



## Changes on the rheological properties of pectin-enriched mango nectar by high intensity ultrasound

Bohui Huang<sup>a,b,1</sup>, Kaili Zhao<sup>a,b,1</sup>, Zhuo Zhang<sup>a,b</sup>, Fengxia Liu<sup>a,b,\*</sup>, Hao Hu<sup>a,b</sup>, Siyi Pan<sup>a,b</sup>

<sup>a</sup> College of Food Science and Technology, Huazhong Agricultural University, Wuhan, Hubei, 430070, PR China

<sup>b</sup> Key Laboratory of Environment Correlative Dietology, Ministry of Education, PR China

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### ABSTRACT

Effects of high intensity ultrasound (US, 5–40 min) on pectin-enriched mango nectar were evaluated, with regards to its rheological properties and related factors, including microstructure, particle size, and water-soluble pectin (WSP) changes. Results showed that the rheological properties of mango nectar exhibited a complex change, with an initial increase and subsequent decrease in apparent viscosity, storage modulus  $G'$ , and loss modulus  $G''$ . All these changes were ultrasonic time dependent. Ultrasound treatment caused significant degradation on suspended particles in mango nectar, as evidenced by optical microscopy, and particle-size distribution. The surface mean diameter ( $D_{[3, 2]}$ ) was reduced to 30.7  $\mu\text{m}$  from an original value of 37.6  $\mu\text{m}$  after ultrasound treatment. Moreover, US further caused the molecular degradation of WSP, with Mw being reduced from 3000 to 367 kDa in mango nectar. The interaction among them during processing determined the final rheological properties.

### 1. Introduction

Mango (*Mangifera indica* L.) is an important tropical fruit, and China is the second largest mango producer in the world (Liu, Wang, et al., 2013). Due to high perishability of the fruit, its value added products such as mango pulp (Liu et al., 2013) and mango nectar (Liu et al., 2014) is of high commercial importance. Mango and its products are sensitive to high temperature treatments, which may result in off-flavor formation and bio-active compound degradation (Santhirasegaram, Razali, & Somasundram, 2013). The introduction of new technologies in food industry might improve the industrial operating conditions, resulting in products with better quality.

High intensity ultrasound (US) is an emerging technology in food science and has generally been used as an alternative to conventional food processing. Frequency in the range of 20–40 kHz and applied power intensities  $> 10 \text{ W/cm}^2$  have been widely used (Bi, Hemar, Balaban, & Liao, 2015), including microbial population reduction in peach juice (Rojas, Leite, Cristianini, Alvim, & Augusto, 2016), and cantaloupe melon juice (Fonteles et al., 2012). Santhirasegaram et al. (2013) reported that US treatment (15 and 30 min at 25 °C, 40 kHz frequency, 130 W) exhibited significant reduction in microbial count of Chokanan mango juice. Thus, US may be an alternative to thermal treatment for mango product in continuous form.

Moreover, several results showed that US treatment can be used to improve the physical properties of different juice by increase the viscosity. For example, Bi et al. (2015) reported that the viscosity of diluted avocado puree after US treatment at 375  $\text{W/cm}^2$  for 1 min was 6.0 and 74.4 times higher than the control samples for dilution levels of 1:2 and 1:9. Similar results were also found in tomato juice (Anese, Mirolo, Beraldo, & Lippe, 2013; Vercet, Sanchez, Burgos, Montanes, & Buesa, 2002), with the apparent viscosity being increased by US treatment, which was reported to be attributed to the decrease of particle size (Anese et al., 2013; Vercet et al., 2002). However, the changes on viscosity of mango nectar after US treatment was found with a much more complex behavior in this study, with an initial increase and subsequent decrease trend. These results were different from most of the findings in the previous literature, which may be attributed to the different food matrix used. Mango nectar was a pectin enriched food system (Liu, Wang, et al., 2013), and the changes of such polysaccharides during processing (Christiaens et al., 2016; Zhou, Wang, Liu, Bi, & Liao, 2014) may also significantly affect the viscosity of the system. However, the mechanism of the complex rheological changes in mango nectar after US were still unclear, which need to be further studied.

Therefore, this paper aimed to study the effect of US treatment on the rheological properties (dynamic and steady-state shear properties)

\* Corresponding author. College of Food Science and Technology, Huazhong Agricultural University, Wuhan, Hubei 430070, PR China.

E-mail address: [liufxia@mail.hzau.edu.cn](mailto:liufxia@mail.hzau.edu.cn) (F. Liu).

<sup>1</sup> Bohui Huang and Kaili Zhao are co-first authors.

## Nomenclature

US	ultrasound
WSP	water-soluble pectin
$G'$	storage modulus
$G''$	loss modulus
$D_{[3, 2]}$	surface mean diameter

$D_{[4,3]}$	volume mean diameter
Mw	molecular weight
MWD	molecular weight distribution
PSD	particle size distribution
AIR	alcohol-insoluble residue
DM	degree of methoxylation

of mango nectar, and the changes of related factors, including microstructure, particle-size distribution and water-soluble pectin (WSP) were also investigated. Since the rheological properties plays an important role in process design and optimization (such as pumps and pipelines) and shows high correlation with product stability and sensory quality, this study could help to provide some guides for the mango nectar industry.

## 2. Materials and methods

### 2.1. Chemicals

D-galacturonic acid and 3-phenylphenol were purchased from Sigma–Aldrich (St. Louis, USA). All the other chemicals were of analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China).

### 2.2. Preparation of mango nectar

Mature green mango fruits (*Mangifera indica* L., cv. Kate) were purchased from a local market in Wuhan, China. The fruit samples were stored at room temperature (20–25 °C) for approximately four days until fully ripened. After washing, peeling, and deseeding, the flesh was cut into 1 cm-thick slices. To prevent all enzymatic reactions during processing (Liu et al., 2014), mango slices were immediately blanched for 2 min in a steamer (SZ26B5; Zhejiang Supor Co., Ltd., Zhejiang, China). The slices were pulped with a 2:1 ratio of mango flesh (g) to water (mL) and then homogenized by a colloid mill at 2900 r/min for 2 min to obtain mango nectar with a uniform particle size. The pH of the final juice was  $4.00 \pm 0.03$ , and the total soluble solid was 10.0 °Brix.

### 2.3. US treatment

US treatment of mango nectar was performed using ultrasound processor model JY92-2D (NingBo Scientz Biotechnology Co. Ltd, Ningbo, Zhejiang, China) with a 0.636 cm diameter titanium probe. Forty grams of mango nectar were treated at 20 kHz at 400 W for 0, 5, 10, 15, 20, 25, 30, 35, and 40 min with pulse duration of on-time 5 s and off-time 5 s. Moreover, an ice bath was used to maintain uniform mixture temperature. The sonication probe was maintained at a constant depth of 3 cm for all US treatments. After ultrasound treatment, the samples were stored at 4 °C and analyzed within 12 h. The ultrasonic intensity was 105–110 W/cm<sup>2</sup> in this study (Zhang et al., 2016).

### 2.4. Rheological characteristics

Rheological measurements were conducted using AR2000 rheometer (TA Instruments, New Castle, DE, USA) with its accompanying computer software (Rheology Advantage, TA Instruments, Waters Co., Ltd., New Castle, DE, USA). Samples were placed between parallel plates (40 mm diameter) using a gap size of 1 mm (Liu, Wang, et al., 2013). For steady flow studies, approximately 3 mL of sample was applied for each measurement at 25 °C controlled by circulating water in a thermostatic system. The shear rate ranged exponentially from 1 to 100 s<sup>-1</sup>.

For dynamic rheological studies, stress sweep tests were first performed to ensure that the following measurements were performed within the linear viscoelastic region. Based on these results, the stress amplitude of 1.0 Pa was selected for frequency sweep tests. Storage modulus ( $G'$ ) and loss modulus ( $G''$ ) were obtained from the software over the angular frequency range of 0.1–10 rad/s.

### 2.5. Microscopy analysis

The microstructures of mango nectar were vividly shown by a light microscope Vision-21S-D (Jinan Qiangsheng photoelectric instrument co., Ltd, Jinan, China). A drop of each sample was placed on a glass slide, pressured by the cover glass, and then observed with under a 10 × objective lens and a 10 × eyepiece lens. Microphotographs of different mango nectar were obtained by the accompanying software equipped with the microscope.

### 2.6. Particle size distribution (PSD)

The PSD of mango nectar was measured by Mastersizer APA2000 (Malvern Instruments Ltd., Malvern, UK), according to Zhou, Zhang, Leng, Liao, and Hu (2010). Laser light diffraction was used to measure particles from 0.05 to 2000 μm at room temperature (25 °C). The refractive index was 1.73 and the absorbance was 0.01. The stirring speed was 3000 g, and the samples were added into the stirred tank filled with distilled water until an obscuration of 5%–10%. Six readings were obtained from each sample and the average of the readings was calculated. The PSD, surface mean diameter ( $D_{[3, 2]}$ ), and volume mean diameter ( $D_{[4,3]}$ ) for each sample were recorded for analysis.

### 2.7. Properties of WSP from mango nectar

#### 2.7.1. Isolation of WSP

Alcohol-insoluble residue (AIR) of mango nectar was isolated as described by Zhou et al. (2014). Fifteen grams of mango nectar were homogenized with 60 mL ethanol (950 mL/L) using a mixer and then boiling for 30 min. The mixture was filtered with G<sub>4</sub> Buchner funnel, and the insoluble solids were collected. After washing with the boiling ethanol (950 mL/L) again, the insoluble solids were then homogenized in 60 mL acetone for 10 min followed by drying overnight under vacuum at 40 °C.

AIR was suspended in 50 mL of distilled water and stirred at 40 °C for 15 min. After being centrifuged at 12850 g for 20 min, the supernatant was dialyzed (40 kDa MWCO) against distilled water for 48 h. WSP extract was then freeze dried and stored in a dryer until further analysis.

#### 2.7.2. Uronic acid content

The uronic acid content of WSP was determined based on the method described by Blumenkrantz and Asboe-Hansen (1973). Six mL of 0.0125 mol/L sodium tetraborate (dissolved in sulfuric acid) and 1 mL of D-galacturonic acid solution or WSP extract were added to a glass tube and cooled in an ice bath. The mixture was then boiled for 5 min and cooled immediately, 0.1 mL of *m*-phenylphenol (1.5 g/L, dissolved in 5 g/L sodium hydroxide) to the mixture. After reacting for 15 min in the dark, uronic acid content was quantified by the

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