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## Rheology and composition of citrus fiber

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#### 1. Introduction

Appearance, flavor, texture, and nutrition are known factors that play a role in creating high quality foods (Bourne, 1992). Although there are many definitions, texture is defined by the International Organization for Standardization as properties that encompass all the rheological and structural attributes of a food product that are perceptible by mechanical, tactile, and when appropriate, visual and auditory receptors. Thus, the assessment of texture begins when a consumer opens a food product container, handles it, and prepares it, all the way through consuming the product. Consumer textural preference is based on the attributes at each point in the process starting when the consumer first sees the product through the point when the product is consumed and the food product undergoes many changes in that process. Therefore, producing food products with favorable textural qualities has many dynamic aspects and can be complex (Borwankar, 1992).

Because fibrous plant cell walls typically play an important role in determining the quality characteristics of the foods that they go into, there is further need to understand not only how they function but also why fibrous materials function the way they do (Van Buren, 1979). Important factors determining food texture are composition and rheological properties (Bourne, 1992). Thus,

#### ABSTRACT

While fibrous byproducts are abundant, using them in food products to improve food nutrition and quality without degrading taste or texture can be challenging. Citrus fiber has been shown to have high water holding capacity and apparent viscosity. However, to better incorporate citrus fiber into foods, their rheological properties, and composition, need to be better understood. Pectin was found to be 42% of the composition of the citrus fiber evaluated in this study. The rheological properties of citrus fiber solutions were clearly non Newtonian and the type of model that best fit the citrus fiber varied depending on its particle size. Particle size of citrus fiber also significantly changed the apparent viscosity of their solutions. As citrus fibers hydrate, the fibers swell, which was illustrated by microscopic imaging.

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knowing information related to the rheology, function, and composition of ingredients is important for a food product engineer as well as quality control, process control, and design of process equipment (Saravacos, 1970; Ofoli et al., 1987).

One method used to enhance the functionality of fibrous materials, e.g. food processing byproducts, is to expand their internal surface area, which increases water binding capacity and/or apparent viscosity so they are better suited to improve the nutritional and/or guality of foods (Turbak et al., 1983). Ruan et al. (1996) made a product similar to microfibrillated cellulose, called highly refined cellulose, utilizing agricultural residues and food processing byproducts. Citrus fiber also has high internal surface area, water holding capacity and apparent viscosity (Lundberg, 2005). Its high water holding capacity, ability to increase yields and retain moisture means that citrus fiber has many food applications in baked products, meats, dairy products, sauces and dressings. Moreover, because of the neutral color, taste, and odor, processed citrus fiber make products suitable for many applications where other fibrous products would otherwise not be considered (Garau et al., 2007; Grigelmo-Miguel and Martin-Belloso, 1999). One of the unique challenges of citrus fiber is how to dry it while maintaining functional properties upon rehydration (Braddock, 1999). Although overcoming the challenge of producing citrus fiber in a dried form while maintaining functional properties has been overcome, more research is needed to better understand its functionality. Improving this understanding will not only expand the ability to utilize plant cell walls in more applications, but also it will help create healthier foods, which is an underlying goal of





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this study. There is also a lack of information documenting the rheology of citrus fiber and the effect of its composition on the rheology. Knowing more about the rheology of citrus fiber will enable manufacturers to be better informed in designing as well as manufacturing food products that contain citrus fiber ingredients. The objective of this research is to study the composition and rheology of citrus fibers to gain an understanding of how to better apply the product in food applications.

#### 2. Materials and methods

#### 2.1. Raw materials

The citrus fiber studied in this project was obtained from orange (*Citrus sinensis*) pulp or juice vesicles, which is a byproduct of orange juice production. The production process and source of citrus fiber were consistent with those previously described by Fiberstar, Inc. (River Falls, WI) (Lundberg, 2005). Chemicals used in this study were purchased from Sigma–Aldrich (St. Louis, MO) and used as received.

#### 2.2. Compositional analysis

Protein was measured using the Dumas method as outlined in AOAC International Method 992.15, where the sample was combusted and nitrogen content measured. Protein was calculated from the nitrogen content by a timing factor of 6.25. Total fat was measured following AOAC method 996.06, where lipids were extracted and quantified using capillary column gas chromatography. Moisture was determined by heating the sample in a vacuum oven for 18 h following AOAC method 925.09. Total carbohydrates were found by subtracting fat, protein, ash and moisture from 100%.

Cellulose and hemicellulose were quantified after acid hydrolysis, and the sugars released in the hydrolysate were determined using the HPLC method described in the Laboratory Analytical Procedure NREL LAB method #002 (Sluiter et al., 2006). From the HPLC data, cellulose was determined by counting the weight of glucan measured. Hemicellulose was calculated by adding xylan, glucomannan, 36% of the galactan and 9.5% of the arabinan found in the monosaccharides. These percentages of galactan and arabinan were used because they represent the relative amounts previously found in orange pulp as reported by Ting (1970).

For pectin and galacturonic acid (GA) analysis, the degree of esterification (DE), degree of acetylation (DA), and extractable carbohydrate analysis was conducted according to Yoo et al. (2003). Pectin content in the carbohydrate fraction was found by adding the arabinose, galactose, rhamnose, and galacturonic acid found from the monosaccharide analysis using the percentages reported in orange pulp by Ting (1970). For instance, Ting (1970) found that 90% of arabinose, 63.8% of galactose, 99% of galacturonic acid, and 100% of rhamnose made up pectic substances in orange pulp and the remaining amount of each monosaccharide made up hemicellulose. Total pectin in the citrus fiber was calculated by multiplying the pectin content found from the monosaccharide analysis amount by the total extractable carbohydrates as determined by the methods described in Yoo et al. (2003).

#### 2.3. Water holding capacity

As a measure of the samples' degree of hydrophilic properties, the water holding capacity was measured following American Association of Cereal Chemists (AACC) Method 56-30, which included centrifuging the sample at 2000g, with a minor modification needed to accommodate a high water holding capacity material. The modified method involved weighing 2.5 g of dry material into the centrifuge tube for the measurement instead of 5.0 g that the standard procedure called for.

#### 2.4. Swelling capacity

Swelling capacity, defined as the ratio of the volume occupied when the sample is immersed in excess of water after equilibration to the sample weight, was measured by the method of Raghavendra et al. (2004). To 0.2 g of dry sample placed in a graduated test tube; around 10 mL of water was added to hydrate the sample for 18 h; then the final volume attained by fiber was measured. The swelling capacity was then calculated using the following equation:

Swelling Capacity 
$$\left(\frac{mL}{g}\right) = \frac{\text{final volume occupied by sample (ml)}}{\text{original sample weight (g)}}$$
(1)

#### 2.5. Hydration procedure

To prepare the samples for rheological measurements, dried (4–8% moisture content) samples were blended in water for 3 min in a Waring blender on low speed to begin the hydration process. After blending, they were allowed to sit for 30 min to allow for the solution to come to equilibrium. Unless otherwise mentioned, the citrus fiber samples were hydrated at 3% solids and at room temperature (25 °C).

#### 2.6. Rheological measurement

A Brookfield DV-II+ viscometer with cylindrical spindles (Brookfield spindle types: #1 LV, #2 LV CYL, #3 LV CYL, #4 LV) was used for all the rheological work and apparent viscosity measurements. The range of speed varied from 0.5 rpm up to 200 rpm. However, when apparent viscosity measurement was taken, 10 rpm was used. A 250 mL sample at 3% solids and a temperature of 25 °C was used for apparent viscosity measurement unless noted otherwise.

#### 2.7. Particle size

Homogenous citrus fiber was ground to different degrees in order to obtain a range of particle sizes. Dynamic light scattering was used for measuring the particle width of the citrus fiber samples. The range of lengths that could be determined using dynamic light scattering is limited to  $300 \mu$ m; thus, fiber length was determined using laser diffraction with a Microtrac 3500 (Montgomeryville, PA) particle size analyzer. The length and width measurements used in this study are shown in Table 1.

#### 2.8. Statistics

Where error bars are shown in the graphs, they were calculated using a t-distribution with a 95% confidence interval and Excel software.

Table 1Fiber width and length measurements for the various samples in this study.

Sample ID	Width (µm)	Length (µm)
F33	33.6	363
F27	30.8	180
A01	27.2	76.2
A02	19.0	41.5
J01	11.2	12.7

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