Programmable droplet manipulation by a magnetic-actuated robot

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Droplet manipulations are fundamental to numerous applications, such as water collection, medical diagnostics, and drug delivery. Structure-based liquid operations have been widely used both in nature and in artificial materials. However, current strategies depend mainly on fixed structures to realize unidirectional water movement, while multiple manipulation of droplets is still challenging. Here, we propose a magnetic-actuated robot with adjustable structures to achieve programmable multiple manipulations of droplets. The adjustable structure redistributes the resisting forces from the front and rear ends of the droplets, which determine the droplet behaviors. We can transport, split, release, and rotate the droplets using the robot. This robot is universally applicable for manipulation of various fluids in rough environments. These findings offer an efficient strategy for automated manipulation of droplets.

INTRODUCTION
Controllable manipulation of droplets is critical for a wide variety of applications (1–5), such as water collection, medical diagnostics, and drug delivery. Structure-based liquid operations have been widely used both in nature and in artificial materials. However, current strategies depend mainly on fixed structures to realize unidirectional water movement, while multiple manipulation of droplets is still challenging. Here, we propose a magnetic-actuated robot with adjustable structures to achieve programmable multiple manipulations of droplets. The adjustable structure redistributes the resisting forces from the front and rear ends of the droplets, which determine the droplet behaviors. We can transport, split, release, and rotate the droplets using the robot. This robot is universally applicable for manipulation of various fluids in rough environments. These findings offer an efficient strategy for automated manipulation of droplets.

RESULTS
The droplet manipulation system consists of two steel beads and a magnetic control system (Fig. 1A). Here, we name the two steel beads as the “robot” (22–24). The diameter of the beads is 1.2 mm, and the robot is actuated by a magnetic control system, as shown in fig. S1. We can design both the movement and the structure of the robot by the magnetic control system. The sequenced images in Fig. 1 (C to F) show the multiple droplet manipulations using the magnetic-actuated robot (movie S1). We color the water droplets with food dyes for distinction. Different droplet behaviors can be realized by adjusting the robot structure. Figure 1B displays the key parameters of the robot structure, including the diameter of the beads (d) and the distance between the beads (D). The robot is hydrophilic (the characterizations of the beads and the substrate are shown in fig. S2) and can easily capture the droplet after contact. We can transport the droplet using the robot with a proper structure (Fig. 1C). To split a daughter droplet, we adjust the structure of the robot by reducing or increasing the distance between the beads (Fig. 1, D and E). Revolving the robot can facilitate the rotation of the droplet, which greatly accelerates the material mixing in the droplet (Fig. 1E). Besides manipulating large water droplets (250 μl), we also realize the control of micro-drops with volume smaller than 10 μl (fig. S3).

The droplet manipulation processes mainly rely on the structure of the robot, while the volume of the droplet (V) will surely influence the manipulating results. To exhibit the droplet manipulation ability of the robot, we systematically investigate the dependence of the droplet behaviors on the robot structure and the droplet volume and summarize the results in Fig. 2A. Here, the structure of the robot is quantified by the ratio of the beads’ center-to-center distance to the diameter (D/d), as shown in Fig. 2A. In general, droplet transport is achieved with moderate V and D/d; split of a daughter droplet occurs when reducing D/d and enlarging V, while increasing D/d and/or V contributes to the release of the droplet from the robot. For example, the robot with a D/d of 1.67 can transport a 150-μl water droplet, while a daughter drop will be split and moved if the droplet volume

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enlarges to 350 μl. When increasing D/d to 3.33, the 350-μl droplet will be released by the robot, although the smaller droplet (150 μl) can still be transported (fig. S4 and movie S2).

To understand the origin of the diverse droplet behaviors performed by the robot, we conduct the mechanical analysis of this system, including the beads, the droplet, and the substrate, as shown in Fig. 2B. According to the droplet shape evolution (movie S1), the droplet behaviors are determined by the movement of the three-phase contact line (TCL) at the front end (the portion between the beads, as shown in Fig. 2B, top) and the rear end (Fig. 2B, bottom). Three forces decide the movement of the droplet at the front end. The driving force is the adhesion force between the beads and the droplet (F_a), the resisting forces include the elastic force due to the droplet deformation (F_e) and the adhesion force between the droplet front end and the substrate (f_front) (25). The net force at the front end of the droplet is

$$ F_{\text{front}} = 2F_a - F_e - f_{\text{front}} = \int_0^\delta 2\gamma \cos \theta \left( \pi d \cdot \sin \frac{\beta}{2} \right) dl - E \cdot e \cdot A + \gamma \cos \theta_{\text{adv}} \cdot L_1 $$

where γ is the liquid surface tension; δ is the angle between the liquid and the moving direction (Fig. 2B); E is the elastic modulus of
the droplet; $\varepsilon$ is the tension rate of the droplet, which is equal to the stretched length of the droplet to the maximum stretched length (26); $l$ is the contour of the TCL around the bead; $A \approx d \cdot D$ is the cross-sectional area of the liquid between the beads; $\theta_{\text{adv}}$ is the advancing contact angle of the droplet on the substrate; and $L_1 = D - d \cos \alpha$. If the resisting force cannot be overcome by the driving force ($F_{\text{front}} \leq 0$), the TCL between the beads will be pinned, and the droplet will be released by the robot. Otherwise ($F_{\text{front}} > 0$), the front end will be pulled by the robot, and the droplet split or transport will occur, depending on the force balancing at the rear end. Two forces determine the movement of the rear end of the droplet: the driving force arising from the moving of the droplet front end ($F_3$) and the resisting force arising from the liquid-solid adhesion on the rear end ($F_{\text{rear}}$). The net force at the rear end ($F_{\text{rear}}$) is given by

$$F_{\text{rear}} = F_3 - F_{\text{rear}} = E \cdot \varepsilon \cdot A - \gamma (\cos \theta_{\text{rec}} - \cos \theta_{\text{adv}}) L_3 - \cos \theta_{\text{adv}} L_2$$

(2)

where $\theta_{\text{rec}}$ is the receding contact angle of the droplet on the substrate, $L_2$ is the width of the thinnest liquid bridge behind the beads, and $L_3$ is the maximum width of the TCL contour between the droplet and the substrate. The droplet can be transported if the net force ($F_{\text{rear}}$) is positive; otherwise, a daughter drop will be split and transported. The detailed mechanical analysis and the phase diagram analysis are provided in sections S1 and S2. In addition, other factors, such as the droplet surface tension, the substrate fraction, and the size of the beads, also influence the droplet manipulation functions. Detailed analyses are shown in section S3.

Besides controlling the water droplet behaviors in the air, we systematically investigated the generality of the robot under different conditions (movie S3). Manipulation of oil or water drops under liquid environment is crucial to micro-organogel printing (27), soft robot fabrication (24), and emulsion reactions (28), especially because the reagents are sensitive to the atmospheric environment. After proper surface modification, the robot can be used to transport oil drops under water, move water drops under oil, and even collect gas bubbles under water. As shown in Fig. 3A, carbon tetrachloride (oil) droplets dissolved with bromine and styrene are placed under water. Dragged by the superhydrophobic beads, the two droplets approach and coalesce for the unsaturated bond detection (29).

![Fig. 3. Generality demonstration of the robot.](image)

**Fig. 3. Generality demonstration of the robot.** (A) Oil droplet manipulation under water. The oil droplets (100 $\mu$l) are CCl₄-dissolved with Br₂ (left) and styrene (right). The robot transports the left droplet to mix with the right one. (B) Water droplet manipulation under oil (n-heptadecane). The droplets (50 $\mu$l) are water-dissolved with KSCN (left) and FeCl₃ (right), respectively. The left droplet is captured by the robot and transported to the right one. (C) Gas bubble manipulation under water. A superhydrophobic robot can successively collect the gas bubbles (20 $\mu$l). The white dotted line indicates the trajectory of the robot. (D) Manipulation of a water droplet on the upright surface. The robot transports a 20-$\mu$l water droplet to move up and down with a speed of 2 mm/s. (E) Manipulation of a water droplet inside a tube. A droplet (20 $\mu$l) is actuated by the robot to capture the impurity inside a tube. After 1 min, the impurity is dissolved and taken away by the droplet. The red dashed circle indicates the location of the impurity. The black arrow indicates the movement of the droplet. Scale bars, 10 mm.
with another aqueous droplet (Fig. 3B). In addition, Fig. 3C shows that the superhydrophobic robot can also successfully capture and collect gas bubbles distributed under water, which shows potential for removing bubbles in microfluidic devices (30). The key principle for specific surface modification of the robot is that the contact between the robot and the manipulated fluid (fluid-2) cannot be replaced by the contact between the robot and the bulk fluid (fluid-1). The quantitative judgment criterion for the effective surface modification is

$$\cos \alpha = \frac{\gamma_{1,s} - \gamma_{2,s}}{\gamma_{1,2}} > 0, \alpha < 90^\circ$$  \hspace{1cm} (3)

where $\gamma_{1,s}$, $\gamma_{2,s}$, and $\gamma_{1,2}$ are the interface energies of the fluid-1/bead, fluid-2/bead, and fluid-1/fluid-2 interfaces and $\alpha$ is the contact angle. This means that if we intend to manipulate fluid-2 in the bulk fluid-1, the contact angle between fluid-2 and the bead should be smaller than 90° (see the detailed analysis in section S4).

Moreover, we demonstrate the applicability of the robot in rough environments, as most of the reported droplet manipulation methods become invalid because of the uneven surface or the limited operating space (31). Figure 3D displays the droplet manipulation on an upright surface. A colored water droplet can be dragged to move up and down by overcoming the gravity and adhesion force between the droplet and the substrate. The robot can also be applied in limited spaces, such as boxes and tubes, which is extremely important in microfluidics and clinical medicine (30). As shown in Fig. 3E, a tube is stained with impurities (NaCl) adhering to the tube. By manipulating a droplet of water as the washing agent, the impurity can be easily dissolved and wiped off from the tube.

Lossless transport and precise control of reagents are notable in quantitative chemical reactions, especially in the micro-reaction, which is widely used in analytical chemistry, diagnostics, and biotechnology (32). For illustration, we perform sequential acid-base neutralization reactions through the programmable droplet manipulation using the robot, as shown in Fig. 4A. First, we split a daughter drop with a volume of 1.5 $\mu$l from a NaOH droplet (left) and transport it to a neutral phenolphthalein droplet (middle) by the robot with a proper structure of $D/d = 1.67$. The effect of the experimental parameters on the daughter drop volume is analyzed in section S5. After being merged and mixed with the daughter droplet, the phenolphthalein droplet turns from colorless to pink, indicating that the solution is alkaline. Then, we take a tiny drop of HCl solution from a mother droplet (right) and mix it with the alkaline phenolphthalein droplet (middle). The color becomes colorless because of the neutralization reaction (movie S4). It is noteworthy that we can accelerate the mixing of liquid by rotating the droplet, and the quantitative mixing efficiency evaluation is shown in section S6. Our robot provides a versatile route to automatically control the droplets for micro-reactions, especially when the reagents are toxic, radioactive, or explosive. In fig. S6 and movie S5, we show that the toxic luminol reagent is prepared and manipulated by the robot for ferrum detection (33).

Last, we explore the potential of the robot in in vivo medical applications by simulating typical biomedical processes. Calculi are minerals deposited in organs such as kidneys and gallbladders (34).
In clinical medicine, it is difficult to remove such structures surgically when their size is too small (35). In Fig. 4B, we show that the robot may be used to collect and remove the simulated calculi pieces. A drug droplet is dragged to the calculi that are randomly placed on the substrate. Because of the interface compatibility between the drug and the calculi, we can easily collect and take the calculi away from the substrate (movie S6). In addition, we demonstrate that the robot has potential for clearing the blood vessels. A surfeit of cholesterol can build up in coronary arteries and increase the chances of a stroke or heart attack (36). As shown in Fig. 4C, we use a tube filled with water to imitate the coronary arteries. The robot transports a drug droplet and captures the simulated plaque deposited inside the tube. After 1 min, the plaque is dissolved in the drug and is taken away by the robot. Furthermore, the demonstration of the drug delivery is given in fig. S7 and movie S8. These demonstrations may provide a new idea for in vivo medical applications (22, 37).

DISCUSSION

We propose a simple and general strategy for manipulation of droplets by a magnetic-actuated robot made of two steel beads. We can control the structure of the robot by the magnetic field, which determines the distribution of the resistive forces at the front and rear ends of the droplet. Multiple behaviors of droplets, including transport, split, release, and rotation, are realized by adjusting the robot structure. In addition to manipulating water droplets in air, the robot is effective for complex liquid systems such as oil in water, water in oil, and gas in water. The robot can manipulate droplets in limited spaces, on inclined surfaces, and under harsh conditions such as toxic and radiative environments. It shows great potential in the fields of device fabrication, sensing and bioassay, and in vivo medical applications. In the future, smaller droplet manipulation (in the nano- or picoliter scale) and the biocompatibility of the system should be considered.

MATERIALS AND METHODS

Preparation of the magnetic-actuated robot

The magnetic-actuated robot consists of two components: a couple of steel beads and a magnetic control system. First, we placed two magnets on a motorized precise translation stage (stage I), which can control the distance between the magnets. Stage I is fixed on a rotary stage (stage II), which can control the distance between the magnets. Stage I is fixed on a rotary stage (stage II), which can control the magnets to rotate with adjustable speeds. Stage II is placed on a two-dimensional motorized precise translation stage that can drive the system to move in $X/Y$ directions. Then, we placed a silicon plate on top of the magnets, and the steel beads were attracted by the magnets through the silicon plate. The cylindrical magnets are 5 mm in length and 1 mm in diameter. The intensities of the magnetic field on the surfaces of the magnet and the silicon plate are 150 and 25 mT, respectively. The diameter of the beads is 1.2 mm.

We prepared three kinds of beads with different surface modifications to manipulate the water droplets, the oil droplets, and the gas bubbles. The beads for the manipulation of water droplets were washed by ethanol and deionized water, with a receding contact angle of 10.5 ± 1°, indicating that they are highly adhesive. Note that we used the steel sheets that are of the same material with the beads for the contact angle measurement. These beads were used for the experiments in Figs. 1 (C to F), 2B, 3D, and 4 (A and B). The beads for water manipulation under oil, displayed in Fig. 3B, are superhydrophilic. To fabricate superhydrophilic beads, we first used the mixed solution of hydrochloric acid (40 ml), hydrofluoric acid (2.5 ml), and deionized water (12.5 ml) to etch the beads. Then, the beads were washed with ethanol, acetone, and deionized water and blow-dried with nitrogen. The beads for oil and gas manipulation under water, in Figs. 3 (A and C) and 4C, are superhydrophobic with a receding contact angle of 157.8° ± 3°. These beads were obtained by modification of the superhydrophilic beads with $\text{1H,1H,2H,2H-}$perfluorodecyltrimethoxysilane (PFOTS) by chemical vapor deposition (CVD) at 80°C for 4 hours.

Preparation of the substrates

The hydrophobic substrates used in Figs. 1 (C to F), 3D, and 4A are silicon wafers modified with PFOTS by CVD at 80°C for 4 hours. The advancing and receding contact angles of these substrates were 115.5° ± 2° and 86.3° ± 2.8°, respectively. The test of oil droplet manipulation shown in Fig. 3A was carried out under water on commercial filter paper. The tests in Fig. 3 (B and C) were conducted in a polystyrene plastic dish. The substrate in Fig. 3E is a hydrophilic glass tube, which is pickled with sulfuric acid and H$_2$O$_2$ at 220°C for 6 hours and then modified with PFOTS by CVD at 80°C for 4 hours. The substrate in Fig. 4C is a superhydrophilic glass tube, which is pickled with sulfuric acid and H$_2$O$_2$ at 220°C for 6 hours.

Various chemical reactions

The droplets displayed in Figs. 1 (C to F), 2B, and 3 (D and E) are water droplets colored by edible dyes with a volume ratio of 1:100 (dye/water). In Fig. 4A, the NaOH droplet and the HCl droplet were colored by yellow and green edible dyes with a concentration of 1 M, respectively. The concentrations of the Br$_2$/CCl$_4$ and styrene/CCl$_4$ droplets in Fig. 3A are 0.1 M. In Fig. 3B, the concentrations of the KSCN/H$_2$O and FeCl$_3$/H$_2$O droplets are 0.1 M.

Simulated medical applications

The experiment of the simulated calculi removal, shown in Fig. 4B, was conducted on a polypropylene substrate. The simulated calculi are made of calcium carbonate. The drug droplet is a water droplet colored by yellow edible dyes with a volume ratio of 1:100 (dye/water). The volume of the drug droplet is 50 μl. The experiment of the vascular clearance was conducted in a glass tube (Fig. 4C). The tube was filled with water. The drug droplet is a CCl$_4$ droplet with a volume of 5 μl. The simulated plaque is a mixture of cholesterol and butter.

SUPPLEMENTARY MATERIALS

Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/6/7/eaay5808/DC1

Section S1. Mechanical analysis of the droplet behaviors manipulated by the magnetic-actuated robot

Section S2. Detailed analysis of the phase diagram

Section S3. Analysis of factors affecting droplet manipulation behaviors

Section S4. Principle of the bead surface modification

Section S5. Analysis of factors influencing the volume of daughter drops

Section S6. Quantitative evaluation of the mixing efficiency

Fig. S1. Scheme of the magnetic-actuated robot.

Fig. S2. Contact angle characterization.

Fig. S3. Micro-droplet manipulation.

Fig. S4. Demonstration of the droplet behaviors influenced by $D/d$ and $V$.

Fig. S5. Demonstration of the luminol reaction.

Fig. S6. Demonstrations of the drug delivery using the magnetic-actuated robot.

Movie S1. Typical behaviors of the droplets manipulated using the magnetic-actuated robot.

Movie S2. Demonstration of the droplet behaviors influenced by $D/d$ and $V$.
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