

Chitosan Polymer and Composite Hydrogels: An Emerging Approach for Microplastic Elimination from Water

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Abstract

Hydrogel is a polymer network which can retain huge volumes of water but still retains its shape because of its special chemical formula. Recently, hydrogels have become an object of great interest for environmental remediation due to the possibility of controlling their physicochemical properties in the case of removal of microplastics. The current work reviews and analyzes the efficiency of application of conventional coagulation systems based on chitosan and new hydrogel-based systems for microplastic separation. Chitosan is a degradable natural polymer which provides successful microplastic separation using electrostatic charge neutralization and interparticle bridging with removal efficiency from 68.3% to 99.9%. Moreover, a three-dimensional pGel\@IPN hydrogel system based on chitosan, polyvinyl alcohol, and polyaniline showed up to 95% removal of polyvinyl chloride and polypropylene microplastics at around pH 6.5, along with retention of its effectiveness in multiple reutilization cycles. Besides, the photocatalytic degradation efficiency of copper-substituted polyoxometalate (Cu-POM) hydrogel systems are very high when used under ultraviolet radiation; additionally, the unique property of such hydrogels is their conversion into valuable carbon nanomaterials following utilization. From the comparative study, it can be concluded that although chitosan-coagulation methods are cheap and effective in water treatment applications, hydrogels are more reusable, environmentally friendly, and can be utilized for removal and degradation of microplastics simultaneously.

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INTRODUCTION

Indeed, hydrogels are among the most versatile classes of materials developed today. They are constituted by the structures containing hydrophilic materials. Such materials absorb large amounts of water without changing their shapes. Hydrogels are very important in the modern world. The combination of their characteristics – such as a high degree of water content, variable mechanical behavior, biocompatibility, and stimuli-responsiveness – make them perfect materials to meet the challenges of the 21st century [1, 2].

Hydrogel evolution history can be sketched back to over an era ago. Hydrogel as a colloidal system formed from inorganic salts was first mentioned in 1894. Nonetheless, the current

definition of hydrogel as a network of polymers having the capacity to swell without dissolving arose through several decades after this time. The earliest notable artificial hydrogel formulation involved cross-linking of poly(vinyl alcohol) with formaldehyde in 1949 resulting in development of a product dubbed Ivalon – hence the birth of hydrogels for biomedical purposes [1, 2].

This study reviews and compares the performance of conventional chitosan-based coagulation systems and advanced hydrogel-based materials for microplastic removal. Chitosan, a biodegradable natural polymer. The issue of pollution is harmful to the planet as small plastic particles are present in the water we consume and in the surrounding water sources. Microplastics exist in our water. This is a significant problem. The study checks out what chitosan/PVA/PANI hydrogels are made of how they are made and how they can soak up microplastics. It also looks at what other researchers have found out about using these hydrogels to clean up water. The goal is to see if chitosan/PVA/PANI hydrogels can be a way to get rid of microplastics from water without hurting the environment. There are still some problems that need to be solved, and more research is needed to make this technology better. Through analysis of synthesis procedures, compositions, performance and applications, this research paper presents an exhaustive insight into advanced hydrogels.

BACKGROUND AND MOTIVATION

The term microplastics describes plastic pieces smaller than 5 mm in diameter and either purposely created or derived from further degradation of other plastic materials. Microplastics turned out to be one of the most widespread pollutants in our world.

Industrial artificial plastics have been used since the beginning of the twentieth century after the invention of Bakelite in the early decades of the 1900s. In recent decades, the production of such materials has been growing rapidly, going from about 1.5 million metric tons at the start of the post-war period to an incredible 368 million metric tons in 2019 [2, 3]. It became clear that plastics are flexible, durable, and cheap in manufacturing. Various application of removing microplastic shown in Figure 1.

Scientific reports concerning the presence of microplastics in the environment are not numerous and appeared in the 1970s after scientists discovered the smallest plastic fragments in sea water and marine life [1, 2, 4]. The very term microplastics appeared relatively recently in 2004 [1, 2]. There are

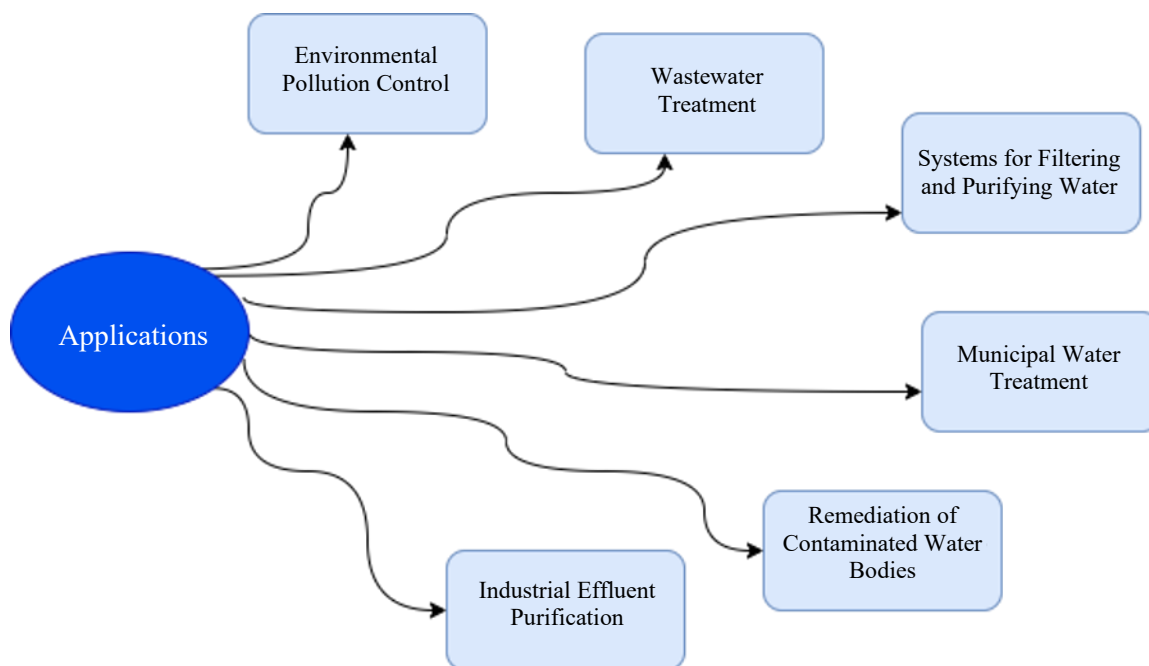


Figure 1. Illustrate the various application of removing microplastic.

two forms of microplastics. Primary microplastics are produced specifically in microplastic form and can be exemplified by microbeads utilized in cosmetics and nurdles used in industrial production. On the other hand, secondary microplastics originate because of mechanical and photochemical decomposition of plastic products via processes such as weathering and abrasion. Such microplastics have been found not only in marine sediments, but also in freshwaters, soil, food, and even human tissue samples, raising serious doubts about their environmental and health implications. Some of the issues that have been identified include bioaccumulation, reduced food consumption among marine organisms, genetic modifications, and respiratory hazards [3, 5].

Traditional methods for water purification are unable to efficiently address the problem of microplastics due to the very nature of such contaminants – their tiny size and wide variety in terms of chemistry and spatial occurrence. The utilization of advanced hydrogels seems to provide a more effective solution. In this research paper we are considering two methods for removal of microplastics.

CHITOSAN, POLYVINYL ALCOHOL, POLYANILINE (IPN)

Chemical Composition

The following chemical compounds are found in the hydrogel with microplastic absorber having a triple network of IPN, 3D pGel@IPN, which is a high-tech composite having a triple interpenetrating polymer network hydrogel that contains polyoxometalate nanoclusters and is mainly used in the adsorption and photodegradation of microplastics in polluted water.

- *Chitosan (CS)*: Bio-polymer derived from chitin; functionality stems from reactive amine groups that allow pollutant absorption, biodegradability, and anti-bacterial effect of the material.
- *Polyvinyl Alcohol (PVA)*: Synthetic polymer which helps increase hydrogel stability, moisture-retention properties and overall strength.
- *Polyaniline (PANI)*: Conductive polymer which promotes pollutants' oxidation as well as hydrogel surface properties.
- *Cross-Linker (For Example, Tetraethylene Glycol Dimethacrylate, TEGDMA)*: Allows to bind polymer chains through a covalent bond.
- *Photoinitiator*: Under the action of UV light, initiates radical polymerization (for example, 2,2-Dimethoxy-2-phenylacetophenone, DMPAP).
- Nanoclusters of copper-containing polyoxometalate (Cu-POM); additional components that make it possible to perform microplastic degradation under UV irradiation.

Preparation of the Pre-Gel Solution

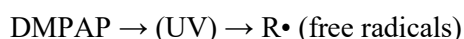
The first step involves dissolving chitosan in an aqueous acetic acid solution to obtain a homogeneous solution of chitosan (CS_{sol}). The next step involves dissolving polyvinyl alcohol in distilled water by heating to obtain a PVA solution. Polyaniline can be obtained through the oxidative polymerization of aniline in hydrochloric acid solution:

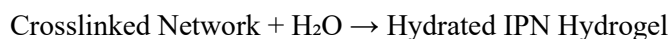
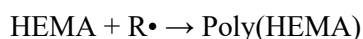


The three polymer solutions are then combined in controlled proportions along with the crosslinker (TEGDMA) and photoinitiator (DMPAP) to form the pre-gel mixture.

Free Radical Polymerization and Crosslinking

On being exposed to UV rays, the photoinitiator produces active free radicals, which initiate the crosslinking reaction within the polymer chains. Crosslinker TEGDMA forms links with existing double bonds present within the mixture, ensuing in the formation of a three-dimensional matrix. The reactions proceed as follows:





Chemical Crosslinking via Glutaraldehyde or MBA

Instead, in conjunction with UV cross-linking, cross-linking can also be done chemically with the help of glutaraldehyde or methylenebisacrylamide (MBA), thus forming an interpenetrating network as follows:



In this way, the formed hydrogel will undergo hydration at controlled temperatures and time parameters, leading to the establishment of a porous system that can trap microplastics.

Performance and Characterization

Both the 3D pGel@IPN hydrogel material and the Cu–POM loaded version have been tested under various environmental conditions.

Table 1. Behaviour observation of microplastic-absorbing hydrogels.

Hydrogel type	Composition	Microplastic types removed	Efficiency	Conditions tested	Reusability
3D pGel@IPN	Chitosan, Polyvinyl Alcohol, Polyaniline	Polyvinyl Chloride (PVC), Polypropylene (PP)	Up to 95% removal at pH ~6.5	Various pH levels, temperatures, and concentrations	Effective for up to 5 cycles.
Copper Substitute POM	Infused with Cu–POM nanoclusters	PVC and PP	High degradation efficiency with UV light	Tested under different environmental conditions	Can be repurposed into carbon nanomaterials after use.

The important performance characteristics are highlighted in the following (Table 1).

The 3D pGel@IPN material shows up to 95% removal capacity for PVC and PP microplastics at pH ~7.0 and can be used up to 5 cycles of adsorption/desorption without losing functionality. The Cu–POM composite variant extends performance further by enabling photocatalytic degradation of captured microplastics under UV irradiation, with the spent material subsequently repurposable as carbon nanomaterials, thereby establishing a zero-waste approach to microplastic remediation [6–10].

USING CHITOSAN AS A COAGULANT FOR THE REMOVAL OF MICROPLASTICS OF POLYETHYLENE (PE) FROM THE WASTEWATER

In this research, we reported the removal of polyethylene (PE) microplastics from artificial wastewater by the utilization of chitosan as a coagulant. Due to the abundance of PE microplastics in the environment, it was selected for use in this study as a model microplastic. The effect of chitosan concentration and coagulation pH on the removal of microplastics was assessed. Furthermore, chitosan is believed to coagulate with microplastics [11–13].

Methodology

Chitosan was prepared in 0.1 N HCl for the purpose of preparing chitosan stock solution (3000 mg/L). Preparation of a 200 mg/L solution required PE microplastics (34–50 µm) to be sonicated for one hour in distilled water. 0.1 N HCl or NaOH was used to adjust the pH of 500 mL of microplastic solution from 5–9. Doses of 25–150 mg/L of chitosan were added, and coagulation process was done using Jar Test involving quick mixing at 250 rpm for 2 min and slow mixing at 60 rpm for 30 min.

The solution was left to settle down for 60 min. The efficiency of removal of microplastics was evaluated based on the turbidity before and after treatment. Due to the electrostatic interaction between the positive charges of chitosan and negative charges of PE microplastics, the process of flocculation was facilitated. The efficiency of microplastics removal was maximum at pH 6 (77.35%).

Influence of Coagulant Dosage on the Microplastic-Removal Rate

The low coagulant dosage led to low efficiency of removal because the insufficient amount of coagulating agent was in the water.

The improvement of microplastic removal was caused by an increase in coagulant dosage, with the maximal efficiency of 81.5% obtained at the coagulant dosage of 100 mg/L. However, a decrease in coagulation efficiency occurred when the coagulant dosage increased up to 150 mg/L (Table 2 and Figure 2) [14–19].

Table 2. The comparison of coagulation efficiency of chitosan in our study and in literature.

Coagulant	Microplastic type	Removal efficiency	Dosage	pH	Reference
Chitosan	Polyethylene (PE) (200 mg/L)	81.5%	100 mg/L	6.0	D. W. Skaf [14].
FeCl ₃ + Chitosan	Polystyrene (25 mg/L)	99.8%	FeCl ₃ : 2 mg/L; Chitosan: 7 mg/L	6.3	Raj et al. [16].
Chitosan	Beringin River water sample (21 mg/L)	68.3%	30 mg/L	8.1	Putranto et al. [15].
Polyaluminium Chloride + Chitosan	Polyethylene terephthalate (PET) (100 mg/L)	90.0%	PAC: 50 mg/L; Chitosan: 50 mg/L	7.0	Huang et al. [17].
Al ₂ (SO ₄) ₃ + Chitosan	Low-density polyethylene (LDPE) (40 mg/L)	92.1%	Al ₂ (SO ₄) ₃ : 20 mg/L; Chitosan: 1 mg/L	5.0	Eamrat et al. [18].
Hydrophobic Chitosan	Polyethylene terephthalate	99.9%	20 mg/L	7.5	He et al. [19].

Microplastic Removal Efficiency with Different Coagulants

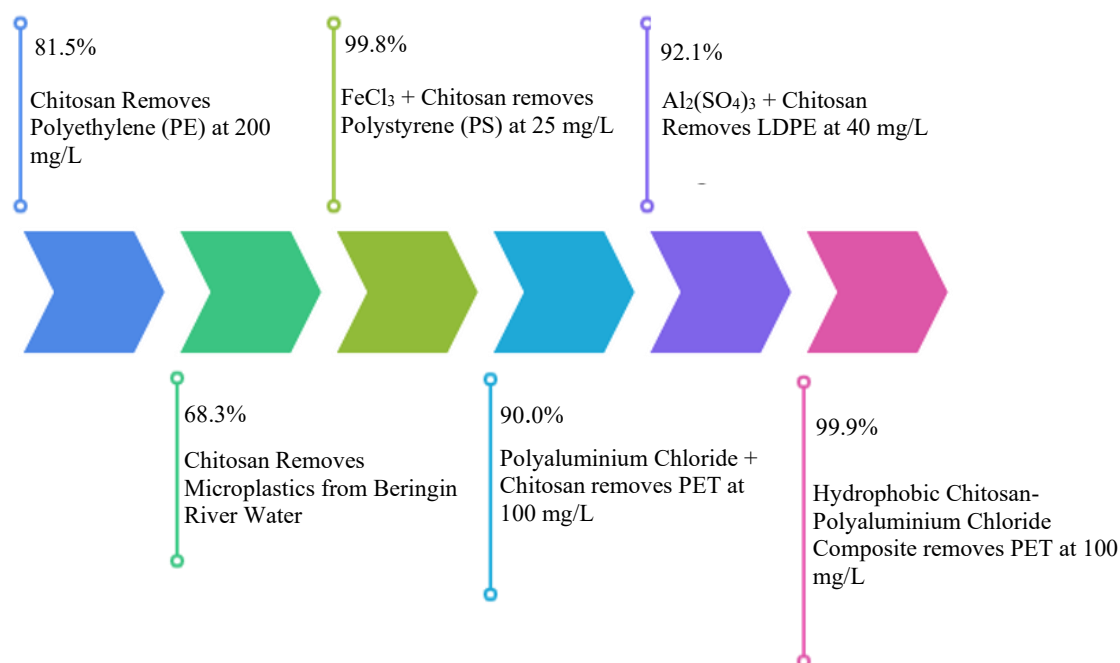


Figure 2. Coagulation efficiency of chitosan.

Summary of the Process

The charge neutralization and bridging of the polymers are the two major processes involved in the removal of the microplastics through chitosan. This is because the negatively charged microplastic particles get attracted to the positively charged chitosan molecules, and in turn destabilized due to the neutralization process. Further, the polymer chain nature of the chitosan results in the formation of large flocs by joining particles together through bridging. But due to the steric repulsion between the polymer chains on the particle surface, the formation of large flocs can become unstable.

The efficiency in the microplastic removal in this study is lower than some previous studies. The absence of inorganic coagulants, such as FeCl_3 , $\text{Al}_2(\text{SO}_4)_3$, or poly aluminum chloride, which are usually used together with chitosan and have an additive effect that improves the coagulation process, may explain this difference. On the other hand, this study performs better in microplastic removal efficiency compared to the study by Putranto et al. due to differences in water samples.

DISCUSSION

The chitosan-based coagulation technology, via interparticle bridging and electrostatic charge neutralization, thus producing flocs and settling, can remove microplastics primarily. The maximum removal rates (up to 99.9%) can be achieved when chitosan is added into the reaction mixture along with some inorganic coagulants such as ferric chloride, alum, and polyaluminum chloride with synergetic coagulation effect.

In turn, the adsorption and degradation techniques are characteristic of hydrogels-based materials, for instance, Cu-POM composites and 3D pGel@IPN. The key features of such technologies are reusability, low sludge production, and microplastic degradation rather than separation although they are quite similar to the chitosan technology (up to 95%). Particularly attractive is the Cu-POM hydrogel because it can break down microplastics with UV-light and then use them to synthesize nanomaterials based on carbon (Table 3).

Table 3. Comparison between hydrogel-based microplastic removal systems and chitosan-based coagulation systems.

<i>Parameter</i>	Chitosan-based coagulation (previous Table)	Hydrogel-based systems (current Table).
<i>Material Type</i>	Natural biopolymer coagulant, often combined with FeCl_3 , $\text{Al}_2(\text{SO}_4)_3$, or PAC	Advanced hydrogel/composite adsorbent and photocatalytic materials.
<i>Removal Mechanism</i>	Charge neutralization, flocculation, and polymer bridging	Adsorption, entrapment within porous network, and photocatalytic degradation.
<i>Microplastics Targeted</i>	PE, PET, PS, LDPE	PVC and PP.
<i>Removal Efficiency</i>	68.3–99.9%	Up to 95% (3D pGel@IPN); high degradation efficiency (Cu-POM hydrogel).
<i>Optimal pH</i>	Typically, 5–8, maximum removal around pH 6	Around pH 6.5 for 3D pGel@IPN; broader environmental conditions for Cu-POM.
<i>Dosage Requirement</i>	1–100 mg/L depending on coagulant combination	No specific dosage reported; material acts as reusable adsorbent.
<i>Need for Auxiliary Chemicals</i>	Often enhanced by metallic coagulants (FeCl_3 , PAC, $\text{Al}_2(\text{SO}_4)_3$)	Generally, operates independently without additional coagulants.
<i>Reusability</i>	Limited; chitosan flocs are typically discarded after use	Reusable up to 5 cycles (3D pGel@IPN); Cu-POM can be converted into carbon nanomaterials.
<i>Environmental Impact</i>	Biodegradable and environmentally friendly, but sludge generation occurs	Sustainable, reusable, and capable of reducing secondary waste generation.
<i>Application Scale</i>	Suitable for conventional water and wastewater treatment processes	More suitable for advanced remediation and circular economy applications.
<i>Best Reported Performance</i>	99.9% removal using hydrophobic chitosan-PAC composite	95% removal using 3D pGel@IPN; additional degradation capability with Cu-POM hydrogel.

Hydrogel systems can be considered more environmentally friendly and reusable options with the ability to degrade the microplastics, thus, becoming promising options for the next generation of water purification techniques, whereas chitosan coagulation may be used for the industrial water treatment due to its simple and cheap nature.

COMPARATIVE ANALYSIS OF COAGULATION AND HYDROGEL METHODS FOR MICROPLASTICS REMOVAL

One rationale behind the inclusion of both the coagulation- and hydrogel-based systems into this literature review is not to create a direct comparison between the efficiencies of the two technologies quantitatively but rather provide some case examples of the various treatment technologies used for the removal of microplastics.

It is crucial to emphasize the difficulty of making any direct comparison of the removal efficiencies of the two systems under consideration because of different physicochemical principles underlying these methods. Namely, microplastics in coagulation-based methods are removed by charge neutralization, flocculation, and sedimentation. In other words, removal efficiency in this case means the decrease in the concentration of suspended microplastic particles after the process of coagulation and sedimentation. Hydrogels, in turn, remove microplastics via adsorption and physical entrapment, while some also perform photocatalytic degradation.

In addition, the two techniques are tested under different experimental parameters such as pH, contact time, type and size of the microplastics, concentration, dose, and characteristics of the water matrix. Coagulation can be considered as a kinetic process where there is destabilization of particles followed by solid/liquid separation whereas adsorption of hydrogels is controlled by the adsorption equilibrium. Hence, identical percentage removal cannot be considered to reflect identical efficiency in both techniques.

MECHANISMS OF ADSORPTION AND SELECTIVITY OF CHITOSAN/PVA/PANI INTERPENETRATING POLYMER NETWORKS

The selectivity of chitosan/PVA/PANI interpenetrating polymer network (IPN) towards PVC and PP is probably achieved via a combination of electrostatic interactions, hydrogen bonding, hydrophobic interactions, and physical entrapment and not because of any one particular adsorption mechanism. Electrostatic interactions due to the presence of positively charged amino groups in chitosan can take place with polar and negatively charged PVC. Hydrophobic domains in PANI are more likely to interact with nonpolar PP. The porous nature of hydrogels improves the efficiency of microplastics capture physically. Unfortunately, the available study lacks enough evidence to prove selectivity of adsorption by reporting the overall removal efficiencies only.

DIFFERENTIATION BETWEEN PHOTOCATALYTIC DEGRADATION AND FRAGMENTATION OF CU-POM HYDROGELS

We concur with the opinion that reduction in the amount of microplastics does not automatically translate to complete degradation. In the case of Cu-POM hydrogels, the reduction in the amount of microplastics might not be because of total degradation to carbon dioxide and water but rather fragmentation into smaller forms such as nanoplastics, nanoplates, or oligomers. Since there was no report on carbon mass balance, total organic carbon removal, carbon dioxide formation or even identification of the products formed, the results obtained can only be considered as apparent degradation efficiencies.

IMPACT OF COMPONENTS IN THE WASTEWATER MATRIX ON CHITOSAN-BASED COAGULATION PERFORMANCE

While recognizing that the coagulation experiment was carried out using artificial wastewater with clearly defined PE microplastics at constant pH conditions via jar testing, the presence of dissolved organic matter (DOM), competing colloids, multivalent ions, and different ionic strengths in wastewater can greatly impact the performance of chitosan-based coagulation. Multivalent ions

can act to either increase or decrease coagulation performance by altering surface charge and bridging mechanisms, whereas DOM and colloids can compete for chitosan binding sites and reduce charge neutralization. Likewise, ionic strengths can impact the electrical double layers around the particles and influence floc formation. As a result, removal efficiencies measured in laboratory settings may not directly correlate with those in complex wastewater matrices. The findings presented here, therefore, need to be considered only as proof-of-concept evidence of the use of chitosan in removing microplastics from wastewater.

ROLE OF PH IN CONTROLLING THE REMOVAL OF MICROPLASTICS BY CHITOSAN AND HYDROGEL

The optimum pH of around 6.0–6.5 could be explained because of an optimal combination of physical and chemical properties of chitosan, microplastics, and the hydrogel. Under these conditions, chitosan is highly protonated but maintains enough positive charges for electrostatic interactions with negatively charged microplastic surfaces and proper polymer bridging. Lower pH would lead to overprotonation of the chitosan, which might increase the solubility of the polymer and destabilize the flocs, whereas higher pH leads to a lower degree of protonation and hence weaker charge neutralization and adsorption. In the case of the chitosan/PVA/PANI hydrogel, the effect of pH is also associated with hydrogel swelling, adsorption site availability, and the oxidation level of the polyaniline, which influence the interactions between pollutants and hydrogel. Therefore, it could be assumed that the optimum pH value is because of the maximum electrostatic attraction, polymer bridging, hydrogel swelling, and adsorption. Unfortunately, no information on the kinetic and thermodynamic properties was given in the original research paper; hence, the above explanation is only mechanistic in nature.

FUTURE PROSPECT

The future research work related to the treatment of microplastics should focus on the development and design of hybrid systems using chitosan, hydrogels, nanomaterials, and photocatalysts to enhance the efficiency of both adsorption of microplastics and their decomposition. The principles of the physicochemical process involved in the removal process – like the selectivity of adsorption, neutralization of charge, polymer bridge bond formation, interactions of hydrogels and microplastics, and effect of pH – should be studied in more detail. Moreover, thermodynamic and kinetic studies are needed to correlate the properties of the material and the removal efficiency of microplastics.

In addition, the efficiency of such hybrid systems in wastewater containing dissolved organic matter, multivalent ions, competing colloids, and various ionic strengths should be investigated, as all these factors have great effects on the mechanism of removal. Finally, testing procedures should be developed for the study of such systems in presence of different types of microplastics.

The future work should consider the requirement to distinguish between mineralization and fragmentation by including the carbon mass balance, TOC, CO₂ generation, and degradation product analysis in their research in photocatalytic treatment systems.

Moreover, the efforts should be made to develop reusable and responsive to stimuli hydrogels, to conduct pilot testing, and to employ a multifunctional technology based on coagulation, adsorption, membrane, and photocatalysis techniques. Finally, the future work should include a life cycle assessment, ecotoxicity evaluation, and resource recovery to ensure that the technologies developed for microplastic elimination have sustainable environmental and economic impacts.

CONCLUSION

According to the reviewed literature, the use of chitosan-based coagulation techniques and hydrogels as a material appears to be a promising solution for the microplastic removal in aquatic systems. Chitosan is a highly efficient sustainable coagulant that can remove up to 99.9% and 68.3% of microplastics via charge neutralization, flocculation, and interparticle bridging. Higher efficiency of chitosan application is often achieved by applying it as a constituent of the combination with such inorganic coagulants as FeCl₃, Al₂(SO₄)₃, and polyaluminium chloride.

Also, the sophisticated hydrogels – like 3D pGel@IPN and Cu–POM-based hydrogels – possess the advantages of exhibiting high adsorption capacity, being reusable, and even possessing the capacity for photocatalytic degradation of microplastics. However, any comparison in terms of removal efficiency between the coagulation process and the hydrogels is not suggested since both these technologies have different working mechanisms and conditions.

Moreover, the efficiency of both technologies is usually estimated through the outcomes of laboratory experiments carried out based on artificial microplastics and aqueous matrices. Such parameters as dissolved organic matter, ionic strength, competing colloids, and multivalent ions, which can be found in natural wastewater, can have a significant impact on the process of removal. The same applies to photocatalytic hydrogel systems; their degradation efficiency should be considered very carefully without mineralization and carbon balance being provided.

In general, the technology involving biopolymers, nanomaterials, and functional hydrogels appears to be quite a promising direction in the field of microplastic removal. Future research dedicated to clarifying the mechanism of action and verification of the findings in more real-life conditions is essential.

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