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Removal of Microplastics from Water by Bare Magnetic Nano-γ-Fe_2O_3 under Ultrasound

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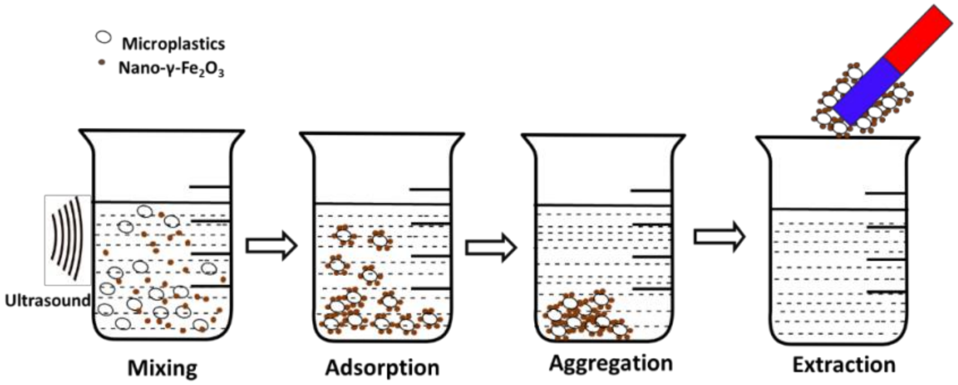
Abstract

Microplastics (MPs) have been widely detected in aquatic environments as a new type of pollutant. There is an urgent need to develop effective methods for removing microplastics from water. This study establishes a novel method utilizing commercial bare nano-γ-Fe_2O_3 to efficiently remove microplastics from water under ultrasound. Under ultrasonic vibration, the microplastics rapidly adsorbs bare nano-γ-Fe_2O_3 and aggregate consequently. Microplastics with nano-γ-Fe_2O_3 can be efficiently removed using a magnet. This method exhibits high removal rates (up to 96%) for four common types of microplastics (polyethylene, polypropylene, polystyrene and polyethylene terephthalate) found in water, with particle sizes of 300 μm and 900 μm, respectively. The method is efficient, cost-effective, non-toxic, and does not cause secondary pollution.



Key words

Nano-γ-Fe_2O_3, Microplastics removal, Ultrasonic vibration, Water treatment

Abbreviations

MPs: Microplastics

PP: Polypropylene

PET: Polyethylene terephthalate

SEM: Scanning electron microscope

PE: Polyethylene

PS: Polystyrene

DMSO: Dimethyl sulfoxide

OM: Optical microscope

rpm: round per minute

Biography:

Max Peiyuan Wu

Max is a 17-year-old rising senior studying at Shanghai High School (SHSID). Through research and innovation, he strives to contribute to solving environmental issues. Max has given speeches in UN China Youth Environmental Forum and China (Shanghai) International Technology Fair, raising awareness about the issue of microplastics in water and his research journey to contribute to this cause.

Max is a persistent tutor, volunteering to teach in schools, the elderly community, and online free of charge in various STEM fields. Max is also an enthusiastic swimmer, winning medals in backstroke and freestyle. Besides his studies, Max has a passion for marine biology, palaeontology, and evolutionary history, self-studying these topics outside of the classroom.

Xindi You

Cindy is a 16-year-old girl from Shanghai High School International Division. Her love for Chemistry drives her academic journey. She joined a chemistry club in her school, where she has learned a lot about chemistry. After she read about the severe threat of microplastics, it sparked her research interest.

Outside of school, she is a girl with a passion for singing. She has participated in several singing competitions before, and each experience has been a thrilling journey for her to express herself through music.

Contents

1. Background

Microplastics (MPs) are defined as "plastic fibers, particles, or films with a diameter less than 5 millimeters" [1]. Microplastics have been found in various aquatic environments worldwide, including rivers, lakes, oceans, and even in surface and deep-sea sediments in polar region [2]. Furthermore, microplastics have been widely detected in drinking water globally [3]. Studies estimate that the average weekly intake of microplastics ranges from 0.1 to 5.0 g [4]. Recently, multiple reports have indicated that microplastics may have serious implications for human health [5, 6]. To address the impact of microplastics in drinking water on human health, the World Health Organization (WHO) has called for the development of effective methods to remove microplastics from drinking water [7, 8, 9].

Currently, microplastics removal methods includes biodegradation, chemical catalytic degradation, physical coagulation, sedimentation, and filtration [10]. Biodegradation is typically slow and relies on microorganisms. Efficient strains are rare and require specific environments, resulting in challenges for industrial use. Activated sludge technology is commonly used in urban wastewater treatment plants. Researchers found removal rates of MPs using activated sludge treatment of 54.5% and 28%, respectively [11, 12]. However, this method can only retain MPs in the sludge, requiring further treatment for secondary pollution. Chemical removal techniques mainly rely on the strong oxidative free radicals generated by various oxidants under specific conditions to degrade MPs into smaller organic molecules. However, the use of costly oxidants often results in low efficiency during the decomposition of MPs [13, 14]. Physical methods include coagulation, sedimentation, and filtration. The removal efficiency of coagulation for MPs is generally below 60%. If the loading of coagulants is not high, the removal effect is unsatisfied [15]. Sedimentation is suitable for high-density MPs, but not for low-density MPs. Thus, the removal efficiency of different types and sizes of MPs varies greatly [16]. The pore size of the membrane is a determining factor for filtration's efficiency. In theory, smaller pore size membranes can retain more small-sized MPs, but in practice, issues as membrane clogging and fouling arise [17].

Recently, magnetic separation technologies have gained considerable attention as an efficient method for removing microplastics from water [18, 19]. These techniques leverage the large specific surface area of magnetic nanoparticles and their capacity for rapid and efficient magnetic recovery of microplastics from water. However, nearly all reported magnetic nanoparticles were functionalized and prepared on a small scale[18,19]. These synthesized magnetic nanomaterials tend to be costly and have limitations for large-scale industrial implementation. Only a few papers have reported that bare nano-Fe_3O_4 could be used on the removal of MPs in water [20, 21, 22]. Nano-γ-Fe_2O_3 is the most chemically stable among the iron-based magnetic nanoparticles and demonstrates effective reusability for adsorbing water pollutants [23, 24, 25]. Moreover, it is commercially available at a large scale and has been utilized in various industries. However, to our knowledge, there are currently no reports on the removal of MPs in water using bare nano-γ-Fe_2O_3. In this study, we aim to develop an efficient and effective method for removing MPs in water using bare nano-γ-Fe_2O_3.

2. Materials and Methods

2.1 Experimental Materials

Polyethylene (PE), Polypropylene (PP), Polystyrene (PS), Polyethylene terephthalate (PET) MP particles (purity of 98%, particle sizes of 900 μm and 300 μm) and nano-γ-Fe_2O_3 (purity of 98%, diameter< 10 nm) were purchased from Aladdin Chemical Reagent Co., Ltd. (Shanghai, China). The surface morphology was characterized by an optical microscope (OM, SMZ-171) and a scanning electron microscope (SEM, GeminiSEM 560). Zeta potential was tested by a Zeta Potential Analyzer (Brook Haven 90Plus PAL 20191753).

2.2 Calculation of Removal Rate of MPs

The removal rate was calculated according to mass of MPs by the similar methodology reported by Prof. He's group [20]. In the following formula (1), Ƞ represents the removal rate of MPs (%); m represents the mass of MPs removed from the water; M represents the initial mass of MPs added into the experimental solutions.

$$Ƞ = (m/M) \times 100\% \quad (1)$$

2.3 Removal Rate Optimization by Shaker Vibration

To determine the appropriate loading of nano-γ-Fe_2O_3, a concentration gradient experiment was carried out. The concentration of PE (particle size: 900 μm) in water was set at 0.5 g/L, and the concentrations of nano-γ-Fe_2O_3 were set at 0.7, 0.9, 1.1, 1.3, and 1.5 g/L, respectively. Three parallel experiments were performed at each concentration group.

The designated amount of nano-γ-Fe_2O_3 was added to 100 mL water containing PE (particle size: 900 μm) with a concentration of 0.5 g/L. The mixture was subsequently placed in a shaker and agitated at 180 rpm at 25 °C for 3 h (or at 50 °C for 1 h) (Fig. 2-1). Then the mixture was allowed to rest for 1-5 min. (Fig. 2-2). The PE particles with adsorbed nano-γ-Fe_2O_3 particles was removed from the water using a magnet (magnetic force: 20 kg) (Fig. 2-3). The PE particles on the magnet was detached from the surface of the magnet and collected in another beaker by rinsing with pure water. After filtration and drying in an oven at 30 °C until the sample weight no longer changed (Fig. 2-4), the removed MPs on magnets were weighed and the removal rate was calculating by formula (1). To minimize potential secondary pollution, the remaining nano-γ-Fe_2O_3 in the mixture after MP removal was recovered using a magnet (magnetic force: 20 kg). The average removal rates at each concentration were compared, and the optimal loading of nano-γ-Fe_2O_3 was determined.

After determining an appropriate loading of nano-γ-Fe_2O_3, a time gradient experiment was carried out for the optimal treatment time. The treatment time was designed as 30 min, 60 min, 90 min, 150 min, 210 min, and 240 min. For each treatment time, three parallel experiments were performed. The removal rates were calculated and compared to determine the optimal treatment time.

Fig. 2–1

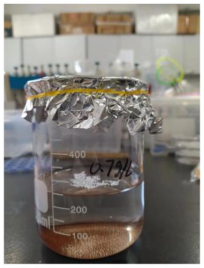


Fig. 2–2

Fig. 2–3

Fig. 2–4

2.4 Removal of MPs with Different Particle Sizes by Shaker Vibration

Four types of MPs (PE, PP, PS and PET) were chosen to prepare MPs-contained solutions. Each type of MP, two particle sizes (900 μm & 300 μm) were used. Three parallel experiments were carried out for each polymer and particle size.

50 mg of MPs and 150 mg of nano-γ-Fe_2O_3 were weighed and added to 100 mL of pure water. The mixture was placed in a shaker and agitated for 1 h at 50 °C and 180 rpm. The rest of the experimental procedure is similar as described in section 2.3.

2.5 Removal Rate Optimization by Ultrasonic Vibration

To determine the appropriate loading of nano-γ-Fe_2O_3, a loading gradient experiment was conducted with PE particles (particle size: 900 μm). The concentration of PE (particle size: 900 μm) in water was set at 2.5 g/L, and the loadings of nano-γ-Fe_2O_3 were 2%, 5%, and 20% of the weight of PE particles added, respectively. Each loading was tested in three parallel experiments, and the average removal rates were calculated to determine the optimal loading of nano-γ-Fe_2O_3.

The designed amount of nano-γ-Fe_2O_3 was added to 100 mL of water containing PE with a concentration of 2.5 g/L. The mixture was placed in an ultrasonic cleaning machine (KUDOS 20104708) and vibrated under ultrasound for 20 min at room temperature. The rest of the experimental procedure is similar as described in section 2.3.

After determining the optimal loading of nano-γ-Fe_2O_3, further investigations were conducted to determine the treatment time. Based on preliminary results, treatment times of 10 min, 20 min and 40 min were designed. For each different treatment time, three parallel experiments were performed, and the average removal rates were calculated to determine the optimal treatment time.

2.6 Removal of MPs with Different Particle Sizes by Ultrasonic Vibration

Four types of MPs (PE, PP, PS and PET) were used to prepare MPs-contained solutions. Each type of MP, two particle sizes (900 μm & 300 μm) were used. Three parallel experiments were carried out for each polymer and particle size.

375 mg of MPs (particle size: 900 μm) and 18.75 mg of nano-γ-Fe_2O_3 were added to 150 mL of pure water [26]. The mixture was placed in an ultrasonic cleaning machine (KUDOS 20104708) and vibrated under ultrasound for 20 min at room temperature. The rest of the experimental procedure is the same as described in section 2.3.

2.7 Desorption of MPs Adsorbed with nano-γ-Fe_2O_3

An appropriate amount of PE particles (particle size: 900 μm) adsorbed with nano-γ-Fe_2O_3 particles was added to 10 mL of 5 mol/L urea solution, 3 mol/L NaCl aqueous solution, 50% DMSO aqueous solution, and pure water at a concentration of 0.5 g/L, respectively. The mixtures were then vibrated with a shaker overnight at room temperature. The PE particles was filtered from the solutions, and observations were made regarding the color changes in each solution.

3. Results and Discussion

3.1 Removal of MPs by Shaker Vibration

We started investigating the removal of MPs from water using nano-γ-Fe_2O_3, following similar experiment protocols reported by Prof. He's group [20]. Preliminary results indicated that nano-γ-Fe_2O_3 can adsorb onto MPs similarly as nano-Fe_3O_4 does [20]. At room temperature, as the concentration of nano-γ-Fe_2O_3 gradually increased (0.7 g/L, 0.9 g/L, 1.1 g/L, 1.3 g/L, and 1.5 g/L), the removal rate of PE particles (particle size: 900 μm) also increased gradually and showed a linear increasing trend (Table 3-1, entry 1-5). Extrapolation suggested that achieving a target removal rate of 95% may require a nano-γ-Fe_2O_3 concentration of 2.3 g/L.

To our knowledge, no papers has been published on the temperature effect using magnetic materials for removal of MPs from water. However, we reasoned that temperature might have impact on the removal efficiency, so we screened different temperatures and were delighted to find that the removal rate of PE (particle size: 900 μm) reached 99% at 50 °C, while the removal rate was 78% at room temperature (Table 3-1, entry 5 & 7). Additionally, treatment time could be reduced to from 3h to 1 h at 50 °C (Table 3-1, entry 7-11).

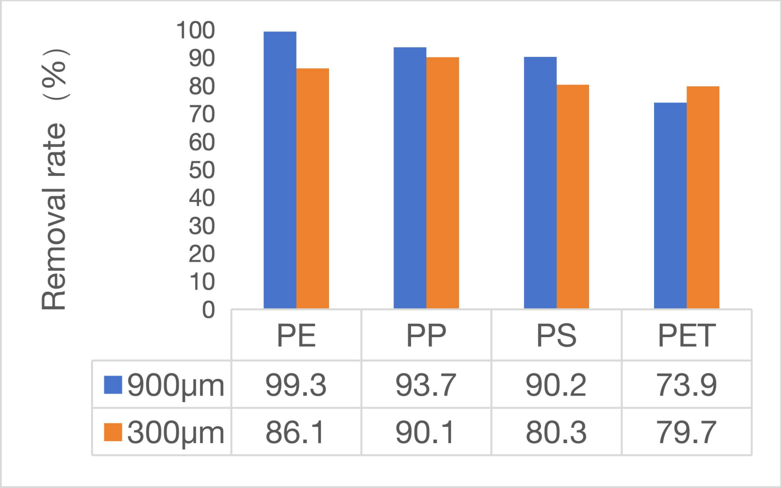
Table 3-1: Removal rate of PE particles at different removal conditions

Entry	Con. of nano-γ-Fe_2O_3 (g/L)	Treatment time (h)	Temperature (°C)	Ave. removal rate (%)
1	0.7	3	R. T.	59
2	0.9	3	R. T.	66
3	1.1	3	R. T.	68
4	1.3	3	R. T.	74
5	1.5	3	R. T.	78
6	1.5	0.5	50	73
7	1.5	1	50	99

8	1.5	1.5	50	94
9	1.5	2.5	50	98
10	1.5	3.5	50	99
11	1.5	4	50	97

These results indicated that increasing the removal temperature had a significant impact on removal efficiency. When the temperature increased from room temperature to 50 °C, the loading and treatment time of nano-γ-Fe_2O_3 can be greatly reduced. We reason that raising the temperature helps increase the Brownian motion of nano-γ-Fe_2O_3 and MPs in water, accelerating adsorption. This lowers the loading of nano-γ-Fe_2O_3 and improves removal efficiency.

Under optimized experimental conditions (temperature: 50 °C, concentration of nano-γ-Fe_2O_3: 1.5 g/L, treatment time: 1 h), we investigated the removal rates of four common MPs in water, PE, PP, PS, and PET. Fig. 3-1 summarizes the removal efficiency of MPs with shaker vibration. The experimental results demonstrated that nano-γ-Fe_2O_3 exhibited excellent removal rates for various MPs in pure water.



	PE	PP	PS	PET
🟦 900μm	99.3	93.7	90.2	73.9
🟧 300μm	86.1	90.1	80.3	79.7

Fig. 3-1: Summary of removal rates of different types and sizes of MPs

3.2 Removal of MPs by Ultrasonic Vibration

During above experiments, we observed that most of the nano-γ-Fe_2O_3 did not adsorb onto the MPs, but instead gradually self-aggregated and settled down in the water due to its high surface free energy. The SEM images of nano-γ-Fe_2O_3 showed that the nano-γ-Fe_2O_3 particles used in our experiments were mostly aggregated at dry powder state (Fig. 3-2). In pure water (pH = 7), the zeta potential of the nano-γ-Fe_2O_3 particle was -7 mV. Due to the relatively low zeta potential, the dispersion stability of nano-γ-Fe_2O_3 in water was poor and it was prone to self-aggregation. Those testing results confirmed our assumption that the commercial nano-γ-Fe_2O_3 was prone to aggregate in both dry powder state and in water.

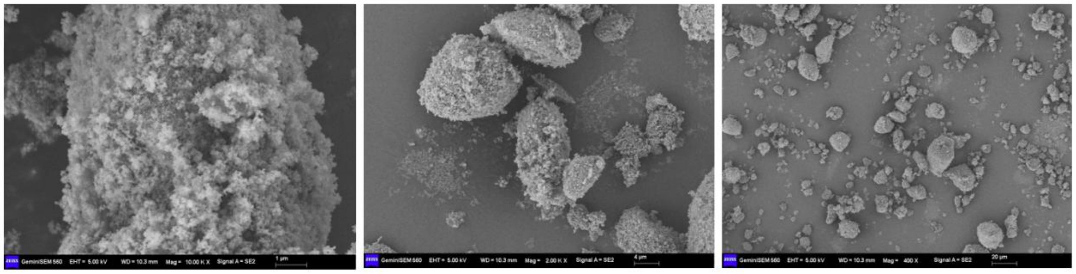


Fig. 3-2 SEM images of nano-γ-Fe_2O_3

We envisioned that the removal efficiency might be improved if nano-γ-Fe_2O_3 particles could be redispersed to create a suspension in water. We tried to increase the shaking frequency from 180 rpm to 300 rpm and adjust the amplitude; and we also used strong mechanical stirring equipment to improve the stirring efficiency, but failed to reduce the self-aggregation of nano-γ--Fe_2O_3 in water.

Ultrasonic vibration has been reported as an efficient approach for the disaggregation of nanomaterials [27, 28]. We were delighted to find that a suspension of nano-γ-Fe_2O_3 was formed under the vibration in an ultrasonic cleaning machine. Under ultrasonic vibration, removal conditions were screened by use of PE particles (particle size: 900 μm). The experimental results indicated that the loading of nano-γ-Fe_2O_3 can be significantly reduced. Only 5% w/w loading of nano-γ-Fe_2O_3 (w/w: nano-γ-Fe_2O_3 / PE particles) was needed to achieve a removal rate of 94% (Table 3-2, entry 2). Compared to the shaker vibration method, using ultrasonic vibration with nano-γ-Fe_2O_3 requires only 1.6% of the loading used by shaker vibration (Table 3-2, entry 2 & 6). The treatment time was significantly shorter compared to shaker vibration, reducing from 1h to 20 min. And the treatment temperature could also be reduced to room temperature.

Table 3-2 Removal rate of PE particles at different removal conditions

Entry	Loading (w/w, %)	Treatment time (min)	Ave. removal rate (%)
1	2	20	91
2	5	20	94
3	20	20	99
4	5	10	84
5	5	40	93
6^a	300	60	99

a. best conditions by shaker vibration

Under optimized experimental conditions (loading of nano-γ-Fe_2O_3: 5% of the weight of MPs; treatment time: 20 min; room temperature), the removal rates were examined for four commonly found MPs in water (PE, PP, PS and PET; particle sizes: 900 μm and 300 μm) were examined. The results indicated use of 5% weight loading of nano-γ-Fe_2O_3 achieved excellent removal rates for various MPs in pure water (Fig. 3-3). Compared to shaker vibration, ultrasonic vibration not only reduced the loading of nano-γ-Fe_2O_3 by 98.4% but also shortened the treatment time by 66.7%. This new method could significantly enhance the removal efficiency.

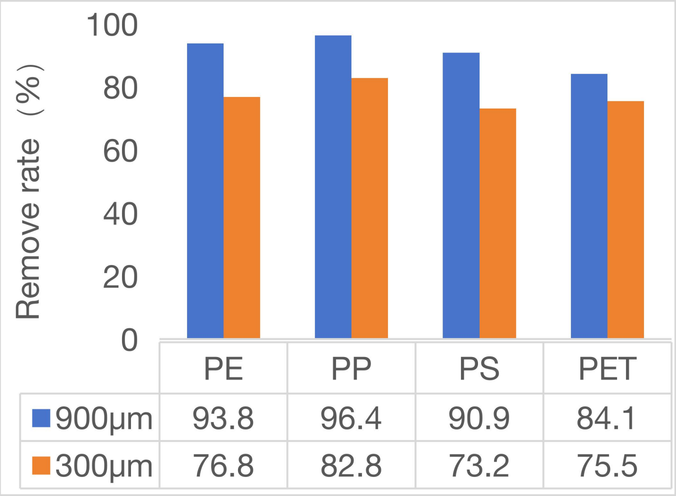


Fig. 3-3 Summary of removal rates of different types and sizes of MPs

3.3 Mechanisms discussion of Nano-γ-Fe_2O_3 and MPs

3.3.1 Surface Morphological Characteristics of MPs

The optical microscope (OM) images of all four types of MPs appear as white or translucent particles (Fig. 3-4, left). After adsorption of nano-γ-Fe_2O_3 particles, the MPs changed to brownish yellow (Fig. 3-4, right) the adsorption of nano-γ-Fe_2O_3 on the surfaces of various MPs. The SEM images further demonstrated that numerous of nano-γ-Fe_2O_3 particles were adsorbed onto the surface of MPs (Fig. 3-5).

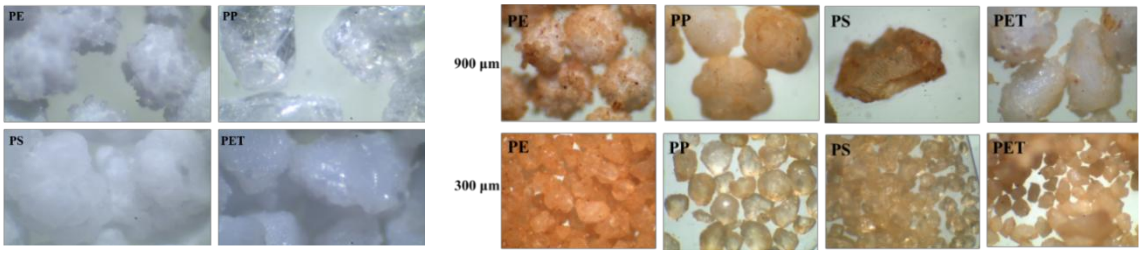


Fig. 3-4 OM images of MPs before & after adsorption of nano-γ-Fe_2O_3 particles

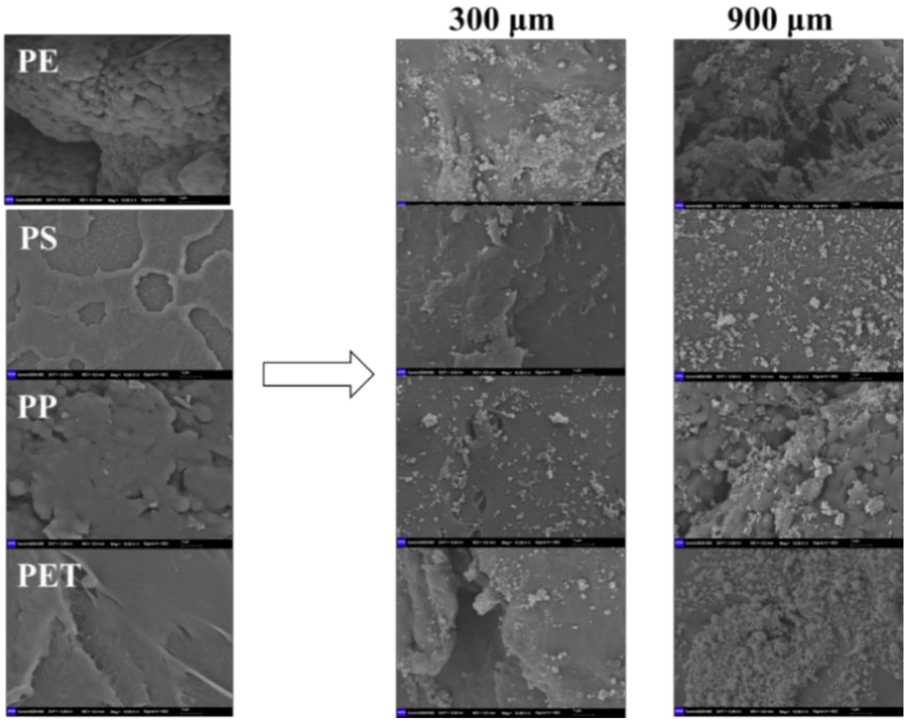


Fig. 3-5: SEM images of MPs before & after adsorption of nano-γ-Fe_2O_3 particles

3.3.2 Adsorption Mechanisms of MPs and nano-γ-Fe_2O_3

There have been few studies investigating the removal of MPs from water using magnetic nanomaterials. To our knowledge, no published reports have addressed using bare nano-γ-Fe_2O_3 to remove MPs from water. To study the possible adsorption mechanism between MPs and nano-γ-Fe_2O_3, we followed the research methodology developed by Prof. Liu's research group [21].

The zeta potentials of four types of MPs in pure water ranged from -13 mV to -24 mV, while the zeta potential of nano-γ-Fe_2O_3 in pure water was -7 mV (Fig. 3-6). These results indicated that both MPs and nano-γ-Fe_2O_3 particles carried negative charge in pure water. Therefore, the adsorption between nano-γ-Fe_2O_3 and MPs is unlikely to be driven by electrostatic attraction.

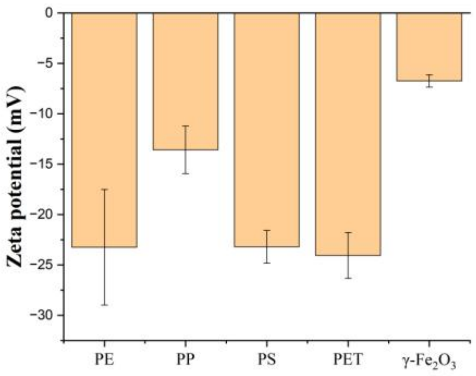


Fig. 3-6 Zeta potential of MPs and nano-γ-Fe_2O_3

To further examine the nature of the adsorption, various agents were used to induce desorption of nano-γ-Fe_2O_3 particles. The desorption was tracked visually (Fig. 3-7). As a control, when water was added to the pre-adsorbed nano-γ-Fe_2O_3, the supernatant remained clear, indicating no release of nano-γ-Fe_2O_3. Then, 5 mol/L urea was added to probe hydrogen bonding (urea is a hydrogen bonding disruptor). The urea solution became light brown, suggesting desorption occurred. These results suggested contribution of hydrogen bonding in adsorption. Hydrogen bonding likely occurred between the C-H and O-H bonds on MPs as donors and the -OH on iron oxide as acceptors. 50% DMSO solution was used to determine the effect of hydrophobic interactions. The solution turned a brown color, suggesting desorption of nano-γ-Fe_2O_3. This revealed that hydrophobic interactions played a crucial role in adsorption. Moreover, we screened the charge interactions by adding 3 mol/L NaCl, but negligible desorption happened suggesting the electrostatic interactions were less important than hydrogen bonding for the adsorption. Thus, hydrogenation bonding was the main force responsible for the adsorption of the bare nano-γ-Fe_2O_3 particles.

Fig. 3-7 Desorption of γ-Fe_2O_3 from PE pre-adsorbed nano-γ-Fe_2O_3

(from left to right: 50% DMSO, 5 mol/L Urea, 3 mol/L NaCl, control group)

Based on the above experimental and results, we reason that both nano-γ-Fe_2O_3 and MPs particles carry negative charge in water, while nano-γ-Fe_2O_3 particles are prone to self-aggregation due to its low zeta potential in water. Under ultrasonic vibration, nano-γ-Fe_2O_3 particles are dispersed to form a suspension in water. Nano-γ-Fe_2O_3 and MPs particles undergo Brownian motion in water, and nano-γ-Fe_2O_3 particles adsorb onto the surface of MPs particles by hydrophobic effects and hydrogen bonding. MPs adsorbed with nano-γ-Fe_2O_3 particles become magnetic and can be efficiently removed from water by a magnet (Fig. 7).

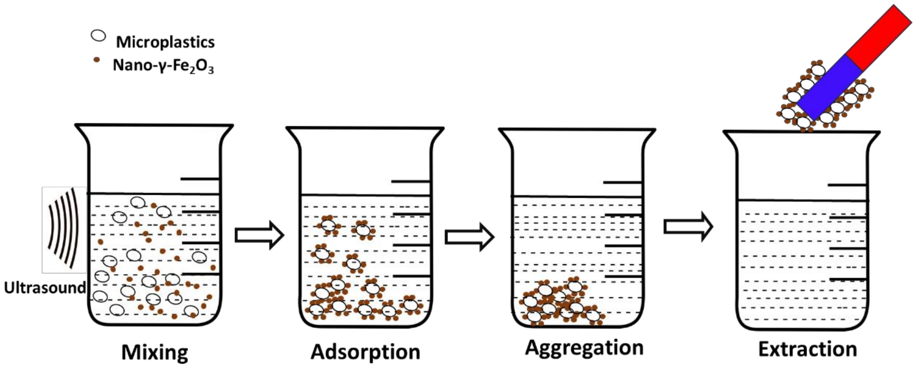


Fig. 3-7 Removal of MPs by nano-γ-Fe_2O_3

4. Conclusion and Outlook

4.1 Conclusion

Our research demonstrated for the first time that microplastics in water could effectively adsorb commercial nano-γ-Fe_2O_3 particles and be easily removed using a magnet. We investigated the influence of vibration mode and removal temperature on the adsorption effectiveness. Our experiments revealed that ultrasonic vibration outperformed shaker vibration, reducing the loading of nano-γ-Fe_2O_3 by 98.6% and decreasing the treatment time by 66.7%. Under shake vibration, appropriately increasing the removal temperature, we observed a decrease in the loading of nano-γ-Fe_2O_3 and the treatment time. Upon conducting desorption experiments, we identified hydrophobic effects and hydrogen bonding between nano-γ-Fe_2O_3 and MPs particles as the primary forces driving the adsorption of nano-γ-Fe_2O_3.

This study introduced a novel method for efficient and cost-effective removal of microplastics from water, demonstrating a high removal rate and a short operation time of just 20 min. In contrast to traditional filtration techniques, this method eliminates the need for costly filter membranes and reduces the risk of secondary membrane contamination. Furthermore, the use of commercially available nano-γ-Fe_2O_3 offers cost advantages, making it a promising solution for implementation in water treatment plants.

4.2 Outlook

During a visit to a wastewater treatment plant, we noticed that many waste water treatment processes take place in different pipelines. Given that our developed removal method has a relatively short treatment time, we are planning to further investigate the effectiveness of removal in flow equipment, which is more likely to be implemented in water treatment industry (Fig. 4-1).

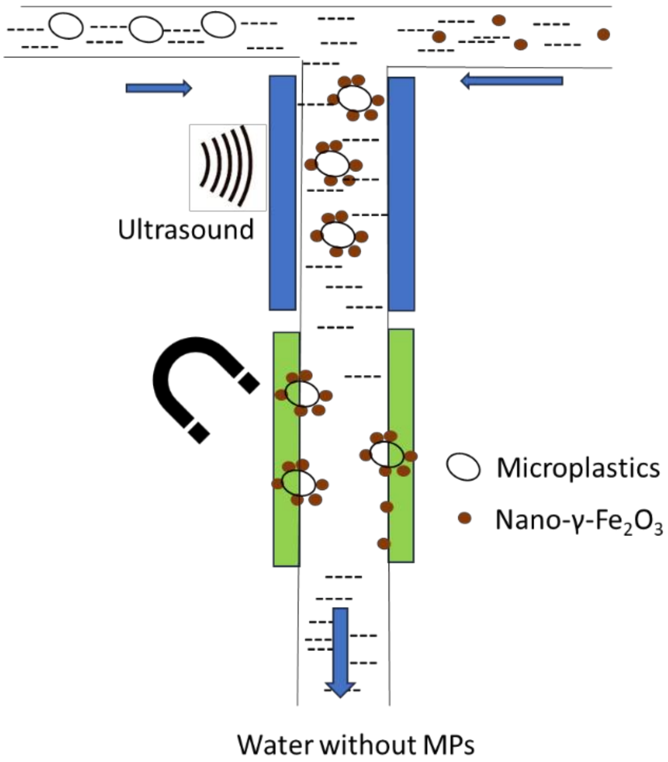


Fig. 4-1 Proposal: Removal of microplastics in flow equipment

5. Acknowledgments

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