



Stability of active components of cardamom oleoresin in co-crystallized sugar cube during storage

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ABSTRACT

Cardamom oleoresin was co-crystallized to formulate flavoured sugar cubes for table top use in tea. The flavoured sugar cubes were packed in two-layer composite and three-layer metalized laminate packaging materials and evaluated for 5 months at relative humidities of 33%, 63% and 93% and temperatures of 5 °C, 25 °C and 45 °C, respectively. The major active components of cardamom oleoresin viz. 1,8-cineole and α -terpinyl acetate in the co-crystallized sugar cubes was quantified by gas chromatography throughout the storage period. The major active components of cardamom in the oleoresin, freshly prepared flavoured sugar cubes, and in sugar cubes stored under extreme condition of storage at 45 °C under all the relative humidities were identified by GCMS. The rate of degradation of 1,8-cineole was higher than α -terpinyl acetate as seen from the kinetic study and activation energy in both the packaging materials under all conditions of storage.

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1. Introduction

Cardamom is indigenous to the evergreen rainforests of Western Ghats of southern India from where it spread to other tropical countries such as Sri Lanka, Tanzania and a few Central American countries. It is well known for its contribution to pleasant and characteristic aroma and flavour. These are broadly grouped into two categories as small cardamom which is popularly known as 'Queen of Spices' (*Elettaria cardamomum maton*) or the true cardamom, and large cardamom (*Aframomum* and *Amomum* species) (Peter, 2003). It is widely used as spice and medical herb, and is also incorporated in foods by either adding ground seeds to coffee and tea since ancient times. The pleasant aroma profile of cardamom oleoresin is attributed to the volatile essential oils that have marked antispasmodic, analgesic and anti-inflammatory activity (Nirmala, 2000) as well as antifungal effects (Al-Zuhair et al., 1996; Utta-Ur et al., 2000).

Cardamom is used as extracts from hydrodistillation or solvent extraction in the form of essential oil or oleoresin. The functionally important constituent of cardamom is the essential oil which has a penetrating, irritating, cineolic, camphoraceous, warm, sweet, spicy, aromatic, pleasing, and citrus like odour due to the volatile constituents present therein. Cardamom essential oil is used in pickles, chewing gums, alcoholic beverages, pharmaceutical syrups as well as in the perfume industry (Farrell, 1985). Oleoresin is a

concentrated form of the spice containing the volatile essential oils as well as non-volatiles such as fixed oils, antioxidants, and pigments materials. The main constituents in cardamom essential oil have been identified by gas chromatography to be 1,8-cineole, α -terphenol, α -terpinyl acetate, limonene, and sabinene by Guenther (1975), of which 1,8-cineole and α -terpinyl acetate are the major character impact compounds. The variation in flavour characteristics from different sources of cardamom have been attributed to the proportion of the esters such as α -terpinyl acetate, linalyl acetate and 1,8-cineole (Korikanthimath et al., 1997; Lewis et al., 1966; Salzer, 1975). Cineole is reported to contribute to pungency while terpinyl acetate is known for its pleasant aroma (Kari-basappa, 1987).

Terpenoids, more specifically α -terpinyl acetate in cardamom oil could undergo hydrolysis, rearrangement, polymerisation, and oxidative reactions and thereby affect flavour due to its vulnerability to acid, light, oxygen or heat. These changes increase the content of *p*-cymene which is also a terpene but with petroleum-like aroma (Brennand and Heinz, 1970). These changes can be arrested by proper encapsulation of cardamom oleoresin.

Encapsulated cardamom oleoresin prepared by spray drying using a blend of gum acacia/emulsifying starch solution as wall materials is reported by Krishnan et al. (2005). Although encapsulation has become a common technique in the food industry, aspects related to their handling during processing and storage stability of the products in which they are incorporated should be simple and also application focused. Cardamom oleoresin itself has good solubility but the stability of its active components in

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presence of oxygen, light and moisture is rather poor. Oleoresin could be stabilized and result in uniform dispersing of flavour on incorporating it in sugar matrix to prepare co-crystallized cardamom flavoured sugar cubes for use in beverage and other food products. In this context, co-crystallization can be promising for preparing flavoured sugar cubes containing entrapped cardamom oleoresin for use in ready to drink beverage like tea.

Encapsulation by co-crystallization in sucrose matrix is a relatively new and simple method which offers an economical and flexible alternative for handling and preserving various active components used in the food industry (Jackson and Lee, 1991). In this process, the active ingredient is incorporated in supersaturated sucrose syrup to attain simultaneous crystallization of both components as well as entrapment of active ingredient in the sucrose matrix respectively (Hartel, 1993). On co-crystallization, the crystalline structure of sucrose is modified from a perfect to an irregular agglomerated crystal to provide a porous matrix where the active ingredient can be incorporated (Chen et al., 1988).

We have previously reported on solubility and hygroscopicity of co-crystallized sugar cubes containing cardamom oleoresin (Sardar and Singhal, 2013). This work reports on the thermo-oxidative stability of active components of cardamom oleoresin encapsulated within the sugar matrix of the flavoured cubes. The study was carried out using two different packaging materials and storage conditions of different relative humidities and temperatures by determining the activation energy required for oxidation using Arrhenius equation. The kinetic parameter of flavour degradation of cardamom oleoresin under isothermal conditions over a temperature range of 5–45 °C and the relative humidities of 33–93% were analysed according to the first order kinetic equation. The oxidation was followed by periodic analysis of 1,8-cineole and α -terpinyl acetate during storage. Structure identification of the major aroma components of cardamom oleoresin of the initial and terminal storage in the co-crystallized sugar cubes was done by gas chromatography mass spectra (GCMS).

2. Materials and methods

2.1. Materials

Food grade crystal sugar was purchased from Sahakari Bhandar, Reliance Retail, Mumbai, India. Food grade gum acacia powder was purchased from Sigma Aldrich Ltd., Mumbai, India. Cardamom oleoresin was obtained as a gift sample from Plant Lipids Ltd., Kerala, India. Standard 1,8-cineole and α -terpinyl acetate was gifted from Symrise Pvt. Ltd., Mumbai. The internal standard, pentadecane, was purchased from Merck Chemicals, Mumbai, India. GC grade hexane was procured from SD Fine Chemicals, Mumbai, India and analytical grade ethanol from Changshu Yangyuan Chemicals, China. Two different packaging materials viz. two-layer composite (12PET/50PE) and three-layer metalized laminate (12PET/12MET-PET/75PE) obtained as a gift sample from Shako Flexipack Pvt. Ltd., Mumbai, India. The PE in both the packaging materials is a three-layer co-extruded PE which is a combination of (LDPE/LLDPE/HDPE). All other chemicals used in this work were of AR grade and procured from reliable suppliers.

2.2. Preparation of co-crystallized sugar cubes containing cardamom oleoresin

This was adapted from the work by Bhandari and Hartel (2002) and modified as reported and carried out with 150 g batches of sucrose in three steps as detailed in our earlier work (Sardar and Singhal, 2013). In the first step, cardamom oleoresin was emulsified with gum acacia to make a stable flavour emulsion. Gum acacia

solution 40% (w/v) was prepared to which 1.2 g cardamom oleoresin/kg of sucrose was added, subjected to shear homogenisation, and then kept undisturbed to form a stable emulsion. The gum acacia solution was used to prepare sucrose cubes containing 2.5 g gum acacia/100 g of sucrose. In the second step, sucrose syrup of 75°brix was concentrated by heating in a metallic vessel on a heating mantle with continuous stirring until a concentration above 95°brix was attained. When slight sand like turbidity was detected in the syrup, indicating the beginning of crystallization process, emulsified cardamom oleoresin was added rapidly to the syrup (Bhandari and Hartel, 2002). The semi-solid product was then poured into trays and moulded into of small cubes of 3.38 cm³ at 30 ± 2 °C. It was then vacuum dried at 60 °C.

2.3. Retention of 1,8-cineole and α -terpinyl acetate in co-crystallized sugar cubes containing cardamom oleoresin

Storage stability of the co-crystallized cardamom oleoresin sugar cubes was carried out at 5 °C, 25 °C and 45 °C for 5 months and relative humidities of 33%, 63% and 93%. Saturated salt solutions of magnesium chloride, sodium nitrite, and potassium nitrite were used to maintain relative humidities of 33%, 63% and 93%, respectively (Karel et al., 1996).

Initially, we evaluated the encapsulation efficiency of two major active flavour components viz. 1,8-cineole and α -terpinyl acetate in co-crystallized sugar cubes containing cardamom oleoresin. The co-crystallized sugar cubes were then quantified for entrapped 1,8-cineole and α -terpinyl acetate every 15 days by gas chromatography for a period of 5 months under varying conditions of temperature and relative humidity and in both the packaging materials selected in the study (Sardar and Singhal, 2013).

2.4. Analysis of entrapped 1,8-cineole and α -terpinyl acetate within the co-crystallized cardamom oleoresin sugar cubes

Quantification of the two major active components, 1,8-cineole and α -terpinyl acetate of the optimised product was done by gas chromatography (Guenther, 1975 and Marriott et al., 2001) with slight modifications. Co-crystallized sucrose cubes were dissolved (25 g) in distilled water (30 ml) in a beaker. Ethyl alcohol (60 ml) was then added and the solution was transferred to a separating funnel. A measured amount of hexane (15 ml) was added to the solution and shaken well. The non-polar top layer of hexane containing the active flavour components was quantitatively transferred to a 25 ml volumetric flask. The aqueous layer was washed twice with hexane and the organic layer transferred to the same volumetric flask. An internal standard, pentadecane (1 ml), was added in the volumetric flask and made to volume by hexane. The concentration of 1,8-cineole and α -terpinyl acetate in cardamom flavoured sucrose cubes at the beginning and after storage under defined conditions was estimated by gas chromatography (Agilent Technologies 7890A, GC, San Francisco, USA) using a flame ionising detector (FID) and a copper made Amkett CB-5 capillary column of 30 m length, 250 μ internal diameter and 0.25 μ m film thickness. The oven temperature was maintained at 40 °C holding for 1 min, raising the temperature to 103 °C at the rate of 15 °C/min with holding for 3 min, and then again raising the temperature to 115 °C at the rate of 5 °C/min with holding for 6 min, followed by raising to 133 °C at 3 °C/min with holding for 3 min, and then finally raising the temperature to 250 °C at the rate of 18 °C/min with holding for 1 min. The peaks at 103 °C, 115 °C and 133 °C corresponded to 1,8-cineole, α -terpinyl acetate, and the internal standard (pentadecane), respectively. The injection port and detection port (FID) temperature was 270 °C and 280 °C, respectively. The volatile compounds passing across the column were carried by a dry carrier gas (nitrogen) at a flow rate of 30 ml/min

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