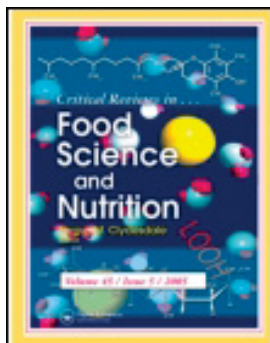


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### Microencapsulation in the food industry

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Petition for Inter Partes Review  
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Exhibit

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## MICROENCAPSULATION IN THE FOOD INDUSTRY

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### INTRODUCTION

Anyone who has contemplated the mysteries of a bird egg, the durability of a bacterial spore, or the complexities of a living red blood cell, has, perhaps without realizing it, thought about microencapsulation.

The variety and versatility of natural cellular membranes are well known and, indeed, the presence of such membrane structures was necessary before life could evolve and progress to its present complex forms.

Many types of foods familiar to us utilize exterior cellular membranes, commonly called skins. Seeds, grains, fruits, and berries all have exterior skins which protect the interior oils or fruit juices from the harsh and unpredictable outside environment. These membranes control the loss of water, regulate the transfer of gases, provide isolation from airborne contamination, function as a temporary temperature insulation, and also often furnish protective supportive structures.

Man-made microcapsules are similar to nature's in that they also consist of an outer film or skin

and an inner "core" of liquid or solid material - the active material requiring protection.

Microencapsulation technology has given man a means to duplicate some of the protective and selective properties of natural membrane films. Through a choice of different capsular film coatings, both gaseous and liquid transfer can be controlled. Oxygen transfer and resultant oxidation can be reduced. The transfer of salts or organic molecules can be regulated. By varying the wall thickness, or the physical properties, e.g., toughness, flexibility, etc., of the capsule wall, strength and durability can be built into the capsule structure.

A choice of capsule solubility and meltability is even possible. Thus, a capsule can be made to dissolve in hot water, but not cold; or to melt at a certain temperature, but not before. Even pH changes can be utilized for the controlled release of encapsulated materials.

While there is some discussion concerning what exactly should be defined as a microcapsule, or a microencapsulation process, in order to include those processes most used in the food industry, this review uses the broadest interpretation of microencapsulation.

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Schematic diagrams of microcapsules are shown in Figure 1, and 1A.

The applicability and the utility of micro-encapsulation in the food industry are becoming generally recognized. Besides the examples previously cited, utilization of sensitive or active problem materials is often enhanced by changing the apparent physical form. For instance, liquid flavor aromas and oils encapsulated in an edible starch or gelatin matrix result in dry pourable powders, which can be used directly in dry food

products. The encapsulated materials show reduced reactivity with other food ingredients and have a lower susceptibility for oxygen-induced rancidity.

Capsules can be produced in sizes varying from sub-micron to pea-sized particles.

Where attractiveness is important, it is also possible to add food grade colors or pigments, either to the capsule wall or to the core liquid within.

### A. SINGLE CAPSULES

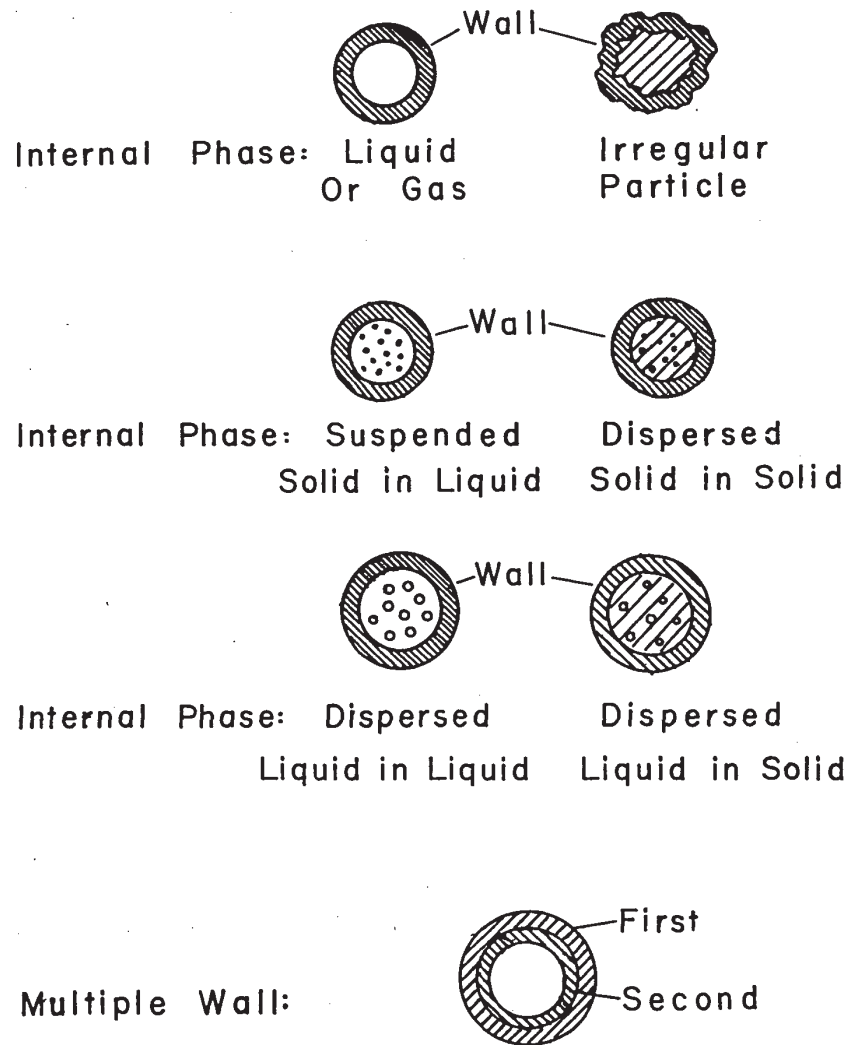


FIGURE 1. Capsule Configuration.

## MICROENCAPSULATION IN THE FOOD INDUSTRY

The food industry is constantly interested in improving the flavor, aroma, stability, nutritive value, and appearance of its products. Microencapsulation has much to offer to food producers

in these areas. While some applications have been utilized for a considerable time, e.g., vitamin protection, many developments are quite recent. Past studies have shown that encapsulation could furnish increased stability to vitamins, which are normally sensitive to UV radiation, light, oxygen, metals, and humidity. More recent work has

### B. MULTIPLE CAPSULES (AGGREGATE)

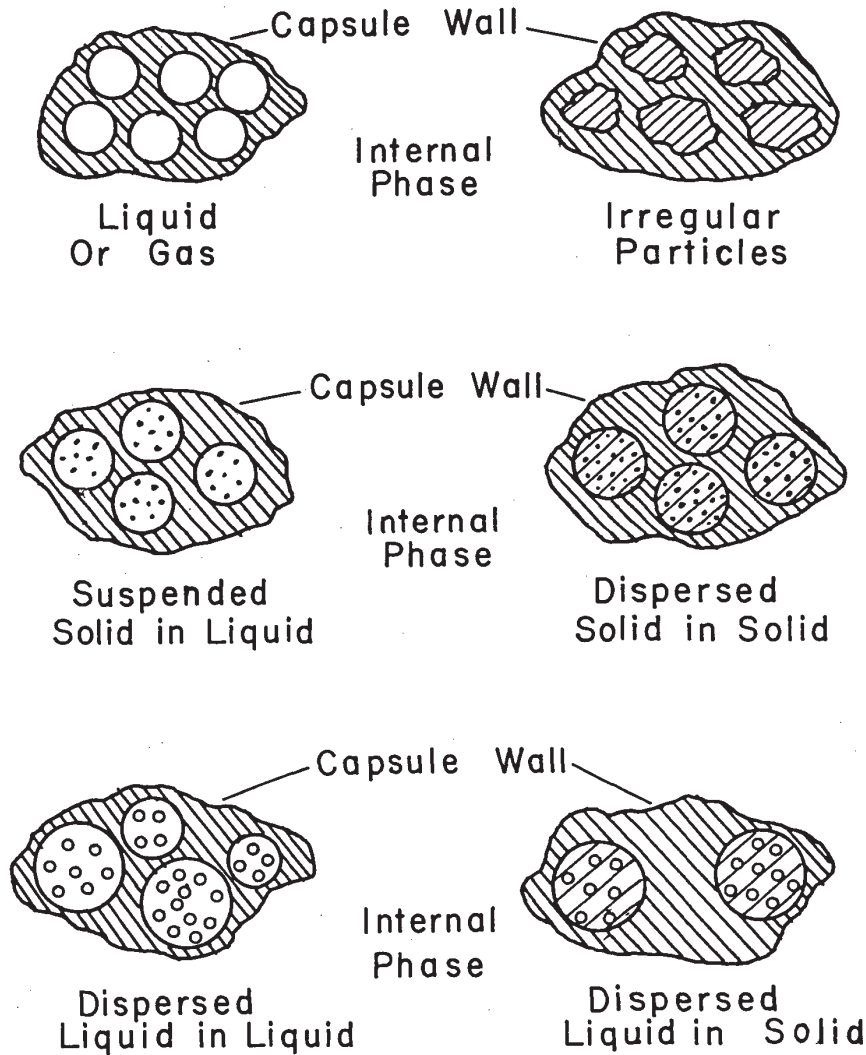


FIGURE 1A. Capsule Configuration.

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shown that citrus oils and aromas can be successfully protected from oxidative and even thermal degradation.

Diet in the industrialized countries consists increasingly of processed food. As the length of time for storage and transit of foods from the place of manufacture to the market place increases, insuring a nutritive value equal to or surpassing that in the original food becomes both more important and more difficult.

Essential or desirable components of foods such as fugitive aromas, flavors, vitamins, oxidizable constituents, and other ingredients sensitive to heat all tend to be affected to some degree by processing techniques.

Microencapsulation can offer the food producer:

1. a means to store sensitive food components for protection from other food ingredients during storage,
2. a means of adding nutritive materials to foods after processing and insuring that the original nutritive levels are not lost on extended storage under the expected storage conditions,
3. a method of utilizing novel, fugitive, or otherwise sensitive components to prepare new and highly nutritional foodstuffs,
4. unusual or time-saving release mechanisms for foods and food products,
5. additional attractiveness for the display and merchandising of food products.

The present review covers the most generally used encapsulation techniques and systems in the food industry as well as the more recently introduced or proposed techniques.

### SPRAY DRYING

Spray drying is one of the oldest and certainly the most generally used method of encapsulation in the food industry. Spray drying is a form of gas solids contacting in which the solids phase usually contains a solvent or diluent, e.g., water, which is removed in the drying process.

Spray drying achieves the same or similar results as most other encapsulation processes. That is, it covers an active material with a protective coating which is essentially inert to both the material being encapsulated and to the drying medium.

In the encapsulation of food additives, the "solids phase" may be either a liquid or even a volatile food flavor combined with a capsule wall material. As a rule, the wall-former is chosen from a group of food grade hydrocolloids such as gelatin, vegetable gum, modified starches, or dextrin. The solvent or diluent liquid is usually water in which the "solids phase" is dissolved, emulsified, or dispersed. This "solids phase" is essentially non-volatile under the conditions of the process.

In the case of food flavors or oils, the hydrocolloid capsule wall formers are first dissolved in water. The oils or flavors, composed largely of essential oils, aldehydes, ketones, alcohols, esters, and organic acids, are then either emulsified (if water insoluble), or dissolved (if water soluble) in the hydrocolloid solution.

Spray drying and other "spray" systems involve three basic steps:

1. liquid atomization
2. gas-droplet mixing
3. drying from liquid droplets

Atomization is accomplished by one of three atomizing devices:

1. high-pressure nozzles (single-fluid nozzles)
2. two-fluid nozzles
3. high speed centrifugal disks

With these atomizers low viscosity solutions of up to 2000 cP (Brookfield) may be broken up into droplets as small as  $2\mu$ . The largest drop sizes rarely exceed  $500\mu$  (35 mesh). Because of the large total drying surface of the small droplets, the actual drying time in a spray dryer is in the range of fractions of seconds to a few seconds at most.

The total residence time of a particle in a spray drying system is, on the average, less than 30 sec.

Spray drying produces spherical shaped particles, the result of the free suspension of the liquid droplets in a gaseous medium in which the droplets acquire a spherical shape. These dried particles may be either solid or hollow depending on the characteristics of the material processed and on the drying conditions.

A typical spray dryer is shown in Figure 2. Most spray dryers consist of a large, usually vertical, cylinder chamber (A), into which material

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