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Review

Manipulation of microfluidic droplets by electrorheological fluid

Microfluidics, especially droplet microfluidics, attracts more and more researchers from diverse fields, because it requires fewer materials and less time, produces less waste and has the potential of highly integrated and computer-controlled reaction processes for chemistry and biology. Electrorheological fluid, especially giant electrorheological fluid (GERF), which is considered as a kind of smart material, has been applied to the microfluidic systems to achieve active and precise control of fluid by electrical signal. In this review article, we will introduce recent results of microfluidic droplet manipulation, GERF and some pertinent achievements by introducing GERF into microfluidic system: digital generation, manipulation of "smart droplets" and droplet manipulation by GERF. Once it is combined with real-time detection, integrated chip with multiple functions can be realized.

Keywords:
Droplets / Electrorheological / Microfluidics

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1 Introduction

1.1 Microfluidics

Microfluidics deals with the behavior, precise control and manipulation of fluids that are geometrically restricted to small, typically sub-millimeter scales. From the time of the field's origin approximately two decades ago, many studies and investigations have been carried out intensively [1]. As it is multidisciplinary, intersecting engineering, physics, chemistry, microtechnology and biotechnology, microfluidics generates an equally diverse array of applications ranging from drug delivery, point-of-care diagnostic chips to organic synthesis and microreactors [2–4]. Great promise has been shown in that it enables integration of multiple steps of complex analytical procedures, microliter consumption of reagents and samples and portability [5–7]. Also, synthesis in microfluidic reactors provides a powerful strategy for continuous, reproducible and scalable production of inorganic, organic and bio-organic products [8–13]. Microfluidic technology has been employed in the development of inkjet print-heads [14], lab-on-a-chip technology [15, 16] microthermal technologies [17], *etc.* Many applications have been developed, such as microlenses [18], high-throughput screening [19, 20], nano-/microparticle separation [21], on-chip NMR [22], cell culture [23], DNA and protein study [24–27] as well as many other biology analysis [28, 29] and testing.

Microfluidic systems are characterized by the low-Reynolds number ($Re = \frac{\rho v D}{\mu}$; ρ: density; v: velocity; D: characteristic length μ: dynamic viscosity) flow regime under which all fluid flow is essentially laminar. Continuous-flow-based systems exploit this phenomenon, allowing for the creation of many novel microenvironments [30]. Many applications are based on this phenomenon [31–33]. Although continuous-flow devices offer fine control of flow characteristics, scaling up is a challenge, as the size of devices increases almost linearly with the number of parallel experiments. For this reason, droplet-based microfluidics, with which a large number of reactions can be run without having to increase device size or complexity, has attracted more and more interest [34]. Certainly, chemical reactions and biological testing have greatly benefited from the unique advantages afforded by tiny drops ranging from nano- to pico-liter size [35, 36].

1.2 Droplet-based microfluidics

In the form of droplets, reagents are conveyed precisely in discrete volumes, enabling high-throughput chemical reactions [2, 37] and single-cell manipulation in bio-testing [35, 38, 39]. It provides a promising avenue for spatially and temporally resolved chemistry [40], which can be used for inexpensive and potentially improved measurements of kinetic and binding constants, as well as measuring aspects of phase and reaction diagrams of multicomponent systems [40, 41]. Furthermore, mixing of reagents in droplets has

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Abbreviations: ER, electrorheological; **ERF,** electrorheological fluid; **GER,** giant electrorheological; **GERF,** giant electrorheological fluid; **MFF,** microfluidic flow-focusing been proved to be achievable within milliseconds, making multistep chemical reactions *via* droplet microfluidics possible [42]. Nowadays, it is not only applied in DNA and protein analysis [43–47], immunoassay [48] and chemistry [49, 51], but also can achieve some physicial measurement [52], bubble logic [53] and clinical application [54–56]. Droplet-based microfluidics involves the generation, detection and manipulation of discrete droplets inside microdevices [42, 57]. A detailed introduction can be found in Ref. [34].

1.2.1 Droplet generation

The special utility of droplet-based microfluidic systems lies in the formation of uniform droplets and particles; thus, intrinsic to such systems, and of utmost importance, is precise control of the size, shape and monodispersity of droplets. Howard A. Stone's group integrated flow-focusing geometry into a microfluidic device. Thereby they could form drops much smaller than the orifice, and could produce both mono- and polydisperse emulsions [57–59]. This microfluidic flow-focusing (MFF) method is often used in droplet/bubble formation [60, 61], the generation of double emulsions [62, 63], multifunctional particles and microbeads [64]. It could also perform as a bioreactor for bacteria with long-term culture and monitoring [65]. In our previous work, we employed this method, together with polymerization processing and droplet encapsulation, for targeted delivery and release [66]. Another geometry-based generation method uses T-junction, by which two immiscible fluids are brought together. Basic research into this method has been conducted by Quake's group [67] and others [68]. Also, many passive generation control methods have been developed, such as external force [69], acoustic wave [70], electrolysis [71], high-voltage pulses [72], electrowetting [73] and thermocapillary [74].

1.2.2 Manipulation of droplets

Besides geometry-based pressure control for droplet mixing [75], merging [76, 77] and sorting [78, 79], many active control methods have been realized recently, such as hydrostatic pressure [80], temperature gradient [81], thermal expansion [82], optical approaches [83–85], magnetic field [86] and electrical control [87–89], including electrostactic [90], electrokinetic effect [91], dielectrophoresis [92] and electrowetting [93–98]. Among them, electrowetting on dielectric [99–101] has been proved to be very effective for cutting, merging, creating and transporting liquid droplets, while its disadvantage is that many electrodes should be fabricated with metallic thin film, causing droplet moving and device fabrication complex, while, with GERF, one can control and manipulate the droplets by much fewer electrodes [102, 103].

Moreover, it is noteworthy that the size of droplets in the microfluid is small enough to reduce fluid volume consumption, and large enough for the participation of nano-/microparticles. Also, the additional functional nano-/microparticles add new functionalities and make some processes easier. Many types of smart materials have been reported, such as magnetorheological fluid and electrorheological (ER) fluid, which can respond quickly to magnetic or electric fields. Some preliminary work has been done with magnetorheological fluid or ferrofluid for the microfluidics. Hatch *et al.* [104] used ferrofluid to design a micropump, while Hartshorne [105] used it as a valve and pump to control water/air. However, few results have been reported to date for the microfluidic applications of ER fluid.

1.3 GERF

ER fluids are a type of smart material composed of dielectric particles suspended in insulating oil and can be treated as a two-phase system. Owing to the dielectric constant contrast between the solid particles and the liquid in a colloid, each solid particle is polarized under an electrostatic field, with an effective dipole moment. The resulting (induced) dipole–dipole interaction determines that the particles tend to aggregate and form columns along the applied field direction. This formation of columns explains why the high-field state of an ER fluid exhibits increased viscosity or even solid-like behavior that can sustain shear in the direction perpendicular to the applied electric field [106–114] (as shown in Fig. 1). The response time of electrorheological fluids (ERFs) can be as short as a few milliseconds. Given such marvelous features, ERFs can serve as an electric–mechanical interface, and when coupled with sensors to trigger an controllable electric field, can render many devices such as clutches, valves, dampers and others active mechanical elements capable of responding to environmental variations – hence the denotation, ''smart'' fluid. Such a diverse applications potential [106–112] has made ERFs a perennial area of study in soft matter research, ever since their discovery six decades ago. Despite the broad interest, however, applications have been retarded by the inadequate yield stress of conventional ER fluids. Considerable improvement efforts have gone into the preparation of alternative suspended particles. These include semiconductive polymers [115–118], metal- or metal-oxide-doped titania or titanate [119–122], intercalated clay composites [123, 124], organic-layer-modified titania or silica [125–127], mesoporous composites and nanocomposites, and still others [128–130]. A breakthrough has been achieved in recent years, with the discovery of the GER effect, which represents a different paradigm from the conventional ER mechanism, offering a GER yield stress value up to 300 kPa, which provides some potential realistic applications in various devices [131–135].

2 Droplet manipulation by GERF

Some studies have applied GERF to microfluidic chips such as micropumps [136] and mixers [137]. What is more, a new

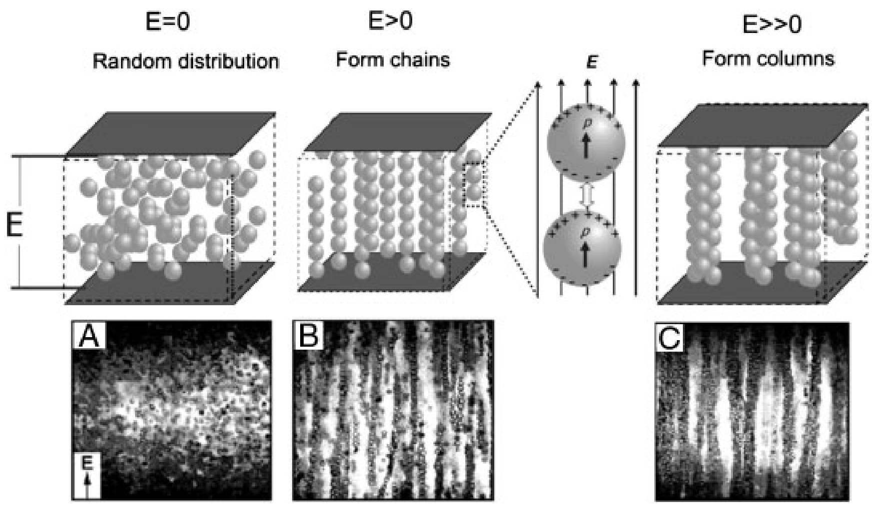

Figure 1. The structural evolution of dielectric microspheres under an increasing electric field, from (A) no field, to (B) a moderate field of 500 V/mm, to (C) a strong field of 900 V/mm. Here the ER fluid consists of 1.5 micron glass spheres suspended in silicone oil.

kind of material, a PDMS-based conducting composite [138] composed of nano/micro conductive particles in a PDMS matrix, has been used in the fabrication of microheaters [139] and microthermo-indicators [140]. It provides the embedded electrodes for applying an E-field in channels.

Two methods of applying the GERF in microfulidics have been developed: The first approach is that GERF is used as flowing droplets carried by another fluid, while the second one is that GERF is used as a carrier fluid to control other liquid droplets or bubbles. A schematic view of the two cases is shown in Fig. 2.

2.1 GER smart droplets [102]

With the generation method shown in Fig. 2A, controlled generation according to different sizes, frequencies and phases is possible by applying an electric field through the control electrodes. Without electrical control, GER droplets can be generated in a passive scheme, which is dependent on the flow rate and the oil to GERF ratio, as shown in Figs. 3A and b. However, by applying an electrical control in this design, very uniform GER droplets – stable over a wider range of flow rates were obtained. This is seen in Figs. 3C and d, where the generation period is obtained under the same flow rate ($R_{GER}=0.6$ mL/h). A series of experiments was carried out to investigate the relationship between the droplet length and the frequency (and duty cycle) of the electric pulse at different GERF flow rates. The results are shown in Fig. 3. The period T of the electric pulse is noted to be a very critical parameter for stable GER droplet production. For example, smart droplet generation becomes unstable when T is beyond 100–1000 ms working range for the flow rate of $R_{GER} = 0.4$ mL/h [141]. However, by controlling two or more GER inlets independently in the stable region, GER droplet synchronization and relative phase variation were easily achieved using the same approach. The generated GER droplets are further controllable by the electrodes in the downstream. In this experiment, it is found that the

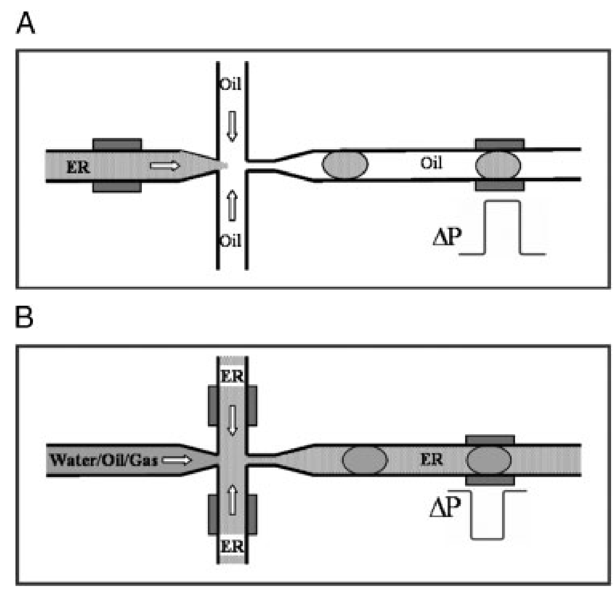

Figure 2. Schematic view of GER droplet generation and control in microchannels.

differential pressure induced by the GER droplets can be adjusted readily by varying the strength of the E-field, drop size and GER particle concentration. Thereby, the channel flow can be slowed down or fully stopped. By trapping or releasing GER droplets in different branches, the encoded droplets can be stored or displayed in the desired channels, forming certain patterns.

Moreover, it is well known that many chemical reactions as well as bio-processing usually require accompanying water or other liquids. Therefore, control and sorting of water droplets are very critical and challenging functionalities. Smart droplets offer an alternative to water droplet control and labeling by injecting GER droplets among water droplet chains. The design of such a chip is showed in [102]. By adjusting the injection frequency and phase of GER droplets, one GER droplet can lead to various numbers of water droplets. Thus, by controlling the number of GER droplets, the number of water droplets can be precisely controlled, and thereby, indirect control of water droplets can be realized. Together with further manipulations, such as droplet separation and merging, it will be helpful in multistep chemical reactions and analysis.

2.2 Droplet control through GER carrier fluid [103]

With the second-generation method shown in Fig. 2B, GERF is used as a carrier fluid. The stable generation range at different flow rates with both the MFF and T-junction approaches are investigated, and it was found that at the same flow rate, droplet control through GER carrier fluid could generate stable droplet chains over a larger frequency range, whereas passive generation could include only one frequency. Droplet control through GER carrier fluid can control not only one kind of droplet generation but also the phases of different kinds of droplets. Figure 4 shows two kinds of droplets generated in the same phase or contrary phases. Another advantage is that, by controlling the carrier fluid, droplets can be encoded and stored. Such controlled droplets can consist of different fluids or even gas.

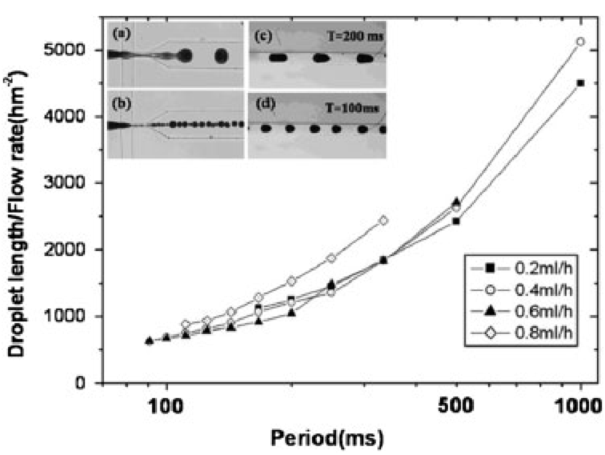

Figure 3. Smart droplet generation under different GERF flow rates, with droplet length (normalized by flow rate) plotted as a function of the period *T* of the electrical control signals (applied to electrode 1). Insets (a)–(d) are images of smart droplet generation in which (a) and (b) show the stable and unstable droplet generation with no electrical signals applied. Here (a) shows stable generation under a low flow rate of 0.2 mL/h and (b) shows unstable generation under a high flow rate of 4 mL/h for the GERF. (c) and (d) show stable generation under both low and high flow rates to be achievable with the application of electrical control signals (ref. [102]).

The use of flow rate control to switch the order(s) in a train of droplets is also illustrated. If one regards a train of different droplets as a coded message, such switching implies the ability to revise or correct the message. The detection circuit and the parallel electrode fabrication method, which will be briefly introduced, are integrated together. If the droplets are detected, the control box will be triggered to send control signals to the electrodes located on the downstream branches. The on/off control signals on these electrodes can thus be varied according to the requirements. Once the two droplets enter the exchange loop, their direction and relative residence time in the exchange loop can be controlled by the E-field status on the electrodes of the branches. So, by properly controlling the on-off duration of the applied voltages, the droplets resume their movement into the downstream main channel, but with their order changed. Moreover, not only will the order be changed, but also the distance between the droplets can be adjusted on demand. Such mutability would be very useful in droplet control for biosystems and in microfluidic computing.

Bubble generation control is an interesting topic, especially as pertains to digital microfluidics [142], and it is also very useful to biosystems applications. With this approach, bubble size, flow direction as well as the separation distance between bubbles can be controlled using a similar MFF generation device.

3 Droplet detection

Usually, droplet generation rates could be higher than 2000/s. Besides, generation and manipulation of droplets also involve discontinuous pressure changes (*e.g.* during

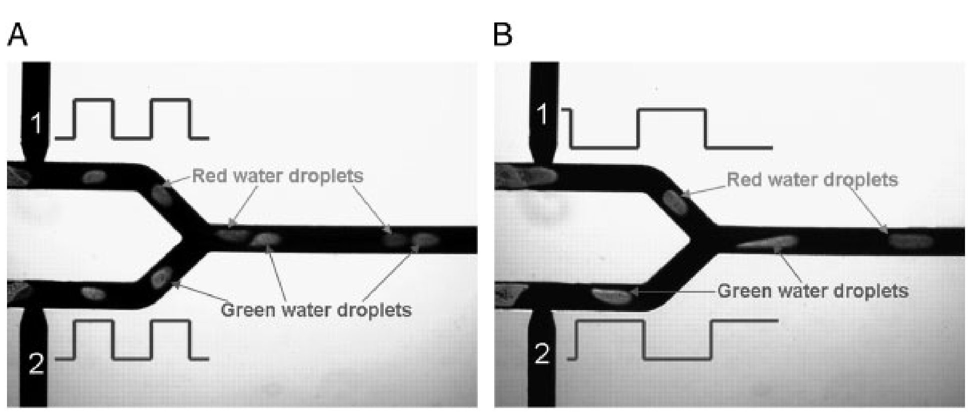

Figure 4. Controlled generation of two types of water droplets: (A) with the same phase of the control signals and (B) with opposite phase of control signals. The solid lines represent the control electric signals applied to the electrodes located next to channels 1 and 2 [103].

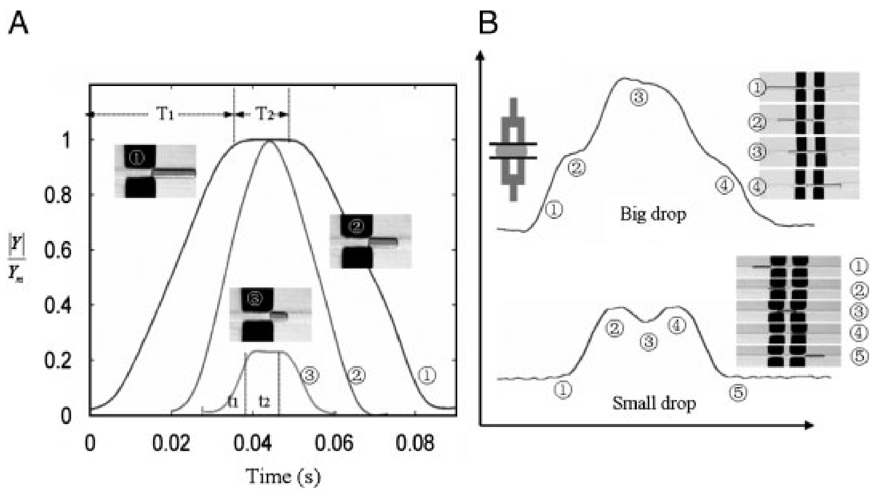

Figure 5. (A) Detected capacitive signals corresponding to droplets with lengths that are larger than (case 1), equal to (case 2) and smaller than (case 3) the electrode width. The insets are the corresponding micrographs. (B) The detected voltage signals for big and small droplets passing through fork-shaped electrodes. The left upper inset is the schematic illustration. The right insets are the optical images and are marked according to droplets' positions.

droplet fusion, fission or the droplet formation process) from the continuous phase [143, 144]. In the method introduced above, droplet size and distance vary spatially and temporally. Therefore, a real-time detection of droplets is crucial for accurate feedback generation controlling and manipulation. Sensitive detection with microfluidic analytical devices is very challenging since extremely small detection volumes are usually available for the experiments [145]. Many methodologies have been introduced other than laser and fluorescence [146, 147], such as optical methods including absorbance [148, 149], chemiluminescence [150–152], refraction and thermo-optics, as well as electrochemical methods [153] including amperometry [154], impedance sensing [155] capacitor detection [156], *etc.* There are also some new developments in progress, such as miniaturized plasma-emission spectrometry and sensitive detection for gas-chromatographic separation. General introductions and comparisons can be found in Ref. [157]. Semiconductor techniques have also been introduced to the microfluidic system for detection and active control [158, 159]. However, not all of them are suitable for droplet detection.

A detection method based on capacity determination mechanism is delivered together with an associated electronic circuit based on the resonance mechanism: R-L-C resonance [160]. Droplets and the carrier fluid have different dielectric constants ε, and thus the capacitance difference can be determined with and without desirable droplets passing through the two parallel electrodes. The detected signals are shown in Fig. 5A. From such signals, droplet length and velocity can be derived directly. To measure the signals more precisely, fork-shaped electrodes were brought forward, resulting in the new waveforms for the different droplet sizes, shown in Fig. 5B. That type of electrode could effectively distinguish the sizes by the amplitude and shape of the waveforms of the detected signals.

An important material used in this detection is a new type of conducting composite that is a mixture of micro-/nano-Ag/C particles and PDMS material [138]. A multistage process is employed to simplify the fabrication incorporating the two embedded parallel electrodes on opposite sides of microchannel to achieve both good detection and fine observation.

As the GERF, water and carrier fluid always have different dielectric constants, capacitances and resonances reflecting different droplet compositions, they can thus give rise to significantly different signal amplitudes. This design is *in situ*, real-time, sensitive, accurate, fast (>1100 Hz) and requires no special sample preparation. GERF, together with the detection circuit, could be very useful to achieve accurate control of droplet microfluidics. One of such applications, droplet's order exchange, has been shown in Ref. [103].

4 Concluding remarks

In this article, we reviewed the application of GER fluid in droplet microfluidics. Conbined with a new kind of real-time detecton approach, active accurate droplet generation and manipulation could be realized. Although only a few works have been carried out by applying this smart material into microfluidics, the realistic devices like ER-activited microvalve, pump, mixer, *etc.* provide the potential utilizations of smart materials. As the better part of droplet manipulation can be effectively controlled, we believe that many additional chemical reaction, bio-testing as well as precise element analyses could be performed by integrating these functions into a micro total analysis system.

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