bound to polymers [46]. Heterogeneous catalysis, on the other hand, stands out in many industrial processes because it uses a catalyst in a phase distinct from the reactants, usually a solid, facilitating the separation and reuse of this substance [47].

Among the various types of heterogeneous catalysis, photocatalysis has gained prominence, particularly in activating catalysts such as TiO_2, which are highlighted for their non-toxicity, low cost, and effectiveness in degrading organic pollutants under irradiation. Recent studies of the application of polysaccharide-based biopolymers as supports in photocatalytic water and wastewater treatment have proven that heterogeneous photocatalysis using TiO_2, ZnO, and g-C_3N_4 is a promising and environmentally friendly technology for removing resistant pollutants [48–50]. This technology benefits from the ability of photocatalysts to generate highly reactive free radicals. Chitosan offers an ideal matrix for immobilizing these photocatalysts, not only by improving stability and reducing semiconductor accumulation but also by increasing the efficiency of photocatalysis due to its adsorption. The efficiency of this system can be further enhanced by incorporating glycerol, where chitosan acts as an adsorbent for the dyes, and glycerol reduces electron recombination, thereby increasing the production of free radicals [51].

Nitrogen doping is an effective technique for improving the catalytic properties of materials by incorporating nitrogen atoms into their structure during the carbonization process. In the case of chitosan, this doping increases electronic conductivity and creates new active sites, optimizing catalytic efficiency, especially in photocatalytic and electrocatalytic reactions. Khan et al. [52] developed nitrogen-doped chitosan derivatives into carbon materials for catalytic applications. Doping was performed during carbonization, and the results showed a significant increase in photocatalytic efficiency with increasing pyrolysis temperature, demonstrating the potential of these materials in more efficient and sustainable catalytic processes.

The active sites in chitosan provide excellent catalytic support that can immobilize catalytically active species and generate biodegradable material [53]. According to Jiménez-Gómez et al. [54], chitosan has a promising chemical reactivity due to these functional groups in its structure, which act as efficient selective sites for metals and other catalytically active species. When used as a porous structure, whether as a sponge, scaffold, or foam, chitosan offers a high surface area and pore volume, which is fundamental for heterogeneous catalysis. These characteristics allow for the better dispersion of catalytic species and enhance the accessibility of reagents to active sites.

Modified chitosan has gained prominence as a promising material in catalysis for pollutant removal. Chemical modifications significantly improve chitosan's stability, such as cross-linking with glutaraldehyde, nitrogen doping, or incorporating metallic nanoparti-

cles. These modifications enhance its effectiveness in processes such as the degradation of organic compounds, the removal of textile dyes, and the extraction of heavy metals from aqueous solutions, as illustrated in Table 1.

Table 1. Application of chitosan-based materials in catalysis for pollutant removal.

Catalyst	Chitosan Modifiers	Pollutant	Major Achievement	Ref.
Pd NPs	Carbon nanotubes + Glutaraldehyde	Nitroaromatic and CR, MO, MR dyes	High activity provides 85% removal after 6 cycles.	[55]
ZnO	MOFs/Polypropylene	MB	97% removal; reuse: 6 cycles	[56]
TiO_2	Glycerol	AB 193	85% removal	[51]
TiO_2/Ag NPs	Ultrasound radiation	Cu(II), MO, MB, and microbial inhibition	95% removal Cu(II); reuse: 6 cycles	[57]
FeOOH CeO_2	Glutaraldehyde	OTC	98% removal; reuse: 5 cycles	[58]
Cu_2O/Fe_3O_4 NPs	Glutaraldehyde	Cr(VI)	91% removal	[59]

NPs (nanoparticles); Methyl Orange (MO); Methyl Red (MR); Congo Red (CR); Methylene Blue (MB); metal–organic frameworks (MOFs); Acid Blue 193 (AB 193); oxytetracycline (OTC).

From Table 1, it can be seen that several studies highlight the potential of chitosan-based catalysts in removing pollutants and their efficiency. Sargin et al. [55] indicated that palladium nanoparticles (Pd NPs) demonstrate a high catalytic activity in environmental remediation, especially in reducing toxic pollutants. The modification of chitosan with glutaraldehyde was essential because it created a standard support that efficiently immobilizes the Pd NPs, improving the dispersion and stability of the catalyst. This resulted in a highly effective and reusable catalytic system, maintaining excellent performance over multiple cycles. Therefore, the modification of chitosan with glutaraldehyde occurs when the glutaraldehyde reacts with the amino groups (-NH_2) of chitosan, forming Schiff bonds (-C=N-) [60]. This cross-linking process creates a standard three-dimensional polymer matrix, enhancing the immobilization and dispersion of palladium nanoparticles and increasing the catalytic efficiency in environmental remediation. Furthermore, another study also indicated that the presence of Au NPs plays a crucial role in improving the catalytic activity of the chitosan composite [61].

Hoang et al. [51] showed that the TiO_2/chitosan ratio and the irradiation time are critical parameters that directly influence the removal efficiency of the azo dye Acid Blue 193 (AB 193). Like other complex pollutants, this dye has a complicated structure, high resistance in aquatic environments, and is difficult to remove using conventional methods. The authors found that acidic pH conditions were most favorable for removing the anionic dye, in which the protonation of the $–NH_{3+}$ groups of chitosan occur, and the positive charge of TiO_2 enhanced dye adsorption, leading to a greater degradation efficiency. At higher pH levels, the negative charge of TiO_2 and the repulsion between charged surfaces reduce dye adsorption, resulting in a lower efficiency. Furthermore, the study tested blending the chitosan with glycerol, which helps the system act as an electron donor, increasing the photocatalytic efficiency and removing about 70% from AB 193.

Rehan et al. [57] developed nanocomposites of chitosan, TiO_2, and silver nanoparticles (Ag NPs) to remove Cu(II) and dyes, achieving 95% removal of Cu(II), with a slight decrease in efficiency after 5 cycles. Shen et al. [59] presented a promising study in which they developed a bifunctional catalyst for removing Cr (IV), which is highly toxic and carcinogenic, even at low concentrations. The authors manufactured chitosan beads incorporated with Cu_2O and Fe_3O_4 nanoparticles. They also used glutaraldehyde as a cross-linking agent to ensure the catalyst's structural integrity, particularly in experiments with pH variations, which is essential in wastewater treatment. The results were highly significant due to the catalyst's dual functionality, acting in oxidizing Cr (III) organic complexes and capturing and reducing Cr (VI). The effectiveness of removal across a wide pH range was also highlighted. Shen et al. [58] created chitosan microspheres modified with FeOOH and CeO_2, removing 98% of oxytetracycline (OTC) and maintaining efficacy for 5 cycles.

4. Synthesis of Chitosan-Based Catalysts

Using natural polymers as catalytic substrates is of great importance in organic chemistry. Chitosan can be used in several reactions, as the interactions between its hydroxyl and amino groups to metals can lead to the development of different and complex materials [62,63]. The amino group imparts several properties to chitosan, including solubility in acid, adhesion to negatively charged surfaces, and cationic polyelectrolyte behavior that allow for the formation of stable complexes with metal ions, preventing agglomeration during catalysis [17,26].

Therefore, the incorporation of metallic catalysts to chitosan leads to composite materials with the dual function of adsorption and catalysis—the absorbent accelerates the migration of pollutants due to the presence of amino and hydroxyl groups that act as active sites, and the catalyst converts the contaminants into readily biodegradable intermediates or simple inorganic substances, such as CO_2 and H_2O [64].

The synthesis of chitosan-based materials typically involves different steps—chemical or physical modification by different organic and inorganic ligands; the incorporation of metallic catalysts, such as TiO_2, CuO, and Bi_2WO_6; cross-linking to increase the stability of chitosan and/or improve the binding of the catalytic material; and the final steps comprise shaping and drying the material into powder particles, beads, films, fibers, flakes, or sponges. Different methods, such as electrospinning, coating, casting, and phase inversion, can carry out these stages. Figure 2 illustrates each technique, indicating the final shapes of the chitosan-based material produced by each method. The following discussion explores the details of these techniques.

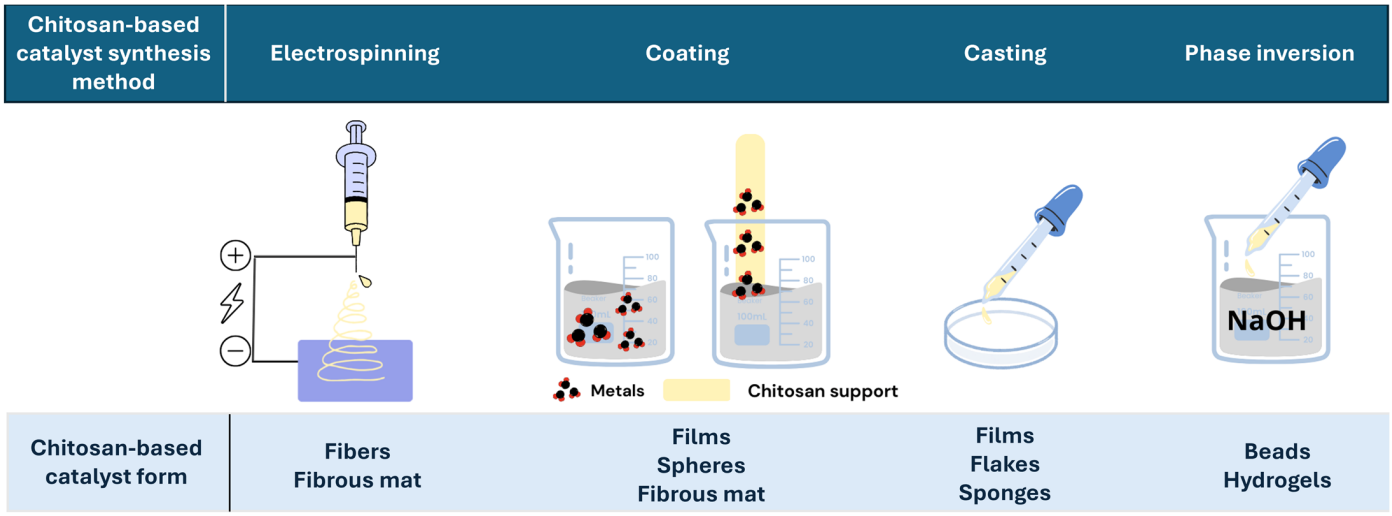


Figure 2. Synthesis methods for chitosan-based catalysts and the resulting material forms.

4.1. Synthesis of Chitosan-Based Catalysts by Electrospinning

Electrospinning is a technique used to prepare fibers with micro- to nanometer diameters that involves the application of a high-voltage electric field to a polymeric solution. Instability is produced in the polymer because of the electric field application, and charge repulsion creates a force that resists surface tension, deforming spherical droplets and creating the Taylor cone [65]. The evaporation of the solvent dries the polymer, producing the nanofibers collected on a positively charged flat surface or in a revolving drum. This technique produces fibers with a high surface area within a low volume and interfibrous pores sizes. These properties increase mass transfer rates, making these fibers attractive for adsorption applications. The electrospinning equipment comprises a high-voltage power supply (10–20 kV); a syringe pump; a spinneret, where the polymeric solution is placed; and a collector [66,67].

The production of nanofibers from chitosan by electrospinning requires the application of high electric field intensities due to chitosan's properties (high viscosity, large surface

tension, and cationic nature). Therefore, low molecular weight chitosan is preferred to produce these materials [65]. The first step in producing chitosan materials by electrospinning is dissolving this polymer in acids or organic solvents. The second step involves solubilizing the catalyst in the chitosan solution. The last step is the electrospinning process of the material.

ZabihiSahebi et al. [18] produced chitosan nanofibers with carbon nanotubes and used Fe_3O_4 and TiO_2 for the removal of Cr (VI), As(V), methylene blue, and Congo red from aqueous solutions. The authors used a polymer blend (60:40, *w*/*w*, chitosan/cellulose acetate) dissolved in trifluoroacetic/dichloromethane (70:30, *v*/*v*). To this solution, various concentrations (5–20%, *w*/*w*) of carbon nanotubes/Fe_3O_4/TiO_2 composite were dispersed through sonication for 1 h, followed by stirring for 3 h. The electrospun fibers were produced using 20 kV, with a tip–collector distance of 10 cm and a flow rate of 0.25 mL/h. The researchers concluded that the adsorbent was more effective for removing Cr(VI) and As(V) at lower concentrations via adsorption. For higher concentrations of these metal ions, photocatalytic reduction was more effective. Additionally, the photocatalytic method effectively degrades the methylene blue and Congo red dyes at both low and high concentrations.

Rabanimehr et al. [68] produced chitosan/Bi_2WO_6/carbon tubes/TiO_2 nanofibers for cephalexin removal. Chitosan powder was dissolved in trifluoroacetic acid and dichloromethane. The catalysts were added to this solution, and the mixture was sonicated for 1 h and fed into the spinneret for electrospinning. Electrospinning was performed at 22 kV, 0.2 mL/h, and a distance of 8 cm between the needle tip and the collector. The nanofibers were collected on a stainless-steel plate collector and dried at 60 °C for 24 h. The nanofibers were treated with glutaraldehyde at 25 °C for 24 h for cross-linking, and samples were heated at 60 °C for 12 h to remove residual glutaraldehyde. The results showed that the synthesized material was effective for cephalexin removal, with a 99% degradation of the pollutant.

In other studies, electrospinning was used to produce chitosan nanofibers. Once the nanofibers were created, the catalyst was added to the polymeric structure by immersion in a solution containing the catalyst or electrospray deposition. Alabduljabbar et al. [69] prepared chitosan membranes by electrospinning to remove methylene blue dye in aqueous media. The authors dissolved chitosan powder in trifluoroacetic acid (6% *w*/*w*) and stirred the solution for 5 h. The solution was then filtered to remove undissolved particles, and electrospinning was performed under a voltage of 22 kV, a flow rate of 0.4 mL/h, a distance of 100 mm between the needle tip and the collector, and a needle diameter of 0.8 mm. After electrospinning, the nanofibers were dried in a vacuum oven at 60 °C and −0.1 MPa. TiO_2 nanoparticles were suspended in trifluoroacetic acid and sonicated for 5 min. The suspension was electrosprayed onto the chitosan membrane. The authors observed a higher degradation efficiency (89%) of this material compared to a similar membrane obtained by the electrospinning of a suspension containing chitosan and TiO_2 (40%).

4.2. Synthesis of Chitosan-Based Catalysts by Coating

Coating refers to the immersion of the chitosan support into a solution containing the catalyst. This process allows for the uniform deposition and bonding of the catalyst to the chitosan surface. Abdelwahab et al. [19] developed a material using chitosan, polyacrylamide, Fe_3O_4, and TiO_2 for Congo red dye treatment. The Fe_3O_4 spheres were prepared by dissolving $FeCl_3 \cdot 6H_2O$, sodium citrate, and sodium acetate in ethylene glycol at 200 °C for 10 h. The precipitate was filtered, washed, and dried at 80 °C for 12 h. Carboxymethylated chitosan was dissolved in water and added to the Fe_3O_4 dispersion. TiO_2 was coated on the modified chitosan by dissolving titanium tetraisopropoxide in isopropanol. The resulting material was washed and dried at 60 °C for 6 h. The results showed that the solution was 92% decolorized for 3 h.

Chitosan–bismuth–tungstate composite coated with silver composite was synthesized and used for the adsorption and photocatalytic removal of Cu (II) from aqueous solu-

tions [70]. Ag/Bi_2WO_6 was synthesized by dissolving citric acid in water and adding $Bi(NO_3)_3.5H_2O$, $Na_2WO_4 \cdot 2H_2O$, and $NaHCO_3$. After stirring, silver nitrate was instilled in the solution. This mixture was heated at 180 °C for 24 h. After cooling, the precipitate was rinsed with ethanol and water, and was then dried at 70 °C for 12 h to obtain the Ag/Bi_2WO_6 powders. The material presented a desirable adsorption ability of Cu(II) after 5 cycles.

Other researchers used coating to prepare the chitosan support. Ali et al. [71] prepared eggshell membranes coated with chitosan to support nanocatalysts to reduce different organic pollutants. The material was prepared by mixing eggshell membranes with a chitosan solution, forming strips. These strips were templated with Cu and Fe nanoparticles by immersing them in aqueous solutions to adsorb the ions. The metal ions were subsequently reduced to zero-valent metal nanoparticles using an aqueous $NaBH_4$ solution. The synthesized materials were utilized as catalysts to evaluate their performance in reducing nitroaromatic compounds and decolorizing dyes. The results showed that when using eggshell membrane with 80% chitosan, the reduction reaction was completed after 7, 6, and 7 min, for 4-nitroaniline, 4-nitrophenol, and methylene blue, respectively.

Haider et al. [72] developed a chitosan-based catalyst using a cellulose microfiber mat and chitosan as support for Cu nanoparticles. The material was applied to treat 2-nitrophenol, 4-nitrophenol, and cresyl blue. For that, chitosan was diluted in acetic acid, and this solution was coated into a cellulose mat by dipping. The support of cellulose and chitosan was mixed with a $CuSO_4$ solution, and after 2 h, the material was washed and immersed in a $NaBH_4$ solution for 4 h to reduce Cu^{2+} to Cu. The material was effective for the reduction of nitroaromatic compounds and cresyl blue dye compared with other supported Cu nanoparticles.

Chitosan, *S. cordifolia* aqueous leaf extract, and CuO were used to synthetize a composite used for the degradation of crystal violet and malachite green dyes [7]. For that, $CuSO_4 \cdot 5H_2O$ solution was mixed with the leaf extract, stirred at 70 °C for 3 h, and the pH was adjusted to 9 with NaOH. Separately, chitosan was dissolved in acetic acid and heated at 65 °C until dissolution. Then, chitosan solution was gradually added to the $CuSO_4$–leaf extract solution, stirred constantly, cooled, washed, and dried at 60 °C. According to the authors, the composite was considered suitable for industrial wastewater treatment.

Veisi et al. [73] produced a chitosan/starch material with Fe_3O_4 and Pd for 4-nitrophenol treatment. Chitosan and starch were modified with acetic acid, and this material was added to an Fe_3O_4/water solution. This mixture was sonicated and cross-linked with glutaraldehyde, and a Na_2PdCl_4 solution was gradually added. The catalysts were removed from the solution, washed, and dried, and the material was influential in the reaction with 4-nitrophenol.

4.3. Synthesis of Chitosan-Based Catalysts by Casting

Chitosan films are commonly produced with additives to improve their mechanical properties. Poly(vinyl alcohol) (PVA) is a common additive due to its hydrophilic nature, film-forming properties, and resistance to different solvents. The film process is usually performed by stirring at ambient temperature, ultrasonic application, and/or the utilization of moderate to high temperatures (60–80 °C). After mixing, the film solution is poured into a mold and allowed to dry.

Habiba et al. [17] developed a chitosan/PVA/Na–titanate/TiO_2 composite film for methyl orange and Congo red adsorption. Chitosan was hydrolyzed to enhance its solubility using a NaOH solution (33.5% *w*/*w*), stirred at 90 °C for 12 h, and then dried at 70 °C for 7 h. The material was then dissolved in acetic acid, and this solution was mixed with an aqueous PVA solution (8% *w*/*w*) in various ratios (60:40, 80:20, and 90:10). TiO_2 (0.5%, *w*/*w*) was added to the obtained solutions. The blended solutions were cast, dried at 70 °C for 7 h, treated with NaOH, and dried for 2 h. The material presented efficient absorptivity and photocatalytic properties for dye degradation.

Soltaninejad et al. [74] prepared a bionanocomposite of PVA, TiO_2, chitosan, and chlorophyll for degrading methylene blue, Congo red, and 4-chlorophenol. For that, titanium isopropoxide was gradually added to deionized water and stirred at 50 °C for 1 h. TiO_2 nanoparticles were formed by the hydrolysis of the alkoxide and precipitated as a white solid. The nanocomposite was prepared with a solution of PVA in distilled water performed at 80 °C with ultrasonication. TiO_2 nanoparticles and chitosan were dispersed in distilled water and sonicated at 80 °C for 2 h. The two solutions were then combined and heated to 200 °C for 2 h. After cooling, chlorophyll was added and thoroughly mixed. The resulting mixture was poured onto a glass plate and dried at room temperature to form the film. The material was effective for the removal of the pollutants.

Souza et al. [64] prepared a catalyst consisting of copper nanoparticles supported in a chitosan/PVA film to remove aromatic nitro compounds. Chitosan was dissolved in 1% acetic acid solution under magnetic stirring at room temperature for 5 h. Separately, PVA was dissolved in distilled water at 80 °C. The chitosan and PVA solutions were then blended in a 1:2 mass ratio and stirred for 30 min. Glutaraldehyde (25% solution, 80 µL) was added dropwise, the resulting solution was poured into Petri dishes, and the solvent was evaporated at 40 °C for 24 h. The films were immersed in a $CuCl_2{\cdot}2H_2O$ solution to introduce Cu^{2+} ions and were dried at 40 °C for 24 h. Afterward, the material was immersed in $NaBH_4$ solution at room temperature to produce a film entrapped with Cu nanoparticles. The results showed that the material was influential in reducing aromatic nitro compounds to aromatic amines.

Majnis et al. [75] prepared a ZrO_2/TiO_2 composite using a sol–gel method with different TiO_2:ZrO_2 weight ratios. The material was applied in the catalytic degradation of malachite green dyes. $ZrOCl_2{\cdot}8H_2O$ and TiO_2 powders were mixed in deionized water at 65 °C to form a solution, which was stirred for 30 min. The pH of the solution was raised to 10 to precipitate the gel. The gel was filtered, washed, dried, and calcined at 500 °C for 2 h to obtain ZrO_2/TiO_2 powder. Acetic acid and sodium chloride were mixed, then chitosan flakes were added and stirred for 12 h. TiO_2 or ZrO_2/TiO_2 powder was added to this solution, followed by acetic acid, and stirred for 24 h to obtain a chitosan–TiO_2 solution. The material was poured into glass plates and dried at 100 °C for 4 h. The authors observed that that the degradation efficiency improved with the increase in ZrO_2 content in TiO_2.

4.4. Synthesis of Chitosan-Based Catalysts by Phase Inversion

Phase inversion is a technique used to create porous structures, such as hydrogels, membranes, or beads. This process involves the transformation of a liquid or gel solution into a solid or semi-solid form by changing the solvent composition or introducing a non-solvent [76]. A chemically modified chitosan and catalyst solution is usually dropped into an alkaline solution for bead formation. Eroğlan et al. [77] developed a catalyst of Pd nanoparticles fixed on NiO-modified chitosan beads to reduce organic pollutants. The material was synthesized with chemically modified chitosan with acetic acid. NiO was added to the mixture, and the solution was dropped into an alkali solution to allow for the formation of chitosan–NiO gelatinous microspheres. The microspheres were recuperated by filtration, washed, and added to a solution of ethanol and glyoxal. The mixture was heated to allow for the cross-linking. The solution was filtered, and the microspheres were washed and dried. The tests demonstrated that the synthesized Pd/Chitosan–NiO was an effective catalyst for the reduction of 4-nitro-o-phenylenediamine, 4-nitrophenol, 4-nitroaniline, 2-nitroaniline, methylene blue, methyl orange, and rhodamine B in an aqueous medium at room temperature. The time required to achieve complete reduction ranged from 65 to 145 s for nitroarenes, 0.023 s for methylene blue, and 55 and 40 s for methyl orange and rhodamine B, respectively.

Balakrishnan et al. [78] produced beads of TiO_2 and chitosan for the degradation of 2,4-dichlorophenoxyacetic acid. Chitosan flakes were dissolved in acetic acid, TiO_2 was added, and this suspension was added to a NaOH solution to form the beads. The beads

were washed and cross-linked with glutaraldehyde to enhance the texture, properties, and stability. The results showed a 92% degradation of 2,4-dichlorophenoxyacetic acid.

Other researchers have synthesized a chitosan-based catalyst in the form of a hydrogel. A hydrogel is a two-phase material composed of a mixture of porous, permeable solids and at least 10% (by weight or volume) of intercellular fluid consisting entirely or predominantly of water. These materials have a porous structure and soft surface properties, closely mimicking biological cells and tissues. Chitosan hydrogels can be synthesized using only chitosan or in combination with another polymer [79].

A chitosan–gelatin hydrogel with CuO particles was synthesized to be used in the production of polyhydroquinoline [63]. The chitosan support was synthesized with acetic acid and glutaraldehyde and was continuously added to the solution to form the hydrogel. After preparing the support, CuO was dispersed in distilled water and the hydrogel solution was added to this mixture under agitation. The material was then submitted to in situ magnetization with the addition of $FeCl_3.6H_2O$ and $FeCl_2.4H_2O$ at 70 °C. Afterward, ammonia was added to the mixture; the material was cooled, washed, and dried. According to the authors, the material can be used for various purposes, including removing pollutants from water bodies, such as drugs and dyes.

5. Biocatalysts Using Chitosan-Based Supports

Biocatalysts comprise enzymes that degrade pollutants by biochemical reactions. Biocatalysts are promising alternatives to chemical catalysts due to their selectivity, non-toxicity, and biodegradability. Enzymes are globular proteins that enhance the rate of biochemical reactions by reducing the activation energy. The selectivity and function of enzymes depend on their active sites' specific composition and conformation. The types of donor groups present and the spacing among amino acids are also important characteristics of the active site. It should be mentioned that the presence of metal ions can facilitate the orientation of functional groups, increasing the reactivity of chemical bonds. Moreover, the three-dimensional shape of an enzyme is essential for adequate substrate binding and effective catalysis. For instance, the complexity of enzyme structure can lead to selective degradation of many pollutants [22,80–82].

Different groups of enzymes can be employed in biocatalysis. However, oxidoreductases, especially peroxidases and oxidases, are the most frequently used. These enzymes have drawn attention due to the substrate specificity and minimal byproduct release. Therefore, these enzymes have been extensively studied in wastewater treatment for the degradation of different types of pollutants, such as dyes, pharmaceuticals, personal care products, and pesticides [83,84]. Oxidoreductases can degrade pollutants by catalyzing oxidation–reduction reactions, which involve transferring electrons from electron donors to electron acceptors [2,82,85].

In the biocatalysis of pollutant molecules, hydrogen peroxide or hydroperoxides act as electron acceptors for peroxidase enzymes. The active sites of peroxidases may contain iron proto-porphyrin; nonmetallic prosthetic groups, such as those in thiol peroxidases or alkyl hydroperoxidases; or metals, as seen in vanadium haloperoxidases or manganese catalases. Unlike peroxidases, laccases do not rely on peroxide molecules for their action. These enzymes use oxygen molecules as electron acceptors. Laccases are mainly multicopper oxidases, whose mechanism of action involves four copper ions—one type 1, one type 2, and two type 3. Both peroxidases and laccases are used for the oxidation of pollutants, such as phenolic compounds, aromatic amines, azo dyes, polycyclic aromatic hydrocarbons, and chlorinated compounds [2,86–90].

5.1. Biocatalyst Immobilization in Chitosan Support

The use of soluble enzymes in biocatalysis can have some disadvantages, such as high costs and lower effectiveness compared to inorganic compounds, such as metal oxides. These drawbacks could be related to their instability under different operating conditions and the need for additional separation operations post-process. Therefore,

enzyme immobilization can create a microenvironment that protects enzymes against extreme conditions and facilitates their separation and reusability. These advantages can enhance the cost-effectiveness of the process. Furthermore, enzyme immobilization can improve end-product separation, which is important for efficient effluent disposal. Moreover, different enzymes can be immobilized for multiple pollutant targets, which is essential for treating complex wastewater in industrial applications [22–24].

The material used for enzyme immobilization supports should have suitable physicochemical characteristics, such as functional groups that can be used for chemical modifications to improve interaction and maximize immobilization efficiency. Indeed, chitosan is a promising support material due to its polycationic nature, with amino and hydroxyl groups within its structure. These functional groups can undergo chemical modifications, such as grafting and cross-linking, to enhance the interaction between the enzyme and the support. These interactions can increase immobilization efficiency and protection against different operating conditions and harmful agents or inhibitors [91–94].

Grafting methods can introduce distinct functional groups into the chitosan structure, improving interactions between the enzyme and support and enhancing enzyme immobilization. These functional groups include carboxyl, alkyl, sulfonate, fatty acyl, thiol, quaternary ammonium, and phosphonic groups [22,95–99]. Cross-linking methods encompass compounds to promote ionic (e.g., tripolyphosphate) or covalent bonds (e.g., glutaraldehyde). The cross-linker interacts with functional groups from chitosan to create new intermolecular interactions through biopolymer structure. These modifications can improve its mechanical and thermal stabilities [100–102]. Chitosan is also an exciting material for enzyme support, considering environmental aspects. As mentioned above, this biomaterial is biodegradable and has abundant sources due to its production of natural sources.

In addition to the support biomaterial, the immobilization techniques affect the final physicochemical properties of the biocatalysts. These methods include entrapment, adsorption, and cross-linking. The most suitable immobilization method depends on the physicochemical characteristics of the support, enzyme, and target pollutant [22,103,104]. The entrapment method involves the adsorption of chitosan onto the enzyme surface, which creates a protective shell. However, this method can have mass transfer limitations, restricting substrate diffusion to the enzyme's active site and compromising the biocatalysis [94,105]. Furthermore, adsorption methods involve the diffusion of enzymes onto the chitosan surface or into its inner structure, depending on the morphology of the support. Porous or nano-sized materials provide higher surface areas, with additional inner surfaces and functional groups for enzyme adsorption. Indeed, adsorption can involve physical interactions (e.g., van der Waals forces) or chemical interactions (e.g., covalent bonds). Due to weaker interactions, physical adsorption is advantageous for its simplicity and minimal enzyme conformational changes. However, these interactions can lead to increased enzyme leaching, affecting the cost-effectiveness of the process. On the other hand, chemical adsorption reduces enzyme leakage and improves reusability due to stronger interactions, but it may cause conformational changes, which can compromise enzyme activity [22,106,107].

The cross-linking method, similar to the previously discussed method, usually uses glutaraldehyde to promote intermolecular interactions among enzymes by Schiff's base reaction, resulting in cross-linked enzyme aggregates (CLEAs). Indeed, chitosan can be combined with enzymes to create coaggregation enzymes and chitosan. The cross-linking method can also be used after adsorption to create new covalent bonds between the functional groups of chitosan and the enzyme to reduce enzyme leaching. The covalent bonds can improve the attachment of enzymes in chitosan support, enhancing the reusability cycles. However, the operational parameters should be carefully studied to avoid conformational changes in the active site of the biocatalyst [25,108,109]. Each immobilization technique has advantages and disadvantages, which can preserve or alter enzyme conformation, thus affecting biocatalytic chemo-selectivity (ability to favor a reaction pathway) and specificity (ability to recognize a substrate). Therefore, it is essential to study the effect

of different immobilization techniques on the interaction between chitosan and enzymes and the biochemical reactions between biocatalysts and target pollutants.

5.2. Advances in Chitosan-Based Biocatalyst Immobilization for Wastewater Treatment

The advantages of chitosan as an enzyme support and the efficiency of oxidoreductases in degrading pollutants have led to more studies focused on chitosan-based biocatalyst immobilization for wastewater treatment. Therefore, Table 2 summarizes chitosan-based supports used for biocatalyst immobilization.

Table 2. Immobilized biocatalysts on chitosan-based supports for wastewater treatment.

Chitosan-Based Support	Biocatalyst	Target Pollutant	Major Achievement	Ref.
Chitosan beads	Manganese peroxidase	Textile effluent	97.31% of decolorization	[110]
Chitosan beads	Horseradish peroxidase	Textile dyes	Reduction in total organic carbon of 78.58; 84.03; 77.61; and 76.16% for remazol brilliant blue R; reactive black 5; Congo red; and crystal violet dyes, respectively	[111]
Dopamine-modified cellulose–chitosan composite beads	Horseradish peroxidase and glucose oxidase	Acridine	99.5% of aridine degradation	[112]
Chitosan beads	Laccase	Textile dyes	Reduction in chemical oxygen demand of 93.91; 93.47; 94.67; 94.94; 93.01; and 91.90% for drimaren red; drimaren black; drimaren yellow; drimaren turquoise; foron turquoise; and foron blue dyes, respectively	[113]
Chitosan and alginate composite hydrogel	Laccase	Bisphenol A	93.4% of degradation	[114]
Chitosan–clay composite beads	Laccase	Textile effluent	78% of decolorization	[115]
Chitosan beads	Laccase	Bisphenol A	100% of bisphenol A degradation	[116]
Calcium-modified chitosan–alginate	Laccase	Phenanthrene	94.4% of phenanthrene degradation	[117]
Chitosan–clay composite beads	Laccase	Textile dyes	Decolorization of 82%; 85%; and 69% for methyl red; remazol brilliant blue R; and reactive black 5 dyes, respectively	[3]

One of the significant drawbacks of non-immobilized enzymes is their lack of stability and the difficulty of separating them for reuse. Bilal et al. [110] developed chitosan beads to immobilize manganese peroxidase by cross-linking. The immobilized manganese peroxidase retained its catalytic activity for up to five cycles without significant reduction and maintained 49% of its initial catalytic activity after ten cycles. Therefore, the cross-linking provided the strong interactions required to form a protective shelter around the enzyme.

Bilal et al. [111] developed chitosan beads to immobilize horseradish peroxidase by encapsulation. The authors studied the effect of denaturant compounds on free and immobilized enzymes. The exposure of urea, EDTA, cysteine, Triton X-100, and 1,4-dithiothreitol promoted a reduction of more than 50% in the catalytic activity of the free enzyme. On the other hand, immobilized enzymes remained with 76.4, 92.6, 69.2, 71.4, and 74.6% of the original activity, respectively. Therefore, chitosan successfully formed a surface around horseradish peroxidase, avoiding the diffusion of these compounds into the enzyme's active site.

Co-immobilization of enzyme–enzyme or enzyme–redox mediator systems within the same support has been studied to improve enzyme efficiency and reduce toxic intermediates. Gu et al. [112] developed dopamine-modified cellulose–chitosan composite beads to immobilize horseradish peroxidase and glucose oxidase by chemical adsorption. The

authors aimed to address the limitations of using only horseradish peroxidase, which requires hydrogen peroxide as a hydrogen donor, raising environmental concerns. Both enzymes played an important role in the degradation of acridine, achieving 99.5% degradation compared to the use of immobilized enzymes separately, horseradish peroxidase (93.8%), and glucose oxidase (15.8%).

The enzymes acted synergistically in degrading harmful intermediates, increasing the reaction rate and cost-effectiveness, considering that glucose oxidase is less expensive. Initially, glucose oxidase oxidized β-D-glucose in the presence of oxygen, producing hydrogen peroxide. Subsequently, horseradish peroxidase reached an oxidized state by reducing hydrogen peroxide. 1-hydroxybenzotriazole (HBT), used as a redox mediator, was oxidized by horseradish peroxidase, resulting in the free radical HBT. Therefore, this radical acted in the oxidation of acridine by transferring a hydrogen atom, returning to its initial state.

Chen et al. [117] developed a Ca-modified chitosan–alginate system to immobilize laccase and soybean meal extract, as a redox mediator using an encapsulation method. The immobilized enzyme achieved a 94.4% degradation of phenanthrene, which was 20–30% higher than non-immobilized laccase and immobilized laccase beads without soybean meal extract. The authors suggested that the phenolic compounds in the soybean meal extract contributed to stronger interactions with laccase, resulting in intermediates with increased redox potential. These intermediates played an important role in degrading phenanthrene via a non-radical pathway, which was identified as the primary mechanism.

6. Adsorption of Emerging Pollutants Using Chitosan-Based Materials

Emerging pollutants in water bodies such as pharmaceutical residues, personal care products, microplastics, perfluoroalkyl and poly-fluoroalkyl substances, and agricultural pesticides, as well as persistent organic pollutants, pose environmental and health challenges because they persist in an unmetabolized state, remaining active and persistent even at low concentrations. Pharmaceutical residues, for example, cause microbes and pathogenic bacteria to induce antibiotic resistance, thus resulting in potential health risks to humans and other living organisms [118]. To ensure the safety and sustainability of water resources, the efficient removal of these contaminants from water should be a priority. In this context, studies on chitosan-based materials applied as adsorbents for these emerging pollutants will be presented below.

6.1. Pharmaceutical Adsorption with Chitosan-Based Materials

Pharmaceuticals are considered emerging contaminants due to their extensive use, continuous release into the environment, and physiological effects on organisms [119]. Chitosan contains functional groups that facilitate interactions with these types of molecules that are present in wastewater [120]. Studies of Shahrin et al. [121] have proposed a mechanism of interaction between chitosan and antibiotics such as rifampicin and streptomycin, as well as the anti-inflammatory ibuprofen. This mechanism involves intermolecular dipole–dipole forces and hydrogen bonding due to the amino (-NH_2) and hydroxyl (-OH) groups of chitosan acting as hydrogen donors.

In another study, Soares et al. [122] demonstrated that chitosan adsorbents form hydrogen bonds with non-steroidal anti-inflammatory drugs, such as diclofenac, naproxen, and ketoprofen. This is due to the -OH groups of trimethyl chitosan and the positively charged trimethylammonium units interacting with the carboxylate groups of the non-steroidal anti-inflammatory drugs. A case study conducted in Brazil detected high concentrations of caffeine in surface waters, of around 28,242.45 ng/L and 18,828.00 ng/L [55]. Other researchers [123] from the same country observed maximum caffeine concentrations ranging from 122 to 2769 ng/L in drinking water. Stefanowska [124] presented promising isotherms for caffeine adsorption with chitosan films and attributed the positive results to the presence of chitosan's functional groups, which facilitated intermolecular interactions with caffeine.

Ko et al. [10] investigated chitosan sponges (CSs) modified with graphene oxide (GO) and genipin (GP) to remove diclofenac, triclosan, and polystyrene microplastics. The GO/CS/GP sponges removed 97.7% of diclofenac and 98.1% of triclosan in 30 min. The removal percentage was 73% for nanoplastics and 41.5% for microplastics. The removal remained stable for five adsorption/desorption cycles for diclofenac and microplastics. The authors concluded that electrostatic and hydrogen bonding interactions dominated diclofenac adsorption, while triclosan adsorption involved hydrophobic, $\pi-\pi$, and hydrogen bonding interactions. The GO/CS/GP sponge showed potential for removing the studied pharmaceuticals, microplastics, and nanoplastics. Table 3 shows other studies related to pharmaceutical adsorption.

Table 3. Chitosan-based materials for pharmaceutical removal.

Chitosan-Based Support	Target Pollutant	Major Achievement	Ref.
Chitosan, activated carbon sodium hydroxide (KOH), and carbon dioxide (CO_2) composite beads	Caffeine	q_e = 39.53 mg/g for chitosan/CO_2 and $q_{e\,=}$ 121.90 mg/g in batch. q_e = 83.88 mg/g in fixed bed for chitosan/KOH. %R = 72.84% and 77.22% for chitosan/activated carbon, and their activation in the composites increased efficiency by approximately 4 times.	[125]
Activated carbon/chitosan beads	Diclofenac	q_e = 99.29 mg/g.	[126]
Magnetite/chitosan and *Chlorella vulgaris* (MCC) or *Arthrospira platensis* (MCA)	Tetracycline (TC), ciprofloxacin (CIP), and amoxicillin (AMX)	q_e = 744.3 mg/g (TC), 327.8 mg/g (CIP), 125.6 mg/g (AMX) for MCC; q_e = 753.2 mg/g (TC), 339.0 mg/g (CIP), 135.4 mg/g (AMX) for MCA.	[127]
Chitosan nanoparticles	Ethinylestradiol	q_e = 5.79 mg/g and %R = 93.7% of degradation.	[128]
$NiFe_2O_4$–COF–chitosan–terephthalaldehyde nanocomposite film (NCCT)	Tetracycline (TC) and cefotaxime (CTX)	q_e = 388.52 mg/g for TC and q_e = 309.26 mg/g for CTX.	[129]

$q_{e\,=}$ maximum adsorption capacity; %R = percentage removal.

6.2. Personal Care Product Adsorption with Chitosan-Based Materials

Personal care products include disinfectants, shampoos, toothpastes, soaps, perfumes, and sunscreens. Emerging pollutants commonly found in these products are polydimethylsiloxane, nano titanium dioxide, butylated hydroxyanisole, UV filters, insect repellents, microplastics, butylated hydroxytoluene, triclosan, and triclocarban. Fragrances (galaxolide, tonalide, celestolide, and phantolide) and preservatives (diethyl phthalate, nano zinc oxide, benzophenone, parabens, and octocrylene) are also present. Among these, triclosan, a synthetic antimicrobial, has been identified as a significant environmental contaminant. Its residues, even at low concentrations, can accumulate in the fatty tissues of fish, affecting marine biodiversity [130]. The removal of triclosan was reported by Almeida et al. [131] using graphene oxide, magnetic chitosan, and organophilic clay as adsorbents, achieving a removal efficiency of 98% and an adsorption capacity of 40.7 mg/g, even at low concentrations (0.140 mmol/L). Vakili et al. [132] conducted an adsorption test to remove tonalide using a fixed bed of chitosan/zeolite, with an initial concentration of 1.636 mg/L, achieving an 86.3% removal.

On the other hand, the authors Kavianinia et al. [133] used the contaminant benzophenone-3,3,4,4-tetracarboxylic dianhydride (BTDA) copolymerized with chitosan to produce hydrogels and adsorb heavy metals. This was the authors' strategy for the reuse of BTDA; however, other applications that minimize environmental impacts should be explored. In the same context, Machado et al. [134] used chitosan with triclosan to create antibacterial adhesive resins, demonstrating reliable physical and chemical properties and maintain-

ing high antibacterial activity over time. These results suggest that similar approaches, such as the reuse of contaminants in combination with chitosan in support of catalysis or adsorption, could be used. This highlights the need for further research on this issue.

6.3. Microplastic Adsorption with Chitosan-Based Materials

In 2020, global plastic production reached 367 million tons [135], and projections indicate a 29% increase by 2028 [14]. In the aquatic environment, exposure to sunlight, weathering, mechanical wear, and the action of microorganisms break down plastics, resulting in the formation of microplastics (MPs) and nanoplastics (NPs) [15,136]. MPs, ranging in size from less than 5 mm to more than 0.1 µm, and NPs, with sizes smaller than 100 nm or 0.1 µm, are considered toxic emerging pollutants. This is due to their peculiar physicochemical characteristics, chemical stability, and non-biodegradable nature [15,137].

MPs are prone to aging, resulting in a rough surface and surface properties charged with functional groups such as –COOH and $–NH_2$. Due to their high surface area, small size, and strong hydrophobicity, MPs can adsorb toxic contaminants such as antibiotics, pharmaceuticals, heavy metals, pesticides, plasticizers, and pathogenic organisms [12]. Notably, protonated chitosan enables the selective adsorption of negatively charged MPs, making it an attractive alternative for removing these contaminants [11].

In this context, in the study by Sun et al. [11], sponges composed of chitin/graphene oxide (GO)/oxygen-doped carbon nitride ($O\text{-}C_3N_4$) showed great potential for the removal of microplastics due to the strong interfacial interactions between the graphene oxide and chitin chains, which significantly improved the mechanical properties of the material. Chitin-based sponges removed 71.6–92.1% of microplastics (carboxylate-modified polystyrene (PS-COOH), amine-modified polystyrene ($PS\text{-}NH_2$), and polystyrene (PS)) at a concentration of 1 mg/L in water. The sponges are reusable for up to three cycles due to their compressibility, good biocompatibility, and biodegradability in natural soil. Complementing this approach, Zheng et al. [138] developed a strategy to create magnetic chitosan aerogels enhanced with polydopamine (PDA-MCSs). These aerogels showed high efficiency in removing polyethylene terephthalate (PET) microplastics from water, achieving a removal efficiency of 91.6%. Even after three cycles of reuse, they maintained an efficiency of 83.4%, demonstrating the potential of these materials in environmental remediation. Implementing these technologies offers a sustainable and efficient solution to mitigate environmental impacts, protecting aquatic ecosystems and human health.

6.4. PFASs Adsorption with Chitosan-Based Materials

Perfluoroalkyl and polyfluoroalkyl substances (PFASs) are persistent organofluorine compounds characterized by partially or entirely substituting hydrogen atoms with fluorine in their carbon chains. PFASs have been widely used as surfactants in industrial and commercial applications, such as cosmetics, lubricants, paints, firefighting foams, non-stick cookware, and paper products. These substances are emerging contaminants due to their environmental persistence, toxicity, and bioaccumulation potential [139–141].

Although adsorbents like granular and powdered activated carbon, ion exchange resins, montmorillonite, and graphene oxide have been studied for the removal of PFASs, these materials face challenges such as their low adsorption capacity, prolonged times to reach equilibrium, low selectivity, and difficulties in structural modification [141].

Granular activated carbon, while effective at removing long-chain PFASs in pilot-scale applications, proves ineffective and with slow kinetics for short-chain PFASs. Additionally, its large-scale application is hindered by the frequent need for reactivation or replacement, especially in water containing natural organic matter, making the process less efficient and more costly [142].

With its protonated $-NH_2$ groups in an acidic solution, chitosan can attract the anionic groups (COO^- and $SO_3{}^-$) of PFASs through electrostatic interactions. Additionally, its active amino and hydroxyl groups make it an efficient matrix for immobilizing covalent organic frameworks (COFs), which enhances the adsorption capacity [139,142]. Studies

have indicated that chitosan-based materials are effective in removing these pollutants. He et al. [143] synthesized a fluoro-functionalized covalent organic framework coated with chitosan, which showed a higher efficiency in PFAS removal due to the increased hydrophilicity provided by chitosan.

Shahrokhi et al. [141] developed crushed chitosan spheres (CBs) grafted with polyethyleneimine (GCBs) to remove four types of PFASs—perfluorooctanoic acid (PFOA), long-chain perfluorooctane sulfate (PFOS), perfluorobutanoic acid (PFBA), and short-chain perfluorobutano sulfate (PFBS). Adsorption tests, conducted at pH 7.0 to simulate environmental conditions, showed that chitosan rapidly adsorbed long-chain PFASs, reaching equilibrium in 5 min, while short-chain PFASs took up to 3 h. After four adsorption/desorption cycles, the removal capacity for long-chain PFASs was maintained. Still, the efficiency for short-chain PFASs decreased after the first cycle, likely due to their hydrophobic nature. Chemical modifications to the grafted chitosan spheres (GCBs) significantly increased adsorption capacity, resulting in a greater affinity and selectivity for efficiently removing PFASs of both chain lengths.

6.5. Glyphosate Adsorption with Chitosan-Based Materials

Glyphosate (N-(phosphonomethyl)glycine) is a systemic and non-selective organophosphorus herbicide that is widely used in agricultural and non-agricultural contexts. The main concern associated with glyphosate is its infiltration into effluents, waterways, and aquifers. This contamination is persistent and cumulative, posing a risk to the survival of aquatic ecosystems, as well as flora, fauna, and environmental matrices such as water and soil [144]. Exposure to glyphosate, whether direct or indirect, has been associated with genotoxic, nephrotoxic, and hepatotoxic effects, and the International Agency for Research on Cancer (IARC) has classified it as a "probable carcinogen" [145].

In the literature, magnetic nanosorbents, such as magnetite (Fe_3O_4) or maghemite (γ-Fe_2O_3), have been synthesized due to their desired functionality for magnetic separation and contaminant adsorption [146]. However, iron oxide nanoparticles tend to lose reactivity due to oxidation, so they can be coated with chitosan and its derivatives to improve their stability. On the other hand, the glyphosate molecule has at least two functional groups—phosphonic acid and carboxylic acid—which allow for the formation of stable bonds with the surface of metal oxides. Thus, the binding between glyphosate and the oxide can occur through covalent solid interaction with the phosphonic acid and weaker covalent and/or non-covalent interactions with the carboxylic acid. These interactions contribute to glyphosate's efficient adsorption on metal oxides' surfaces [147].

The study by Briceño and Reinoso [148] analyzed a simple magnetic nanocomposite based on graphene, chitosan, and $CoFe_2O_4$ nanoparticles as an adsorbent for glyphosate remediation in water. The combination of graphene with chitosan enhances its biocompatibility and hydrophilicity and facilitates dispersion in aqueous solutions. The study showed that the maximum glyphosate removal (48%) was achieved with 10 ppm of $CoFe_2O_4$ nanoparticles due to stable dispersion, increased surface area, and number of active sites, with graphene contributing oxygen-containing functional groups that enhanced adsorption and electrostatic/ionic interactions.

Another study conducted by Aksu Demirezen et al. [147] investigated magnetic hydrogel spheres of chitosan/calcium alginate (MCAH) with different contents of iron oxide nanoparticles (MIONPs), finding that glyphosate removal increased with MIONP content, reaching up to 43% after 120 min. Although the adsorption rate increased by 13% with more MIONPs, the final removal efficiency remained similar across the three compositions tested. The increase in MIONPs affects the surface area and porosity of the beads, limiting the adsorption capacity. This is because the nanoparticles occupied more space in the hydrogel matrix, resulting in fewer available pores and, consequently, a smaller surface area, negatively affecting the adsorption qualities. However, increasing the MIONPs improved magnetization, making removing the beads with a magnet easier. However, the water content in the nanoparticles affects the mixing, leading to the formation of elongated hydrogels

instead of spheres, which limits the integration of more MIONPs into the structure and affects the removal efficiency. Therefore, the authors suggest improving the composition and manufacturing process to improve glyphosate removal efficiency.

7. Conclusions

Chitosan modification has emerged as a promising solution for improving catalytic and adsorption processes in wastewater treatment due to its versatility and unique functional properties. Considered one of the most attractive subclasses of functional materials, modified chitosan offers excellent potential for environmental applications. Nitrogen doping, for example, is a technique that improves electronic properties and creates new active sites, making chitosan a highly efficient material in photocatalytic and electrocatalytic reactions. Chemically modified chitosan by cross-linking with agents such as glutaraldehyde increases stability and the ability to immobilize catalytically active species. Combining chitosan with metal nanoparticles or enzymes creates materials that perform catalytic and adsorption functions, providing efficient pollutant removal in multiple treatment cycles. These improvements increase the degradation capacity of pollutants such as persistent organic compounds, textile dyes, heavy metals, pharmaceuticals, microplastics, and perfluorinated compounds. Furthermore, using chitosan supports in photocatalytic systems with catalysts such as TiO_2 and silver nanoparticles has proven to be a promising strategy for degrading resistant pollutants. A notable example is the development of chitosan-based bifunctional catalysts, such as those combining Cu_2O and Fe_3O_4. These catalysts play a dual role in the treatment of pollutants, facilitating the oxidation of organic Cr(III) complexes and inhibiting the accumulation of Cr(VI) using the Fenton reaction. The synergy between the nanoparticles and the chitosan matrix not only improves the catalytic efficiency, but also promotes the stability and reusability of the system, expanding its potential application in sustainable wastewater treatment. These advances demonstrate that when modified and incorporated into catalytic and adsorption systems, chitosan offers an effective and sustainable solution for wastewater treatment and paves the way for developing new high-performance functional materials capable of addressing global environmental challenges with greater efficiency and lower environmental impact.

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