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Preparation of microencapsulated xanthophyll for improving solubility and stability by nanoencapsulation

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ABSTRACT

A microencapsulated xanthophyll preparation method, using an ultrasonic cell grinder synchronizing emulsification inclusion procedure, was developed for improving the stability and water solubility of xanthophyll. The microencapsulated xanthophyll was analyzed, tested and characterized by methods, including high performance liquid chromatography, Fourier transform infrared spectrum, high resolution scanning electron microscopy, thermo-gravimetric analysis, and X-ray powder diffraction. The half-life (t ½) of the microencapsulated xanthophyll against light, heat and oxygen was 7.1 weeks, 5.1 h and 9.2 weeks, respectively. Compared with non-encapsulated xanthophyll, the stability of the microencapsulated xanthophyll against light, heat and oxygen was improved by 5.6 times, 1.9 times and 7.7 times, respectively. The results also showed that the fat soluble xanthophyll was successfully converted into microencapsulated xanthophyll with good water solubility (over 0.125 g/g). The investigation can be of a great interest for food, drink, pharmaceutical, cosmetic and related scientists considering the health benefit effect of xanthophyll and the unsuccessful attempts hitherto to render it completely water-soluble for a full use.

1. Introduction

Xanthophyll is a non-provitamin A carotenoid and yellowish pigment. Its chemical and biochemical activities are related to the unique structure extended system of conjugated double bonds. The high resolution scanning electron microscope microstructure-images of xanthophyll are presented in Fig. 1A and B, and chemical structure of xanthophyll is presented in Fig. 1C. Xanthophyll is fat-soluble pigment found profusely in marigold, spinach, cabbage, broccoli, corn, oranges, peaches, mangoes, tangerines and egg yolk (Khachik et al., 1992; Bhosale et al., 2004; Aman et al., 2005). This bright organic-coloured phytochemicals have been found to have many health beneficial effects. In fact, it has been shown from many studies how this important xanthophyll may help improve visual function (Johnson et al., 2000; Bone et al., 2003), reduce the risk of atherosclerosis (Kritchevsky et al., 2000; Berendschot et al., 2000), prevent cardiovascular hardening caused by ageing, coronary heart disease and cancer (Michaud et al., 2000; Slattery et al., 2000), and may be important for skin health resulting in reduction of UV induced damages (Dwyer et al., 2001). Xanthophyll is also a potent antioxidant (Zhang et al., 1991) and is found to enhance immune function, suppress mammary tumor growth and enhance lymphocyte proliferation (Chew et al., 1996; Hadden et al., 1999).

Xanthophyll is, however, unstable against heat, light and oxygen, fat-soluble rather than water-soluble and not synthesized in body. The applications of xanthophyll are being limited. Therefore, the improvement of the water solubility and stability of xanthophyll is necessary. Recently, microencapsulation technology is widely used to prepare water-soluble and stable products in food and pharmaceutical science (Desai and Jin Park, 2005; Zuidam and Shimoni, 2010; Munin and Edwards-Lévy, 2011; Agnihotri et al., 2012). Microencapsulation is a technique whereby liquid droplets or solid particles are packed into continuous individual shells. The shells, or "walls" as they are called, are designed to protect the encapsulated material from factors that may cause its degradation. Important applications of microencapsulation in the food industry involve materials such as volatile compounds, essential oils, oleoresins and unstable compounds. Encapsulation of these sensitive materials makes it possible to incorporate them in dry form so that they are protected by the walls against oxidation, chemical reaction, and light. In the case of unstable compounds microencapsulation, the full value properties of the products are thus preserved, and high quality and commercial value can be ensured (Rosenberg et al., 1990). Numerous techniques for various

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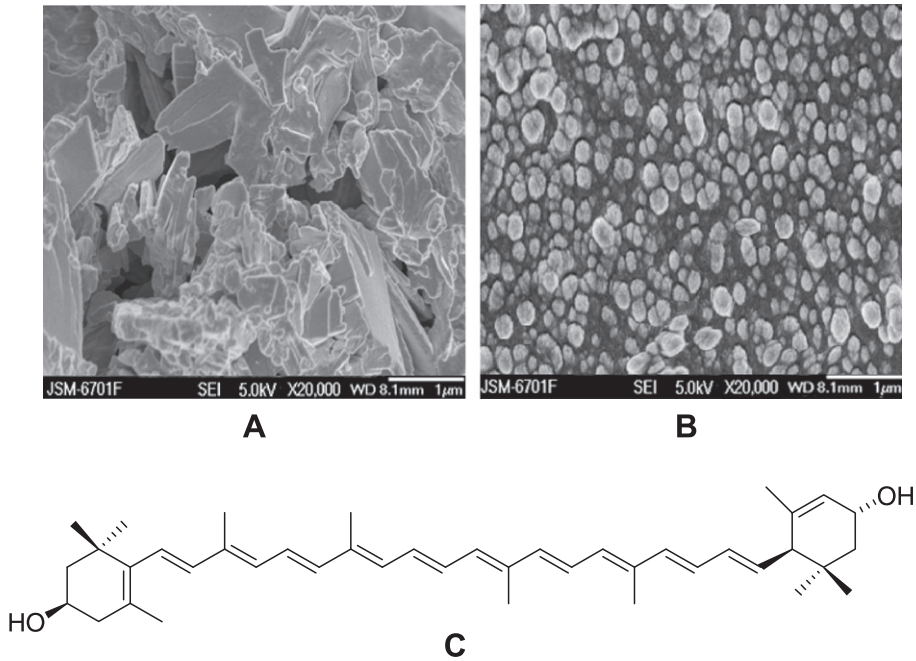

Fig. 1. High resolution scanning electron microscopy image of xanthophyll (A), microencapsulated xanthophyll (B) and chemical structure of xanthophyll (C).

materials have been developed (Dziezak, 1988). Techniques for microencapsulation of food ingredients and the potential use of the technology in the food industry have been discussed in the literatures (Todd, 1970; Chen et al., 1988) and others. At present there are few studies using the polyvinylpyrrolidone and sodium alginate to prepare the water-soluble and stable microencapsulated xanthophyll.

The aim of this work was to develop a method for preparing the water-soluble and stable microencapsulated xanthophyll via an ultrasonic cell grinder synchronizing emulsification–inclusion procedures. Four main major procedures were involved, including (1) fusion and homogeneous process, (2) ultrasonic cell grinder emulsification and inclusion-forming process, (3) drying and molding process, and (4) determination and characterization process. Ultrasonic cell grinder was applied in the emulsification–inclusion processes for assisting the formation of the microencapsulated xanthophyll. Through the nanoencapsulation technology, a completely water-soluble and stable xanthophyll (against heat, light and oxygen) was successfully prepared and analyzed by high performance liquid chromatography (HPLC), Fourier transform infrared spectrum analysis (FT-IR), high resolution scanning electron microscopy (HRSEM), thermo-gravimetric analysis (TGA) and X-ray powder diffraction analysis (XRD).

2. Materials and methods

2.1. Chemicals and apparatuses

The xanthophyll was extracted from the dried marigold flowers by the supercritical fluid extraction and saponification procedures in our laboratory (the details of extraction and saponification conditions of xanthophyll see "Section 2.2"). Polyvinylpyrrolidone (PVP) and Sodium alginate (SA) were purchased from Shanghai Chemical Corporation (Shanghai, China). The water used was double-distilled. Main apparatuses were used in the experiments, including ultrasonic cell grinder (UCG) (Xinzhi Corp., Ningbo, China), high performance liquid chromatography system (Waters Corp., Massachusetts, USA), Fourier transformation infrared spectrometer (Nicolet Corp., USA), high resolution scanning microscopy (Electron optics Corp. Japan), thermo-gravimetric analysis (Netzsch Corp. Germany), and X-ray powder diffraction (Rigaku Corp. Japan).

2.2. Supercritical fluid extraction and saponification conditions of xanthophyll

A 5 l pilot scale supercritical fluid extractor (Model HA121-50-01) with maximum working pressures of 100 MPa was used for the supercritical extraction of xanthophyll esters. The marigold meal pellets were ground to a particle size of 0.5 mm and dried in a rotary drier. The supercritical fluid used in the extractor was CO_2 without co-solvent, and the rate of flow was 16.0 ml/min. 1 kg of the powdered marigold meal was loaded into a cylindrical basket and both the ends were secured with fine steel meshes and clamped tightly. The basket was then placed inside the extractor and closed. The pressure used in the extractor was 45 MPa and the temperature was 47 °C. The pressure developed in the first separator was 12 MPa and the temperature was 51 °C. The pressure and temperature in the second separator were 5.5 MPa and 51 °C respectively. The extraction time was 150 min. The first fraction (120 g) collected in the first separator is called the total extract and it contained xanthophyll ester. For saponification 120 g of the xanthophyll ester resin was mixed with about 1000–1500 ml of isopropanol. The mixture was continuously agitated and kept at a temperature of about 55 °C until the solution became homogenous. Then 50 ml of an aqueous solution equivalent to 15% potassium hydroxide was added slowly to the reaction mixture over a period of 35–65 min. The reaction is carried over for a period of 8–8.5 h to ensure complete saponification. An aliquot of 1 ml was drawn from the reaction mixture every 1 h and the sample was analyzed by HPLC to determine the completion of saponification which is indicated by the complete disappearance of the xanthophyll ester peaks. After saponification the reaction mixture is cooled to about 50 °C and neutralized with a 20% aqueous acetic acid. Then distilled water was added to the reaction mixture and the temperature was increased to about 65 °C and this mixture is