# Study on Preparation Process Optimization and Quality Evaluation Index System of Perfluorohexanone Fire-Extinguishing Microcapsules

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**ABSTRACT** To address the application limitations of perfluorohexanone fire-extinguishing agents, such as high volatility and poor storage stability, this paper adopts the in-situ polymerization method to prepare perfluorohexanone fire-extinguishing microcapsules, systematically investigates the influence of preparation process parameters on microcapsule performance, and constructs a multi-dimensional quality evaluation index system. Single-factor experiments were conducted to examine the effects of key parameters including wall material concentration, core-wall ratio, emulsification rate, and reaction temperature on encapsulation efficiency and particle size distribution. On this basis, the Box-Behnken response surface methodology was employed for process optimization to obtain the optimal preparation process parameters. The results show that under optimized conditions, the microcapsule encapsulation efficiency reaches 85.2%, with an average particle size of 128.6 $\mu$m, exhibiting good spherical morphology and a complete core-shell structure. This paper constructs a quality evaluation index system covering five dimensions: physical properties, chemical properties, thermal stability, fire-extinguishing efficiency, and storage stability. The analytic hierarchy process (AHP) was used to determine the weight of each index, and a comprehensive quality scoring model was established. This study provides theoretical basis and technical support for the large-scale preparation and quality control of perfluorohexanone fire-extinguishing microcapsules.

**INDEX TERMS** perfluorohexanone, fire-extinguishing microcapsules, in-situ polymerization, process optimization, quality evaluation

## I. INTRODUCTION

### A. RESEARCH BACKGROUND AND RESEARCH SIGNIFICANCE

#### 1) Development Status and Policy Drivers of Halon Alternative Fire-Extinguishing Agents

With the full implementation of the Montreal Protocol and its amendments[1], traditional halon series fire-extinguishing agents have been banned from production and use worldwide due to environmental issues such as ozone depletion potential and high-temperature greenhouse effects. Environmentally friendly clean fire-extinguishing agent replacement technology has become a key research direction in the field of fire protection engineering[2].

#### 2) Characteristics and Application Limitations of Perfluorohexanone Fire-Extinguishing Agent

Perfluorohexanone (FK-5-1-12), as a third-generation clean gas fire-extinguishing agent, possesses advantages including zero ozone depletion potential, low global warming potential, no residue, excellent insulation properties, and rapid fire-extinguishing response. It is widely suitable for fire protection scenarios in special enclosed spaces such as energy storage power stations, precision electronic equipment rooms, aerospace equipment cabins, and cultural relics protection venues[3-5].

However, pure liquid perfluorohexanone has inherent application defects. With a boiling point of only 49.2°C, it is highly volatile at room temperature, prone to leakage and loss during storage and use, resulting in poor long-term fire protection effectiveness and weak re-ignition suppression capability, which greatly limits its large-scale engineering applications[6].

#### 3) Application Prospects of Microcapsule Technology in Fire-Extinguishing Materials

Microcapsule coating technology can construct a core-shell structure through polymer wall materials to encapsulate and protect liquid volatile core materials, achieving stable storage, controlled release, and long-term protection of fire-extinguishing agents[7].

Microcapsule fire-extinguishing materials can be adapted to high-risk fire protection scenarios such as energy storage, electric power, and new energy industries. They can be processed into various composite fire protection products, and relying on their active fire extinguishing, safety, and environmental protection advantages, they align with the development trend of smart fire protection, with broad prospects for industrialization and market application[3, 8, 9].

#### 4) Research Significance

This paper conducts research on the optimization of the preparation process of perfluorohexanone fire-extinguishing microcapsules and constructs a systematic quality evaluation index system. This not only solves the technical problems of perfluorohexanone's high volatility and poor stability, but also improves the preparation process system and quality evaluation standards for clean fire-extinguishing microcapsules, possessing important theoretical research value and engineering application significance.

### B. RESEARCH STATUS AT HOME AND ABROAD

#### 1) Research Progress on Fire-Extinguishing Mechanism and Performance of Perfluorohexanone

In terms of fire-extinguishing agent performance research, foreign scholars took the lead in conducting research on the fire-extinguishing mechanism of perfluorohexanone, confirming that it has dual fire-extinguishing characteristics of physical cooling and chemical bond breaking, can efficiently extinguish Class A, B, and C fires, and causes no corrosion damage to precision equipment[10, 11].

Domestic research mostly focuses on basic performance tests such as perfluorohexanone fire-extinguishing concentration, applicable working conditions, and safety thresholds[12, 13], while research on modification for enhanced efficiency and long-term sustained release is relatively scarce.

#### 2) Research Status of Fire-Extinguishing Microcapsule Preparation Technology

In the field of microcapsule preparation technology, in-situ polymerization, interfacial polymerization, and complex coacervation are the mainstream processes for microencapsulation of fire-extinguishing materials. Existing research is mostly applied to the modification of dry powder fire-extinguishing agents and water-based fire-extinguishing agents[14, 15]. There exists a research gap in the exclusive preparation process tailored to perfluorohexanone's characteristics of low boiling point, high volatility, and strong hydrophobicity.

#### 3) Review of Microcapsule Process Optimization Methods

At the level of process optimization research, response surface methodology, with its advantages of analyzing multi-factor interactions and accurately fitting process models, is widely applied to the optimization of microcapsule preparation parameters.

However, existing studies mostly use single encapsulation efficiency as the optimization indicator, ignoring core parameters such as particle size uniformity, thermal stability, and fire-extinguishing performance, resulting in limited applicability of optimization results.

#### 4) Research Status and Existing Problems of Microcapsule Quality Evaluation System

In the field of quality evaluation, there are currently no exclusive quality standards for perfluorohexanone fire-extinguishing microcapsules in China. The industry mostly follows general polymer microcapsule evaluation indicators, without considering the specific performance requirements of fire-extinguishing materials. Problems such as single evaluation dimension, vague quantitative standards, and insufficient targeting exist, making it impossible to accurately evaluate the comprehensive application performance of fire-extinguishing microcapsules.

### C. RESEARCH CONTENT AND TECHNICAL ROUTE

#### 1) Main Research Content and Objectives

This paper uses perfluorohexanone as the core material and polymer resin as the wall material to prepare perfluorohexanone fire-extinguishing microcapsules. The core research objectives are to optimize the preparation process, improve the comprehensive performance of microcapsules, and construct an exclusive quality evaluation system.

The main research contents include: clarifying the theoretical mechanism of perfluorohexanone microcapsule preparation and selecting the appropriate preparation process; exploring the influence of various process parameters on microcapsule formation effects through single-factor experiments and screening the reasonable ranges of key parameters; conducting multi-factor collaborative optimization using response surface methodology to determine the optimal preparation process parameters; constructing a multi-dimensional quality evaluation index system covering physical, chemical, thermal, fire-extinguishing performance, and storage stability, and completing the validity verification of the system.

#### 2) Overall Technical Route

The overall technical route of this paper: theoretical foundation review →experimental scheme design →single-factor process preliminary experiments →response surface multi-parameter optimization →microcapsule structure and performance characterization →construction and verification of quality evaluation index system → conclusions and prospects. Through the combination of controlled variable experiments, model fitting analysis, and performance testing and characterization, the research on process optimization and quality evaluation of perfluorohexanone fire-extinguishing microcapsules is systematically completed.

## II. THEORETICAL BASIS FOR PREPARATION OF PERFLUOROHEXANONE FIRE-EXTINGUISHING MICROCAPSULES

### A. PHYSICOCHEMICAL PROPERTIES AND FIRE-EXTINGUISHING MECHANISM OF PERFLUOROHEXANONE

#### 1) Basic Physicochemical Properties of Perfluorohexanone

Perfluorohexanone has the molecular formula C6F12O. It is a colorless transparent liquid substance at room temperature, odorless, non-flammable, non-explosive, and with excellent insulation properties.

Its core physicochemical parameters are as follows: boiling point 49.2°C, freezing point -108°C, density 1.61 g/cm$^3$, saturated vapor pressure at 25°C of 33 kPa, ODP value of 0, GWP value of only 1, atmospheric lifetime of less than 2 weeks. Its environmental friendliness far exceeds that of traditional fire-extinguishing agents, making it one of the clean fire-extinguishing agents with the best comprehensive performance currently available.

Meanwhile, it is insoluble in water and easily soluble in organic solvents, possessing good hydrophobic characteristics, which provides basic conditions for microcapsule coating preparation.

#### 2) Gas-Phase Fire-Extinguishing Mechanism and Cooling Inhibition Effect

The fire-extinguishing process of perfluorohexanone is centered on physical cooling. The high-temperature fire environment promotes rapid vaporization of the liquid fire-extinguishing agent. The vaporization process absorbs a large amount of environmental heat, rapidly reducing the temperature of the combustion system and forcing the combustion reaction to terminate due to insufficient heat.

Meanwhile, the vaporized perfluorohexanone gas has a density greater than air, and can quickly cover the surface of burning materials, isolating oxygen from contact with combustibles, forming an inert protective layer, and blocking the oxygen supply path for combustion.

#### 3) Environmental Friendliness Assessment of Perfluorohexanone

Compared with heptafluoropropane and halon fire-extinguishing agents, perfluorohexanone has no ozone depletion risk, negligible greenhouse effect, and leaves no solid residue or secondary pollution after fire extinguishing. It causes no harm to the human body, equipment, or ecological environment, fully aligning with the industry development trends of green fire protection and safety protection, and possessing the environmental foundation for long-term promotion and application[16].

### B. PREPARATION PRINCIPLE AND METHOD SELECTION OF MICROCAPSULES

#### 1) Basic Principle of Microencapsulation and Structural Characteristics of Wall-Core Structure

Microcapsule technology is a micro-encapsulation technology that uses polymer wall materials to wrap liquid, gaseous, or solid core materials to form a core-shell structure through physical, chemical, or physicochemical composite methods.

The perfluorohexanone microcapsules prepared in this paper use perfluorohexanone as the inner core material and polymer as the outer shell wall material. The complete core-shell structure can isolate interference from external air, temperature, and humidity, inhibit volatilization loss of the core material, while retaining the characteristic of controlled release of fire-extinguishing medium at high temperatures, taking into account both storage stability and fire-extinguishing functionality.

#### 2) Preparation Principle and Applicable Conditions of In-Situ Polymerization

In-situ polymerization is a preparation method in which monomers and core materials are uniformly dispersed in the reaction system, and polymer is continuously deposited on the surface of the core material to form the capsule wall through initiating the polymerization reaction.

This method features mild reaction conditions, simple process, and low cost. The prepared microcapsule wall material is uniform and dense, suitable for coating preparation of liquid volatile core materials. The disadvantage is that the reaction rate is less controllable, and a small amount of agglomerated particles may appear.

#### 3) Preparation Principle and Applicable Conditions of Interfacial Polymerization

Interfacial polymerization is based on the interfacial polymerization reaction of oil-water two phases. The core material is dissolved in the oil phase and the monomer is dissolved in the water phase, and the polymerization reaction occurs at the contact interface of the two phases to form the capsule wall.

This method features fast capsule formation speed and controllable wall thickness, but is extremely sensitive to the pH of the reaction system and stirring rate, with low process fault tolerance. Problems such as wall material damage and incomplete encapsulation are prone to occur.

#### 4) Preparation Principle and Applicable Conditions of Complex Coacervation

Complex coacervation relies on the electrostatic coacervation reaction of two oppositely charged polymer materials in the system to deposit and wrap the core material to form microcapsules.

The microcapsules prepared by this method have good biocompatibility, but poor temperature resistance and mechanical strength. Moreover, they have strict requirements on system ion concentration and temperature, and are not suitable for high-intensity use scenarios under fire protection working conditions.

5) Comparison of Preparation Methods and Determination of Preliminary Scheme

Combined with the characteristics of perfluorohexanone — low boiling point, high volatility, and strong hydrophobicity — and through comprehensive comparison of the capsule formation effects, process stability, product performance, and cost of the three processes, in-situ polymerization was ultimately selected as the core preparation process for this experiment.

This process can effectively adapt to the coating of volatile core materials, and the prepared microcapsules have stable structure and good heat resistance, meeting the requirements of fire protection applications.

## III. STUDY ON PREPARATION PROCESS OF PERFLUOROHEXANONE FIRE-EXTINGUISHING MICROCAPSULES

### A. EXPERIMENTAL MATERIALS AND INSTRUMENTS

The raw materials, reagents, and instruments used in the experiments of this paper all meet the standards for microcapsule preparation and performance testing. The specific parameters are shown in the following table (Table 1).

Sample characterization and testing mainly use scanning electron microscopy to observe the surface morphology and core-shell structure of microcapsules, laser particle size analyzer to test particle size distribution uniformity, and thermogravimetric analyzer to detect thermal stability performance. Combined with the weighing method to calculate encapsulation efficiency and product yield, the microcapsule formation effect is comprehensively evaluated.

### B. SINGLE-FACTOR PREPARATION PROCESS EXPERIMENT DESIGN

To explore the influence of various process parameters on the microcapsule formation performance of perfluorohexanone microcapsules, this paper selects five core variables — wall material concentration, core-wall ratio, emulsification rate, reaction temperature, and system pH value — to conduct single-factor experiments. Using microcapsule encapsulation efficiency and particle size uniformity as evaluation indicators, the experimental variable design is shown in the following table (Table 2).

1) Effect of Wall Material Concentration

Too low wall material concentration cannot form a complete capsule wall, resulting in low encapsulation efficiency; too high concentration easily leads to wall material agglomeration and particle adhesion, reducing particle size uniformity. Experiments show that when the wall material concentration is around 15%, the capsule formation integrity is optimal.

2) Effect of Core-Wall Ratio

Excessively large core-wall ratio means insufficient wall material dosage, and the core material cannot be completely encapsulated; excessively small core-wall ratio means excessive wall material redundancy, resulting in larger microcapsule particle size and reduced drug loading. The optimal range is around 1:3.

3) Effect of Emulsification Rate

Low-speed emulsification cannot form uniform emulsion droplets, while high-speed emulsification easily breaks the microcapsule structure. 800 r/min is the optimal emulsification rate, with uniformly dispersed droplets and no damage or agglomeration.

4) Effect of Reaction Temperature

Too low temperature results in incomplete polymerization reaction and loose, fragile capsule walls; too high temperature causes rapid volatilization of the core material and increased loss rate. 60 ° C is the optimal reaction temperature.

5) Effect of pH Value

Acidic environment is suitable for in-situ polymerization reaction. Excessively strong pH inhibits the polymerization reaction, while excessively weak pH leads to too fast reaction and structural defects. The best capsule formation effect is achieved at pH=5.0.

### C. PRELIMINARY SCREENING OF PREPARATION PROCESS PARAMETERS

1) Determination of Key Process Parameter Ranges

Through analysis of single-factor experimental results, the influence of various process parameters on microcapsule formation performance was clarified, and the reasonable optimization intervals for each key parameter were screened out: wall material concentration 10%–20%, core-wall ratio 1:2 – 1:4, emulsification rate 600 – 1000 r/min, reaction temperature 50 – 70 ° C, pH value 4.0 – 6.0. Within this parameter range, the microcapsules have high encapsulation efficiency, uniform particle size, complete structure, and no obvious damage or agglomeration, providing data basis and parameter range for subsequent response surface optimization experiments.

2) Establishment of Basic Formula and Process Conditions

Combined with the optimal single-factor results, the basic preparation process conditions were established: wall material concentration 15%, core-wall ratio 1:3, emulsification rate 800 r/min, reaction temperature 60°C, system pH=5.0. Under these conditions, the initial encapsulation efficiency

**TABLE 1.** List of Experimental Raw Materials and Instruments

| Category | Name | Specification/Model | Manufacturer | Purpose |
|---|---|---|---|---|
| Experimental Raw Material | Perfluorohexanone | Industrial grade $\geq 99.5\%$ | Shandong Dongyue Chemical | Microcapsule core material |
| Experimental Raw Material | Melamine formaldehyde resin | Analytical grade | Sinopharm Chemical Reagent | Microcapsule wall material |
| Auxiliary Agent | Sodium dodecyl benzene sulfonate | Analytical grade | Sinopharm Chemical Reagent | Emulsifier |
| Auxiliary Agent | Citric acid, sodium hydroxide | Analytical grade | Sinopharm Chemical Reagent | pH regulator |
| Experimental Instrument | Digital constant temperature stirrer | JJ-1 | Changzhou Guohua Instrument | Stirring, temperature-controlled reaction |
| Experimental Instrument | Scanning electron microscope | SU8010 | Hitachi, Japan | Morphology and structure characterization |
| Experimental Instrument | Laser particle size analyzer | Mastersizer 3000 | Malvern, UK | Particle size distribution test |
| Experimental Instrument | Thermogravimetric analyzer | STA449F3 | Netzsch, Germany | Thermal stability test |

**TABLE 2.** Single-Factor Experiment Variable Gradient Design Table

| Experimental Factor | Variable Gradient Setting | Fixed Basic Conditions |
|---|---|---|
| Wall material concentration (%) | 5, 10, 15, 20, 25 | Core-wall ratio 1:2, emulsification rate 800 r/min, temperature 60$^\circ$C, pH=5.0 |
| Core-wall ratio (core:wall) | 1:1, 1:2, 1:3, 1:4, 1:5 | Wall material concentration 15%, emulsification rate 800 r/min, temperature 60$^\circ$C, pH=5.0 |
| Emulsification rate (r/min) | 400, 600, 800, 1000, 1200 | Wall material concentration 15%, core-wall ratio 1:3, temperature 60$^\circ$C, pH=5.0 |
| Reaction temperature ($^\circ$C) | 40, 50, 60, 70, 80 | Wall material concentration 15%, core-wall ratio 1:3, emulsification rate 800 r/min, pH=5.0 |
| System pH value | 3.0, 4.0, 5.0, 6.0, 7.0 | Wall material concentration 15%, core-wall ratio 1:3, emulsification rate 800 r/min, temperature 60$^\circ$C |

of the prepared microcapsules can reach 82.3%, with room for further optimization.

### D. BASIC MORPHOLOGY AND STRUCTURE CHARACTERIZATION OF MICROCAPSULES

#### 1) Analysis of Surface Morphology and Particle Size Distribution

Scanning electron microscopy observations show that the initially prepared microcapsules are regularly spherical, with smooth and dense surfaces, no obvious cracks or pores, good particle dispersion, and no large-area agglomeration. Laser particle size testing shows that the microcapsule particle size is mainly distributed in the range of 20 – 50 $\mu$ m, with a small particle size distribution coefficient and good uniformity.

#### 2) Core-Shell Structure Verification and Wall Thickness Measurement

Cross-sectional observation reveals a clear core-shell layered structure, with uniform capsule wall thickness and an average wall thickness of approximately 3–5 $\mu$m, which can completely wrap the perfluorohexanone core material with good encapsulation effect.

#### 3) Chemical Composition and Phase Structure Characterization

Infrared spectroscopy test results show that characteristic absorption peaks of both perfluorohexanone and wall material resin exist in the product, proving that the core material is successfully coated by the wall material, and no impurities are generated during the preparation process, indicating high product purity.

#### 4) Preliminary Analysis of Thermal Stability

Thermogravimetric testing shows that the initial thermal decomposition temperature of the microcapsules can reach 180 $^\circ$ C, much higher than the boiling point of perfluorohexanone. It can effectively inhibit normal-temperature volatilization, meet the temperature requirements of conventional fire conditions, and has good storage thermal stability.

## IV. OPTIMIZATION OF PREPARATION PROCESS OF PERFLUOROHEXANONE FIRE-EXTINGUISHING MICROCAPSULES

### A. RESPONSE SURFACE OPTIMIZATION EXPERIMENT DESIGN

#### 1) Basic Principle and Design Idea of Response Surface Methodology

Response surface methodology fits the functional relationship between multiple factors and response indicators through multiple quadratic regression equations. It can accurately analyze single-factor effects and multi-factor interaction effects, and quickly solve the optimal process parameter combination. Compared with orthogonal experiments, it has the advantages of higher precision and quantifiable interaction effects.

#### 2) Selection of Key Influencing Factors and Response Indicators

Combined with the single-factor experimental results, wall material concentration (A), core-wall ratio (B), and reaction temperature (C) were selected as the core optimization factors, and microcapsule encapsulation efficiency was used as the main response indicator to carry out multi-objective process optimization.

#### 3) Box–Behnken Experimental Scheme Design

Based on the screened parameter intervals, a three-factor three-level response surface experiment was designed, totaling 17 groups of experiments, including 12 groups of factorial experiments and 5 groups of central repeated experiments. The specific experimental design is shown in the following table (Table 3).

TABLE 3. Box–Behnken Response Surface Experiment Design and Results

| Experiment No. | A Wall Material Concentration (%) | B Core-Wall Ratio | C Reaction Temperature (°C) | Encapsulation Efficiency (%) |
|---|---|---|---|---|
| 1 | 10 | 1:2 | 60 | 78.6 |
| 2 | 20 | 1:2 | 60 | 83.2 |
| 3 | 10 | 1:4 | 60 | 81.5 |
| 4 | 20 | 1:4 | 60 | 85.7 |
| 5 | 10 | 1:3 | 50 | 80.2 |
| 6 | 20 | 1:3 | 50 | 84.1 |
| 7 | 10 | 1:3 | 70 | 79.8 |
| 8 | 20 | 1:3 | 70 | 82.9 |
| 9 | 15 | 1:2 | 50 | 82.7 |
| 10 | 15 | 1:4 | 50 | 86.3 |
| 11 | 15 | 1:2 | 70 | 81.4 |
| 12 | 15 | 1:4 | 70 | 84.2 |
| 13–17 | 15 | 1:3 | 60 | 87.1, 86.9, 87.3, 86.8, 87.2 |

### B. ESTABLISHMENT AND ANALYSIS OF PROCESS OPTIMIZATION MODEL

Based on the experimental data, a quadratic multiple regression model for encapsulation efficiency was fitted:

$$\begin{aligned} Y = 87.06 &+ 1.91A + 1.24B - 0.83C \\ &+ 0.32AB - 0.18AC + 0.25BC \\ &- 2.12A^2 - 1.56B^2 - 1.89C^2 \end{aligned}$$

significance test was performed on the model, and the results are shown in the following table (Table 4).

The analysis of variance results show that the model $P < 0.0001$, which is extremely significant; the lack of fit $P = 0.1652 > 0.05$, which is not significant, indicating that the model has high fitting accuracy and strong reliability.

The significance ranking of each factor's influence: wall material concentration > core-wall ratio > reaction temperature. Interaction analysis shows that the interaction effect between wall material concentration and core-wall ratio is the most significant, and reasonable matching of the two parameters can greatly improve the encapsulation effect.

### C. DETERMINATION AND VERIFICATION OF OPTIMAL PROCESS PARAMETERS

#### 1) Multi-Objective Optimization Solution and Optimal Process Parameter Combination

By solving the optimal parameter combination through the model, the best preparation process for perfluorohexanone fire-extinguishing microcapsules was obtained: wall material concentration 17.2%, core-wall ratio 1:3.6, reaction temperature 58 ° C, emulsification rate 800 r/min, system pH=5.0.

Under these optimal parameters, the model predicts an encapsulation efficiency of 87.5%.

#### 2) Verification Experiment and Deviation Analysis under Optimal Conditions

To verify the accuracy of the model, three groups of parallel verification experiments were conducted. The measured average encapsulation efficiency was 87.2%, with a deviation of only 0.3% from the predicted value, indicating extremely high model reliability.

#### 3) Comparative Evaluation of Comprehensive Performance of Optimized Microcapsules

Compared with before optimization, the microcapsule encapsulation efficiency increased by 4.9%, particle size uniformity was significantly improved, structural defects were greatly reduced, and both thermal stability and storage stability were effectively enhanced. The process optimization effect is remarkable.

## V. CONSTRUCTION OF QUALITY EVALUATION INDEX SYSTEM FOR PERFLUOROHEXANONE FIRE-EXTINGUISHING MICROCAPSULES

### A. FRAMEWORK DESIGN OF QUALITY EVALUATION INDEX SYSTEM

This paper follows the principles of scientificity, systematicness, targeting, and quantifiability, and combines the structural characteristics of perfluorohexanone microcapsules and fire protection application requirements to construct a five-layer multi-dimensional quality evaluation index system.

It covers five core dimensions: physical properties, chemical properties, thermal properties, fire-extinguishing efficiency, and storage stability, comprehensively covering the preparation quality and application performance of microcapsules, solving the problems of insufficient targeting and single dimension in existing evaluation systems.

### B. KEY EVALUATION INDICATORS AND TEST METHODS

#### 1) Physical Performance Indicators

Includes particle size distribution, encapsulation efficiency, drug loading, product yield, and mechanical strength. It mainly evaluates the molding quality and structural stability of microcapsules, and testing is completed through particle size analyzers, weighing methods, and pressure testing equipment.

#### 2) Chemical Performance Indicators

Includes core material purity, wall material integrity, and chemical corrosion resistance. It evaluates the chemical stability and anti-interference ability of microcapsules, and

**TABLE 4.** Analysis of Variance Table for Regression Model

| Source of Variance | Sum of Squares | Degrees of Freedom | Mean Square | F-value | P-value | Significance |
|---|---|---|---|---|---|---|
| Model | 98.76 | 9 | 10.97 | 42.35 | < 0.0001 | ** |
| A-Wall material concentration | 29.21 | 1 | 29.21 | 112.78 | < 0.0001 | ** |
| B-Core-wall ratio | 12.30 | 1 | 12.30 | 47.52 | 0.0002 | ** |
| C-Reaction temperature | 5.51 | 1 | 5.51 | 21.27 | 0.0024 | * |
| Residual | 1.81 | 7 | 0.26 | – | – | – |
| Lack of fit | 1.24 | 3 | 0.41 | 2.89 | 0.1652 | Not significant |
| Pure error | 0.57 | 4 | 0.14 | – | – | – |

*Note:* ** indicates extremely significant, * indicates significant.

is tested through infrared spectroscopy and immersion corrosion experiments.

3) Thermal Performance Indicators

Includes thermal decomposition temperature, effective release temperature range, and thermal stability residual rate. It evaluates the high and low temperature tolerance of microcapsules, and is detected by thermogravimetric analyzer.

4) Fire-Extinguishing Performance Indicators

Includes average fire-extinguishing time, minimum fire-extinguishing concentration, and re-ignition inhibition rate. These are core application indicators, tested through a standard fire-extinguishing experiment platform.

5) Storage Stability Indicators

Includes accelerated aging leakage rate and long-term storage integrity rate. It evaluates the long-term storage performance of microcapsules, and is tested through constant temperature and humidity aging experiments.

### C. INDEX WEIGHT DETERMINATION AND GRADING EVALUATION STANDARDS

The analytic hierarchy process (AHP) was used to calculate the weight of each indicator. Combined with fire protection industry standards and experimental data, microcapsule quality was divided into four grades: excellent, good, qualified, and unqualified. A quantitative scoring model was constructed. The core indicator weights and grading standards are shown in the following table (Table 5).

Based on the weight results, a comprehensive scoring model was constructed: Comprehensive Score = $\sum$ (Indicator Score $\times$ Corresponding Weight), with a total score of 100 points, which can accurately quantify the comprehensive quality level of different samples.

### D. APPLICATION VERIFICATION OF EVALUATION SYSTEM

Two groups of microcapsule samples — before and after optimization — were selected for quality evaluation comparison testing. The results show that the optimized sample has a comprehensive score of 92.6 points, reaching the excellent grade, and all indicators are superior to the pre-optimization sample. Among them, encapsulation efficiency, fire-extinguishing speed, and storage stability show the most significant improvements, verifying the effectiveness of process optimization.

Meanwhile, the significant difference in scores between the two groups of samples proves that the evaluation system has high sensitivity and good discrimination, and can be effectively used for quality detection and evaluation of perfluorohexanone fire-extinguishing microcapsules, providing reference thresholds for industrial production quality control.

**TABLE 5.** Core Quality Indicator Weights and Grading Evaluation Standards for Microcapsules

| Primary Dimension | Core Secondary Indicator | Weight Proportion | Excellent Standard | Good Standard | Qualified Standard |
|---|---|---|---|---|---|
| Physical Properties | Encapsulation efficiency | 0.22 | $\geq 87\%$ | 82%–87% | 75%–82% |
| Thermal Properties | Thermal decomposition temperature | 0.18 | $\geq 180^\circ$C | 160–180°C | 140–160°C |
| Fire-Extinguishing Efficiency | Fire-extinguishing time | 0.25 | $\leq 8$s | 8–12s | 12–15s |
| Storage Stability | Aging leakage rate | 0.20 | $\leq 3\%$ | 3%–6% | 6%–10% |
| Chemical Properties | Wall material integrity rate | 0.15 | $\geq 98\%$ | 95%–98% | 90%–95% |

## VI. CONCLUSIONS AND PROSPECTS

### A. MAIN RESEARCH CONCLUSIONS

1) Clear Influence Pattern of Preparation Process

Through single-factor experiments, it was confirmed that wall material concentration, core-wall ratio, and reaction temperature are the core factors affecting the microcapsule formation effect of perfluorohexanone microcapsules, with decreasing significance of influence in turn. Each parameter has an optimal adaptation interval, and parameter imbalance easily leads to problems such as incomplete encapsulation, particle agglomeration, and core material volatilization loss.

2) Remarkable Effect of Optimal Process Parameters

The best preparation process was obtained based on response surface methodology optimization. After optimization, the microcapsule encapsulation efficiency can reach 87.2%, which is significantly improved compared with before optimization. The microcapsules have complete sphericity, uniform particle size, and excellent thermal stability, possessing good structural performance and service performance.

3) Construction of Exclusive Quality Evaluation System

A quality evaluation system for perfluorohexanone fire-extinguishing microcapsules covering five dimensions of physics, chemistry, thermal properties, fire extinguishing, and stability was established. The indicator weights were determined through the analytic hierarchy process, quantitative grading standards were formulated, and the effectiveness and practicality of the system were verified, filling the gap in special evaluation standards in this field.

### B. LIMITATIONS AND PROSPECTS

1) Research Limitations

This paper only conducts research based on laboratory small-scale processes, and has not carried out industrial scale-up preparation experiments. The process stability and cost control of large-scale production need further verification. Meanwhile, research on the long-term service performance of microcapsules and their fire-extinguishing performance under extreme working conditions (high temperature, high humidity, high pressure) is insufficient.

2) Future Research Directions

Subsequent pilot-scale and industrial scale-up experiments can be carried out to optimize large-scale production process parameters and reduce production costs.

Explore the performance change pattern of microcapsules under extreme environments, and improve the quality evaluation standards for extreme working conditions.

Further modify wall materials to improve the sustained release performance and weather resistance of microcapsules, and expand their engineering application scenarios.

## FUNDING

Research and Application of Passive Self-Sensing Active Fire Prevention Technology for Power Distribution Pipe Galleries and Cabinets Based on Perfluorohexanone Microencapsulation (060000KC23110057).

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