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Enhancing safety in small confined spaces with thermally triggered fire-extinguishing microcapsules from microfluidics†

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Fires in small confined spaces have problems such as difficulty extinguishing, fast burning speed, long duration, strong concealment, and untimely warning. Perfluorohexanone-based fire-extinguishing microcapsule technology provides an important solution to overcome these problems. However, due to the poor solubility and high volatility of perfluorohexanone, the preparation of perfluorohexanone fire-extinguishing microcapsules (FEMs) with a high encapsulation rate, good homogeneity, and low processing costs is still a great challenge. Here, we propose a microfluidic flow-focusing technique to realize efficient encapsulation of perfluorohexanone. It is shown that FEMs can spray fire-extinguishing agents at high speeds in the presence of external heat, and only one FEM is needed to extinguish a candle flame much larger than its size. Meanwhile, the extension of FEMs to two-dimensional fire-extinguishing patches (FEPs) has achieved significant results in suppressing fire and preventing fire spread, which is expected to further expand its application in various fire suppression scenarios.

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1. Introduction

The demand for fire safety technology in small confined spaces, such as aerospace and lithium battery boxes, is increasing rapidly.^{1,2} Fire in space microgravity conditions is often confined to a small area with no open fire because of the lack of natural air circulation, making it hidden and difficult to find.^{3–5} Even if the fire signal is captured, astronauts cannot extinguish the fire in a timely manner due to the tight environment of the spacecraft and the constraints of the zero-gravity factor in space. At present, fire suppression by means of water mist is mainly studied in manned spacecraft. The microgravity environment is conducive to the formation of water mist and the expansion of fog fields, but microgravity will also reduce the momentum of fog, resulting

in a decrease in the ability of fog droplets to penetrate the flame. In addition, it has been reported that more than 10% of fire accidents in nuclear power stations and 73.08% of photovoltaic power station accidents are caused by initial thermal anomalies in small and confined spaces that cannot be promptly warned about or extinguished.^{6–8} Fire in limited space is difficult to fight due to its fast burning speed, long duration, high concealment, and lack of timely warning.

In recent years, fire detection and automatic fire-extinguishing devices have received widespread attention. However, conventional temperature and smoke sensors typically activate the alarm only after the fire has reached a certain degree of development.^{9–11} The storage or non-storage type has a large volume and is difficult to apply in small confined spaces. The use of heptafluoropropane has been significantly reduced or even banned in some cases due to its high latent heat value and contribution to global warming. The spray mode based on atomizing nozzles makes it difficult for the extinguishing medium to accurately reach the intended extinguishing position due to factors such as the injection angle, distance, or retarder under actual working conditions.^{12,13} To improve the fire-extinguishing performance in small confined spaces, it is urgent to study a fire-extinguishing technology that considers high-efficiency fire extinguishing, cost-effective storage, automatic temperature response, and efficient release of fire-extinguishing agents.

Perfluorohexanone microcapsule fire-extinguishing technology provides an important technical solution to solve

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the problem of fire in small confined spaces.^{14–19} Perfluorohexanone, with its non-conductive, volatile, non-residual, easy photolysis, zero ozone depletion, low global warming potential, and other excellent performance, is considered to be the best alternative to halon fire-extinguishing agents. In 2001, the company 3 M took the lead in the introduction of Novec-1230, a halogenated hydrocarbon fire-extinguishing agent whose main component is perfluorohexanone.²⁰ The agent achieves an efficient fire-extinguishing effect through the dual role of physical cooling and chemical inhibition. It is worth noting that the microencapsulation of perfluorohexanone further solves the problems of storage and application and also eliminates the dependence on sensors and space limitations. However, research on microencapsulation technology for perfluorohexanone and similar liquid fire-extinguishing agents is still at an early stage.^{21,22} Due to the poor solubility and high volatility of perfluorohexanone, traditional microencapsulation methods, such as *in situ* polymerization, interfacial polymerization, and composite condensation, are facing great challenges in terms of encapsulation efficiency, homogeneity, controllability, and processing cost.^{23–25} Therefore, exploring new preparation methods is crucial to promoting the application of perfluorohexanone microencapsulation in fire suppression.

Microfluidic technology serves as a versatile platform for the meticulous manipulation of small volume fluids, affording precise control over the generation of uniform emulsions, particles, and microcapsules.^{26–30} Its applicability extends to a diverse array of encapsulation materials, effectively addressing challenges in microencapsulation. The utilization of microfluidic methods facilitates accurate fluid control, yielding benefits such as uniform droplet size and structured microdroplets. The microfluidic techniques employed in our work, including flow focusing, not only boast high throughput suitable for industrial production but also permit meticulous adjustment of capsule size and shell thickness by effectively manipulating parameters such as flow rates. This customization is tailored to specific practical applications.

Here, a novel approach is proposed to achieve the high-loading-rate microencapsulation of perfluorohexanone fire-extinguishing agent with retardant photocurable resin in a one-step method through flow-focusing microfluidic technology.^{31–39} The process allows regulation of the size and shell thickness of nano-liter perfluorohexanone fire-extinguishing microcapsules (FEMs), providing a new research idea for the high-efficiency microencapsulation of perfluorohexanone, as shown in Fig. 1(a). The coaxial flow-focusing nozzle is schematically depicted in Fig. 1(b). When the local temperature abnormally rises to the set detonation temperature, the internal pressure of perfluorohexanone is heated to vaporize and increases until it breaks through the capsule wall and releases the extinguishing agent to achieve a cooling and fire-extinguishing effect. In addition, fire-extinguishing patches (FEPs) have been prepared based on

FEMs, which are suitable for adhering in small confined spaces to achieve effective fire extinguishing, as shown in Fig. 1(c). When an electrical socket burns due to a short circuit or aging, the FEP releases the extinguishing agent upon a thermal trigger. This research will provide an important theoretical basis and technical support for fire suppression research in narrow-confined spaces, such as aerospace and lithium battery boxes, among other fields.

2. Results and discussion

2.1 Preparation and characterization of FEMs

Flow focusing has made significant advancements in the large-scale preparation of microcapsules by microfluidic technology, involving the interface flow of a three-phase, two-layered fluid. The coaxial stretching of the inner and outer layer interface makes the outer phase fluid wrap the inner phase fluid, forming a coaxial micro-jet through the focusing hole. As the interfacial disturbance propagates and grows, the coaxial jet breaks up downstream, resulting in the generation of double-emulsion droplets with a core-shell structure. In this research, the influence of process factors such as fluid flow on microcapsule shape was investigated. This paper proposes to complete the high loading rate microencapsulation of perfluorohexanone fire-extinguishing agent with retardant photocurable resin in a one-step method through flow-focusing microfluidic technology. The morphological characteristics of perfluorohexanone FEMs prepared through coaxial flow focusing under various three-phase flow rates are depicted in Fig. 2(a)–(c). Fig. 2(a) illustrates that, while maintaining a constant focusing phase flow rate of 700 mL h^{−1}, adjustments to the inner and outer phase flow rates (ranging from 10 mL h^{−1} to 60 mL h^{−1}, respectively) enable control over the size of the FEMs. Similarly, Fig. 2(b) demonstrates that with fixed inner and outer phase flow rates of 30 mL h^{−1}, the microcapsule diameter decreases as the driving flow rate increases from 900 mL h^{−1} to 1500 mL h^{−1}. Fig. 2(c) presents the flexibility to adjust the ratio of inner and outer phase flow rates under a constant focusing phase flow rate of 1200 mL h^{−1}, allowing control over the FEM shell thickness while maintaining a constant total of the inner and outer phase flow rates.

The statistical compilation of capsule data from the aforementioned flow combinations is presented in Fig. 2(d). Previous reports have indicated a relationship expressed as $D \sim \alpha[(Q_i + Q_o)/Q_f]^{1/2} D_{\text{orif}}$, where α is a constant related to parameters such as the type of liquid, D and D_{orif} represent the diameters of droplets (microcapsules) and holes, respectively, and Q_i , Q_o , and Q_f denote the flow rates of the inner phase, outer phase, and focusing phase, respectively.²⁶ In Fig. 2(d)(i), maintaining a constant Q_f at 700, 900, and 1500 mL h^{−1}, with $Q_i = Q_o$, leads to an increase in microcapsule size as the flow rates of the inner and outer phases increase. When $Q_i = Q_o = 30$ mL h^{−1} is fixed (Fig. 2(d)(ii)), an increase in the driving flow rate from 700 mL h^{−1} to 1500 mL h^{−1} results in a decrease in the average



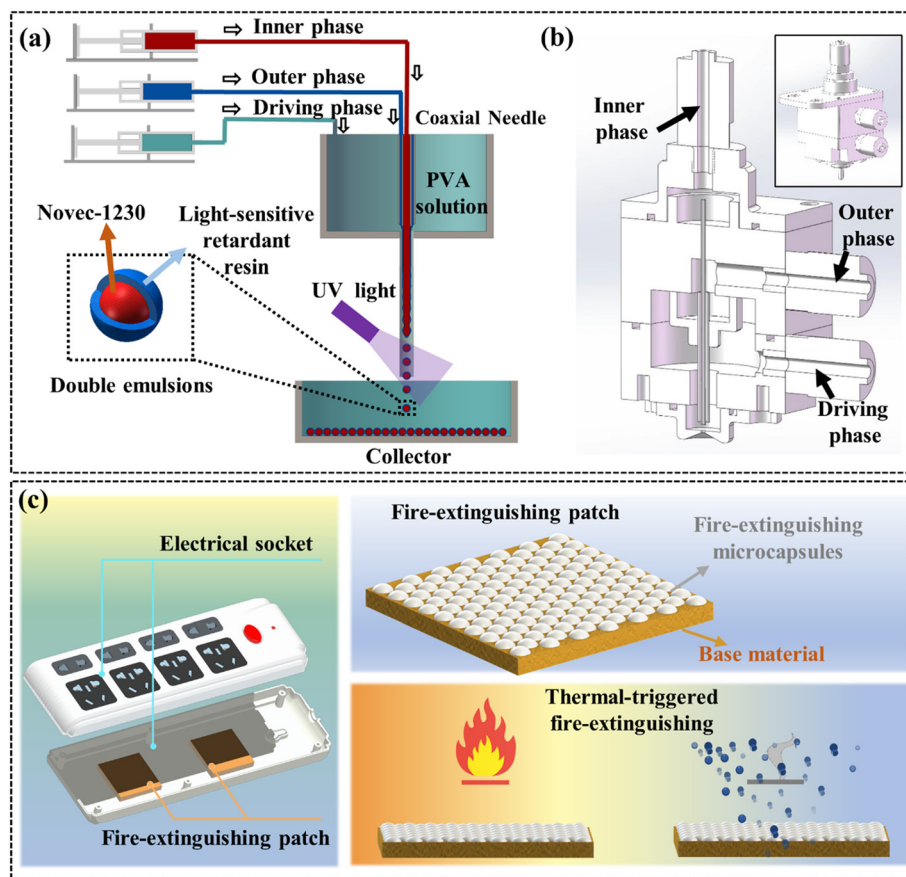


Fig. 1 (a) Schematic diagram of the experimental system for preparing perfluorohexanone FEMs by coaxial flow focusing. It consists of several components, including a nozzle, high-precision syringe pumps, illumination apparatus, and a UV lamp. (b) Schematic diagram of the coaxial flow-focusing nozzle. The coaxial flow-focusing nozzle ensures that the fluids converge at the desired point, leading to the creation of well-defined core-shell structures in the resulting FEMs. (c) Schematic diagram of the thermally triggered FEP for enhanced safety in electrical sockets. By incorporating a thermally triggered FEP into the socket design, it acts as a protective layer, preventing further temperature increases and reducing potential risks.

diameter of the microcapsules from 538.85 μm to 321.05 μm . Furthermore, by keeping a constant $Q_i + Q_o$ at 60 mL h^{-1} , Q_f at 1000 mL h^{-1} , and adjusting Q_i upwards, there is an increase in microcapsule drug loading and thinning of the outer shell, as illustrated in Fig. 2(d)(iii). The loading rate of perfluorohexanone was as high as 67% when the inner and outer phase flow rate ratio was 1:0.8.

After curing by UV irradiation, the prepared microcapsules were washed with distilled water and dried naturally. The SEM image of FEMs, as shown in Fig. 2(e), reveals that the prepared FEMs possess a smooth surface and a uniformly dense shell, which can efficiently encapsulate perfluorohexanone. Thermogravimetric analysis experiments of perfluorohexanone FEMs were conducted to quantitatively reveal the cause of microcapsule micro-explosions. The fire-extinguishing medium in FEMs undergoes a phase transition, resulting in volume expansion and the formation of one or more gas nuclei locally. This hypothesis was confirmed in subsequent high-speed imaging experiments. The volume expansion in the constrained space caused by the expanding gas nuclei causes a rise in internal pressure, which subsequently causes the capsule wall

to rupture and disintegrate. The specific experimental steps were as follows: 5 mg of FEMs were sampled for thermogravimetric analysis (TGA). The temperature was increased from room temperature to 800 $^{\circ}\text{C}$ at a rate of 20 $^{\circ}\text{C min}^{-1}$ in a nitrogen environment. According to the thermogravimetric analysis, the point of mass decline indicates the critical point of the FEM. Thermogravimetric analysis showed that the structure of the FEM had good temperature response characteristics, as shown in Fig. 2(f). Observations revealed that when the temperature reached approximately 100 $^{\circ}\text{C}$, the perfluorohexanone fire-extinguishing agent in the broken core of the microcapsule was released. Furthermore, when the temperature rose to about 400 $^{\circ}\text{C}$, the remaining 20% of the mass of the shell completely melted.

2.2 High-speed photography analysis of FEM explosion and spray direction control under heated

The complex fragmentation process of the FEM was carefully investigated under stringent heating conditions using advanced high-speed photography for precise analysis and



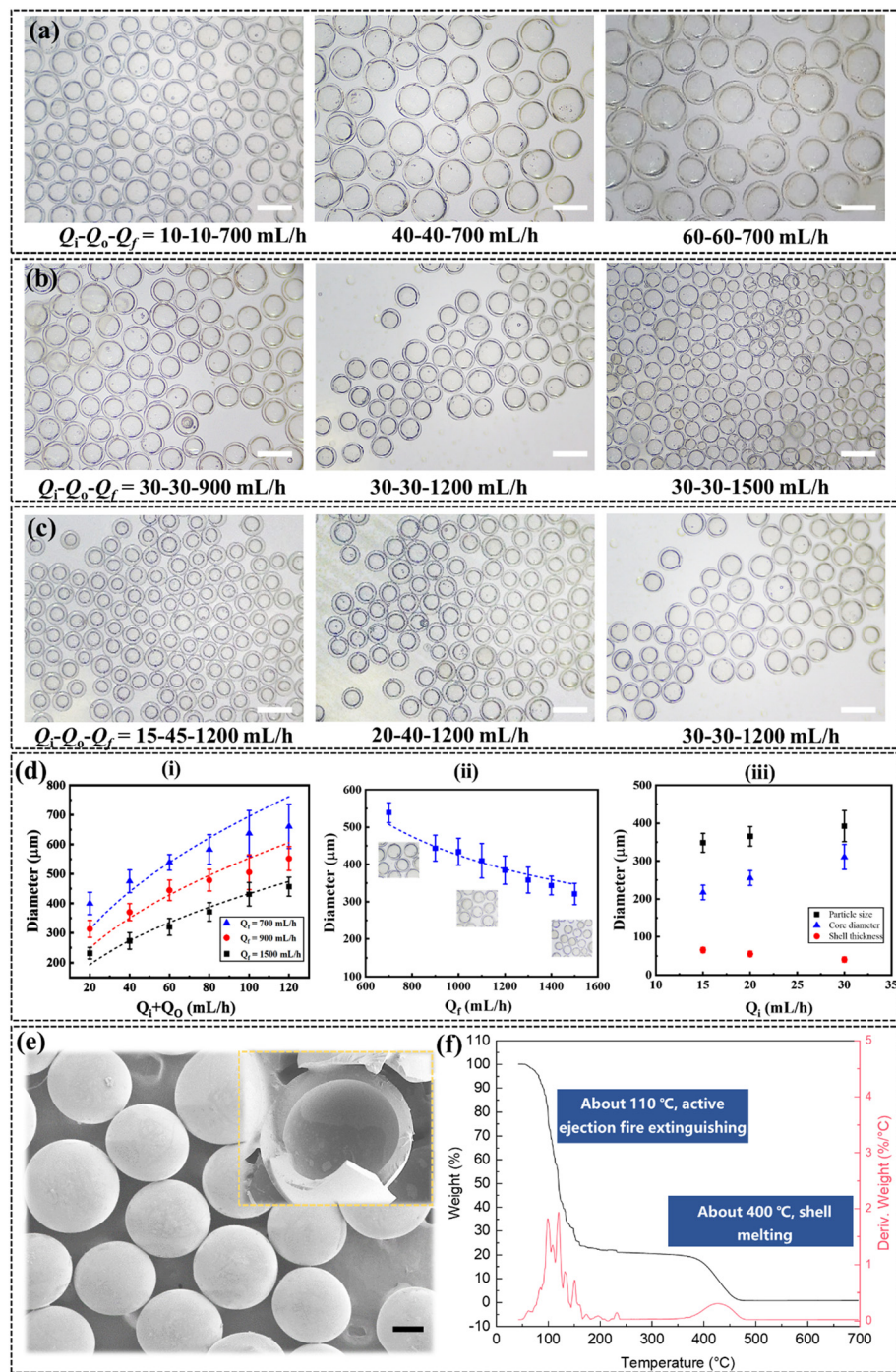


Fig. 2 The morphology with different diameters (a), (b), and thicknesses (c) of perfluorohexanone FEMs prepared by coaxial flow focusing under different three-phase fluid flows (inner phase perfluorohexanone, outer phase photocured resin, and focusing phase PVA water solution). (d) The statistical data on microcapsule size obtained from the above flow combination. (i) $\alpha = 3.679, 3.315, 3.360$, respectively; (ii) $\alpha = 3.467$; (iii) $Q_i + Q_o = 60 \text{ mL h}^{-1}$, $Q_f = 1200 \text{ mL h}^{-1}$. (e) SEM image of perfluorohexanone FEMs. (f) Thermogravimetric analysis of perfluorohexanone FEMs. Scale bars in (a)–(c): $600 \mu\text{m}$.

documentation. Fig. 3(a) visually demonstrates the FEM observation system at elevated temperatures, in which the FEM is fixed on a designated heating plate for controlled and repeatable observation. As the temperature of the heating plate increases, the vaporization of perfluorohexanone within the FEM is evident. Real-time monitoring using high-speed

images demonstrates a steady rise in the internal vapor pressure within the FEM as well as a progressive expansion of the gas core. The culmination of this evolution occurs when the shells of the FEMs finally succumb to the increasing pressure, resulting in a burst, as shown in Fig. 3(b). The entire internal evolution and details of the

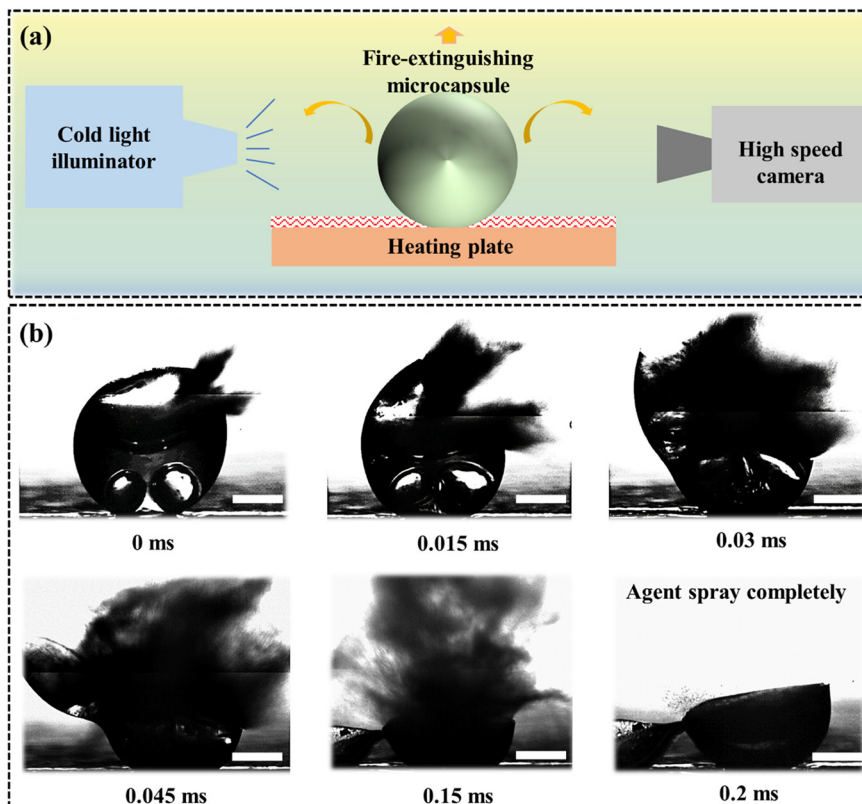


Fig. 3 (a) A high-speed imaging observation system is employed to capture and analyze the explosion process of FEMs triggered by thermal conditions. This system utilizes advanced imaging techniques to record the rapid and transient events that occur during the explosive reaction. (b) Instant high-speed photography of an FEM explosion under thermal triggering, providing valuable insights into their rapid and dynamic reactions. Scale bar: 200 μm .

explosion process were recorded and can be viewed in Movie S1.† A particularly noteworthy finding in the study was that the perfluorohexanone extinguishing agent was released at an alarming rate after the rupture of the FEM shell. In a tiny window of time of less than 0.2 milliseconds, the agent was released very efficiently. It is anticipated that this quick and effective spraying will be crucial in putting out flames. It is worth noting that, under the same internal load, a thicker shell corresponds to a higher response temperature and a faster spraying speed, whereas a thinner shell corresponds to a quicker response rate. In experimental settings, direct contact with the heating plate has the potential to soften the shell layer. To address this concern, a minimal amount of photocurable adhesive is applied to affix the bottom of the capsules, fortifying the mechanical strength below and mitigating the influence of the heating method on the results. The conducted experiments have consistently validated the high reproducibility of these outcomes.

Perfluorohexanone fire-extinguishing agents show impressive capabilities in extinguishing fires with rapid and powerful sprays when triggered by heat. The combination of effective physical cooling and chemical suppression allows for effective fire suppression. We employed various side adhesive thicknesses between the extinguishing microcapsules and the substrate to regulate the direction and coverage of the

perfluorohexanone extinguishing agent. Fig. 4 visually illustrates the thermal excitation process of FEM with varying thicknesses of side adhesive. Comparative analyses were performed to assess their effects on spraying behavior. The results show that as the thickness of the substrate side binder increases, the spray range of the microcapsules decreases while the spray direction becomes more focused. These findings highlight the importance of the side binder in regulating FEM performance.

The customization of the perfluorohexanone extinguishing agent's spray pattern and coverage to meet specific fire suppression requirements is achieved by adjusting the thickness of the binder. In enclosed spaces, such as electrical socket boxes, fire incidents often arise from internal short circuits, leading to a notable temperature increase. Targeting the spray towards potential short-circuit ignition points enhances the precision of the firefighting response. This strategy amplifies the concentration of the localized firefighting agent, effectively extinguishing the fire at its source, thereby elevating the accuracy and effectiveness of firefighting efforts. This degree of control not only significantly enhances the precision and efficacy of fire suppression operations but also holds promise for pivotal roles in furthering the application of FEM in fire safety scenarios.



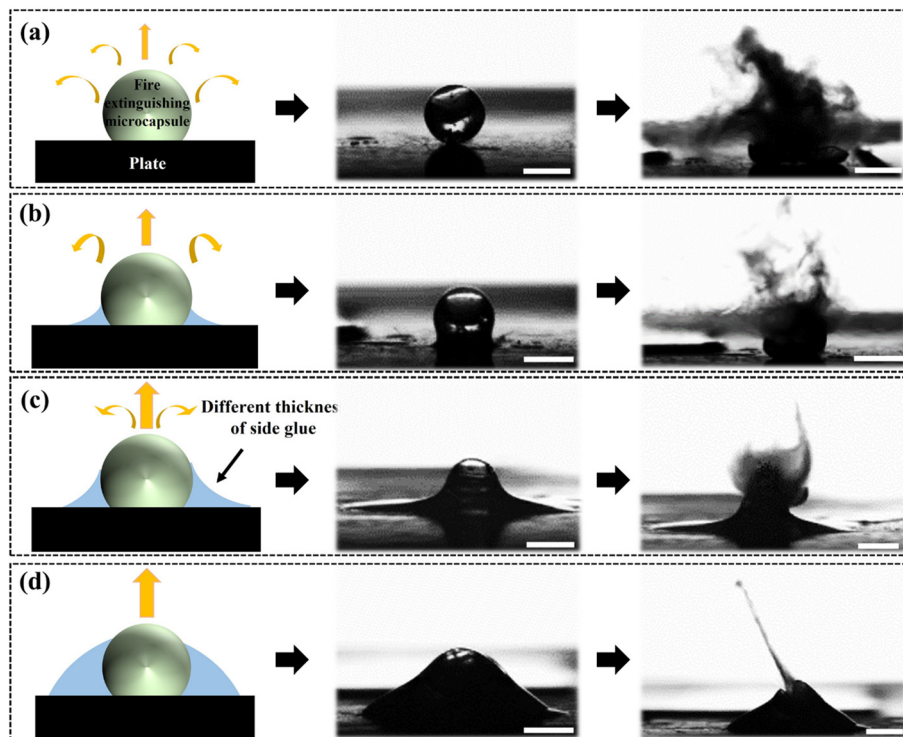


Fig. 4 Comparison of the variability of the FEM explosion process with increasing adhesive thickness from (a) to (d), respectively. By systematically varying the thickness and conducting detailed observations and measurements, the study seeks to understand the relationship between adhesive thickness and the characteristics of the FEM explosion process. Scale bar: 500 μm .

2.3 Fire extinguishment process of FEMs on a candle

Microencapsulating perfluorohexanone protects the agent from external factors such as light, temperature, and humidity, ensuring its long-term stability and effectiveness. This is critical to maintaining the reliability of fire suppression systems, especially in environments where conditions are variable. We conducted tests on the microcapsules by placing them in a constant-temperature oven at 60 °C. After 96 hours, the microcapsules only experienced a minimal decrease in weight of 0.64%. Additionally, we conducted storage tests on the microcapsules in a laboratory under natural environmental conditions for a duration of one year. However, the fire-extinguishing performance of the microcapsules remained virtually unchanged. Therefore, the microcapsules demonstrate excellent heat resistance and stability, maintaining their exceptional fire-extinguishing performance even during long-term storage. In addition, the core-shell structure of the microcapsules plays a vital role in enhancing their performance. This allows FEMs to withstand higher operating temperatures and pressures, making them reliable and durable fire suppression tools. Fig. 5 illustrates a fire-extinguishing experiment using FEMs on a lit candle, which is recorded in Movie S2.† As the flame of the candle approaches the FEM, the internal pressure of the extinguishing agent inside the microcapsule increases due to heating and vaporization. Eventually, the internal pressure reaches a certain threshold and causes the shell to rupture, spraying the internal

extinguishing agent at a high rate. The experimental results show that the spray range of perfluorohexanone is about tens of times the volume of FEM, and the spray time is about 0.28 ms. Since the sprayed perfluorohexanone extinguishing agent has the dual effect of physical cooling and chemical inhibition, only one microcapsule rupture was needed to extinguish the candle flame. It is worth noting that the firefighting performance of FEMs is correlated with the loading rate of perfluorohexanone, where higher loading rates generally result in better firefighting performance. Subsequently, smoke was emitted from the wick of the candle, indicating that the fire had been successfully extinguished. This observation highlights the rapid response and efficient fire suppression capabilities of FEM in the face of fire hazards. The use of perfluorohexanone encapsulated in microcapsules as an extinguishing agent is very effective in extinguishing flames and preventing re-ignition.

2.4 Preparation and application of FEPs for enhanced safety in small confined spaces

To further expand the application range of FEMs, we utilized FEM as the source material and extended it to the two-dimensional field of FEPs (fire-extinguishing patches) research. Larger microcapsules with a higher loading rate result in increased perfluorohexanone spray and improved firefighting efficiency. Conversely, smaller microcapsules offer versatility for precise firefighting in specific areas, leading to a broader range of applications. In general engineering applications, microcapsules



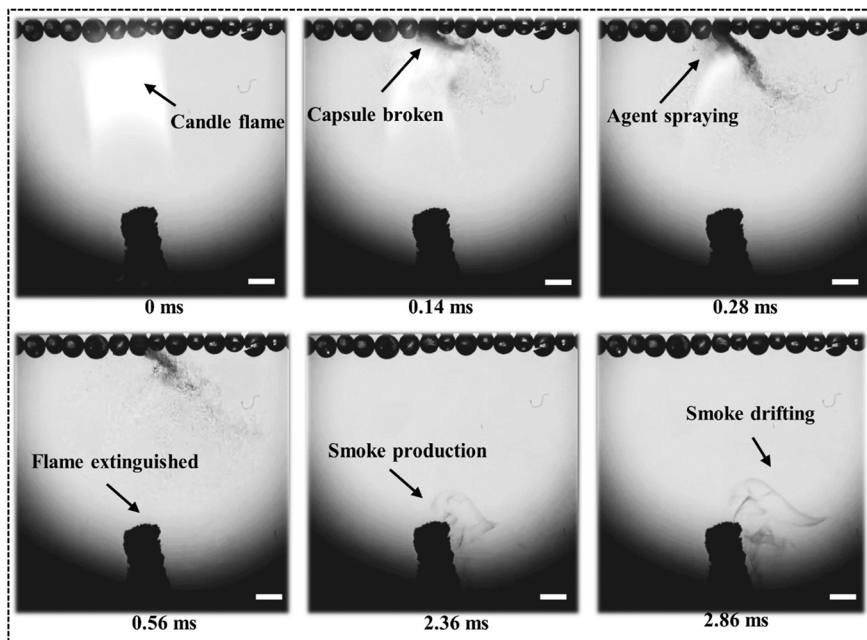


Fig. 5 Instant high-speed photography of the process of extinguishing candle flames using FEMs. The entire process includes the rupture of microcapsules, the spraying of perfluorohexanone, the extinguishing of the flame, and the generation of smoke. Scale bar: 500 μm .

ranging from 400 to 600 μm are commonly used in patch form. The production process from FEMs to FEPs is illustrated in Fig. 6(a). In this process, a flexible and easy-to-release rubber mold was chosen. Firstly, a thin layer of flame-retardant base material, such as UV-curable resin or room temperature-curable AB epoxy resin, was coated on the mold. Then, a layer of FEMs was placed on top of the base material. After the base material solidified, the rubber mold was twisted and removed, resulting in FEPs with a flame-retardant base. To facilitate installation, double-sided adhesive was applied to the back of the FEPs, enabling their wide application in various fire-extinguishing scenarios. Fig. 6(b) shows the prepared FEMs and FEPs, demonstrating the successful implementation of morphology expansion. To validate the effectiveness of FEPs, an experiment involving electrical sockets was conducted as shown in Fig. 6(c). Two sockets, labeled A and B, were used. Socket A did not have a fire-extinguishing patch, while socket B was equipped with an extinguishing patch. Ignited alcohol cotton was placed simultaneously in both sockets to simulate a fire situation. The experimental results clearly indicated a significant difference between the two sockets. As shown in Movie S3,[†] the fire in socket B was rapidly extinguished, while the fire in socket A continued to burn. This verification experiment confirms the efficacy of FEPs in suppressing fire and preventing its spread. The successful morphology expansion of FEMs not only expands their application range but also provides practical solutions for various fire scenarios.

3. Conclusion

In this study, a flow-focused microfluidic technique was proposed to accomplish efficient microencapsulation of

perfluorohexanone by flame-retardant resin in one step. The size and shape of FEMs may be accurately controlled by modifying the process parameters. FEMs can be stimulated by external heat, and the inner core perfluorohexanone can be sprayed at high speed to achieve a significant fire-extinguishing effect. The results show that the detonation temperature of FEMs is about 110 $^{\circ}\text{C}$, and the explosion time is only 0.2 ms. By using different thicknesses of the side binder between the extinguishing microcapsules and the substrate, the direction and coverage of the perfluorohexanone spray can be controlled to meet the specific extinguishing requirements. Fire-extinguishing experiments using FEMs on lit candles show that the microcapsules rupture the spray range up to tens of times their volume, and only one FEM is needed to extinguish a candle flame much larger than its volume. This demonstrates the rapid response and efficient fire-extinguishing capability of FEMs in the face of fire hazards. We further extend the FEMs to two-dimensional FEPs, confirming the efficacy of FEPs in suppressing fire and preventing the spread of fire, and are expected to expand their application to various fire-extinguishing scenarios.

4. Materials and methods

4.1 Materials and reagents

Perfluorohexanone was purchased from 3 M Co., Ltd. Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) supplied the flame-retardant photocurable resin. PVA was purchased from Sigma-Aldrich (St. Louis, MO, USA), $M_w = 13\,000\text{--}23\,000\text{ g mol}^{-1}$, 87–89% hydrolyzed. Perfluorohexanone (density $\rho_i = 1.6\text{ g cm}^{-3}$) was used as the inner phase in the



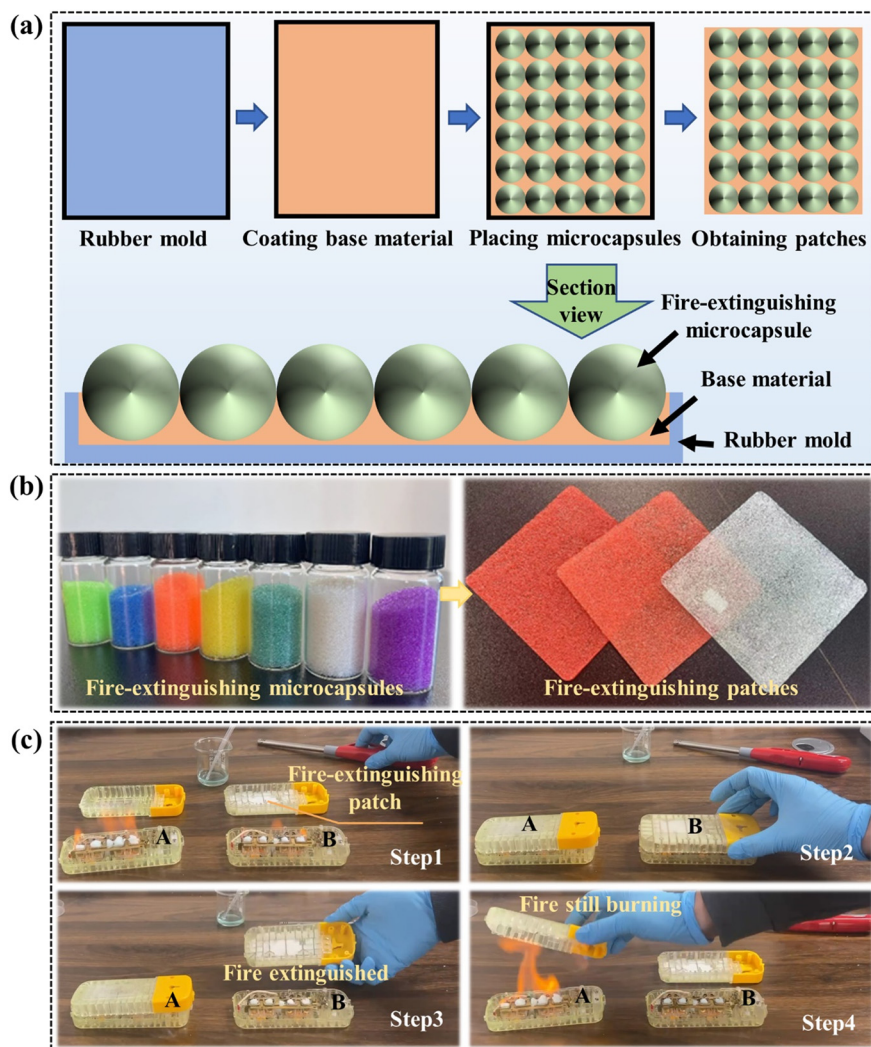


Fig. 6 The comprehensive process of preparation and application of FEP. (a) FEP preparation process, involving several stages to obtain the desired material. (b) The prepared FEMs and FEPs. By staining the shells of microcapsules, FEMs and FEPs can be prepared in various colors. (c) Comparative study of fire-extinguishing patches in electrical sockets. Sockets A and B, without and with an extinguishing patch, respectively, were subjected to ignited alcohol cotton to simulate a fire. Results showed a significant difference, evaluating FEPs as fire suppression agents in electrical socket scenarios.

conventional coaxial flow-focusing method. Resin that can be photocured was used as the outer phase. A 1 wt% PVA water solution was used as the focusing phase.

4.2 Microfluidic system for FEM preparation

To create FEMs, a liquid-driven flow-focusing experimental apparatus was built. The system was made up of a number of essential parts, such as a nozzle, high-precision syringe pumps, lighting, and a UV lamp. Syringe pumps were used to deliver the inner, outer, and driving fluids (WK-101P, Nanjing Anerke Electronics Technology Co., Ltd., China). Using a high-speed camera (FASTCAM SA6, Photron, USA), the FEM's crushing process was observed. A strong continuous DC light source that was purchased from Shanghai Zhaoji Electronic Technology Co., Ltd. in China supplied illumination. The FEMs' dimensions and morphology were examined with an inverted microscope (T2000, Nikon Corp., Japan) for this

study. A UV lamp (UVP60, 365 nm, 8.8 W cm^{-2} , De Sheng Xing Electronics Co., Ltd., China) was used to solidify the double emulsions. The experimental system's schematic design and the FEMs' core-shell structure are shown in Fig. 1(a), which also shows how perfluorohexanone FEMs were created *via* coaxial flow focusing.

Conflicts of interest

The authors declare no conflicts of interest.

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