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Synthesis and characterization of nano-encapsulated catechin by molecular inclusion with beta-cyclodextrin

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ABSTRACT

Nano-encapsulation of catechin (CAT) with beta-cyclodextrin (BCD) at 1:1 ratio resulted in the production of nano-encapsulated powder (BCD + CAT) by molecular inclusion method. The interactions between CAT and BCD were analyzed by differential scanning calorimeter (DSC), Raman laser spectroscopy (RLS), scanning electron microscopy (SEM) and X-ray diffractometer (XRD). The results obtained strongly confirmed supra molecular complex formation. DSC thermograph obtained for nano-encapsulated powder resulted in the appearance of new endothermic melting peak at 250 °C with complete disappearance of peaks at 100, 150 and 200 °C present in catechin and beta-cyclodextrin indicating the inclusion complex formation which was also confirmed by spectral characterization (RLS). Particles ranging from 67 to 470 nm were obtained by SEM micrographs. Structural characterization by XRD at 2θ angle from 0° to 80° for nano-encapsulated powder indicated change of state from crystalline to amorphous phase with average particle size of 518.78 nm.

1. Introduction

The concept of nanotechnology in areas of food and agricultural processing is gaining momentum, as they have great potential in functional foods, delivery systems for therapeutics and for bioactive compounds targeting. Nano-encapsulation is a technique of packaging solids, liquids, or gaseous substances as sealed capsules that can release their contents under specific conditions at controlled rates (Lopez-Rubio et al., 2006; Quintanilla-Carvajal et al., 2010). Heat, temperature and pH sensitive core material can be conveniently incorporated into the food systems after nano-encapsulation. The aim of nano-encapsulation is entrapment of the core and subsequent controlled release of the core under the influence of a specific stimulus at a specified stage inside the human system (Jimenez et al., 2004).

Antioxidants serve to scavenge free radicals and render them harmless. Reducing exposure to free radical initiators and increasing dietary antioxidant intake are advisable strategies for maintaining optimal redox status in human system. With restrictions on use of synthetic antioxidants such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) in foods due to their undesirable effects on human health (Jayaprakasha et al., 2001), a great interest in the investigation of active components especially polyphenols, from natural sources (fruits, vegetables, cereals, herbs) has greatly increased in recent years.

Grape seed extract has a high content of oligomeric proanthocyanidins (OPCs), powerful antioxidants, which are made up of catechin monomers (Prieur et al., 1994; Bozan et al., 2008). Catechin from grape seed has demonstrated high potential to protect against a myriad of diseases such as anti-hyperglycemic effects (Pinent et al., 2004), anti-inflammatory effects (Terra et al., 2007), enhancement of postprandial lipemia (Del Bas et al., 2005), improvement of insulin sensitivity and prevention of hypertriglyceridemia, cardiovascular disease (Al-Awwadi et al., 2005), prevention of *in vitro* LDL oxidation (Meyer et al., 1997), modulation of the expression of antioxidant enzyme systems (Puiggròs et al., 2005). The effectiveness of these polyphenolic antioxidants depends on preserving the bioactivity, stability and bioavailability of these compounds which could be answered by the process of encapsulation (Fang and Bhandari, 2010). A recent review by Fang and Bhandari (2010) on encapsulation of polyphenols clearly explains different techniques of encapsulation of polyphenols and its potential benefits. Hence grape seed which is considered as a waste product after grape processing can in turn potentially be converted into an economically high value product by extracting the catechin and forming nano-encapsulated powders.

Cyclodextrins (CD) are cyclic oligomers of α-D-glucopyranose produced from starch degradation by cyclodextrin glucanotransferase (CGTase) enzyme resulting in intramolecular transglycosylation reaction (Szejtli, 1998; Astray et al., 2009). Supra molecular

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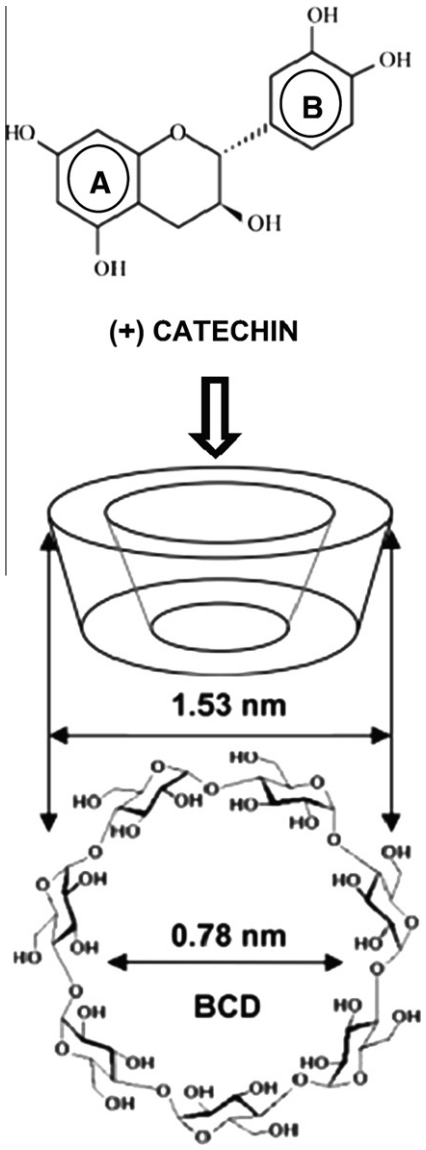

Fig. 1. Molecular inclusion of catechin with beta-cyclodextrin.

self assembly of bioactive compounds can be achieved by cyclodextrin due to its unique structure. It has a truncated cone structure like a torus with a hydrophilic outer surface and a lipophilic inner cavity (Dalmora and Oliveira, 1999). This supramolecular assembly of cyclodextrins aids in the protection of bioactive ingredients against oxidation, light-induced reactions, sublimation, decomposition and loss of volatility. It is also used in the elimination or reduction of undesired taste, odors, microbial contaminations, hygroscopicity and other undesired components. As a result of molecular complexation, cyclodextrins are widely used in industrial and analytical applications (Szente and Szejtli, 2004).

Nano-encapsulation of antioxidants is a promising prospective for delivering target compounds to human systems, to help in preventing the onset of certain human diseases. Nano-encapsulated ingredients can directly be incorporated into solid foods, delivered as liquid in drinks and consumed in our daily diet. Hence, it has numerous benefits and this new encapsulation technology with unique properties will increase the stability and bioactivity of functional ingredients in food, pharmaceutical and biomedical sectors.

Thus in this study, catechin a basic monomer of oligomeric proanthocyanidin, which is a powerful antioxidant present in grape seed was nano-encapsulated using molecular inclusion with beta-cyclodextrin.

2. Materials and methods

Catechin and beta-cyclodextrin obtained from Sigma Aldrich chemicals Co., (St. Louis, MO, USA) were used for nano-encapsulation. Ratio (1:1) of guest molecule (catechin) with host molecule (beta-cyclodextrin) was used for molecular inclusion (Lira et al., 2009; Jullian et al., 2007; Manolikar and Sawant, 2003). In a 50 ml glass beaker, 40 mg of beta-cyclodextrin was dissolved into 10 ml double distilled water at 50 ± 1 °C. This forms the aqueous phase. Catechin (10 mg) was dissolved in 10 ml of ethanol in a centrifuge tube and was sonicated for 5 min for complete mixing of the compound forming the organic phase. The ratio of catechin (MW = 290.26 g/mol) and beta-cyclodextrin ((MW = 1134.99 g/mol)) used was calculated based on their molecular weight. A magnetic stirrer was introduced into the beaker containing the aqueous solution of β-CD to aid complete mixing. The ethanolic solution of catechin was added drop by drop with 5 ml syringe (PrecisionGlide ® Needle, 25 G 1½) into the beaker containing β-CD with magnetic stirring at 200 rpm. After complete addition of the catechin solution, the complex solution was allowed to cool to 20 °C slowly for 4 h. This complex mixture was kept under constant stirring at 50 rpm for 24 h to aid the molecular inclusion of catechin into the β-CD structure. After completion of 24 h, the complex solution was frozen by liquid nitrogen and freeze dried for 24 h to obtain the nano-encapsulated powder. The molecular inclusion complex formation of antioxidant (catechin) with beta-cyclodextrin is given in Fig. 1.

2.1. Thermal characterization by differential scanning calorimeter (DSC)

Thermal behavior of the nano-encapsulated powder was studied using a differential scanning calorimeter (DSC Q-100, TA Instruments, Texas, USA). Indium, platinum and zinc were used for calibration. Accurately weighed samples of 2–3 mg were placed in small aluminum pans. The sample pans were hermetically sealed. The samples were made to cover the entire bottom surface of the pan to ensure good thermal contact. Care was taken to prevent over filling of the pan. Nitrogen was used as the purge gas as it is inert in nature. The rate of flow of nitrogen was 25 ml/min. Temperature was set from 30 to 400 °C. Reproducibility was checked by running

Table 1
Thermal properties of beta-cyclodextrin, catechin and nano-encapsulated powder obtained from DSC.

Beta-cyclodextrin	T_g	T_m	Catechin	T_g	T_m	Nano-encapsulated powder	T_g	T_m
BCD1	187.890	196.160	CAT1	91.140	101.690	BCD + CAT1	242.690	250.800
BCD2	189.330	195.500	CAT2	91.760	101.590	BCD + CAT2	242.680	247.540
BCD3	189.890	195.330	CAT3	92.500	101.820	BCD + CAT3	246.610	252.940
Avg (mean)	189.037	195.663		91.800	101.700		243.993	250.427
Std Dev	1.032	0.438		0.681	0.115		2.266	2.719