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Coprecipitation of turmeric extracts and polyethylene glycol with compressed carbon dioxide

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ABSTRACT

Low bioavailability and poor absorption of turmeric bioactives in human body limit their application. In order to propose products with improved stability we investigate the quality of coprecipitated microparticles of polyethyleneglycol (PEG) and turmeric extracts, which were obtained using dichloromethane (DCM) and dimethylsulfoxide (DMSO) as solvents and compressed carbon dioxide as antisolvent. The effects of pressure, antisolvent flow, concentration of extract and solvent proportion were investigated. The mean size of microparticles varied between 20.52 and 182.74 μm. Particle size distribution indicate irregularities that were confirmed by polydispersive index (PDI) values, and by the images obtained with scanning electron microscopy. The profile of bioactives was analyzed using two approaches, i.e., a qualitative (Thin-Layer Chromatography), which indicated weak presence and a quantitative (Antioxidant Activity), which indicated relevant contribution of these substances present in coprecipitates for the inhibition of β-carotene oxidation, besides evidence the efficiency of PEG as wall material.

Symbols

d_P	Mean particle diameter (μm)
OCY	Overall coprecipitation yield (%)
PDI	Polydipispersity index (–)
Q_{CO2}	Mass flow of carbon dioxide (kg/h)

1. Introduction

The expanding demand for natural bioactive constituents increased the optimization of separation technologies that maximize their usability. Global natural colorants consumption is projected to reach 40,500.00 metric tons by 2020 [1], while the global pharmaceutical excipients market is projected to reach US$ 8.1 billion in 2021 at a compound annual growth rate (CAGR) of 6.1% in the forecast period 2016–2021 [2].

Turmeric (*Curcuma longa* L.) and its commercial extracts are employed in a number of innovative products for food and non-food industries, due to the colorant power with antioxidant effects [3], followed by anticancer [4–6], anti-inflamatory [7] and antimicrobial [8] properties. The major biologically active components in turmeric are curcuminoids, which are yellow phenolic pigments, and volatile oil, an extract composed by turmerones and sesquiterpene alcohols [9–11].

Brazil has favorable conditions for turmeric cultivation and is considered the 14th producer of this crop worldwide [12]. The levels at dry basis of curcuminoid pigments in turmeric cultivated in Brazil ranged from 1.4 to 6.1 g/100 g, while the volatile oil fraction was between 1.0 and 7.6 mL/100 g [13]. Nevertheless, the poor absorption of bioactives from turmeric in human intestine [14], associated to the low water solubility and low bioavailability limit their wide application [15].

Supercritical and compressed fluids-based particle formation techniques have been successfully applied to enhance stability of these ingredients with the employment of biodegradable and biocompatible polymers as carrier agents, such as polyethylene glycol (PEG) [16], which have been widely studied for coprecipitation of extracts with relevant content of bioactives [17–20].

In this context, the goal of this work is to investigate the effects of process conditions on coprecipitation of turmeric extracts in PEG using compressed carbon dioxide as antisolvent with the purpose to provide stable substances for food and non-food applications.

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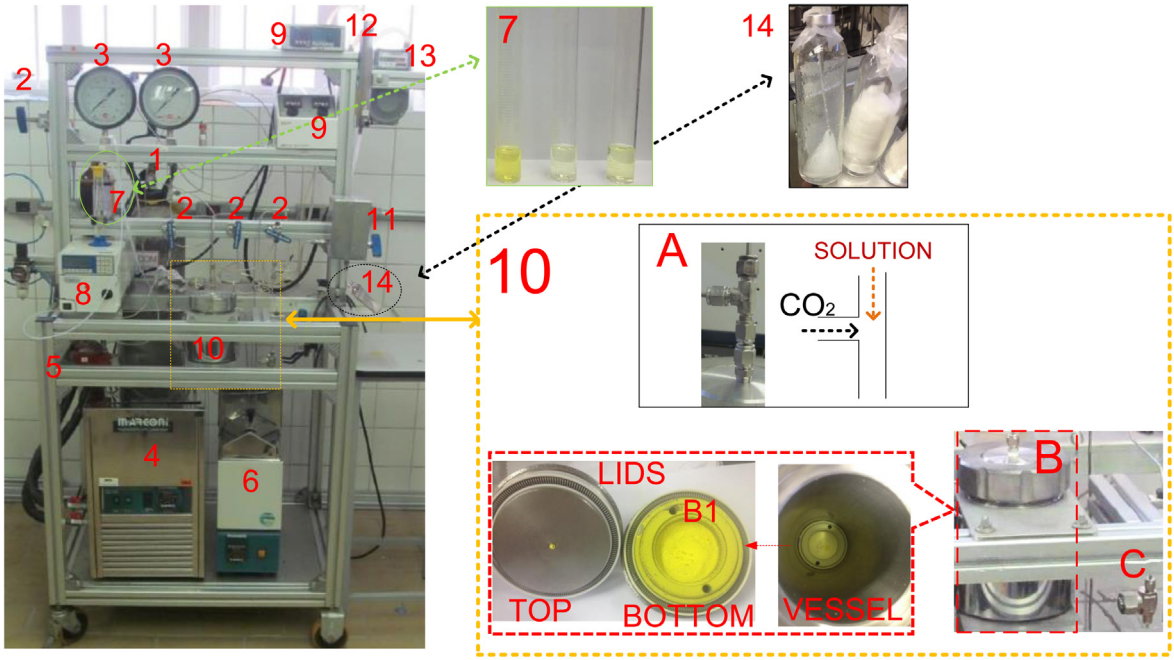

Fig. 1. Schematic diagram of apparatus used in this work.

2. Material and methods

2.1. Materials

Polyethylene glycol (PEG) with a mean molecular weight of 10,000 g/mol (Sigma–Aldrich, Darmstadt, Germany) was used as wall material. Turmeric extracts used in this work were: curcuminoids ethanolic extract (CEE), obtained using pressurized liquid ethanol at 333 K and 10 MPa [21] and turmeric volatile oils in light fraction (LTO) and heavy fraction (HTO), which were obtained using supercritical carbon dioxide at 333 K and 25 MPa [22].

Dichloromethane (DCM), purchased from Synth (99.9% pure, Diadema, Brazil), and dimethylsulfoxide (DMSO), obtained from Dinâmica (99.9% purity, Diadema, Brazil) were selected as solvents because both turmeric extracts and the PEG are soluble.

Carbon dioxide (99% purity) was purchased from White Martins (Campinas, Brazil). For the preparation of the bioactives solutions, PEG was diluted in the solvents until the concentration of 10 mg/mL. Turmeric extracts were diluted in the solution containing PEG until the concentrations of 1 mg/mL (ratio 1/10, w/w) and 5 mg/mL (ratio 5/10, w/w).

Table 1
Summary of operating conditions and experimental results.

Experiment	Substance (s)	Pressure (MPa)	Solvent	Q_{CO2} (kg/h)	OCY (%)	d_P (μm)	PDI (−)
1	PEG	8	DCM	0.5	30.05 ± 9	182.74 ± 1	1.66 ± 0
2	PEG	8	DCM	1	74.75 ± 5	127.51 ± 0	2.37 ± 0
3	PEG	10	DCM	1	74.73 ± 5	26.59 ± 0	3.70 ± 0
4	CEE/PEG (1/10, w/w)	10	DCM	1	70.42 ± 1	21.64 ± 0	3.16 ± 0
5	LTO/PEG (1/10, w/w)	10	DCM	1	59.61 ± 7	20.52 ± 0	3.70 ± 0
6	HTO/PEG (1/10, w/w)	10	DCM	1	55.45 ± 0	25.24 ± 0	3.80 ± 0
7	CEE/PEG (5/10, w/w)	10	DCM	1	52.68 ± 7	30.72 ± 0	3.06 ± 0
8	LTO/PEG (5/10, w/w)	10	DCM	1	50.06 ± 4	23.64 ± 0	2.98 ± 0
9	HTO/PEG (5/10, w/w)	10	DCM	1	51.67 ± 2	23.73 ± 0	2.74 ± 0
10	PEG	10	DCM/DMSO (50/50, v/v)	1	32.05 ± 3	32.98 ± 0	3.17 ± 0
11	PEG	10	DCM/DMSO (90/10, v/v)	1	57.30 ± 4	46.02 ± 1	3.73 ± 0
12	PEG	10	DCM/DMSO (10/90, v/v)	1	5.28 ± 1	*	*
13	PEG	10	DMSO	1	–	–	–

*Unavailability of sample.