



# The effect of ultrafine and coarse grinding on the suspending and precipitating properties of black tea powder particles

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## ABSTRACT

Traditional manufacture of instant tea by extracting and drying results in limited raw material utilization ratio and complicated processing procedures. Here, ultrafine black tea powders (10–50  $\mu\text{m}$ ) and coarse black tea powders (100–500  $\mu\text{m}$ ) were prepared to investigate their properties after brewing for up to 30 min in boiling water. The size of a stably suspending particle was 12–15  $\mu\text{m}$ , which was not affected by the grinding method and particle microstructure. Hence, the particle size criterion to produce soluble ground instant tea is proposed to be < 12  $\mu\text{m}$ . Particles larger than 30  $\mu\text{m}$ , comprised of carbohydrates, lignin, and protein, precipitated. The raw material utilization ratio of ultrafine powder (50.12%) was higher than that of coarse powder (35.70%), resulting in a higher amount of soluble carbohydrates and suspended particles. Moreover, ultrafine grinding reduced the precipitation ratio by delivering more insoluble health-benefiting components to the suspending liquid.

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## 1. Introduction

Tea, as a popular non-alcoholic beverage, is consumed by two-thirds of the world's population (Heneberry, 2006). Based on the different processing technologies, tea is generally divided into unfermented green tea, semi-fermented Oolong tea, and fermented black tea (Sinija et al., 2007). Among these, black tea is the most widely consumed beverage, and accounts for approximately 67% of the global tea production (Xiao et al., 2017). Compared with the traditional loose-leaf tea beverage, instant black tea powder is becoming increasingly popular because of its fast infusion, and easy brewing and disposal (Chandini et al., 2013; Kraujalytė et al., 2016; Someswararao and Srivastav, 2012).

Traditional manufacture of instant black tea involves extracting the brew from tea leaves, and spray-, freeze-, or vacuum-drying of the concentrated extracts (Alasalvar et al., 2013; Someswararao and Srivastav, 2012). However, only approximately 25% of solids are extracted in this manner, resulting in a large quantity of insoluble material waste (Perera et al., 2015). Therefore, a new approach to simplify the processing procedure, leading to a more efficient

material utilization and higher product quality, is needed.

Several studies demonstrated that different mechanical grinding scales induce changes in the physicochemical properties of tea that might influence the quality of tea powder (Hu et al., 2012; Shevkani et al., 2014). Ultrafine grinding is an effective technique for reducing the particle size below 100  $\mu\text{m}$  to produce ultrafine particles with unique properties, which has many potential applications for powder products (Rozalli et al., 2015; Sun et al., 2016; Tkacova and Stevulova, 1998; Zhao et al., 2009; Zhu et al., 2012). Previously, Xiao et al. (2017) developed a method to produce ultrafine black tea powder and demonstrated pronounced changes in the physicochemical properties of ultrafine black tea particles (10–50  $\mu\text{m}$ ), compared with coarse tea particles (100–500  $\mu\text{m}$ ). Specifically, particle size reduction for ultrafine particles led to increased cell wall breakage ratio and higher infusion yields of water-soluble components (tea polyphenols, caffeine, and carbohydrates) in water at room temperature (25 °C). In addition, the authors showed that the combination of homogenization and 0.08% sodium carboxymethyl cellulose (CMC-Na) formulation could reduce the centrifugal sedimentation ratio of ultrafine black tea powder. Furthermore, Zhang et al. (2017) reported particle characterization of ultrafine black tea powder and coarse black tea powder by determinations of particle size, specific surface area (SSA), particle pore distribution, and particle surface element of carbon, nitrogen and oxygen. The authors showed that microstructural changes of ultrafine particles affected the diffusion

Abbreviations: SEM, Scanning electron microscopy; SSA, Specific surface area.

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process of macromolecular phytochemicals in boiling water extraction, leading to higher amount of extractable protein and polysaccharide in water extract, which enhanced the DPPH radical scavenging activity. Further, Park et al. (2001) determined the sedimentation volumes of ultrafine green tea powders with different particle sizes by adding 1.5 g of tea powders to 50 mL of distilled water at 70 °C for 60 min after a 15-s homogenization. The authors reported that the dispersibility of ultrafine green tea powder improved with increasing mill speed (5000, 7500, 10,000, 12,500, to 15,000 rpm) and decreasing particle size (61.70, 23.06, 12.71, 6.94, to 5.14 μm). These three studies showed that particle microstructure change by ultrafine grinding would influence the diffusion property of water-soluble molecules and particle size was an important factor that would affect the amount of precipitates. Nevertheless, the experimental conditions reported in the literature (the tea:water ratio, brewing temperature, and the homogenization and settling times) do not match the usual, real-life, brewing conditions, and do not appropriately reflect the properties of the tea product. In addition, it is not clear whether the microstructural differences between ultrafine tea powder and coarse tea powder would affect the particle suspending and precipitating properties of ground instant tea, which is important for evaluating the quality of the product.

Therefore, in the current study, Qimen black tea leaves were pulverized by ultrafine grinding and coarse centrifugal grinding to produce ultrafine tea powders and coarse tea powders, respectively. Furthermore, by comparing the ultrafine tea powder with coarse tea powder under real-life tea-brewing conditions, changes in particle size, particle morphology, precipitate ratio, and the suspension and precipitate composition were investigated to characterize and explain the changes in particle suspending and precipitating properties upon grinding.

## 2. Materials and methods

### 2.1. Raw materials

Qimen black tea was purchased from the Shenbao Huacheng Company (Shenzhen, China) in 2014. Its moisture content was  $5.2 \pm 0.2\%$  (w/w), and the tea was stored in a refrigerator in a polyethylene bag at  $-4^\circ\text{C}$ . Gallic acid, caffeine, glutamic acid, glucose, xylose, galactose, and arabinose standards were from Sigma Aldrich (St. Louis, MO, USA). Acetonitrile and acetic acid, certified HPLC grade, were from Thermo Fisher Scientific (Waltham, MA, USA). All other chemicals used in this study were of analytical grade.

### 2.2. Methods

#### 2.2.1. Preparation of tea powders by ultrafine and coarse grinding

Ultrafine grinding was achieved by mixing 100 g of the raw black tea material and 2800 g of zirconium oxide milling balls (6–10 mm in diameter) for 8 h, using a CJM-SY-B ball mill (Qinhuangdao Taiji Ring Nano Ltd., Hebei, China). The instrument temperature was maintained below 20 °C by a cold-water recycling system. The obtained ultrafine tea powder was henceforth denoted as “UT”.

Coarse grinding was achieved by passing the raw material through a 0.50-mm screen at 12,000 rpm, using a sieve-based ZM200 centrifugal pulverizer (Retsch, Haan, Germany). The coarse tea powder was hence referred to as “CT”.

All obtained tea powders were sealed in plastic bags and kept in a desiccator at room temperature (25 °C) for further experiments.

#### 2.2.2. Separation of the suspending and precipitating parts

To simulate the real-life conditions of instant tea brewing, according to the Chinese national standard GB/T 23776 (2009) for the sensory evaluation of tea, mixtures of 0.4 g of tea powders in 150 mL of boiling water ( $\leq 100^\circ\text{C}$ ) were prepared in seven 200-mL beakers, stirred for 5 s, and kept at room temperature. A plastic tube with an internal diameter of 3 mm, stuck to the 25-mL tick mark of the beaker, may be used to effectively suck out the suspending liquid without taking out the precipitating particles, by siphon action. Therefore, the separations of the suspending and precipitating parts were achieved by siphoning after brewing for 0.5, 1, 3, 5, 10, 20, and 30 min, respectively. The suspending liquids prepared at the different times for UT and CT samples were denoted as UT<sub>S</sub> 0.5–UT<sub>S</sub> 30 and CT<sub>S</sub> 0.5–CT<sub>S</sub> 30, respectively. Meanwhile, the precipitating parts were referred to as UT<sub>P</sub> 0.5–UT<sub>P</sub> 30 and CT<sub>P</sub> 0.5–CT<sub>P</sub> 30, respectively. UT<sub>S</sub> 0.5–UT<sub>S</sub> 30 and CT<sub>S</sub> 0.5–CT<sub>S</sub> 30 preparations were set up six times in parallel. From these, three parallel preparations were used for particle size determination and the other three were used for the morphology, precipitate ratio, and the suspending and precipitating compositional analyses.

#### 2.2.3. Determination of particle size

Determinations of particle size distributions of the tea powders, and the suspended and precipitating particles were carried out in the 0.01–3000 μm range using a Mastersizer 3000 laser diffraction particle analyzer (Malvern Instruments Ltd., Worcestershire, UK) in wet measurement mode, using distilled water as the dispersant, with a mixing speed of 1000 rpm, following the method described by Zhang et al. (2017). Curves of the particle size distribution were obtained from the mean values from triplicate determinations. Particle parameters, D<sub>10</sub>, D<sub>50</sub>, and D<sub>90</sub>, represented the 10th, 50th, and 90th percentiles of the total volume, respectively. D<sub>50</sub> was used to characterize the mean particle diameter. The particle span was defined as [(D<sub>90</sub>–D<sub>10</sub>)/D<sub>50</sub>].

#### 2.2.4. Measurement of SSA and particle true density

Samples were degassed at 105 °C for 4 h, as a pretreatment, and SSA of the tea powder was determined using an ASAP 2020 Plus Physisorption device (Micromeritics Instruments Ltd., Norcross, GA, USA). The curves of isothermal adsorption of nitrogen gas were analyzed and SSA values were calculated according to the Brunauer-Emmett-Teller (BET) adsorption isotherms (Liu et al., 2011).

Particle true density was determined by an ultrapycnometer 1000 (Quantachrome, Boynton Beach, FL, USA) using the helium gas volume displacement method (Kaialy et al., 2012).

#### 2.2.5. Scanning electron microscopy (SEM)

Suspending and precipitating parts were filtered through 0.22-μm filters (Tianjin Branch Billion Lung Experimental Equipment Co., Ltd, China) to produce suspended and precipitating particles. These were collected following oven drying at 60 °C for 48 h. Images of UT, CT, and the suspended and precipitating tea particles were obtained using S3400 scanning electron microscope (Hitachi, Tokyo, Japan) at 15.0 kV (Ji et al., 2016).

#### 2.2.6. Precipitate ratios of UT and CT

The dried precipitating particles were used to calculate the precipitate ratio according to Equation (1) (Xiao et al., 2017)

$$\Phi = M_2/M_1 \quad (1)$$

where  $M_1$  was the dry weight of the tea powder and  $M_2$  was the dry weight of the precipitating tea particles.

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