Self-driven magnetorobots for recyclable and scalable micro/nanoplastic removal from nonmarine waters

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Micro/nanoplastic (MNP) contamination in nonmarine waters has evolved into a notable ecotoxicological threat to the global ecosystem. However, existing strategies for MNP removal are typically limited to chemical flocculation or physical filtering that often fails to decontaminate plastic particulates with ultrasmall sizes or ultralow concentrations. Here, we report a self-driven magnetorobot comprising magnetizable ion-exchange resin sphere that can be used to dynamically remove or separate MNPs from nonmarine waters. As a result of the long-range electrophoretic attraction established by recyclable ion-exchange resin, the magnetorobot shows sustainable removal efficiency of >90% over 100 treatment cycles, with verified broad applicability to varying plastic compositions, sizes, and shapes as well as nonmarine water samples. Our work may facilitate industry-scale MNP removal with affordable cost and minimal secondary pollution and suggests an appealing strategy based on self-propelled micro/nanorobots to sample and assess nanoplastics in aqueous environment.

INTRODUCTION

Plastic pollution, as an indicator of Anthropocene, has posed a growing threat to global terrestrial and marine ecosystems in consequence of its poor reversibility and continuing accumulation over the past decades (1–3). Compared with 4.8 to 12.7 million tons of plastic emitted to the ocean, annual emissions of plastic on land and into nonmarine waters could be many times more but with less academic investigations (4–6). Plastic debris will be fragmented into microplastics (MPs; particulate size of <5 mm) (7) or nanoplastics (NPs; particulate size of <1000 nm) (8) upon exposure to mechanical, photochemical, thermal, and biochemical factors in nonmarine waters (9–13), bringing increased harms to the health of human and biota after uptake and accumulation of micro/nanoplastics (MNPs) (14–16). However, efficient removal/separation of MNPs remains challenging because of their tiny size and low concentration in contaminated waters. Current approaches to remove MNPs primarily rely on chemical flocculation and passive physical filtration (17), which often incurred MNP-induced membrane fouling and low removal efficiency (table S1) (18–21). Furthermore, physical filtration sampling and microscopy characterization of MPs are not practical for NPs as there are substantial filtering loss and optical diffraction limit (22, 23), which substantially hinder investigations on NP pollution and introduce notable variations between different assessments.

Micro/nanorobot (MNR) recently emerges as a compelling technology that provides the opportunity to actively maneuver micro/nanoparticulates in aqueous medium while promising high interaction efficacy and spatiotemporal precision (24–28). The development of MNRs has enabled a variety of micromachines that can be driven by light (29–32), magnetic field (33, 34), or chemical methods (35) for MNP removal and degradation (36, 37) (table S1). For instance, light-driven MNRs successfully exploited photocatalytic reaction to power MNR motion. As a pioneering work, ultraviolet light–illuminated Au@Ni@TiO2 was used to remove polystyrene (PS) microparticles (1 to 10 μm) with a removal efficiency of 67 to 77%, based on photoretic interaction (38). A series of smart MNRs with multifunctional design were later developed to accomplish dynamic MNP removal and photocatalytic decomposition (TiO2, Fe2O3, Bi2WO6, BiVO4) (29, 31, 39) or electrochemical detection (MXene-derived multilayered γ-Fe2O3/Pt/TiO2) (30). Beyond light-driven MNRs, a magnetic field–powered polydopamine@Fe3O4/lipase microrobot was constructed to closely bind MNPs (<1-μm interaction distance) with mussel adhesive and enzymatically degrade MNPs with lipase (33). A magnetic microsubmarine based on sunflower pollen grain was demonstrated for recyclable removal of large microparticles (100 μm in diameter) by noncontact shoveling with over 70% removal efficiency (34). Moreover, a Fe2O3-MnO2 micromotor was reported to chemically remove more than 10% of MPs (20 to 35 μm in diameter) with an adsorptive bubble separation mechanism (35). However, the interaction distance between MNPs and existing MNRs is primarily dictated by short-range phoretic (typically less than 10 μm) (38, 40, 41) or van der Waals forces (less than 1 nm), which largely restricts the removal efficiency of MNPs (table S1). Separately, the adsorption force between MNRs and MNPs thus far is nonswitchable, which leads to an either too weak force that can only shove adjacent particulates or too strong force that captures MNPs permanently in an irreversible manner. This nonswitchable design results in a poor recyclability of MNRs, impedes the large-scale application of MNRs, and may cause secondary pollution by MNRs themselves. Existing MNRs principally relied on the external actuators (e.g., light or magnetic) fields, which may lead to sophisticated peripheral setup and limited scalability.

Here, we report the design and demonstration of a self-driven magnetorobot (SMR) capable of removing and separating MNPs from nonmarine waters in a recyclable and scalable way (Fig. 1A). SMR consists of an ion-exchange resin microsphere functionalized by superparamagnetic Fe3O4 nanoparticles. The autonomous motion of SMR is driven by asymmetric self-induced ionic gradient and phoretic force while enabling long-range dynamic adsorption of
plastic particulates from the aqueous environment. We demonstrate the utility of SMRs for efficient removal of MPs and NPs, magnetic separation, and controlled MNP release. The broad adaptability of this SMR has been confirmed over plastics with varying compositions [poly(methyl methacrylate) (PMMA), polyamide (PA), PS, polyvinyl chloride (PVC), and poly(1,1-difluoroethylene) (PVDF)], sizes (200 nm to 40 μm), and surface charge conditions (positive or negative zeta potential). After ion exchange, SMRs show over 90% removal efficiency for MNPs in lake water, river water, and sewage wastewater and can sustain performance for 100 treatment cycles. In particular, a removal efficiency over 96% has been accomplished for both MPs and NPs with ultralow concentrations comparable to that in real environmental waters. We further demonstrate the scalability of this SMR technology by building an autonomous closed-loop miniplant that can continuously remove MNPs from contaminated waters, verifying its potential in future industrial applications.

**RESULTS**

**Mechanism description and characterization of SMRs**

The SMR was constructed by decorating ion-exchange resin microsphere with Fe$_3$O$_4$ nanoparticles. As conceptually illustrated in Fig. 1B, the SMR generates long-range electrostatic attraction between SMR and MNPs. Specifically, the fast diffusion of exchanged hydroxide ions released from SMR builds a localized inward diffusion electric field, $E$, which establishes the inward electrostatic flow and provides the long-range attraction. Meanwhile, the initially asymmetric MNP adsorption induces the asymmetric fluidic flow around the SMR that autonomously propels SMR (fig. S2). The motion of SMR promotes its interaction with surrounding particulates and realizes a dynamic removal of MNPs along its moving path (movie S1). Owing to the magnetic property of Fe$_3$O$_4$ particles (fig. S3), SMRs can be simply collected with a magnet (Fig. 1, C to E, and figs. S4 to S6). Furthermore, the addition of magnetic component endows SMR with precise controllability under programmable magnetic field. As shown in fig. S7, the motion direction of SMR can be well controlled by an external magnetic field. Meanwhile, the SMR dynamically adsorbs the MNPs along its migration trajectory (movie S2), where the moving speed is not disturbed by the applied magnetic field (fig. S8). Therefore, the magnetic field here only controls the moving direction of the SMR but is essential for the recycling adsorption process, which will be introduced later. Moreover, the switchable ion-exchange resin enables the desorption of MNPs in sodium chloride (NaCl) salty solution and SMR regeneration via sodium hydroxide (NaOH) treatment. This feature promises a recyclable operation of SMRs, which could facilitate large-scale application of SMRs in a cyclic loop of MNP adsorption, MNP desorption, SMR regeneration, and SMR rinse as shown in Fig. 1A.

Upon exposure to MNP-contaminated water, the anionic ion-exchange resin of SMR captures impurity anions from surrounding aqueous medium and releases OH$^-$, resulting in a long-range pH gradient field and inward electrostatic force to attract negatively charged MNPs. Figure 2A shows declined pH profile from the OH$^-$ ion-exchange resin–based SMR (OH-SMR) surfaces to the bulk
solution that was recorded by a confocal laser microscope with the pH-sensitive dye CF-5 (6) (see Materials and Methods). As the SMR is propelled by the surrounding asymmetric fluid flow, the addition of MNPs triggers the motion (movie S3), and the acceleration process was also observed, which further demonstrates the asymmetric adsorption-induced motion. Three-dimensional (3D) trajectories of PS nanoparticles (nano-PS) were recorded by the confocal laser microscope with time-lapse (2 s) scanning along with the depth of solution (Fig. 2B). As shown in movie S4, the trajectories indicated the attraction of nano-PS from extensive 3D space toward the SMR. These experimental results were corroborated by numerical simulation that showed inward flow to attract adjacent MNPs (Fig. 2C; see note S1 and Materials and Methods). To further differentiate electrostatic from phoretic interaction, the electrostatic/phoretic strength was calculated at various distances from the SMR (fig. S9), in which the electrostatic force limits within 1 μm, while the phoretic force extends to much longer distance. The electrostatic/phoretic strength ratio was also plotted as shown in fig. S10; within a short distance, the electrostatic force dominates the interaction, increases with approaching to the SMR surface, and can be ignored as moving away, while long-range attraction depends on the fluid flow–induced phoretic force. The above simulation results further confirm the mechanism of long-range phoretic flow for MNP transport and electrostatic for final capture. While the experimental speed of
MNs decreased with increasing distance from SMR due to damped phoretic flow (Fig. 2D), the SMR-MNP interaction could still reach ~200 μm (fig. S11). In contrast to previous removal strategies leveraging only short-range (<10 μm) electrostatic adsorption (29, 38, 42, 43), our SMR has improved removal efficiency as it benefits from a combination of long-range electrophoretic attraction and short-range electrostatic adsorption. The ion-exchange capacity determines the strength of surrounding phoretic flow that influences the attraction speed and adsorption range. As shown in fig. S12, different exchange capacities were examined, and the attraction distance and speed increase with a higher exchange capacity.

Electrophoresis is a time-dependent process that dictates the motion behavior of both SMR and surrounding MNPs. Figure 2E shows the change in SMR motion speed by time when adsorption of MNPs first enhances it to 6 μm/s via symmetry breaking and subsequently slows it down with increased payload and decreased symmetry breaking. The SMR motion typically persists for 15 to 20 min with rapid adsorption of MNPs (movie S5), while the adsorption of surrounding MNPs sustains for over 1 hour until the ion-exchange capacity is fully depleted (Fig. 2F and fig. S13). Since the attraction of MNPs is induced by electrophoresis, the effect of ionic strength needs to be taken into account for practical application in nonmarine river and lake waters in which ionic strength is typically around 0.57 to 3.5 mM (44, 45). The motion speed (Fig. 2G) and the attraction distance (fig. S14) of MNPs were tested under varying ionic strength. Even with an ionic strength of 5 mM, MNPs manifested a speed of 1.81 μm/s and an attraction distance of ~24 μm, which could result from the balance between ion-exchange rate and “ion quenching” effect (46). Moreover, the pH influence was also examined by measuring the SMR motion speed at various pH values (fig. S15). Theoretically, the low pH is favorable for the neutralization with hydroxide ions to accelerate the ion-exchange rate, while the ion quenching effect may counteract the flux increase. Thus, still, the neutral pH is favorable for the interaction.

The self-propulsion of SMR, long-range pumping flow, and electrostatic attraction construct the interaction between MNPs with SMR, which can be rationally designed for the efficient MNP removal (fig. S2). To illustrate the difference in electrostatic adsorption and electrophoretic attraction, we compared three types of resin-based SMRs: OH-SMR, Cl− ion-exchange resin (Cl-SMR), and H+-ion exchange resin (H-SMR). The motion speeds of OH-SMR and H-SMR were 3.1 μm/s (in nano-PMMA suspension) and 6.02 μm/s (in nano-PS suspension), respectively. We further examined the effect of SMR size (5 to 70 μm) on SMR motion speed and adsorption range within the PS microparticle (1.5 μm) suspension (Fig. 3E). An optimized SMR speed of 7.12 μm/s was observed when its size approached 20 μm. Since the adsorption range is determined by the ion-exchange capacity, larger SMR size favors long-range electrophoretic flow and adsorption. The MNP removal efficiency should result from the synergistic effect of both motion speed and adsorption range. The optimized SMR size (20 to 25 μm) is selected by evaluating the area clearance rate (fig. S22; see the Supplementary Materials for details).

Motile SMR for dynamic MNP removal
We demonstrated the self-driven motion of magnetorobot and dynamic removal of three representative MNPs. The SMR motion is induced by asymmetric MNP adsorption and, in turn, enhances the dynamic interaction range between SMR and MNPs (Fig. 3A). In all three MNP suspensions, autonomous motion of SMR was observed, which resulted in MNP-depleted regions along the motion trajectories of SMR. With the suspension of PS microparticles (1.5 μm; see Materials and Methods), magnetorobot based on OH-SMR displayed a motion speed of 3.62 μm/s (Fig. 3B and movie S8), which was further improved to 7.12 μm/s in a suspension of irregular PMMA microparticle due to a greater symmetry breaking by adsorption (Fig. 3C and fig. S17). Additional experiments confirmed the motion of SMR across a wide range of plastic compositions and sizes (figs. S18 and S19 and movies S9 and S10).

Figure 3D and fig. S20 show that SMR can migrate with positively charged nano-PS (200 nm; fig. S21) and negatively charged PMMA nanoparticles (nano-PMMA; 550 nm), while naturally fragmented plastics mostly show negative zeta potential values. Negatively charged nanoparticles supported the motion of anionic OH-SMR, while positively charged particles worked with magnetorobot based on H+ ion-exchange resin (H-SMR). The motion speeds of OH-SMR and H-SMR were 3.1 μm/s (in nano-PMMA suspension) and 6.02 μm/s (in nano-PS suspension), respectively. We further examined the effect of SMR size (5 to 70 μm) on SMR motion speed and adsorption range within the PS microparticle (1.5 μm) suspension (Fig. 3E). An optimized SMR speed of 7.12 μm/s was observed when its size approached 20 μm. Since the adsorption range is determined by the ion-exchange capacity, larger SMR size favors long-range electrophoretic flow and adsorption. The MNP removal efficiency should result from the synergistic effect of both motion speed and adsorption range. The optimized SMR size (20 to 25 μm) is selected by evaluating the area clearance rate (fig. S22; see the Supplementary Materials for details).

SMR performance for MNP removal
As mentioned above, the long-range collection of particles by SMR is nonspecific because this process is pumping by the phoretic flow. As shown in movie S11, for different surface-charged particles, the long-range collection is similar, while the final state of attraction and adsorption to the SMR surface depend on the surface charge, in which oppositely charged MNPs with SMR will be attracted and the same charged particles will be rebounded away. Therefore, we can rationally select the anion-exchange resin or cation-exchange resin for the negatively and positively charged MNP removal, respectively. Systematic MNP removal experiments established the applicability of SMR to a broad range of plastic particulates with varying compositions (PS, PVC, PMMA, PA, and PVDF), sizes, shapes, and surface charges (Fig. 4A and fig. S16). Scanning electron microscopy (SEM) images indicated the substantially increased diameters of SMR after MNP adsorption (Fig. 4A, ii). Here, we use an “effective volume ratio” (EVR) to quantify the adsorption capacity of SMR. The EVR is defined as the volume ratio of SMR with MNPs to intact SMR, which yielded a value of 1.3 to 1.7 for different MNPs (table S3) and suggested high-adsorption capacity of our SMR.

MNP adsorption is a dynamic process and therefore depends on the interaction time between SMR and MNPs. Figure 4B shows the removal efficiency of micro-PMMA and nano-PMMA particles, which rapidly ramped up within the first 10 min due to asymmetric
adsorption and gradually saturated after 60 min as a result of ion dissipation. The removal efficiency of micro-PMMA achieved ~97% within 1 hour, while the efficiency of nano-PMMA achieved ~66%. This less efficient removal of nano-PMMA could be ascribed to their stronger Brownian motion and symmetric adsorption on SMR. Therefore, to reach over 90% removal efficiency of NPs, a higher SMR:MNPs ratio as well as increased treatment cycles or longer treatment time should be adopted (Fig. 4C; see Materials and Methods).

In general, plastic particle with larger size and higher surface potential renders a stronger interaction with SMR and thus a higher chance to be removed, which is consistent with the high removal efficiency in irregular PMMA (96.38%) and PA (99.12%) particles (figs. S16 and S19). In addition, the performance of the static and dynamic SMR was compared. The removal efficiency of magnetically immobilized SMR was measured, with all the other conditions kept consistent with mobile SMR. As shown in fig. S23, the removal efficiencies of static SMRs are much lower than those of mobile SMR for both MPs (55.3%) and NPs (47.6%). This result corroborates the importance of dynamic motion for MNP removal. We further demonstrated the utility of SMR in varying nonmarine waters (river, lake, and sewage waters; fig. S24). The SMR in these environmental waters showed similar performance to that in deionized (DI) water for MP

**Fig. 3. Motile SMR for dynamic MNP removal.** (A) Schematic of SMR motion as a result of MNP adsorption–induced symmetry breaking, which in turn enhances dynamic adsorption of MNPs. (B to D) Motion and adsorption processes of an SMR in negatively charged PS MPs (B) and irregular PMMA MPs (C) as well as positively charged PS NPs (D). The SMR adsorbs surrounding MNPs during migration and creates an MNP-depleted region along the path. The trajectories of MNPs are plotted for 5 to 7 s. Trajectories of SMR and MNPs are colored to show the instant speed. Scale bars, 50 μm. (E) Size dependence of the SMR motion speed and adsorption range. Blue and red curves only serve as a visual guide for the change of SMR speed and adsorption range, respectively. Error bars show the mean ± SD (n = 30 data points). OH-SMR, magnetorobot based on OH− ion-exchange resin; H-SMR, magnetorobot based on H+ ion-exchange resin.
removal (micro-PMMA as a representative) (Fig. 4C), verifying the wide applicability of SMR approach across nonmarine waters. Furthermore, we compared the removal efficiency of SMRs based on different ion-exchange resins (see Materials and Methods) and traditional methods (filtration and flocculation) under a plastic (micro-PMMA or nano-PMMA) concentration of 10 and 20 parts per million (ppm) that is ~10 times lower than the concentration of MNPs in real aqueous environment (see Materials and Methods) (47, 48). For the ultralow concentration quantification, colored PMMA micro/nanoparticles by encapsulating Rhodamine B are synthesized and fluorescent quantitation measurement with a microplate reader was adopted to calculate the removal efficiency more accurately (see Materials and Methods for details). As shown in Fig. 4D, OH-SMR provided a removal efficiency of over 96% due to the efficient motile adsorption, while traditional strategies can only remove <40% micro-PMMA and <30% nano-PMMA. The low removal efficiency of CI-SMR is due to the short-ranged phoretic flow and inadequate propulsion originating from its low ion-exchange efficiency (fig. S25 and movie S12).

The stability of magnetic response and ion-exchange capability is crucial for long-term and recyclable operation of SMR. Moreover, along with the adsorption of MNPs, the adsorption of other
micro/nanoparticles will also facilitate the adsorption saturation and may reduce the removal efficiency of MNPs for the disposable SMR; therefore, the recycling adsorption is necessary to thoroughly remove the residual MNPs. Figure 4E shows that SMRs maintained ion-exchange capability over 100 cycles of treatment (adsorption, desorption, regeneration, and rinse) and ensured >90% removal efficiency for micro-PMMA through the cycles. The magnetic response of SMR slightly degraded after the five-cycle treatment (fig. S26), which can be easily refreshed by immersing SMRs in the solution of Fe₃O₄ nanoparticles as in the SMR preparation process (see Materials and Methods). Superparamagnetic property of constituent Fe₃O₄ nanoparticles endowed SMR with remote controllability and enabled magnetic separation of SMR by an external field (figs. S7 and S27 and movie S2), which may facilitate handling of SMRs during cycling and field sampling. Furthermore, the heavy metal ion pollution is another important environmental issue, and the ion exchange–induced propulsion will make the heavy metal ion removal easier. Hereby, we tested the removal of MNPs from the solution with heavy metal ions. As shown in fig. S28, over 99% removal rates were achieved with almost no influence for the MNP removal (movie S13). This result demonstrated the potential of SMR to couple with removal of heavy metal ions due to the ion exchange–based mechanism.

**Closed-loop miniplant for MNP removal**

To demonstrate large-scale decontamination of MNP-contaminated water, we established a closed-loop miniplant that could be adapted for industrial application (Fig. 5A; see the 3D printing code in Supplementary Data). This miniplant comprises four major cells for (i) MNP adsorption by SMRs, (ii) MNP desorption by NaCl (2 M) solution, (iii) SMR regeneration by NaOH (6.25 M) solution, and (iv) SMR rinse by DI water. Owing to the magnetic response of SMRs, they can be easily collected by a magnet under the cell and transferred between cells through the interconnected bridges. Figure 5Ai shows the adsorption cell in which water contaminated by colored micro-PMMA (fig. S29) was cleaned by SMRs after treatment, while the MNPs were carried by SMRs and accumulated...
in the desorption cell as can be visibly detected from the color change over five consecutive cycles (Fig. 5A, v to viii, and fig. S30). After releasing the treated water from the adsorption cell, new contaminated water can be refilled. This refilling and new treatment could proceed in parallel with SMR regeneration and rinse under autonomous control. In this miniplant, SMRs were recycled in the loop of four cells and maintained over 90% removal efficiency, MP desorption rate, and SMR regeneration rate over five cycles (Fig. 5, B to D). For the future real application, the industrial applied electromagnet will be a good option to transfer the SMRs from cell to cell, and programmable automation control system will facilitate the recycling process.

**DISCUSSION**

We have demonstrated an uncomplicated yet effective SMR for recyclable and scalable MNP removal from nonmarine waters. The resulting magnetorobots are constructed by functionalizing ion-exchange resin with superparamagnetic Fe₃O₄ nanoparticles. Upon exposure to MNPs, magnetorobot is driven by asymmetric electrophoretic force that enables dynamic adsorption of MNPs, which thereafter can be magnetically separated and recycled via effortless desorption and regeneration processes. As a result of the unique long-range (up to 200 μm) electrophoretic adsorption, SMRs exhibited a remarkably high removal efficiency over 90% for MNPs in varying nonmarine water samples. This outstanding removal rate for NPs is a remarkably high removal efficiency over 90% for MNPs in varying nonmarine water samples. This outstanding removal rate for NPs is substantially higher than any reported microrobots as well as traditional SMRs. As a result of the unique long-range (up to 200 μm) electrophoretic adsorption, SMRs exhibited a remarkably high removal efficiency over 90% for MNPs in varying nonmarine water samples.

We carried out technoeconomic analysis for MNP removal by our SMR method on the basis of closed-loop miniplant. The unit price of all consumable chemicals was first examined and listed in table S4 (note S3). The costs of ion-exchange resin and iron oxide as the main components of SMR are US$ 0.7 kg⁻¹ and US$ 5.4 kg⁻¹, respectively. Note that SMRs show excellent stability even after 100 cycles, and the average cost of SMR treatment is estimated to be US$ 0.012 for each ton of contaminated water. Compared with the operation cost of sewage water treatment plant (49), only 1.5% increase in cost will be introduced, confirming that the SMR process is an economically feasible strategy for MNP removal. With this low cost and simple scalability, SMR could be an affordable and sustainable alternative to existing MNP removal strategies.

NP pollution in nonmarine aquatic environment has been recognized as an impending threat to human health and biota (8). However, there are few, if any, technologies that can efficiently separate NP particles from environmental aquatic samples, which seriously hinder the analysis of life cycle and impact of NPs. Unfortunately, it is not viable to extrapolate the properties and behavior of NPs from that of their bulk counterparts (e.g., MPs), as both the origin and the pathway to its fragmentation may alter the size-emergent properties (8). Therefore, the knowledge of field-collected NPs is of great value that cannot be replaced by laboratory samples short of diversity in shape, size, composition, and surface properties. Because of the reversible ion-exchange capability of resin, our SMR is capable of interacting with MNPs in a switchable manner, which supported over 100 treatment cycles and may provide a powerful tool for environmental NP sampling.

In summary, attributing to the dynamic and controllable motion property as well as the fruitful design strategies, MNRs may provide an intriguing toolbox for the MNP removal, sampling, and analysis. Previous pioneering studies have demonstrated active removal of MNPs with a laboratory-scale advanced setup. However, from the perspective of practical application, the design of MNRs needs more consideration of the massive synthesis and scalable setup. In this regard, our SMR is propelled by phoretic flow induced by reversible exchange with environmental impurity ions, which circumvents the use of high-energy chemical fuels and sophisticated actuation setup. The uncomplicated structure and facile synthesis of our SMR make industrial production and large-scale application of MNRs possible, which ensures affordable and seamless integration with existing water treatment facilities. Despite the progress made in this work, further work may explore the application of developed SMRs to advanced "capture and digest" tactic (29–35, 38, 39) to decontaminate MNPs in one shot.

**MATERIALS AND METHODS**

**SMR preparation process**

SMRs were prepared by functionalizing anion ion-exchange (IEX) spheres with Fe₃O₄ nanoparticles. Fe₃O₄ nanoparticles were first prepared following a previously reported coprecipitation method (50). Briefly, FeCl₃·6H₂O (6 mmol; Aldrich) and FeSO₄·7H₂O (5 mmol; Aldrich) were dissolved in 40 ml of DI water. Next, 28 weight % (wt %) ammonia solution (5 ml; Aldrich) was added into the solution and heated up to 90°C. Sodium citrate (14.9 mmol; Aldrich) was further added to the solution and stirred for 30 min. After cooling down to room temperature, Fe₃O₄ nanoparticles were separated by a magnet, rinsed by ethanol, and freeze-dried for 24 hours. The synthesized Fe₃O₄ particles manifest a diameter of around 8 to 10 nm with superparamagnetic characteristics (fig. S3). Ten milligrams of Fe₃O₄ nanoparticles was ultrasonically dispersed in 50 ml of DI water to get the stock solution.

To construct magnetorobot based on OH⁻ ion-exchange resin, IEX spheres (Anland, Dowex 1 × 8 chloride chromatography resin, 200 to 400 mesh) of 10 to 100 μm diameters were immersed into 20 wt % sodium hydroxide (NaOH; Aldrich) solution for 24 hours to exchange all chloride ions into OH⁻. For rinsing IEX spheres (pH 7), these resin spheres (50 mg) and Fe₃O₄ nanoparticle stock solution (2 ml) were mixed in a vial with stirring to achieve homogeneous functionalization. The SMRs can be collected by a magnet for MNP removal and separation. The magnetic property of as-prepared and recycled magnetic SMRs can be measured using the equipment of the physical property measurement system (PPMS-9). The magnetorobot based on Cl⁻ ion-exchange resin (Anland, Dowex 1 × 8 chloride chromatography resin, 200 to 400 mesh) of 10 to 100 μm diameters were used as received. Magnetorobot based on inert resin is constructed by Amberlite XAD 1600N (Anland). For the surface modification, 200 mg of resin was dispersed in 2-(acryloyloxy)ethyl trimethylammonium chloride solution (0.3 M) under stirring for 24 hours, then separated, and rinsed by DI water for surface potential measurement and interaction experiments with MNPs.

**The experiment of pH influence examination**

A range of aqueous solutions with pH 3 to 11 was prepared. In detail, for acid solution, HCl was used and the solution of pH 3 was first prepared with around 26 μl of HCl (36 wt %; Macklin) dispersed into 100 ml of DI water. Then, the solution of pH 4 to 6 was diluted by the solution of pH 3. DI water was used as the pH 7 aqueous
solution. For the solution of pH 11, 12.5 mg of NaOH (99%, analytically pure, Macklin) was added into 100 ml of DI water, and subsequently, the solution of pH 8 to 10 can be diluted by pH 11 solution. Here, the pH of the prepared solution was measured by a pH meter (AS600, AS ONE).

MNP particle preparation

**PS-200**

Nano-PS with size around 200 nm were synthesized following reported protocol (51). In a typical procedure, styrene (40 g; Macklin) was added to [2-(acyrloyloxy)ethyl] trimethylammonium chloride solution (2 wt %, 400 g; Macklin) and kept stirring at 800 rpm for 51 h. In a typical procedure, styrene (40 g; Macklin) was added to the mixture. The resultant solution was purged with nitrogen for 20 min and then heated to 90°C with an oil bath. Next, 12 wt % of 2,2′-azobis(2-methylpropionamidine) dihydrochloride solution (10 ml) (V50, >97.0%; Macklin) was added to generate emulsion. The emulsion was kept at 90°C for 24 hours under nitrogen to complete the polymerization. The produced PS particles were washed with ethanol three times and freeze-dried.

**PS-1500**

PS microparticiles with size around 1500 nm were synthesized following the same literature (51). In a typical procedure, 6.0 g of polyvinylpyrrolidone (K-30, >99.5%; Macklin) was mixed with 280 g of 2-propanol and 40 g of DI water to form a clear solution by ultrasonication. 2,2′-Azobisobutyronitrile (AIBN; 1.37 g; Macklin) and styrene (41.6 g) were added into the solution. The resultant solution was purged with nitrogen for 20 min, heated to 70°C, and polymerized for 24 hours with stirring at a rate of 800 rpm. The PS microparticiles were collected after rinsing with ethanol and freeze-dried.

**Positively charged PVDF microparticles**

To demonstrate the broad applicability of our technology, positively charged PVDF microparticles were prepared through polyelectrolyte film modification (52). In a typical procedure, 50 mg of PVDF microparticles (1 to 5 μm, zhonglianshuha) was immersed in the solution containing poly(diallyldimethylammonium chloride) (2 mg/ml, Aldrich) and 0.5 M NaCl for 24 hours. Then, the modified PVDF was rinsed with DI water for three times. The zeta potential of modified PVDF was measured with a zeta potential analyzer (Zetasizer Nano Series; fig. S16).

**Synthesis of colored PMMA MNPs**

**PMMA-550.** The nano-PMMAs with a size around 550 nm were synthesized following the previously reported method (53). Typically, 100 g of methanol (Macklin) and 50 g of distilled water were added into a 250-ml round-bottom flask. Then, 10 g of MMA (Macklin), 0.0375 g of potassium persulfate (KPS, Macklin), and 10 mg of Rhodamine B (Macklin) were added to the mixture. The reaction was carried out with a mechanical stirring at 100 rpm under nitrogen atmosphere at 70°C for 1 hour. The products were rinsed off with DI water and methanol, centrifuged repeatedly to remove the nonreacted materials, and freeze-dried for 24 hours.

**PMMA-3000.** The PMMA microparticles with a size of around 3 μm can be synthesized on the basis of the literature (53). In a typical procedure, 90 g of methanol and 30 g of distilled water were added into a 250-ml round-bottom flask. Then, 10 g of MMA, 0.075 g of KPS, 10 mg of Rhodamine B, and 1 g of poly(vinyl alcohol) (PVA) were added to the mixture. The reaction was carried out with a mechanical stirring at 100 rpm under nitrogen atmosphere at 70°C for 12 hours. The products were rinsed with distilled water and methanol, centrifuged repeatedly to remove the nonreacted materials, and freeze-dried for 24 hours (see fig. S29).

**Experiments of MNP removal**

The removal rate of MNPs was tested in four different water samples: DI water, lake water, Pearl River water, and sewage treatment plant inlet water (fig. S24). MNPs (40 mg; micro-PS, micro-PVC, micro-PMMA, micro-PVDF, micro-PA, nano-PS, and nano-PMMA; 0.2 to 40 μm) were uniformly dispersed in 20 ml of water sample by ultrasonication. For the dynamic adsorption (fig. 4B), 70 mg of SMRs was adopted to identify the dynamic adsorption process. For the adsorption efficiency test, 150 mg of SMRs was added to the MNP-contaminated water for 1 to 2 hours to complete the self-driven cleaning. SMRs carrying MNPs can be easily separated using a magnet, and the captured MNPs can be desorbed by immersing SMRs into a salty solution (2 M NaCl) with gentle stirring or ultrason. The desorbed MNPs can be dried and weighed for the removal rate calculation. Last, the SMRs can be regenerated by immersion in NaOH solution (20 wt%; Macklin) for 0.5 hours followed by DI water rinsing. To demonstrate long-term stability of magnetorobot for potential large-scale application, successive removal experiments of 100 cycles were conducted. The complete cycle includes four major steps: (i) MNP adsorption, (ii) MNP desorption in salty solution, (iii) SMR regeneration in NaOH solution, and (iv) SMR rinse in DI water. For the removal rate evaluation of MNPs in low concentrations, fluorescence quantification was adopted by using a full-wavelength microplate reader (Tecan/Spark 10M). The calibration curve was obtained by recording fluorescence intensity of Rhodamine B–modified MNPs at a series of concentrations (figs. S31 and S32). The removal rate of OH-SMR, CI-SMR, inert resin, filtration, and flocculation was measured with 10 and 20 ppm of colored MNPs.

For the static SMR control experiment, the removal efficiency of magnetically immobilized SMR was measured, with all the other conditions consistent with mobile SMR. As the representative MPs and NPs, micro-PS and nano-PMMA were used for the removal rate measurement.

For the CI-SMR adsorption experiments, PS microparticles were used as an example of MPs and all the other conditions were kept consistent with mobile OH-SMR. We performed multiple experiments to record the motion behavior and adsorption range of CI-SMR as shown in movie S12. The adsorption ranges of CI-SMR and OH-SMR were analyzed on the basis of corresponding videos (fig. S25).

**Low-concentration MNP removal experiment**

For the removal rate evaluation of low-concentration MNPs, fluorescence quantification was adopted by using a full-wavelength microplate reader (Tecan/Spark 10M). The calibration curve was obtained by recording fluorescence intensity of Rhodamine B–modified MNPs at a series of concentrations (figs. S31 and S32). The removal rate of OH-SMR, CI-SMR, inert resin, filtration, and flocculation was measured with 10 and 20 ppm of colored MNPs.

In detail, for the flocculation experiment, the doses of colored MNPs used in the experiment are 10 and 20 ppm, with polyaluminium chloride (PAC; 60 mg/liter) as a coagulation agent. Stirring was maintained at 500 rpm/min for 1 min and subsequently declined to 100 rpm/min for 15 min, with a subsequent 30-min sedimentation. For filtration experiment, the 10- and 20-ppm colored MNPs were
filtered by a funnel with filter membranes (three layers, type: 103, filter speed: 70 to 140 s). Last, the fluorescence intensity of the residual colored MNP supernatant was measured by using a full-wavelength microplate reader (Tecan/Spark 10M). It is worth noting that the concentration of the urban and industrial wastewater varied from 86 to 209 ppm (47).

Magnetic manipulation experiment
The SMR is self-propelled by the unbalanced phoretic flow, while the direction can be controlled by an external magnetic field due to the addition of magnetic component Fe₃O₄ nanoparticles. The precise manipulation of SMR was accomplished with the assistance of an external gradient magnetic field with a tunable strength of 0 to 1 T/m (fig. S27). In detail, the MNP suspension was first dispersed into the PMMA tank (3 cm × 2 cm) on a glass slide, then the SMRs were added, and finally a cover glass was used to prevent the flow of the liquid. Subsequently, the tank was put into the uniform magnetic field space; the direction can be manipulated through the control handle. During the process of MNP absorption, the motile SMR can be maneuvered by the magnetic field and the SMR will migrate along the specific trajectory to adsorb the MNPs.

Closed-loop miniplant experiment
Closed-loop MNP removal was demonstrated in a miniplant composed of four treatment cells (see Supplementary Data for design model). On the basis of the process flow design (Fig. 5A), continuous and large-scale MNP removal can be achieved by repeating the cycle of MNP adsorption, MNP desorption, SMR regeneration, and SMR rinse processes. As shown in fig. 530, 10 ml of contaminated water with dispersed colored PMMA MNPs (2 mg/ml) was first added to cell I for adsorption and then 150 mg of SMRs was added to the MNP-contaminated water. After adsorption, the SMRs were magnetically transferred to cell II for desorption. Next, the SMRs were further transferred to the regeneration cell III, in which OH⁻ in SMRs netically transferred to cell II for desorption. Finally, the SMRs were MNP-contaminated water. After adsorption, the SMRs were magnetically transferred to cell II for adsorption and then 150 mg of SMRs was added to the MNP-contaminated water. After adsorption, the SMRs were magnetically transferred to cell II for desorption. Next, the SMRs were further transferred to the regeneration cell III, in which OH⁻ in SMRs was refreshed by NaOH solution. Last, the SMRs were rinsed by DI water in cell IV, while the reclaimed water in cell I was released. The rinsed SMRs and another batch of contaminated water were added for the next cycle treatment. The desorbed MNPs will be accumulated in cell II as shown by the increased red color. The removal efficiency, MP desorption rate, and SMR regeneration rate (the percentage of SMR after regeneration) were calculated over five cycles.

Confocal imaging of magnetorobot and MP particle
In situ pH profile mapping was recorded with a confocal laser scanning microscope (Leica TCS SP8). 5(6)-Carboxyfluorescein [5(6)-CF; 2 mM] stock solution in DI water was prepared, kept at 4°C in the dark, and can be used for several weeks. The calibration curve of fluorescence intensity as a function of pH was completed by measuring a series of 0.5 μM 5(6)-CF solution with different pH (ranging from 3 to 10) (54). The excitation wavelength was set at 488 nm, and the fluorescence emission spectrum was recorded between 500 and 600 nm (λₘₐₓ = 518 nm). The calibration and pH mapping were conducted with the same setting parameters.

3D motion trajectory of MPs was recorded with a confocal microscope (Leica DMi8). A magnetorobot was fixed on a glass substrate covered by a polydimethylsiloxane (PDMS) chamber that was filled with MP particles (PS with size about 1.5 μm). The microscope was focused at different confocal planes (Z = 0, 4, 8, and 12 μm), and trajectories of particles at each depth layer were traced.

Numerical simulation
The interaction between SMR and MPs was simulated using the commercial COMSOL Multiphysics package (see details in note S1). In the model, Transport of Diluted Species, Electrostatics, and Creeping Flow modules were used to simulate the diffusion field and fluid flow around SMR particles.

Magnetorobot and MP motion measurement and data analysis
The solution with magnetorobots and MP particles was added into a chamber formed by a glass slide and a PDMS reservoir. A bright-field optical microscope (Olympus MX51) and a digital camera (OLYMPUS BX51) were used for video recording. All the videos were recorded with 20x or 50x objective lenses at 1920 × 1080 resolution and 25 frames/s. The motion speed was analyzed by a MATLAB program. Grayscale video frames can be extracted from the video by PhysMo. The centroid coordinates and motion speed of magnetorobot and surrounding plastic particles in each frame were calculated automatically. Average speed and the SD (σₛ) were obtained by analyzing 100 to 200 adjacent speed points. The final speed was given, where z is 1.96 (95% confidence interval) and n is the speed point number. The concentration of salt was measured by a meter for total dissolved solids and electrical conductivity (LD-TDS&EC-001H).

SUPPLEMENTARY MATERIALS
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