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A method for producing superfine black tea powder with enhanced infusion and dispersion property

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ABSTRACT

Tea is rich in healthy components including polyphenols, caffeine, gallic acids, and others. Current technology of tea infusion and extraction leads to more than 40% soluble solids wasted in spent leaf. To increase the bioaccessibility of black tea, we report a method of pulverization treatments including general and superfine grinding to reduce the particle size. In comparison with coarsely ground black tea powders (BTPs), the superfine ground BTP with medium diameter 6.9 μm resulted in significant higher infusion yield of total polyphenols (TPP), caffeine, and water-soluble carbohydrate (WSC). The total water-soluble solids (WSS) of superfine BTP infusion increased markedly by twice due to the accelerated diffusion and enhanced solubility. High correlation between particle size and sedimentation ratio suggested improved dispersion stability of superfine BTP. The optimal dispersion of 0.1% superfine BTP in water was obtained by combination of homogenization and 0.08% CMC-Na formulation with 27.05% centrifugal sedimentation ratio.

1. Introduction

Tea is the most widely consumed beverage in the world after water (McKay & Blumberg, 2002). Apart from being refreshing, tea also confers a number of health benefits including preventing the development of cancer, cardiovascular disease, and other pathologies (Hodgson et al., 2012; McKay & Blumberg, 2002; Pan et al., 2013; Tijburg, Mattern, Folts, Weisberger, & Katan, 1997; Yang & Landau, 2000). The refreshing and health benefits of tea are primarily related to the water soluble components such as polyphenols, caffeine, amino acids (de Mejia, Ramirez-Mares, & Puangpraphant, 2009). Numerous factors are known to affect the release of active components of tea, such as the variety, growing environment, manufacturing conditions, and grade (particle size) of leaf tea particles (Astill, Birch, Dacombe, Humphrey, & Martin, 2001). Among them, the particle size is a key parameter associated with the diffusion of active components from tea leaves.

Superfine grinding has the ability to reduce the particle size of food ingredients to within the range of 1 nm–100 μm (Zhao et al., 2009). Compared with traditional mechanical grinding methods, superfine grinding is reported to increase the extraction and dispersibility (Park, Imm, & Ku, 2001; Zhao, Yang, Gai & Yang, 2009; Zhu, Huang, Peng, Qian, & Zhou, 2010). The underlying mechanism can be integrated from the increase of particle surface area and the breakdown of the cell walls, both leading to improved bioavailability and bioactivity *in vivo* or *in vitro* (Hu, Chen, & Ni, 2012; Tao et al., 2014). It is reported that superfine grinding of green tea markedly increased the extraction of total polysaccharide, leading to improved antioxidant activity against ·OH (Hu et al., 2012). The dispersibility of green tea powder was effectively enhanced by decreasing mean particle diameter to less than 10 μm (Park et al., 2001). Considering that black tea accounts for about 67% of the world's tea production (Chang, 2015), we investigate the effects of grinding on the black tea properties including the morphology, the chemical components, infusion property and the dispersion performance.

2. Materials and methods

2.1. Materials

Raw black tea (Qimen black tea) was purchased from Shenzhen Shenbao Huacheng Company (Shenzhen, China). The moisture content of the original tea sample was 8.0% (w/w). Four stabilizing agents, sodium carboxymethyl cellulose (CMC-Na), sodium alginate, agar, and xanthan gum were purchased from Hefei Siyou Food Industry and Trade (Hefei, China). The standard compounds of caffeine were obtained from Sigma Aldrich (C0750). All other

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reagents (Beijing chemical plant, Beijing, China) were of analysis grade.

2.2. Grinding method

Raw black tea were milled into coarse black tea powder (BTP) particles by a Retsch ZM100 centrifugal grinding mill (Germany) through apertures sized in 1.00, 0.50, 0.25, 0.12 mm at 12,000 rpm. Different particle sizes of coarsely ground BTPs were obtained, denoted as CG1.00, CG0.50, CG0.25, and CG0.12 according to the apertures sizes. CG1.00 was then mixed with zirconium oxide balls (6–10 mm in diameter) in a volume ratio of 1:2 and ground for 8 h to prepare the superfine powder using a vibration ball mill CJM-SY-B (Qinhuangdao Taiji Ring Nano-Products Co. Ltd, Hebei, China). During the ball milling, the tank was cooled to 30 °C with recycled coolant liquid. The BTP samples were air-dried at 45 °C for 48 h and then stored in a refrigerator in polyethylene bags for further experiments and measurements. The moisture contents of the BTPs from coarse to fine were 5.70%, 5.75%, 5.59%, 4.18%, and 6.13% respectively.

2.3. Particle size measurements

The particle size distribution was measured from 0.04 μm to 2000 μm using a LS230 laser diffraction particle size analyzer (Beckman Coulter, Brea, CA, USA) at room temperature with water as solvent.

Particle size distribution is characterized by the median diameter (D_{50}) and by the span factor $[(D_{90} - D_{10})/D_{50}]$, where D_{10}, D_{50}, or D_{90} values represent 10%, 50% or 90% cumulative percentiles of particles (from 0 to 100%) undersize particle size distribution (Mingard et al., 2009). Two measurements were carried out for each sample.

2.4. Cell wall breakage ratios

Generally, a diameter of $10 \sim 20$ μm may be regarded as a typical cell size for plants (Metzler, 2003). When mean particle size $D_{50} > 10$ μm, cell wall breakage ratio Φ was calculated using the following equation (Liu, 2007):

$$\Phi = 1 - (1 - 10/D_{50})^3 \quad (1)$$

When $D_{50} < 10$ μm, Φ = 100%.

2.5. Scanning electron microscope (SEM)

BTPs samples were spread on a conductive adhesive carbon tape, pasted on a sample stub. The morphological characteristics of different sized BTPs were investigated by a Hitachi S3400 model SEM (Hitachi, Japan) at 15 kV.

2.6. BTP infusion preparation and component analysis

To monitor the dissolution of different components during the BTPs infusion, 0.5 g of BTP was put into a glass flask with 75 ml of water (25 °C) and was mixed thoroughly. The flask was placed in a water bath at 25 °C without agitation.

The mixture was filtered through a 0.45 μm films after 5, 10, 15, 30, 45, 60, and 90 min of leaching for the analysis of water-soluble carbohydrate (WSC), total polyphenols (TPP), and caffeine. Meanwhile, the parallel sample solutions were filtered to collect the residue for water-soluble solids (WSS) determination.

WSS were determined by oven drying of the filtered residue at 105 °C to constant weight according to the Chinese National Standard GB/T 8305 (2013).

WSC was determined by the anthrone–sulfuric acid method using glucose as a standard, as described by McDonald and Henderson (1964). A Shimadzu UV–vis 2550 spectrometer (Shimadzu, Kyoto, Japan) was used for the UV–vis spectroscopic analysis. The detector wavelength for water-soluble carbohydrate was 620 nm.

TPP were determined according to the Folin-Ciocalteu method (Singleton & Rossi, 1965) with gallic acid as calibrant. The detector wavelength was set to 765 nm.

The caffeine content of the BTP infusions was determined according to ISO 14502-2 (2005). A Hitachi HPLC system (L-7000, Hitachi, Japan) was used to analyze the solution using binary gradient elution at a flow rate of 1 mL/min. The mobile phase A was 9% (v/v) acetonitrile, 2% (v/v) acetic acid with 20 μg/mL EDTA. The mobile phase B was 80% (v/v) acetonitrile, 2% (v/v) acetic acid with 20 μg/mL EDTA). An Agilent ZORBAX SB-C18 column (4.6×250 mm, 5 μm) was used for separation and the detector wavelength was set to 278 nm.

All the results were expressed as a mass percentage of BTPs on dry basis.

2.7. Dispersion methods and sediment ratios measurement

BTPs were dispersed in deionized water at tea-water ratio of 1:1000 and mixed with agitation for 10 min at room temperature to form 0.1% tea-water mixture. To improve the dispersity of superfine BTP, Four stabilizers were investigated including sodium carboxymethyl cellulose (CMC-Na), sodium alginate, agar, and xanthan gum. The stabilizers were added respectively to the tea-water mixture to reach concentrations of 0.08−0.24% (w/w).

To investigate the effect of homogenization on the dispersion of superfine BTP, an ATS GL-20G-C high-pressure homogenizer (ATS Engineering, Canada) was utilized at 500 bars for the 0.1% tea-water mixture with or without stabilizer.

The mixtures of different treatments were then collected for the measurement of particle sizes and sediment ratios.

The BTPs suspended mixture prepared with above dispersion procedure were allowed to settle by standing 2 h to determine gravitational sediment ratio or centrifuge at 4000 rpm for 10 min to determine the centrifugal sediment ratio. The supernatant was removed and the sediment was dried and weighed to determine the sediment ratio based on the weight of BTPs. The supernatant after centrifugation was also used for particle size measurement.

2.8. Statistical analysis

All experiments were conducted in duplicates. Results were reported as their replicate means ± standard deviation (SD). The differences in mean were calculated using the Duncan's multiple-range tests with 95% confidence limit ($p < 0.05$) by SPSS 10.0 software. The correlation analysis was done by Microsoft Excel 2010.

3. Results and discussion

3.1. Particle size distribution of BTPs and cell wall breakage ratio

Table 1 and Fig. 1 supplementary showed the particle size distributions of ground BTPs. The particle size of the superfine BTP was in the range of 2.4−13.1 μm with a median particle size of 6.9 μm. The particle size distributions of coarsely ground powder fractions of CG1.00, CG0.50, CG0.25, and CG0.12 were much wider, ranging from 9.4 to 1211.0 μm and their median particle sizes were 580.7, 306.5, 242.5, and 154.5 μm, respectively. In addition, the superfine BTP had a significantly narrower particle size distribution (Span = 1.6) than those of coarsely ground BTPs