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Scientific Status Summary

Functional Materials in Food Nanotechnology

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The Institute of Food Technologists has issued this Scientific Status Summary to update readers on the applications of nanotechnology in the food industry.

Keywords: colloids, delivery systems, nano-emulsions, nanolaminates, nanoparticles, nanoscale, nanotechnology

Nanotechnology focuses on the characterization, fabrication, and manipulation of biological and nonbiological structures smaller than 100 nm. Structures on this scale have been shown to have unique and novel functional properties. Consequently, interest and activities in this research area have greatly increased over the past years. According to the National Nanotechnology Initiative (2006), "Nanotechnology is the understanding and control of matter at dimensions of roughly 1 to 100 nanometers, where unique phenomena enable novel applications. Encompassing nanoscale science, engineering and technology, nanotechnology involves imaging, measuring, modeling, and manipulating matter at this length scale."

With the increased funding opportunities and interest in this field, the term "nano" is more frequently and often liberally used, which has led to some criticism within the scientific community. Whether justified or not, it should be understood that the entire field of nanoscience is essentially an eclectic derivative of established disciplines such as chemistry, interface science, microfabrication technologies, and so on. However, use of the term "nano" does allow researchers to highlight the fact that processes (for example, nanomanufacturing) or material structures (for example, nanomaterials) are designed and optimized to use specific properties and behaviors at lengths of 10^{-7} to 10^{-9} m.

The potential benefits of nanotechnology have been recognized by many industries, and commercial products are already being manufactured, such as in the microelectronics, aerospace, and pharmaceutical industries. Developments in these industries are driven by fundamental and applied research in physics, chemistry, biology, engineering, and materials science. In contrast, applications of nanotechnology within the food industry are rather limited. However, achievements and discoveries in nanotechnology are beginning to impact the food industry and associated industries; this affects important aspects from food safety to the molecular synthesis of new food products and ingredients (Chen and others 2006). The fact that systems with structural features on the nanoscale have physical, chemical, and biological properties substantially different from their macroscopic counterparts is changing the understanding of biological and physical phenomena in food systems. Since foods are complex biological systems that are governed by many of the same basic mechanisms and principles that biologists and biochemists study, one would expect that the discoveries made in nanotechnology may eventually also impact the food industry. However, foods undergo a variety of postharvest and processing-induced modifications that affect the biological and biochemical functionality of the system. Nanotechnology allows scientists to measure, control, and manipulate matter at the nanoscale level to change those properties and functions in a beneficial way.

This article provides an overview of some current development efforts in the area of nanotechnology as it applies to food systems. In particular, the article presents some of the morphologically different structures and associated manufacturing technologies that could be used to build functional food systems. Moreover, the article focuses on applications with which the authors have experience and is tailored specifically toward current and emerging technologies that may be used for food formulations, processing, and storage. Although nanotechnology potentially has numerous applications in the food industry, this article does not delve into the areas of food safety and security. These topics will be the subject of a separate article. Interested readers seeking information about nanotechnology in food *and* agricultural systems might additionally refer to a report published by CSREES, USDA (2003).

Nanotechnology: drawing inspiration from nature

Living organisms are not just a collection of nanoscale objects: Atoms and molecules are organized in hierarchical structures and dynamic systems that are the results of millions of years of Mother Nature's experiments. Tenth-nanometer diameter ions such as potassium and sodium generate nerve impulses. The size of vital biomolecules—such as sugars, amino acids, hormones, and DNA—is in the nanometer range. Membranes that separate 1 cell from another, or 1 subcellular organelle from another, are about 5 times bigger. Most protein and polysaccharide molecules have nanoscale dimensions. Every living organism on earth exists because of the presence, absence, concentration, location, and interaction of these nanostructures.

Nature is making extensive use of self-assembly principles to create nanoscale structures. Rather than requiring the expenditure of large amounts of energy for assembly and creation, nanoscale structures are formed through a series of optimized processes that utilize

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the tendency of a system to minimize its overall free energy, thereby minimizing required activation energies. Production of nanoscale structures for use in food science and technology therefore frequently relies on an in-depth understanding of thermodynamically driven self-assembly processes. Areas of research that could prove useful in the near future include molecular design of protective surface systems (Charpentier 2005), surface engineering (Krajewska 2004), and various methods of manufacturing, such as electrospinning (Min and others 2004) and nanofiltration (van der Graaf and others 2005).

Potential Food Applications

Nanotechnology has the potential to impact many aspects of food and agricultural systems. Food security, disease-treatment delivery methods, new tools for molecular and cellular biology, new materials for pathogen detection, and protection of the environment are examples of the important links of nanotechnology to the science and engineering of agriculture and food systems. Examples of nanotechnology as a tool for achieving further advancements in the food industry are as follows:

- Increased security of manufacturing, processing, and shipping of food products through sensors for pathogen and contaminant detection.
- Devices to maintain historical environmental records of a particular product and tracking of individual shipments.
- Systems that provide integration of sensing, localization, reporting, and remote control of food products (smart/intelligent systems) and that can increase efficacy and security of food processing and transportation.
- Encapsulation and delivery systems that carry, protect, and deliver functional food ingredients to their specific site of action.

Most nanotechnological research focuses on the development of applications in biosciences and engineering. Strategies to apply nanoscience to the food industry are quite different from these more traditional applications of nanotechnology. Food processing is a multitechnological manufacturing industry involving a wide variety of raw materials, high biosafety requirements, and well-regulated technological processes. Four major areas in food production may benefit from nanotechnology: development of new functional materials, microscale and nanoscale processing, product development, and methods and instrumentation design for improved food safety and biosecurity. Figure 1 depicts possible applications of nanotechnology in the food industry.

The influence of the material properties of foods at the nanoscale level on their bioavailability and nutritional value has been highlighted (Blundell and Thurlby 1987; Aguilera 2005). In addition, the relationship between the morphology of food materials and their bulk physicochemical properties has been investigated (Losche 1997): for example, biopolymers in solutions, gels, and films (Chinnan and Park 1995; Janaswamy and Chandrasekaran 2005). Functional nanostructures can incorporate individual biological molecules, which is useful in the development of biosensors that can use natural sugars or proteins as target-recognition groups (Charych and others 1996).

In summary, there are a large number of potential applications of nanotechnology within the food industry; however, many of these may be difficult to adopt commercially because they are either too expensive or too impractical to implement on an industrial scale. The subsequent sections focus on a limited number of nanotechnology applications that may have commercial potential in the near future. Most likely, the limited application of nanotechnology to the food industry will change as nanofabrication technologies become more cost-effective.

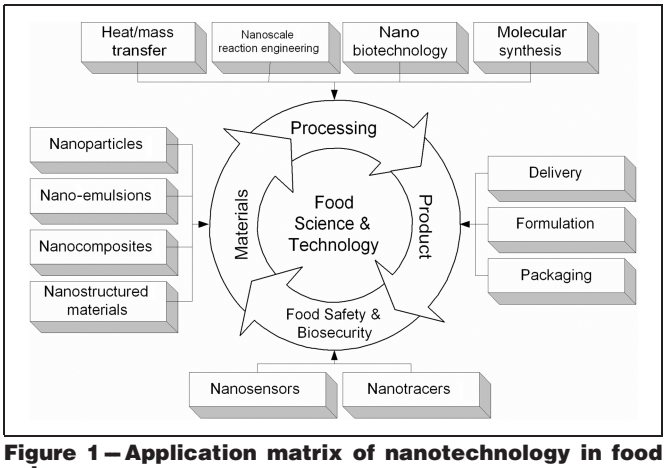

Figure 1—Application matrix of nanotechnology in food science

Nanodispersions and Nanocapsules

Functional ingredients (for example, drugs, vitamins, antimicrobials, antioxidants, flavorings, colorants, and preservatives) are essential components of a wide range of industrial products, including pharmaceuticals, health-care products, cosmetics, agrochemicals, and foods. These functional ingredients come in a variety of different molecular and physical forms such as polarities (polar, nonpolar, amphiphilic), molecular weights (low to high), and physical states (solid, liquid, gas). Functional ingredients are rarely utilized directly in their pure form. Instead, they are often incorporated into some form of delivery system.

A delivery system must perform a number of different roles. First, it serves as a vehicle for carrying the functional ingredient to the desired site of action. Second, it may have to protect the functional ingredient from chemical or biological degradation (for example, oxidation) during processing, storage, and utilization; this maintains the functional ingredient in its active state. Third, it may have to be capable of controlling the release of the functional ingredient, such as the release rate or the specific environmental conditions that trigger release (for example, pH, ionic strength, or temperature). Fourth, the delivery system has to be compatible with the other components in the system, as well as being compatible with the physicochemical and qualitative attributes (that is, appearance, texture, taste, and shelf-life) of the final product.

The characteristics of the delivery system are one of the most important factors influencing the efficacy of functional ingredients in many industrial products. A wide variety of delivery systems has been developed to encapsulate functional ingredients, including simple solutions, association colloids, emulsions, biopolymer matrices, and so on. Each type of delivery system has its own specific advantages and disadvantages for encapsulation, protection, and delivery of functional ingredients, as well as cost, regulatory status, ease of use, biodegradability, and biocompatibility. A number of potential delivery systems based on nanotechnology follow.

Association colloids

Association colloids—such as surfactant micelles, vesicles, bilayers, reverse micelles, and liquid crystals—have been used for many years to encapsulate and deliver polar, nonpolar, and/or amphiphilic functional ingredients (Garti and others 2004, 2005; Golding and Sein 2004; Flanagan and Singh 2006). For example, a nonpolar functional ingredient may be solubilized within the hydrophobic core of a surfactant micelle or as part of the

micellar membrane structure; thus, it can be delivered in an aqueous solution depending on the requirements of the specific application. Association colloids are thermodynamically favorable systems whose formation is normally driven by the hydrophobic effect—that is, the reduction of the contact area between the nonpolar groups of the surfactant that comprise the association colloid and water. The type of association colloid formed and the nature of the resultant structures depend on the concentrations and molecular characteristics of the surfactant and cosurfactant used as well as the prevailing environmental conditions (for example, temperature, ionic strength, and pH). It should be noted that the location of the encapsulated functional ingredient within the association colloid (for example, in the hydrophobic core or as part of the association colloidal membrane) is of particular importance to the functionality of the self-assembled system. The dimensions of many association colloids are in the range of 5 to 100 nm, and these structures are therefore considered to be nanoparticles.

The major advantages of association colloid systems are that they form spontaneously, are thermodynamically favorable, and are typically transparent solutions. On the other hand, the major disadvantage is that a large quantity of surfactant (and in many cases, cosurfactant) is required to form them, which may lead to problems with flavor, cost, or legality. Moreover, because the formation of association colloids is concentration-driven, diluting the solutions containing the colloids can lead to their spontaneous dissociation. Thus, the choice of surfactants and cosurfactants to form colloids is critical in ensuring their functionality over a wide range of environmental conditions.

Nano-emulsions

The use of high-pressure valve homogenizers or microfluidizers often causes emulsions with droplet diameters of less than 100 to 500 nm. In modern literature such emulsions are often referred to as "nano-emulsions." Nano-emulsions have been produced and studied for many years, so a large body of literature dealing with their preparation, characterization, and utilization exists (McClements 2004). Functional food components can be incorporated within the droplets, the interfacial region, or the continuous phase. Encapsulating functional components within the droplets often enables a slowdown of chemical degradation processes by engineering the properties of the interfacial layer surrounding them (McClements and Decker 2000). While it is difficult to engineer the interfaces to be completely impermeable to compounds in the bulk phase that may interact with the encapsulated compounds, the rate of permeation can often be significantly reduced, thus increasing the kinetic stability of the bioactives.

Nanostructured multiple emulsions

The use of multiple emulsions can create delivery systems with novel encapsulation and delivery properties. The most common examples of this are oil-in-water-in-oil (O/W/O) and water-in-oil-in-water (W/O/W) emulsions (Garti and Benichou 2001, 2004). For example, a nanostructured W_1OW_2 emulsion would consist of nanometer-sized water droplets or reverse micelles (W_1) contained within larger oil droplets (O) that are dispersed within an aqueous continuous phase (W_2). Functional food components could be encapsulated within the inner water phase, the oil phase, or the outer water phase, thereby making it possible to develop a single delivery system that contains multiple functional components. This technology could be used to separate 2 aqueous phase components that might adversely react with each other if they were present in the same aqueous phase. Alternatively, it could be used to protect and release an aqueous phase component trapped within the inner water droplets (W_1) to a specific site such as the mouth, stomach, or small intestine.

Nanostructured multilayer emulsions

Recent studies have shown that the use of multilayer emulsions can create novel delivery systems. These systems typically consist of oil droplets (the core) surrounded by nanometer thick layers (the shell) comprised of different polyelectrolytes. These layers are formed using a layer-by-layer (LbL) electrostatic deposition method that involves sequential adsorption of polyelectrolytes onto the surfaces of oppositely charged colloidal particles. Figure 2 shows an example of the LbL approach to encapsulating oil droplets in an O/W emulsion. An ionic emulsifier that rapidly adsorbs to the surface of lipid droplets during homogenization is used to produce a primary emulsion containing small droplets; then an oppositely charged polyelectrolyte is added to the system, which adsorbs to the droplet surfaces and produces a secondary emulsion containing droplets coated with a 2-layer interface. This procedure can be repeated to form oil droplets coated by interfaces containing 3 or more layers. Under certain circumstances, emulsions containing oil droplets surrounded by multilayer interfaces have been found to have better stability against environmental stresses than conventional oil-in-water emulsions with single-layer interfaces (Gu and others 2005; Mun and others 2005; Guzey and McClements 2006). In addition, it is possible to develop smart delivery systems by engineering the properties of the nanostructured shell around the droplets.

This interfacial engineering technology would utilize food-grade ingredients (such as proteins, polysaccharides, and phospholipids) and processing operations (such as homogenization and mixing) that are already widely used in the manufacture of food emulsions. Therefore, this technology should be economically viable and could be easily implemented by the food industry.

A functional component trapped within the core of a multilayer emulsion delivery system could be released in response to a specific environmental trigger by designing the response of the shell to the environment as in the following examples:

1. *Complete Shell Dissociation*: Weakening electrostatic interactions can cause shells to completely dissociate under specific solution conditions (pH, ionic strength). For instance, changing the pH can cause one or more of the polyelectrolytes to lose its charge, or increasing the ionic strength can weaken the electrostatic attraction of a polyelectrolyte to the next layer, thereby promoting desorption.

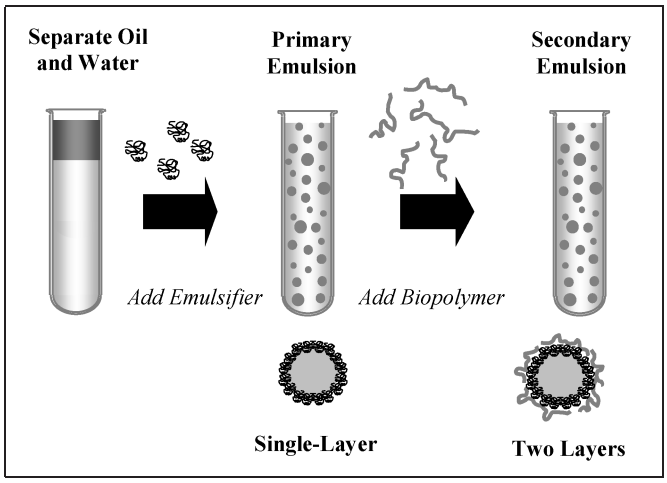

Figure 2—Schematic for formation of a number of nanolayers around particles

2. *Modulation of Shell Porosity*: The thickness and porosity of shells can change with exposure to pH and ionic strength. This determines the rate at which functional components trapped inside the core will diffuse into the surrounding medium. By selecting the appropriate polyelectrolytes to use and the assembly conditions, one could design systems to release, under specific environmental triggers, functional components smaller than some particular dimension.

In principle, one could vary the release of 1 or more encapsulated materials using either of these release mechanisms, either individually or in combination (simultaneously or sequentially).

Biopolymeric nanoparticles

Particles in the nanometer-sized range can often be produced using food-grade biopolymers such as proteins or polysaccharides (Chang and Chen 2005; Gupta and Gupta 2005; Ritzoulis and others 2005). These particles may be formed by promoting self-association or aggregation of single biopolymers or by inducing phase separation in mixed biopolymer systems, for example, using aggregative (net attraction) or segregative (net repulsion) interactions. Functional ingredients can be encapsulated in nanoparticles formed and released in response to specific environmental triggers by altering the solution conditions to induce complete particle dissolution or changes in particle porosity. A more in-depth discussion on the production and utilization of such particles follows.

Nanolaminates

Nanotechnology provides food scientists with a number of ways to create novel laminate films suitable for use in the food industry. A nanolaminate consists of 2 or more layers of material with nanometer dimensions that are physically or chemically bonded to each other (Figure 3). One of the most powerful methods is based on the LbL deposition technique, in which the charged surfaces are coated with interfacial films consisting of multiple nanolayers of different materials (Decher and Schlenoff 2003). Similar to the preparation of multiple emulsions, electrostatic attraction causes polyelectrolytes and other charged substances to be deposited onto oppositely charged surfaces. This LbL technology allows precise control over the thickness and properties of the interfacial films, which in this case enables the creation of thin films (1 to 100 nm per layer).

Nanolaminates can give food scientists some advantages for the preparation of edible coatings and films over conventional technologies and may thus have a number of important applications within the food industry. Edible coatings and films are currently used on a wide variety of foods, including fruits, vegetables, meats, chocolate, candies, bakery products, and French fries (Morillon and others 2002; Cagri and others 2004; Cha and Chinnan 2004; Rhim 2004). These coatings or films could serve as moisture, lipid, and gas barriers. Alternatively, they could improve the textural properties of foods or serve as carriers of functional agents such as colors, flavors, antioxidants, nutrients, and antimicrobials.

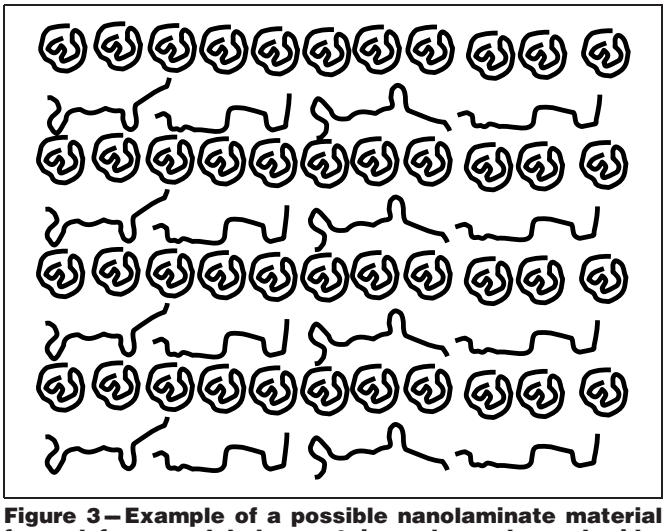

Figure 3—Example of a possible nanolaminate material formed from a globular protein and a polysaccharide. Each layer is approximately 1 to 100 nm.

The basic functional properties of edible coatings and films depend on the characteristics of the film-forming materials used for their preparation. At present, the primary film-forming materials used to construct these edible coatings and films are polysaccharides, proteins, and lipids. Generally, lipid-based films are good moisture barriers, but they offer little resistance to gas transfer and have poor mechanical strength. In contrast, biopolymer-based films are often good oxygen and carbon dioxide barriers, but they offer little protection against moisture migration (Park 1999). Consequently, there has been a great deal of research on identifying additives that can be used to improve the functional properties of edible films and coatings (for example, polyols, emulsion droplets, surfactant micelles, fibers, or particulate matter). To date, most edible films and coatings are formed with little consideration of the internal structure created. Moreover, specific design of nanoscale and microscale internal structures to overcome problems associated with edible films and coatings has not been pursued. Nanolaminates are more likely to be used as coatings that are attached to food surfaces, rather than as self-standing films, because their extremely thin nature makes them very fragile (Kotov 2003).

Figure 4 shows an example of how nanolaminates could encase food objects. The object to be coated with a nanolaminate would be dipped into a series of solutions containing substances that would adsorb to the surface of the object (McClements and others 2005). Alternatively, the solutions containing the adsorbing substances could be sprayed onto the surface of the object. The composition, thickness, structure, and properties of the multilayered laminate formed around the object could be controlled in a number of ways, including (i) changing the type of adsorbing substances in the dipping solutions; (ii) changing the total number of dipping steps used; (iii) changing the order that the object is introduced into the various dipping solutions; or (iv) changing the solution and environmental conditions used, such as pH, ionic strength, dielectric constant, temperature, and so on. The driving force for adsorption of a substance to a surface would depend on the nature of the surface and the nature of the adsorbing substance. The force itself could be electrostatic, hydrogen-bonding, hydrophobic interactive, thermodynamically incompatible, and so on, but it would usually be electrostatic attraction of oppositely charged substances. The influence of the properties of the substrate surface—such as topology and roughness on the structure of the nanolaminates that are built on the substrate surface—has not yet been established. It is possible that nonuniform laminates could be formed that contain microscopic and macroscopic pores that could negate the barrier function of the laminate. Consequently, this would necessitate the formation of a second base biopolymer layer on the food product to form a more uniform substrate surface, followed by deposition of the layer containing the functional ingredient.

A variety of different adsorbing substances could be used to create the different layers (Figure 5), including natural polyelectrolytes (proteins, polysaccharides), charged lipids (phospholipids, surfactants), and colloidal particles (micelles, vesicles, droplets). The choice of the type of adsorbing substances used to create each layer, the total number of layers incorporated into the overall film,

the sequence of the different layers, and the preparation conditions used to prepare each layer will determine the functionality of the final films (for example, their permeability to gases, organic substances, minerals, or water; their mechanical properties, such as rigidity, flexibility, or brittleness; their swelling and wetting characteristics; and their environmental sensitivity to pH, ionic strength, and temperature). In addition, the aforementioned procedure could be used to encapsulate various hydrophilic, amphiphilic, or lipophilic substances within the films by incorporating them, for example, in oil droplets or association colloids (such as micelles or liposomes). As a result, it would be possible to incorporate active functional agents such as antimicrobials, antibrowning agents, antioxidants, enzymes, flavors, and colors into the films. These functional agents would increase the shelf life and quality of coated foods. These nanolaminated coatings could be created entirely from food-grade ingredients (proteins, polysaccharides, lipids) by using simple processing operations such as dipping and washing.

Biopolymeric Nanoparticles

Research into the production and use of biodegradable polymers for use in the manufacturing of dispersed systems began as early as 70 years ago. First developed in 1932, polylactic acid (PLA) is a key component of many biodegradable nanoparticles. However, its high cost and susceptibility to hydrolytic breakdown supposedly made it unsuitable for use in biomedical or agricultural applications; thus, it was used only sparingly in research (Lunt 1998). However, in the 1970s, the use of PLA as an ideal material for sutures was discovered, and in the 1980s a process was developed to produce the polymer via bacterial fermentation, which greatly reduced costs and increased production rate. These 2 developments led to polymers becoming the focus of many studies. Today, PLA is widely available through a number of manufacturers. Biodegradable polymers have found wide applications in the field of biomedicine: for example, eliminating the need for surgical implantation and removal, and encapsulating and delivering a wide variety of compounds (including drugs, vaccines, and proteins). For example, chitosan—a natural antimicrobial polymer obtained by deacetylating chitin extracted from crustacean shells—and synthetic polymers PLA, polyglycolic acid (PGA), and polycaprolactic acid are used to encapsulate and deliver compounds. Copolymers created using combinations of the monomers lactide, galactide, and caprolactone have also been examined.

Because of its excellent encapsulation properties, PLA is one of the principal building blocks of many biodegradable nanoparticles, but it has its limitations. PLA is quickly removed from the bloodstream and sequestered in the liver and the kidneys. Although this is ideal for the treatment of intracellular pathogens that are isolated in these areas, it is less desirable for the delivery of active components to other areas of the body. PLA also breaks down in intestinal fluid, which limits its use as a carrier for oral delivery. These problems can be overcome by associating a hydrophilic compound—such as polyethylene glycol (PEG)—with the hydrophobic PLA nanoparticle. Nanoparticles consisting of a PLA-PEG diblock copolymer form a micellar-like assembly that can entrap a compound that is to be delivered (Riley and others 1999).

The molecular weight of PLA and the ratio of PLA to PEG are key factors in the formation of stable nanoparticles that can avoid the reticuloendothelial system and resist agglomeration and breakdown

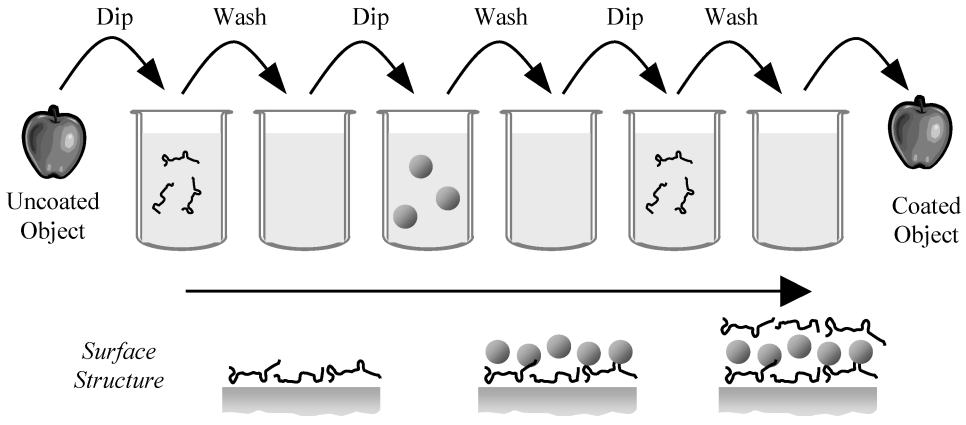

Figure 4—Schematic representation of coating an object with multilayers using a successive dipping and washing procedure

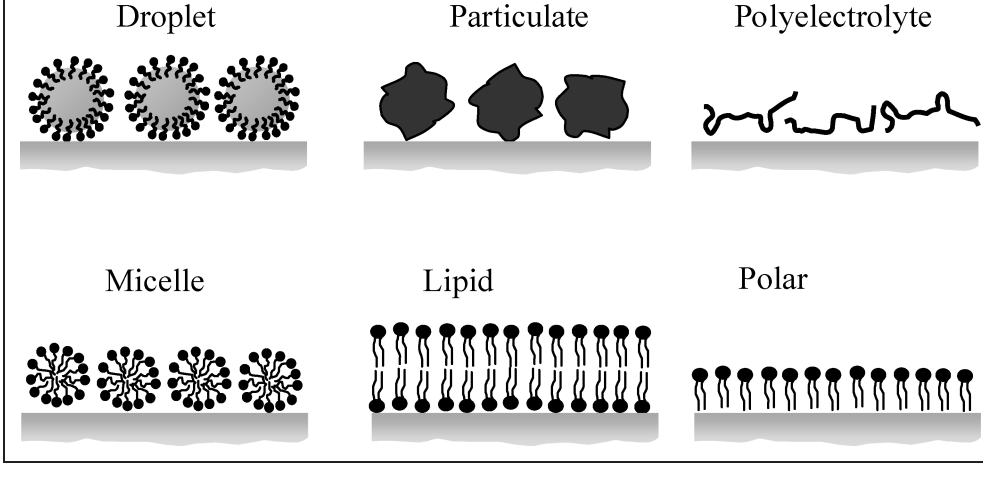

Figure 5—Possible components that could be used to assemble multilayered edible films or coatings

in intestinal fluid. Compared to PLA nanoparticles made with similar amounts of PLA, copolymer nanoparticles are smaller, demonstrating the strong influence of PEG on particle formation (Riley and others 1999). A PEG block seemingly prevents agglomeration of PLA chains, thus reducing the increase in particle size. Using this diblock copolymer makes it possible to create particles in the sub-200 nm range (Tobio and others 2000). PEG also affects the zeta potential of the particles; that is, it has a lower negative surface charge (approximately −6 mV for 30:5 PLA:PEG copolymer, approximately −50 mV for PLA alone) (Riley and others 1999; Tobio and others 2000). This reduction is thought to be due to the capping of carboxy PLA end groups by PEG or by the shifting of the shear plane of the diffuse layer to a greater distance from the nanoparticle. Changes in zeta potential are important because they influence the interaction of particles with other compounds in the food system. The surface charge is of particular importance if particles are to be used as building blocks for more complex structures such as nanolaminates.

One of the main criteria in using nanoparticles as a delivery system for bioactive compounds is that they are nontoxic. Because many early encapsulation procedures did not meet this criterion (Oppenheim 1981), several new methods were developed using less-harsh chemicals and chemicals that were easily removed from the final product. If a method requires using as processing aids organic solvents that are associated with toxicity concerns, then the method may not be suitable for production of nanoparticles that are to be used in foods. When selecting a method to produce nanoparticles, food manufacturers should thus carefully review the solvents required. Ultimately, more development efforts are needed to adapt these methods to strictly use only food-approved processing aids and components. Methods that may fulfill these requirements to produce nanoparticles include salting out, spontaneous emulsification/diffusion, solvent evaporation, polymerization, and nanoprecipitation (Ibrahim and others 1992). Figure 6 illustrates an overview of these methods. In addition, electrospraying has shown to be capable of producing uniform particles of less than 100 nm from polymer and biopolymer solutions.

Salting Out (Ibrahim and others 1992). Salting out involves dissolving a high concentration of a salt and a protective colloid in the aqueous phase, forming a viscous gel. The polymer, which forms the bulk of the particle, and the drug to be encapsulated are dissolved in an organic, water miscible solvent (typically acetone). The 2 solutions are combined with vigorous stirring to form an oil-in-water (O/W) emulsion. Water is added to this emulsion, causing the organic solvent to diffuse into the aqueous phase. The water insoluble polymer will simultaneously aggregate and encapsulate the other compound present in the organic phase, thus forming nanoparticles. Lastly, acetone and salting-out agents are eliminated by cross-flow filtration. While salting out is associated with very high encapsulation efficiencies compared to the other methods, its use is typically limited to the encapsulation of lipophilic compounds.

Nanoprecipitation (Fessi and others 1989). In contrast to the solid nanoparticles that the salting-out method generates, the nanoprecipitation method produces nanocapsules that consist of a central oily core surrounded by a thin polymer wall (Fessi and others 1989; Guterres and others 1995). A polymer and a mixture of phospholipids are dissolved in water-miscible organic solvents such as acetone or ethanol. The compound to be encapsulated or loaded is dissolved in a lipophilic solvent and added to the organic solvent. The organic solution is then added, via stirring, to an aqueous solution containing a surfactant. Addition to the aqueous solution causes the water-miscible solvent to rapidly diffuse into the aqueous phase, which results in the formation of lipophilic nanodroplets that contain the compound to be encapsulated. The water-insoluble polymer now migrates to the O/W interface where it adsorbs to form an interfacial membrane around the lipophilic core. The resultant suspension is then concentrated by evaporating the organic solvent and water under pressure.

Solvent Evaporation (Beck and others 1979). In this method, the polymer and compound to be encapsulated are dissolved into a water-immiscible, volatile organic solvent. This solution is subsequently added to an aqueous solution containing a stabilizing compound and then homogenized to form an emulsion. The formation

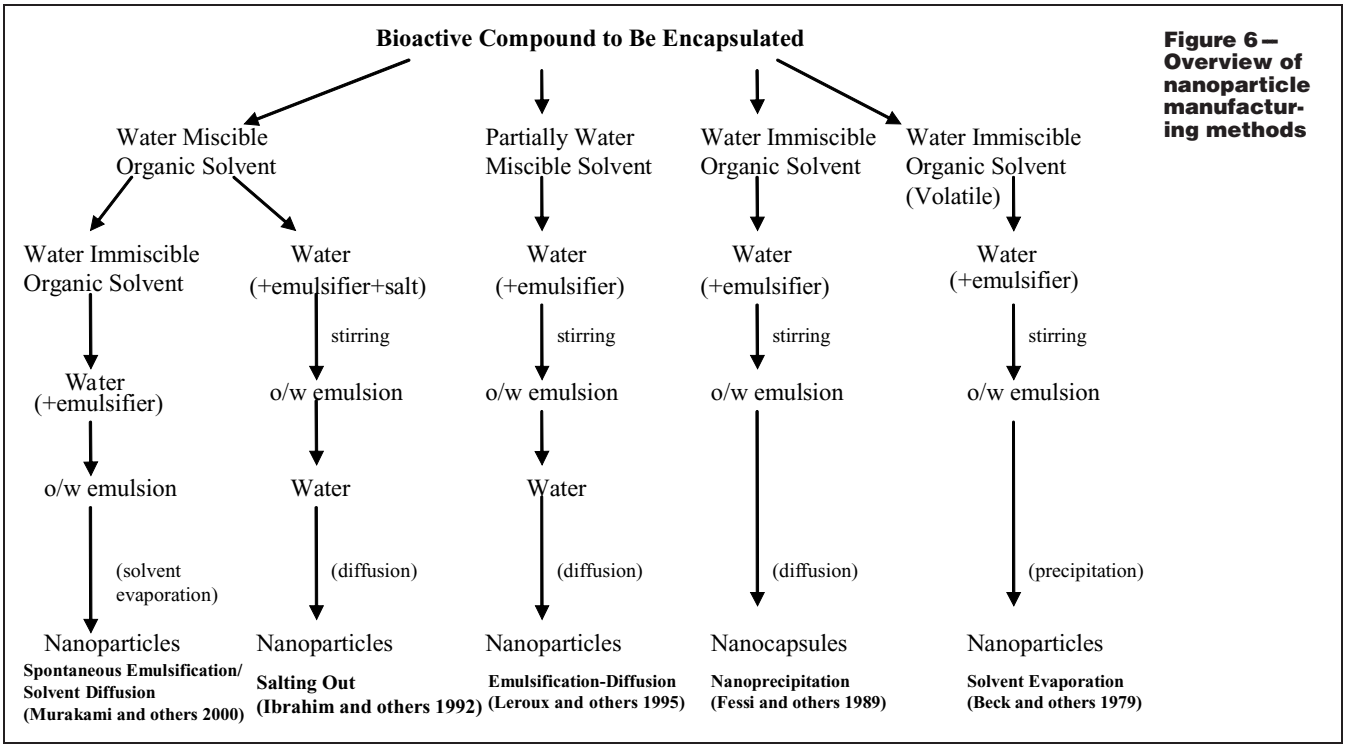

Figure 6—Overview of nanoparticle manufacturing methods

of microspheres is a phase separation process in which the organic solvent diffuses into the aqueous phase from the surface of the droplets in the emulsion. This increases the polymer concentration at the phase boundary and eventually causes the polymer to precipitate, forming the particle (Bodmeier and others 1987a, 1987b). The volatile solvent is evaporated under vacuum and produces encapsulated particles ranging in size from 10 to 250 nm (Beck and others 1979).

Finally, depending on the choice of the base biopolymer used to manufacture the nanoparticles, particle surfaces may be hydrophobic or hydrophilic. Thus, the type of solvent in which the particles are dispersed for their application in food systems may lead to problematic particle aggregation. Aggregation of particles renders them poorly dispersible, and this could negate some advantages of these delivery systems. It is therefore critical to understand particle–particle and particle–solvent interactions to ensure that particles remain dispersed in the solvent as individual particles. Approaches developed in the field of colloid science may be used to predict these interactions and help design systems that are dispersible in the food application.

Nanocomposites

In the late 1980s, a car manufacturer's researchers found that adding 5%-by-weight nano-sized clays increased the mechanical and thermal properties of nylons significantly (McGlashan and Halley 2003). The most widely studied type of polymer-clay nanocomposites, a class of hybrid materials composed of organic polymer matrices and organophilic clay fillers (Kim and others 2003), is montmorillonite (MMT). A hydrated alumina-silicate-layered clay consisting of 2 silica tetrahedral sheets fused to an edge-shared octahedral sheet of aluminum hydroxide, MMT has several advantages. Its high surface area, large aspect ratio (50–1000), and platelet thickness of 10 Å make it suitable for reinforcement purposes (Uyama and others 2003). However, it is not easily homogeneously dispersed in an organic polymer phase due to the hydrophilic nature of the MMT surface (Kim and others 2003). To be compatible with the organic polymer, the organophilicity of MMT must be improved. To increase mechanical and thermal properties, moisture stability, and flame and weather resistances, a small percentage, by weight, of clay in a polymer matrix can be included. Two types of hybrids—intercalation and exfoliation—are ideal nanoscale composites (Figure 7). Intercalation is the state in which extended polymer chains are present between the clay layers, resulting in a multilayered structure with alternating polymer/inorganic layers at a repeated distance of a few nanometers. Exfoliation is a state in which the silicate layers are completely separated and dispersed in a continuous polymer matrix. The structure and properties of the resulting nanocomposites can be altered by controlling subtle polymer-clay interactions (Oya and others 2000).

Polysaccharide/clay nanocomposites

Recently, the preparation of nano-clay containing carbohydrate film has been reported (http://www.pre.wur.nl/UK/Research/Food+Structuring/Microstructured/). Here, carbohydrates are pumped together with clay layers through a high shear cell to produce a film that then contains the exfoliated clay layers. Since these layers are impermeable to water, water can only migrate through the polysaccharide matrix following a torturous path. As a consequence, the nanocomposite carbohydrate film has substantially reduced water-vapor permeability, solving one of the long-standing problems in the production of biopolymer films. Moreover, introduction of the dispersed clay layers into the biopolymer matrix structure has been shown to greatly improve the overall mechanical strength of the film, making the use of these films industrially practicable. For example, Mathew and Dufresne (2002) examined nanocomposites from starch and amorphous poly (beta-hydroxyoctanoate) and from starch and tucinin whiskers. Nanocomposites have also been developed using plant oil-clay hybrid materials (Uyama and others 2003). Park and others (2003) used 2 clays—Cloisite 30B with ammonium cations located in the silicate gallery and one unmodified Cloisite Na^+—to generate thermoplastic starch (TPS)/clay nanocomposites using a melt intercalation method. With 5% by weight inclusion of the clays, strong interactions between TPS and Cloisite Na^+ led to

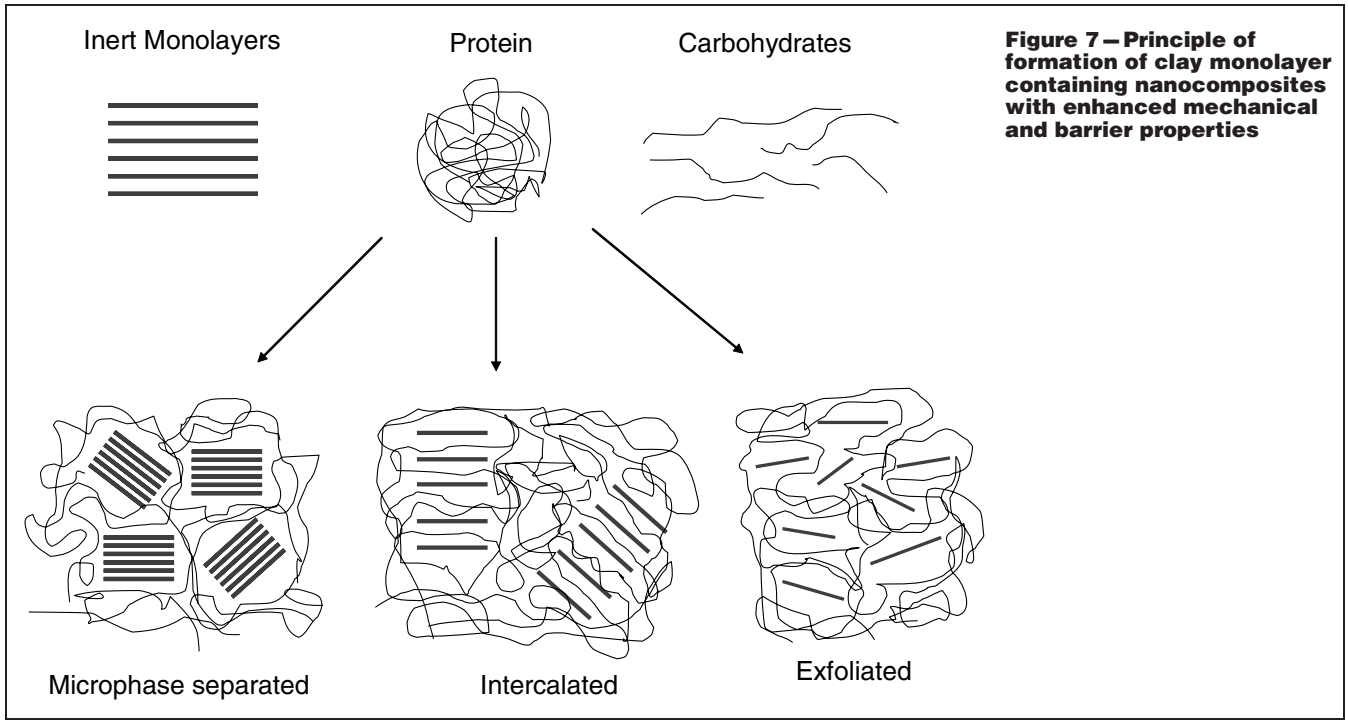

Figure 7—Principle of formation of clay monolayer containing nanocomposites with enhanced mechanical and barrier properties

higher tensile properties and a lower water vapor transmission rate than the pristine TPS.

Chemically derived by deacetylation of chitin, an abundant polysaccharide found in shellfish, chitosan possesses a unique cationic nature relative to other neutral or negatively charged polysaccharides. In an acid environment, the amino group NH_2 in chitosan can be protonated to yield NH_3^+, which yields antifungal or antimicrobial activities since cations can bind to anionic sites on bacterial and fungal cell wall surfaces. Further, chitosan is a nontoxic natural polysaccharide and is compatible with living tissue. These appealing features make chitosan widely applicable in wound healing, production of artificial skin, food preservation, cosmetics, and wastewater treatment (Risbud and others 2000; Juang and Shao 2002). As discussed earlier, chitosan's hydrophilic character and consequently its poor mechanical properties in the presence of water and humidity limit its application. In contrast, chitosan films containing exfoliated hydroxyapatite layers maintain functionality in humid environments, providing good mechanical and barrier properties while having comparable antimicrobial efficacies to solution-cast chitosan films.

Nanofibers

The production of fibers with diameters of less than 100 nm is now feasible with the invention of the electrospinning process. Electrospinning is a manufacturing technology capable of producing thin, solid polymer strands from solution by applying a strong electric field to a spinneret with a small capillary orifice. Generally, electrospun polymer fibers can range in size from 10 to 1000 nm in diameter and may exhibit unusual functionalities with respect to their mechanical, electrical, and thermal properties. Because of the large surface-to-volume ratio, the fibers have shown to be ideal materials to produce tissue templates, medical prostheses, protective clothing, and electronic devices (carbon nanotubes). This processing technology may have some novel applications within the food industry for producing materials with new or improved properties.

The general idea of fiber production from a polymer solution using an electrostatic force was first proposed in patents dating from 1934 to 1944. Polymer filaments are formed from solution between 2 electrodes that are oppositely charged, with 1 electrode being submerged in the polymer solution and the other electrode being connected to a collector. The application of a high-voltage electric field induces a charge on the surface of the liquid inside the capillary tube used to eject the polymer solvent. Mutually charged polymer and solvent molecules subsequently repel each other and are attracted to the oppositely charged electrode. As a consequence, the hemispherical surface of the fluid at the tip of the capillary is distorted to create a Taylor cone, which is "the cone observed in electrospray and hydrodynamic spray processes from which a jet of charged particles emanates above a threshold of voltage" (Wikipedia 2006). If the molecular interaction forces become large enough to overcome the opposing surface tension, a charged polymer fluid jet will be ejected from the tip of the Taylor cone. Upon ejection of the charged solution from the small-diameter spinneret, the solvent rapidly evaporates, forming 10 to 1000 nm diameter solid fibers that are deposited on the collector. Consequently, the mechanical and functional properties of electrospun fine fibers are strongly influenced by the jet path and velocity. The fluid jet is subject to bending instabilities that introduce normal and shear forces, which causes stretching of the polymer jet. This reduces the effective fiber diameter and aligns polymer molecules, thereby improving the mechanical properties of fibers. Because of the tangential force, a rotational element is introduced in the jet velocity vector, which substantially increases the length of the jet path prior to deposition on the grounded electrode. This elongation of the jet path due to bending instabilities is largely responsible for the submicron diameter of electrospun fibers, which is the basis of their remarkable functionalities.

The majority of studies with composite nanofibers has been concerned with the production and reinforcement of carbon nanotubes for use in next-generation microprocessors. However, the majority of filed patents has focused on applications in life sciences. For example, biocompatible nanofibers have been used to produce porous membranes for skin to aid in cleansing, healing, and dressing of wounds; creating tubular fibers for blood vessel and nerve regeneration; 3-dimensional scaffolds for bones and cartilage regeneration; and drug-delivery matrices. In all these cases, the large surface-to-volume ratio of electrospun fibers has been responsible for their superior functional properties. In the case of drug delivery, the dissolution rate of a particular drug increases with corresponding surface area. Hence, the smaller the dimension of the drug delivery vehicle, the better the drug is absorbed by the human body. Because the size is also smaller than that of a typical human cell, these fibers provide ideal templates for cell deposition and proliferation. Beyond biomedical applications, electrospinning has been applied to produce filter media for liquid and gas filtration and protective clothing for the military that is capable of trapping aerosols and large molecular-weight biocidal gases while minimally impeding air flow. Thermal, piezoelectric, and biochemical sensors made from electrospun fibers have demonstrated sensitivities that are 2 or 3 orders of magnitude higher than comparable thin films.

The food industry can use electrospun microfibers in several ways:

- as a building/reinforcement element of composite green (that is, environmentally friendly) food packaging material,
- as building elements of the food matrix for imitation/artificial foods, and
- as nanostructured and microstructured scaffolding for bacterial cultures.

While the number of applications that make use of electrospun fibers is increasing at an exponential rate, the applications for food and agricultural systems are relatively few. This is probably because fibers are not typically composed of biopolymers used in food and agriculture; they are made primarily from synthetic polymers. As progress in the production of nanofibers from food biopolymers is made, the use of biopolymeric nanofibers in the food industry will likely increase.

Nanotubes

Carbon nanotubes have been widely used as a nonfood application of nanotechnology. These structures have been used as low-resistance conductors or catalytic reaction vessels among other uses. It has been shown that certain globular proteins from milk (such as hydrolyzed α-lactalbumin) can be made to self-assemble into similarly structured nanotubes under appropriate environmental conditions (Graveland-Bikker and de Kruif 2005, 2006; Graveland-Bikker and others 2005, 2006). This technique is applicable to other proteins as well and has been explored to assist in the immobilization of enzymes or to build analogues to muscle-fiber structures.

Food Product Innovation

An important area where food nanotechnology is increasingly used is in the design of functional food ingredients such as food flavors (Imafidon and Spanier 1994) and antioxidants. Ultimately,

the goal is to improve the functionality of these ingredients in food systems, which may minimize the concentrations needed. Delivery and controlled release systems for solubilization of nutraceuticals in foods have been previously mentioned (Lawrence and Rees 2000). These new functional ingredients are increasingly integrated into the food matrix development process (Haruyama 2003). Food ingredients such as nanoparticulate lycopene and carotenoids are becoming commercially available. Bioavailability and the ability to disperse these compounds are typically higher than that of their traditionally manufactured counterparts.

Regulations

There are currently no special regulations for the application or utilization of nanotechnology in foods in the United States, and although recommendations for special regulations in the European Union (EU) have been made, laws have yet to be changed. The U.S. Food and Drug Administration (FDA) states that it regulates "products, not technologies," and anticipates that many products of nanotechnology will fall under the jurisdiction of multiple centers within FDA and will therefore be regulated by the Office of Combination Products. FDA regulates on a product-by-product basis and stresses that many products that are currently regulated produce particles in the nano-size range. FDA says that "particle size is not the issue" and stresses that new materials, regardless of the technology used to create them, will be subject to the standard battery of safety tests (http://www.fda.gov/nanotechnology/regulation.html).

In contrast to the FDA view on particle size, a recent report by the Institute of Food Science and Technology (IFST)—a United Kingdom-based independent professional qualifying body for food scientists and technologists—states that size matters and recommends that nanoparticles be treated as new, potentially harmful materials until testing proves their safety. Nonetheless, the European Commission intends to use existing food laws with respect to food products derived through nanotechnology, where applicable, but acknowledges that the technology will likely require modifications to the law. The European Commission plans to use a case-by-case approach to risk assessment.

Recommendations by the Royal Society and the Royal Academy of Engineering, commissioned by the UK government to assess the potential impact of nanotechnology, included a call for identification of the use of nanoparticles in ingredient lists. The UK government agreed that this was necessary for consumers to make informed decisions and that modifications to current labeling requirements would be necessary. The IFST suggested that when nanoparticles are used as food additives, the conventional E-numbering system for labeling be used along with the subscript "n" (IFST 2006). The UK government also agreed to consult with EU partners regarding the IFST report's recommendation that nanoparticle ingredients be subjected to a full safety assessment before use in consumer products.

Conclusion

In addition to the scientific and technical advances needed to continue the application of nanotechnology to foods, regulatory considerations (including safety/toxicology and environmental impact), economics, and consumer acceptance of nanotechnology will ultimately dictate its success in food applications. (A future Scientific Status Summary will address some of these issues in greater detail.) Agricultural producers and food manufacturers could gain a more competitive position through the application of nanotechnology, and in the long term, consumers may benefit from the advances in nanotechnology that contribute to a competitive and innovative domestic agricultural and food system and provide new methods to improve safety and nutritional value of food products. In a recent study by the Helmut Kaiser Consultancy (2004) and the Royal Academy of Engineering, it was estimated that the nanofood market will surge from $2.6 billion today to $20.4 billion in 2010. Currently, over 200 companies are pursuing research in the area of food nanotechnology, examining over 180 potential applications (IFST 2006).

Many of the principles, applications, and techniques that are included in the term "nanotechnology" are the same or fairly similar to those that have already been widely understood and utilized. In particular, there are major areas of overlap between nanotechnology and the more traditional disciplines of colloid, interfacial, and polymer science. However, as this summary shows, one of the defining features of nanotechnology appears to be the emphasis on building structures on the nanoscale rather than on just understanding their properties (which was a major focus of more traditional disciplines). Nanotechnology should probably best be understood as a conceptual and intellectual framework that enables the design of more complex macroscopic structures using nanometer scale building blocks.

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