Research on the Development of Microcapsules and Their Potential Applications in Tibet Plateau

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Abstract. Microcapsule refers to some miniature containers or packages with polymer wall shells developed from natural or synthetic polymer materials. The shape is generally spherical, and the particle size is generally from a few microns to a few hundred. Microcapsule is a micro packaging technology that can wrap solids, liquids, and gases. The process of preparing microcapsules is called microencapsulation, which has the advantages of isolating the core material to reduce the influence of external environmental factors, enhancing the stability of the core material, concealing unpleasant odors, reducing the toxicity of the core material, increasing the utilization rate of the core material. With the continuous development of technology, the application prospects of microcapsules are gradually broad in the fields of food, cosmetics, medical engineering, production and construction, textiles and other fields. In this paper, firstly the development, fabrication techniques of microcapsules, and the materials forming cores and walls were reviewed. Secondly the applications of microcapsules in various areas were summarized. Because of the wide range of application scenarios, they hold great potential for the environmental applications in Tibet Plateau, such as the water treatment, nutrient regulation of the characteristic plants for Tibetan medicine.

1. Introduction

1.1. Research significance

Microcapsule (MC) refers to some miniature containers or packages with polymer wall shells developed from natural or synthetic polymer materials [1], which is used to hold the core materials without altering the properties of the materials inside. The core materials can isolate the capsule core from the external environment, and extend the life of the unstable and volatile material. In addition, microcapsules can protect substances against environment influences, shield odor, reduce toxicity, change the nature or performance of substances, extend the storage time of volatile substances, continue to release substances into the outside, and isolate non-mixable compounds [2,3].

The principle is to wrap solid, gas, or liquid into tiny particles by membrane materials made of natural or synthetic polymers. Its essence is to isolate the core material from the external environment through the closed or semi-permeable wall film, so as to protect and stabilize the core material to
shield the smell and color, and control to release the core material and so on [4]. Microcapsule technology can change the state of matter and convert liquid or gaseous substances into solid particles, which is convenient for storage and application. By coating sensitive ingredients to isolate oxygen, light, ultraviolet rays, etc., to prevent oxidative deterioration and reduce the impact of the external environment on the core of the cyst [5,6]. Although the development time of microcapsule technology is not long, it has gradually been applied to daily life in production to meet the needs of industrial production and daily consumption.

1.2. Brief history of the development of microcapsules

Microcapsules originated in the 1930s and has a history of 90 years. It has been developed rapidly in the mid-1970s. So far, more than 100 research laboratories have been developing and researching microcapsule technology, which has gradually entered various fields of production and life, and has been widely concerned at home and abroad [7].

In the field of microencapsulation, Wuster and Green were two great pioneers.

In November 1936, Atlantic Coast Fishers filed a patent application for preparing gelatin microcapsules containing cod liver oil in liquid paraffin [7].

In 1940, Gelatin Products Co., Ltd. filed a patent application for using a concentric three-layer orifice to create a drug-containing double-arm microcapsule.

In April 1950, Eastman Kodak Company filed a patent application for microencapsulation of emulsions for color photos and three primary color pigments to prepare mixed particles.

In November 1950, General Dunloberge (General Dunloberge) proposed a patent application for alginic acid microcapsules by using a double-layer orifice [8].

In 1953-1954, B.K. Green of NCR (National Cash Register Co.) was inspired by the condensation phenomenon in colloidal chemistry, he invented the method of microgelation in 1953 and successfully applied it to leuco-pressure sensitive carbon paper (NCR paper). This was also the first commercial application of microencapsulation technology [9,10].

In March 1956, NCR Company filed a patent application for the microencapsulation of optoelectronic materials [8].

In April 1957, NCR Corporation filed a patent application for the microencapsulation of compounds for color photography [8].

In August 1957, Moore Business filed a patent application for microcapsules using spray drying technology [8].

In March 1958, the Xerox Company filed a patent application for preparing microcapsules containing liquid phenomenon modifiers [8].

In May 1958, NCR Corporation filed a patent application for preparing heat-sensitive adhesives by microencapsulation. In June of the same year, the company filed a patent application for the preparation method of oily polystyrene microcapsules, in which monomers were used and the in-situ polymerization process was applied [8].

In December 1958, Upjohn filed nearly 20 patent applications. They were all related to the microencapsulation method of "emulsion" [8].

D.E. Wasrter designed an ingenious mechanical embedding method, which was to spray the wall material on the solid heart material suspended in the air to achieve encapsulation.

Then, the microencapsulation technology began to be widely used in pharmaceuticals and production life. The research on microencapsulated drugs, dyes, cosmetic and other products also increasingly grow[9].

2. Preparation of microcapsules

With the advances of science and technology, the preparation of microcapsules has gradually matured. According to statistics, there are more than 200 methods for preparing microcapsules. Using different preparation methods and materials will obtain microcapsules with different properties.
2.1. Selection of wall materials

One of the key factors determining the performance of microcapsules is the material, which is divided into wall material and core material. As the shell of the core material, the wall material plays an important role in the whole microcapsules. In the selection of wall materials should pay attention to the following aspects: (1) it should have good film-forming properties and permeability to meet the application requirements; (2) the wall material must have a certain strength, plasticity, and extrusion resistance; (3) the wall material must not react with the core material, and its solubility is different from that of the core material; (4) the wall material needs to be non-toxic, non-irritating, cheap and easy to prepare [11]. The common wall materials include natural polymer materials and their derivatives and synthetic polymer materials [12], as shown in Table 1.

![Table 1. Common materials and properties of microcapsule wall materials.](image)

| Type                          | Common material                          | Features                                                      |
|-------------------------------|------------------------------------------|----------------------------------------------------------------|
| Natural polymer materials      | Vegetable gum, sodium alginate, gelatin, fatty acid, etc. | Non-toxic or low toxicity, high viscosity, easy to form film, but poor mechanical strength |
| Semi-synthetic molecular materials, cellulose derivatives such as: dextrin, oligosaccharides. | Intolerant to acid, intolerant to high temperature, easy to hydrolyze |
| Synthetic molecular materials | Polyester, polyether, polyamide, etc.    | Good mechanical properties, and easy to control by chemical or physical modification |

Therefore, the environment and required materials should be fully considered when selecting microcapsule wall materials, and the specific microcapsule wall materials should be selected for synthesis. In recent years, some new wall materials have appeared, such as liposomes, which are widely used in food, cosmetics and pharmaceutical industries, because of their non-toxic and good biocompatibility with human body [12]. Wrapping SOD with liposomes and smearing it to the skin, it can increase the oxygen absorption capacity of damaged skin glands and prevent skin aging, light allergies and skin diseases [13]. Among them, the Poly Dimethyl Diallyl Ammonium chloride-sodium cellulose sulfate are a system with relatively good biocompatibility, which is widely favored by people [14].

2.2. Selection of core material

The material wrapped in the wall of the microcapsule is called the core material, and the core material can be liquid, solid or gaseous phase. Generally, the microcapsule core material will be released from the wall material to be effective. There are two kinds of core releasing models, one is instantaneous release and the other is slow release. Instantaneous release means that the wall material ruptures or melts under various forms of external forces such as friction, deformation, mechanical breakage, or the use of chemical methods to make the capsules loose. Slow release means that the core material is gradually released through the diffusion of the wall and the degradation of the wall material. In general the release of core material from microcapsules follows the zero-order or first-order reaction rate equation [12]. Meanwhile, the degradation mode, thickness, wall hole, crosslinking degree, crystallinity deformation mode of wall material will also affect the core material release rate.

2.3. Preparation method of microcapsules

Preparation of microcapsules has been always one of the main research directions of the researchers. So far, there are more than 200 preparation methods. The preparation of microcapsules is usually divided into physical methods, chemical methods, and physical-chemical methods according to the nature and formation mechanism of the wall and core materials. Each method can be divided into several different preparation methods according to its different process principles, and each method has its own advantages and disadvantages.
2.3.1. Physical Methods. The physical spraying-drying method, which is mostly used in the preparation of food industry. The core material is dissolved in the dilute solution of the wall material to form a suspension, and the dispersion is atomized into small droplets by spray drying, then the solvent in the system is quickly evaporated to precipitate the wall material to form microcapsules [11]. Liu Bingqian et al. [15] used pseudo-boehmite as the precursor and nitric acid as the gel solution, by combining sol with spray drying methods to obtain micron-sized porous alumina microspheres with good spheroidization and discussed its adsorption performance. In order to study the effect of iron tannate catalyst on the thermal decomposition properties of common components in solid propellants, Yang Li et al. [16] obtained three submicron composite microspheres by ultrasonic spray drying.

The air suspension method is suitable for the preparation of solid core materials. The strong airflow applied to the fluidized bed suspends the core material particles in the air, and the suspended core material undergoes a fluidization operation to precipitate microcapsules on the wall material. Liu Dongchun et al. [17] prepared taste-masking microcapsules using Wurster fluidized bed and air suspension coating method. Cao Jian et al. [18] used an air suspension method to prepare a fracturing fluid microcapsule delayed breaker with ammonium persulfate (APS) as the active core material.

By applying electrostatic bonding method, the core material and wall material were first made into the aerosols with opposite charges, and then they were held together by electrostatic attraction. Huang Fang et al. [19] used high-voltage electrostatic method combined with freeze-drying (freeze-drying) method and ion cross-linking method to prepare chitosan-gelatin porous microspheres, and then discussed the factors affecting the formation of microsphere porous structure.

In solvent evaporation method, the core material and wall material are dispersed in the organic phase in turn, and then added to a solution that is incompatible with the wall material. When the solvent is evaporated by heating, the wall material is precipitated into capsule [20]. This method is often used in the preparation of microcapsules in agricultural and food fields. Silvia Surini et al. [21] used solvent evaporation (SE) and spray drying (SD) to microencapsulate tocotrienol oil with ethyl cellulose (EC) as coating.

Vacuum evaporation deposition method uses solid particles as the core material, and the vapor of the wall material is condensed on the core material through vacuum evaporation to form microcapsules.

In inclusion complex method, the structural characteristics of β-cyclodextrin hollow and internal hydrophobicity and external hydrophilicity are utilized to form microcapsules at molecular level by forming encapsulated complexes [20].

Compared with spray technology, extrusion method is a new method. According to the principle, the mixture of core material and wall material suspended in liquefied carbohydrate medium passes through the membrane hole, which is squeezed into the solidification bath of wall material by pressure, and the wall material precipitates and hardens into a capsule [20]. This kind of microcapsules must be produced in a low temperature, and its structure stability is higher than that of SD method.

2.3.2. Chemical methods. The chemical methods include:

(1) Interfacial polymerization method. The principle is to dissolve two different active monomers in different solvents. When one solution is dispersed in another, the monomers of the two solutions are polymerized at the phase interface to form a capsule. Shell materials include polyamide, polyurea, etc. The particle size range is 1-2000 µm, and core materials are wrapped in water-soluble or insoluble materials [22]. Rui Wang [23] and others used interfacial polymerization to prepare sulfonamide polyurethane microcapsules in inverse emulsion.

(2) In-situ polymerization method. The monomer components and catalyst are all located inside or outside the core material. By the polymerization reaction it occurs to form microcapsules. The shell materials include polystyrene, polymethyl methacrylate, etc., and the particle size range is 1-2000 µm, while the core material is wrapped in insoluble material [22]. Sofia Tzavidi et al. [24] used this method to successfully prepare poly(urea-formaldehyde) microcapsules with epoxy healing agent.
micro-cores, and studied their morphology to obtain spherical microcapsules with smooth or rough surfaces.

(3) The orifice method. The principle is that the formation of the microcapsule wall is attributed to the solidification of the polymer. This method is simple to operate without organic solvents and high-speed stirring requirement. The obtained microcapsules have high mechanical strength, and the microencapsulated microencapsulation is large in size and low in embedding rate, which is widely suitable for UV-sensitive and biologically active packaging.

3. Application of microencapsulation technology

With the advances of microcapsule technology, they have been gradually applied to industrial production and daily life, such as the areas of food, medicine, agriculture, industry, and life.

3.1. Application of microcapsules in food

Many additives used in food and the nutrients from the outside that are easy to oxidize in the air and lose their activity. Microcapsule technology can wrap these easily oxidized materials into the wall material, which is convenient for transportation and storage.

In order to improve the performance of food ingredients, fixing food ingredients on suitable polymers or adding antibacterial agents are common practices in the food industry [28,29]. Microencapsulation technology is widely used in food industry. For example, lactic acid bacteria, an important bacterium used in the food industry, was first immobilized on a Berl saddle in 1975. A few years later, the lactobacillus was wrapped in alginate gel beads [30]. With the advent of controlled release technology, some heat-sensitive, temperature-sensitive or pH-sensitive additives can be conveniently used in food systems, and these additives are mainly introduced into food systems in the form of microcapsules. The additives in the microcapsules are released under the influence of specific stimuli at a specific stage [31]. For example, flavors and nutrients may be released during consumption, while easily heated sweeteners is released at the end of baking, thereby preventing undesirable caramelization in baked products [32-37].

| No | Microencapsulation technique   | Major steps in encapsulation                                                       |
|----|--------------------------------|-------------------------------------------------------------------------------------|
| 1  | Spray-drying                   | a. Preparation of the dispersion                                                   |
|    |                                | b. Homogenization of the dispersion                                                |
|    |                                | c. Atomization of the infeed dispersion                                             |
|    |                                | d. Dehydration of the atomized particle                                            |
| 2  | Spray-cooling                  | a. Preparation of the dispersion                                                   |
|    |                                | b. Homogenization of the dispersion                                                |
|    |                                | c. Atomization of the infeed dispersion                                             |
| 3  | Spray-chilling                 | a. Preparation of the dispersion                                                   |
|    |                                | b. Homogenization of the dispersion                                                |
|    |                                | c. Atomization of the infeed dispersion                                             |
| 4  | Fluidized-bed coating          | a. Preparation of coating solution                                                  |
|    |                                | b. Fluidization of core particles                                                  |
|    |                                | c. Coating of core particles                                                       |
| 5  | Extrusion                      | a. Preparation of molten coating solution                                           |
|    |                                | b. Dispersion of core into molten polymer                                          |
|    |                                | c. Cooling or passing of core-coat mixture through dehydrating liquid              |
| 6  | Centrifugal extrusion          | a. Preparation of core solution                                                    |
|    |                                | b. Preparation of coating material solution                                        |
|    |                                | c. Co-extrusion of core and coat solution through nozzles                           |
| 7  | Lyophilization                 | a. Mixing of core in a coating solution                                             |
|    |                                | b. Freeze-drying of the mixture                                                    |
| 8  | Coacervation                   | a. Formation of a three-immiscible chemical phases                                |
|    |                                | b. Deposition of the coating                                                       |
3.2. Application of microcapsules in medical treatment
As a new treatment method, immunotherapy has attracted great interest and showed the potential of treating cancer [38, 39]. However, the adverse immune microenvironment seriously affected the vaccination, thus the self-repairing function of microcapsules provides a good example for antigen encapsulation. Xiaobo Xi et al. [27] reported a new type of microcapsule-based high-performance cancer vaccination program with special self-repairing functions, providing a mild and effective paradigm for antigen microencapsulation. After vaccination, the microcapsules can form a good immune microenvironment in situ, in which the kinetics of antigen release. It can effectively improve antigen utilization and antigen presentation, and activate antigen presentation cells. Finally, the various types of antigens are used to achieve effective T cell responses, effective tumor suppression, anti-metastasis effects and prevention of postoperative recurrence, and while, the new antigens are encapsulated and evaluated in different tumor models.

Because of its excellent immune isolation performance and controlled release performance, microcapsules are expected to become an important tool for the practical and industrialization of upstream research results in bioengineering, and promote tissue recombinant cell transplantation technology, gene therapy and protein. Those polypeptide biochemical drugs are applied to clinical application as soon as possible and contribute positively to human health and quality of life [40].

3.3. Application of microcapsules in agriculture
With the further development of the food industry, the pesticide has occupied an important position in agricultural production. The combination of agriculture and microcapsule technology is inevitable to meet the needs of consumers. The slow release of pesticide microcapsules greatly reduces the risk of pesticides and can also promote the long-term growth of crops. Meanwhile, because of the higher demand for green food, the research of pesticide microcapsules has attracted much attention. Shenyang Research Institute of Chemical Industry has begun to study microencapsulation technology since 1970s. In 1982, there was the first commercial pesticide microcapsule-25% parathion microcapsule. Pesticide microcapsules can disperse pesticides or their solutions in immiscible solvents to form particles and then add two monomers (insoluble resin monomers and solvent-soluble monomers) to polymerize to form pesticide microcapsules.

3.4. Application of microcapsules in industrial production
In industrial production, the development of microcapsule technology in the field of coatings is quite fruitful. The core-shell material of the microcapsule can change the structure of coating. While adding the flame retardant and other raw materials into the coating, the performance can be enhanced. Yan Xiaoxing et al. [41] prepared urea-formaldehyde resin coated waterborne coating microcapsules by means of in-situ polymerization, and added the microcapsules to waterborne coatings with different mass fractions, to study the effects of different microcapsule mass fractions on the performance and self-restraint of the water-based crack coating on the wood surface.

3.5. Applications of microcapsules in other areas
As the increase in demand for soil and water treatment in China, the research on microcapsules, which reduce the content of heavy metals, is imminent. Up to now, the research on microcapsules for soil and water treatment is still immature at home and abroad. Only by continuously improving the process and developing new microcapsule materials suitable for different ranges of microcapsules, the scope of applications will be broadened.

Sun Quanping[42] collected 9 soil samples from each of the 3 typical functional areas in Lhasa, Tibet, with a sampling depth of 0-20cm. With determination of 7 heavy metal elements, such as As, Hg, Cr in soil, and analysis of the spatial distribution characteristics of heavy metals, they adopted the single factor pollution index method to judge the pollution degree of the main pollution factors in the environment. Then the potential ecological hazard was evaluated by the potential ecological risk index method to understand the pollution levels of heavy metal in different functional areas around Lhasa.
The results showed that the as enrichment in the sample was the most serious issue with a pollution sharing rate of 62.7%. It can be seen that the as content of Tibetan soil is seriously exceeding the standard, and the removal of heavy metals by microcapsules has become a new method that can be adopted.

3.6. Potential applications in Tibet Plateau

Tibet is a plateau with an average elevation of about 3,000 meters above sea level, being honored as the roof and the third pole of the world. The air in Tibet is thin and the oxygen content is very low, which is only about two thirds of that at sea level. In addition, the weather is cold and dry most of the year. These formidable natural conditions make the ecological environment fragile and the characteristic plants malnourished. On the other hand, Tibet Plateau is rich in mineral and water resources, resulting in high level of heavy metals in the soil and waters.

Through the micro-packing technology, the nutrients needed by the plants are wrapped in the polymer shell to form microcapsules, which are injected to make the plants have sufficient nutrients during the growth process. The controllable release ability of microcapsules can protect plant nutrition and promote the growth and development of characteristic plants. In addition, microcapsule technology can also be utilized to remove heavy metals in soil and water bodies. Other applications for various needs of groundwater pollution prevention and control include: (1) regulation of pH with microcapsule slow-release to maintain the best survival of groundwater microorganisms; (2) heavy metal chelating agent by microcapsule to continuous release of chelating agent to remove heavy metal ions in groundwater; (3) carbon source supply with microcapsule to continuous offer carbon source to microorganisms that treat groundwater to ensure energy supply; (4) microcapsule slow-release reducing material: continuous releasing of reducing agents to reduce heavy metals or nitrates; (5) microcapsule slow-release oxidizing materials: continuous releasing of oxidants to degrade organic pollutants and sterilization; (6) Microcapsule slow-release microorganisms: continuous releasing of microorganisms required for groundwater treatment. Soil remediation agents can be used to remove heavy metals from the soil. Organic waste liquid from the yeast industry is used as one of the main raw materials to reduce environmental pollution and at the same time reduce the cost of soil remediation agents. In addition to effectively controlling heavy metal pollution, it also helps increase crop production and income, and has potential applications.

4. Conclusion

With the advances of science and technology and the improvement of daily life, the application prospects of microcapsules will be gradually broad. It is mainly used in the fields of food, cosmetics, medical engineering, production and construction, textile industry and so on. Tibet is located in a high-altitude area, where heavy metal level exceeds the standard condition in the soil and medicinal materials. According to the related literature, furfuryl alcohol can be used as the wall material of acid aqueous microencapsulation, and methyl fatty acid as solvent. Under the action of catalyst, furfuryl alcohol in fatty acid methyl ester gradually polymerized at the oil-water interface to form a wall material, which is wrapped with an acidic aqueous solution (the combination of sulfuric acid and phosphoric acid improves the overall extraction efficiency of arsenic and heavy metals in contaminated soil samples). The microcapsule has a simple synthesis process, strong product controllability, good safety and environmental friendliness. It is feasible and controllable within a certain theoretical range, and its correlation characteristics and results need further study.

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