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Encapsulation of vitamin E in edible emulsions fabricated using a natural surfactant

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ABSTRACT

Vitamin E is a fat-soluble vitamin widely used in pharmaceutical, supplement, food, and cosmetic preparations that must be encapsulated before it can be dispersed into aqueous-based products. The purpose of this study was to develop "all-natural" oil-in-water emulsions that could be used to incorporate vitamin E into functional food and beverage products. These emulsions were stabilized by a natural food-grade surfactant (Q-Naturale®) isolated from the bark of the *Quillaja saponaria* Molina tree. The influence of oil phase composition (vitamin E to medium chain triglyceride (MCT) ratio), aqueous phase composition (glycerol to water ratio), and surfactant type (Q-Naturale® *versus* Tween 80) on the size of the droplets produced by high pressure homogenization was examined. Small droplets could not be formed using only vitamin E acetate as the oil phase because of its very high viscosity, but they could be formed when ≥20% MCT was incorporated into the oil phase prior to homogenization. In the absence of glycerol, Q-Naturale® was capable of forming emulsions containing relatively small droplets ($d < 400$ nm) from oil phases containing relatively high vitamin levels (60–80%). This droplet size could be decreased ($d < 250$ nm) by incorporating 50% glycerol in the aqueous phase prior to homogenization to increase its viscosity and decrease its interfacial tension. Tween 80 was more effective than Q-Naturale® at producing small droplets when the oil phase contained low levels of vitamin E acetate (≤40%), but the opposite was true at higher vitamin levels. These results indicate that a natural surfactant (Q-Naturale®) is effective at forming edible Vitamin E delivery systems that could be used in functional food and beverage applications.

1. Introduction

There is increasing interest within the food and biomedical industries in the creation of delivery systems to encapsulate, protect, and release lipophilic bioactive compounds, such as ω-3 fatty acids, phytosterols, flavonoids, carotenoids, vitamins, and drugs (Flanagan & Singh, 2006; Kesisoglou, Panmai, & Wu, 2007; McClements, 2010; Taylor, Davidson, Bruce, & Weiss, 2005). These delivery systems should be designed so that they can incorporate appreciable amounts of the bioactive component, protect it from physical or chemical degradation during storage, and deliver it to the appropriate site of action after ingestion. They should also be designed so that they are compatible with the delivery vehicle in which they are to be incorporated, *e.g.,* for food and beverage products they should not adversely affect their appearance, stability, texture, or flavor. In addition, for food and beverage applications delivery systems must be fabricated entirely from food-grade ingredients using economic and commercially viable processing operations. Emulsion-based delivery systems are particularly suitable for this type of application, since they can be fabricated entirely from generally recognized as safe (GRAS) food ingredients and processing operations (McClements, Decker, Park, & Weiss, 2009; McClements & Li, 2010).

The term "vitamin E" refers to a group of fat-soluble vitamins that are widely used as functional ingredients in food, pharmaceutical, and cosmetic preparations (Chiu & Yang, 1992). Vitamin E comes in eight different molecular forms that have a common structural feature: a chromanol ring and a phytol side chain (Gonnet, Lethuaut, & Boury, 2010; Kamal-Eldin & Appelqvist, 1996). The different molecular forms of vitamin E are classified as either tocopherols (α, β, γ, and δ) or tocotrienols (α, β, γ, and δ), with α-tocopherol being the most biologically active form (Brigelius-Flohe & Traber, 1999). The major biological function of Vitamin E appears to be as an oil-soluble antioxidant (Institute of Medicine, 2000). Various other health benefits of vitamin E have also been claimed, such as reducing cardiovascular disease, diabetes and cancer

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(Sylvester et al., 2011; Traber, Frei, & Beckman, 2008; Weng-Yew & Brown, 2011). For these reasons, there has been interest in fortifying many processed foods and beverages with Vitamin E (Sagalowicz & Leser, 2010). Some studies suggest that the bioavailability of Vitamin E may be increased when it is delivered in colloidal form rather than bulk form (Feng, Wang, Zhang, Wang, & Liu, 2009). Vitamin E is unstable to oxidation in many food products and may therefore be lost during processing, storage, and utilization (Gawrysiak-Witulska, Siger, & Nogala-Kalucka, 2009; Yoon & Choe, 2009). For this reason, vitamin E acetate (rather than vitamin E) is used in many food and beverage applications since it has a higher oxidative stability. After consumption vitamin E acetate is broken down to vitamin E in the gastrointestinal tract by the action of pancreatic esterases (Brisson et al., 2008). The recommended daily intake (RDI) of Vitamin E is 15 mg/day, although deficiency only usually occurs in the case of individuals with diseases that inhibit fat absorption or in premature infants (Gonnet et al., 2010; Institute of Medicine, 2000).

Vitamin E is a highly lipophilic molecule that cannot be directly dispersed into aqueous solutions (Sagalowicz & Leser, 2010). Instead, it must be incorporated into an appropriate colloidal delivery system prior to dispersion (Gonnet et al., 2010). A number of previous studies have shown that vitamin E can be successfully incorporated into emulsion-based delivery systems, such as microemulsions (Chiu & Yang, 1992; Feng et al., 2009), nanoemulsions (Hatanaka et al., 2010; Li et al., 2011; Shukat, Bourgaux, & Relkin, 2012), emulsions (Chen & Wagner, 2004; Gonnet et al., 2010) and liposomes (Nacka, Cansell, Méléard, & Combe, 2001). One major problem with vitamin E is that it is only partially absorbed at the intestinal site, which reduces its bioavailability. A number of studies have shown that fat-soluble vitamins, including vitamin E, are better absorbed in the presence of surfactants or emulsions, compared to when they are incorporated into bulk oils (Bateman & Ucellini, 1984; Julianto, Yuen, & Noor, 2000; Nacka et al., 2001). Encapsulation of vitamin E has also been reported to improve its physicochemical stability during storage, in addition to its biological activity after consumption. In the current study, we focus on the development of oil-in-water emulsions suitable for incorporating vitamin E in functional food and beverage products designed to improve human health and wellness.

Recently, there has been increasing interest within the food industry in replacing synthetic emulsifiers with natural "label friendly" alternatives (Guclu-Ustundag & Mazza, 2007; Qian & McClements, 2011). Consequently, there has been a focus on identifying and characterizing natural emulsifiers that can be successfully used in emulsion-based food products. In this study, we investigate the emulsifying properties of a recently developed compound extracted from the bark of the *Quillaja saponaria* Molina tree. The major components within quillaja extract have been reported to be saponins (van Setten, ten Hove, Wiertz, Kamerling, & van de Werken, 1998; van Setten, van de Werken, Zomer, & Kersten, 1995), which are high molecular weight glycosides consisting of a sugar moiety attached to a triterpene or a steroid aglycone (Hostettmann & Marston, 1995). The surface activity of saponins arises from the fact that they contain both hydrophilic regions (sugar groups) and hydrophobic regions (such as quillaic and gypsogenic acids) on the same molecule (Mitra & Dungan, 1997; Sidhu & Oakenfull, 1986). Saponins have previously been shown to form surfactant micelles in aqueous solutions and to stabilize oil-in-water emulsions (Mitra & Dungan, 1997; Waller & Yamasaki, 1996). A food ingredient based on a quillaja saponin extract has recently been marketed by the National Starch Company (Bridgewater, NJ) under the trade name Q-Naturale®.

We investigate a number of factors expected to influence the size of the oil droplets formed in oil-in-water emulsions produced by high pressure homogenization: oil phase composition (Vitamin E to medium chain triglyceride (MCT) ratio); aqueous phase composition (glycerol to water ratio); and, surfactant type (Q-Naturale® *versus* Tween 80®). MCT was used as an example of a food-grade oil that is usually obtained by modification (hydrolysis/esterification) and fraction of natural oils, such as coconut or palm kernel oils. However, we have found that other food-grade oils can also be used in a similar way (such as corn oil). Our aim was to identify optimum conditions for fabricating all-natural emulsion-based delivery systems for encapsulating vitamin E. These delivery systems may be suitable for application in a variety of industrial applications, including food, beverage, pharmaceutical, cosmetic, and healthcare products.

2. Materials and methods

2.1. Materials

The non-ionic surfactant polyoxyethylene (20) sorbitan monooleate (Tween 80) was purchased from Sigma–Aldrich Co. (St. Louis, MO). Quillaja saponin (Q-Naturale® 200) was provided by National Starch LLC (Bridgewater, NJ). The molecular weights of Tween 80 (Sigma–Aldrich, St. Louis, MO) and quillaja saponin (Mitra & Dungan, 1997) have been reported to be 1310 and 1650 g mol^{-1}, respectively. Medium chain triglyceride (MCT) oil (Miglyol 812) was purchased from Coletica (Northport, NY). Vitamin E acetate (Fig. 1) was obtained from BASF (Florham Park, NJ). Glycerol was purchased from Acros Organics Co. (Pittsburgh, PA) and lysolecithin was provided by Compass Food Co. (Charlotte, NC).

2.2. Emulsion preparation

Oil-in-water emulsions were prepared by homogenizing 10 wt% lipid phase (MCT) with 90 wt% aqueous phase. The aqueous phase consisted of surfactant (1 wt% Tween 80 or Q-Naturale®) and buffer solution (10 mM sodium phosphate buffer, pH 7.0). A coarse emulsion premix was prepared by blending the lipid and aqueous phases together using a high-speed mixer (Bamix, Biospec Products, Bartlesville, OK) for 2 min at room temperature. Fine emulsions were formed by passing the coarse emulsions through an air-driven microfluidizer (Microfluidics, Newton, MA, USA). The coarse emulsions were passed through the homogenization for 4 passes at 9000 psi. The commercial Q-Naturale® ingredient contained 14% of surfactant (active ingredient) dispersed within an aqueous solution, and therefore we reported its concentration based on the amount of active surfactant present (rather than the amount of overall ingredient). In future studies, it would be useful to carry out a detailed chemical and structural analysis of this commercial ingredient to better understand how specific components influence its emulsifying properties.

2.3. Particle characterization

The particle size distributions of the samples were measured using a static light scattering instrument (Mastersizer 2000; Malvern Instruments, Malvern, UK) after samples had been diluted with buffer solution.

2.4. Shear viscosity measurements

The shear viscosity of samples was measured using a dynamic shear rheometer (Kinexus Rotational Rheometer, Malvern Instruments, Malvern, U.K). A cup and bob geometry consisting of a rotating inner cylinder (diameter 25 mm) and a static outer