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Producing micron- and nano- size formulations for functional foods applications

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ABSTRACT:

Background: Nutrient deficiencies affect the health and wellness of large populations around the world. For example, the majority suffer from vitamin, essential fatty acid (such as omega-3), dietary fiber, and other important ingredient deficiencies due to their limited supply in the human food chain. Current trends in the nutraceutics industry to place these substances in higher, more-efficiently dispersed quantities in our food have become critically essential to their business plans. Nutrients in the form of small solids or droplets improve bioavailability. However, there remain numerous barriers to successful implementation of cost effective manufacturing processes. These challenges are addressed in the work presented here with particular focus on stability, bioavailability, and consumer acceptance. The goal is to develop large scale manufacturing systems that implement efficient platform technologies, with their respective operational maps, to produce functional food formulations, with particle sizes of these specially formulated nutraceutical ingredients in the micron-and nano- range.

Objective: Demonstrating that stable micron- and nano-size emulsions, liposomes, and aqueous suspensions of functional food formulations can be produced using both "top down" and "bottom up" methods is our main objective. Addressing the challenges associated with the incorporation of these ingredients into large scale manufacturing systems, mainly mechanical stability and related shelf-life issues, is also a focus. That is, to develop proper processing protocols providing improved quality foods enriched with ingredients that are in limited supply in our food chain; to enhance human health and wellness world-wide.

Methods: The formulations considered here typical of those used for increasing bioavailability of the infused, specially formulated ingredients with anti-cancer, anti-aging, and in-general wellness properties, lowering fat content and enhancing the shelf-life stability. Included are (a)

an oil-in-water (fish oil/omega-3) emulsion, (b) liposome chaperones to vitamin C, and (c) aqueous suspensions (curcumin crystals, lutein/carotenoids, and fiber in soy milk). The production techniques include both "top-down" particle size reduction and "bottom-up" formation of crystals/precipitates via solubility adjustments. Both techniques are based on high shear processing of multiple liquid feeds. Using an impinging jet system, micro-mixing scales less than 100 nm were obtained.

Results: (a) All nano-emulsion types, single, double and larger, either as oil-in-water and water-in-oil, can effectively be produced from various formulations using "top-down" methods. Illustrated here are single, oil-in-water systems; concentrations of 12-14 wt. % fish oil/omega-3 were mixed with water containing food grade surfactants. The high shear processing produced stable, submicron particles; with median particle sizes of 119-163 nm, no particles larger than 1 micron, and the "fish" odor was suppressed. Pertinent discussions related to the other types are also given as suggested path forward approaches for the development of nutrient enriched functional foods. This includes water-in-oil formulations for reduced fat content and the delivery of multiple species via double and triple emulsions, as compared to liposome configurations.

(b) Although liposomes may be used to encapsulate both hydrophobic and hydrophilic substances, we selected liposomal vitamin C as our initial proof-of-concept system since it is absorbed into the body over four times more easily than its non-encapsulated form. After top down processing, the median size was 200 nm, compared to a median size of about 5 microns obtained by traditional self-assembly protocols. (b) Aqueous suspensions of micron- and nano-size formulations were also accomplished. The top down size reduction technique was used for processing soy bean fibers and lutein and the bottom-up method used for curcumin crystals. The fibers initially had a median size of 150 microns and a bi-modal distribution was obtained after processing; 99% of the particles were smaller than 15 microns with median sizes at 10 microns and the larger peak at about 200 nm. The curcumin submicron particles were formed via anti-solvent crystallization; with stable particles in the range of 300-500 nm.

Conclusions: Our study demonstrates that stable micron- and nano-size emulsions, liposomes, and aqueous suspensions can be produced using both "top down" and "bottom up" methods. The formulation properties, in terms of particle size and stability, strongly depend on the processing parameters used in terms of energy input and temperature history. The energy requirements of the "bottom up" methods may be substantially lower than those of "top down" methods. Although some of the processes presented here have been scaled up to commercial levels, more work is needed in terms of fully assessing the bioavailability of the produced formulations and optimizing the processes to minimize cost.

Key words: nano-emulsion, nano-suspension, high-shear processing, crystallization, curcumin, fish oil, liposomal vitamins: C and E, lutein, nutraceuticals, omega-3, soybean fiber.