



## Research paper

## Correlation analysis between chemical or texture attributes and stress relaxation properties of 'Fuji' apple

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## ABSTRACT

Rheological properties of fresh apple have been studied extensively, but the available information concerning the correlation between the rheological properties and quality characteristics is still limited. This study mainly aimed to investigate the feasibility of predicting physicochemical attributes of 'Fuji' apple using stress relaxation parameters. Relaxation tests were performed on the intact 'Fuji' apple to define the proper relaxation model and parameters through multi-exponential regression. The same sample was described by simultaneous instrumental profiling analyses including texture profile analysis, chemical measurements and stress relaxation test. Models for quality prediction of 'Fuji' apples were established and verified based on the variables of relaxation property. A three-term Maxwell model was used to satisfactorily describe the relaxation behaviors of intact 'Fuji' apples, and its coefficients of determination were over 0.9995. Significant relationships ( $p < 0.01$ ) were found between elastic or viscous components and chemical or texture attributes, while the correlations was not significant ( $p > 0.05$ ) between relaxation time ( $T_i$ ) and chemical or texture parameters. In regression analyses, effective predictive models were established for all chemical and texture attributes ( $R^2 > 0.80$ ), except for adhesiveness, springiness and cohesiveness, which were less effective ( $R^2 > 0.60$ ). All these results indicate that the measurement of relaxation properties can be deemed as an innovative, reliable and simple method to predict the chemical composition and texture characteristics of 'Fuji' apple.

## 1. Introduction

Apple is the fruit with the third highest yield (FAO, 2014) in the world and usually keeps fresh for sales in the market throughout the year. Thus, it is a crucially commercial issue to consider the postharvest quality of apples during storage and shelf life. As for customers, quality of apples is deemed as the most important motivation of their choices. At present, fruit wholesalers employ instrumental methods to measure apple quality based on consideration of appearance attributes (such as fruit shape, size, color), chemical properties (such as soluble solids content, titratable acidity, vitamin C), and texture attributes (Harker et al., 1997; Hoehn et al., 2003; Stow, 1995; Daillat-Spinnler et al., 1996; Jaeger et al., 1998). Texture is deemed as a multi-trait feature (Bourne, 2002) which is composed of two fundamental types of components, both mechanical evaluation (such as hardness and elasticity) and sound characteristics (such as crispness and crunchiness). Texture Profile Analysis (TPA) has been commonly used during evaluation of mechanical texture properties, wherein these properties are measured by the compression test and represented by force displacement based parameters. However, these instrumental measure-

ments of chemical and texture properties are time-consuming and destructive, and cannot measure quality properties in real time (Corollaro et al., 2014). In order to overcome these limitations, it is necessary to find a fast, nondestructive and accurate analytical method to evaluate the quality of fruit for the routine control of food industry.

Fundamental rheological tests are based on physicochemical theories, wherein, the chemical properties of molecules can be linked with bulk rheological properties of the system. Rheology studies have been extensively performed on fruit and vegetables to understand the relationship among the structure, texture and the changes induced by processing (Edwards, 1999; Jack et al., 1995). It is well known that the mechanical properties of biological tissues depend on the contributions made by different levels of structures (i.e., the molecular, cellular and organ level) and their physicochemical interactions (Jackman et al., 1992; Waldron et al., 1997). Dynamic rheology is frequently used for studying the viscoelastic behavior of apple. They allow to assess the storage and loss moduli which characterize the elastic and viscous behavior of a viscoelastic solid. For example, Dynamic Mechanical Analysis has been applied to investigate the effects of osmotic dehydration and turgor changes (Martinez et al., 2007; Winisdorffer et al.,

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2015). Nevertheless, the static rheological behaviors of apple have rarely been studied.

A stress relaxation experiment, as a typical static rheological test, is one of the most important evaluation tools to study the viscoelastic properties of materials (Campus et al., 2010; Andres et al., 2008). In a stress relaxation test, a constant strain is assigned to a sample, and the stress which is required to maintain the deformation is observed as a function of time (Steffe, 1992). When a stress relaxation test is carried out on viscoelastic solids, it can be observed that the material gradually relaxes and reaches an equilibrium stress larger than zero. Furthermore, as a typical experiment is restricted to small deformations within the linear viscoelastic range of the sample, stress relaxation makes it feasible to reflect indirectly the microstructure changes at the cellular level without disrupting them during the processing (Pitt and Chen, 1983). Information about the phenomena involving fruit production such as measurement of ripening (Hassan et al., 2005) and firmness (Blahovec, 1996), and prediction of mechanical damage during storage and transportation (Li et al., 1991) can be provided by the stress relaxation data.

Several investigations on relationship between rheological properties and texture attributes or chemical properties have been published. Wu and Abbott (2002) found that the firmness was closely related with an elastic behavior, and some relaxation parameters were related to ripeness. Ballabio et al. (2012) established the relationships between crispness and rheological parameters, predicting the apple crispness through multivariate analysis. This study can be considered as a more efficient exploitation of fruit texture in apple. Nevertheless, the rheological properties of the fruit flesh, especially the shape and outer pericarp, need to be measured for evaluation of apple quality and physiological studies. There is a considerable amount of literature on rheological tests of fruit tissue, but few articles about measurements involving intact apple have been published. Firmness or crispness of apple has been proved to be related to rheological properties, but similar studies on other texture attributes were rarely available. Furthermore, no attempt was made to apply rheological parameters of intact apples to evaluate and predict apple quality.

This work aimed to study the rheological properties of the intact apples by stress relaxation and correlate the relaxation parameters with texture attributes (obtained by texture profile analysis) and chemical compositions (solids soluble content, titration acid, vitamin C). Apples were stored in different periods to obtain a large spread of data suitable for establishing scientific and reasonable prediction models. The relaxation data generated was fitted with three different models in order to realize the best possible interpretation of the relaxation curves obtained. Finally, effective models were proposed to predict texture and chemical properties through rapid rheological characterization of apple.

## 2. Materials and methods

### 2.1. Samples

‘Fuji’ apples were harvested at commercial maturity in the mid-October 2014 from an orchard in Shaanxi Province, China. These fruit were selected based on uniform size, same ripeness stage and absence of obvious visible defects or blemishes, and consistent environmental conditions including temperature of 10 °C and relative humidity of 95%. Prior to the experiment, fruit were placed at temperature of 20 °C for one hour. Stress relaxation behaviors, texture and part chemical parameters (soluble solids concentration, titratable acidity and vitamin C) were measured in succession. In this study, five apples were taken each time for the analysis at intervals of a week, wherein the total duration was 8 weeks. A total of 40 apples were separated into two groups, wherein 32 fruit were chosen for establishment of the prediction models for TPA and chemical characteristics based on the relaxation parameters, while the other 8 fruit were characterized to verify the

prediction models.

### 2.2. Stress relaxation test

#### 2.2.1. Assay

Stress relaxation tests were performed on the intact apples at a room temperature using a TA-XT plus Texture Analyzer (Stable Microsystems Ltd., Godalming, UK) with a 5 kg load cell to obtain the relaxation data. Samples were compressed by 1.5 mm using a 10 mm cylinder probe at a crosshead speed of 1 mm/s. The compression was maintained for 100 s so as to balance the stress. In that period, the relaxation of stress was measured at a rate of 1 mm s<sup>-1</sup>. Measurements were carried out around the equator of each fruit so as to align the carpodium of the fruit parallel to the contact surface of the probe. Three measurements were carried out for each apple.

#### 2.2.2. Data analysis

Generalized Maxwell model is frequently used to interpret stress relaxation data of viscoelastic materials. The model consists of  $n$  Maxwell elements and a free spring in parallel, wherein each Maxwell element refers to one spring and one dashpot in series (Steffe, 1992). During the compression, the compression area of intact apples varied under loading, so the force-time curves could be obtained to analyze the stress relaxation behavior. The force-time relationships in relaxation were fitted to a modified version of the generalized Maxwell model as follows (Nussinovitch et al., 1989):

$$F(t) = D_0 E_0 + \sum_{i=1}^n D_0 E_i \exp(-t/T_i)$$

where  $F(t)$  is the decaying force (N) at a given time,  $D_0$  is the constant strain (mm),  $E_0$  denotes the equilibrium elastic modulus,  $E_i$  are the elastic modulus of the ideal elastic element,  $n$  is the number of Maxwell elements,  $T_i$  are the relaxation times of the  $i$ -th Maxwell element. The viscosity of element  $i$  can be calculated according to the following equation:

$$\eta_i = E_i T_i$$

In this study, one-element, two-element and three-element Maxwell models were used to analyze the force-time curves. Analysis of maximum relative difference (MRD) and the coefficient of determination ( $R^2$ ) were used as the criteria to find the best fitness which could describe the relaxation curves most effectively. For each curve fitting, the estimated parameters of elements were obtained and the values were used for subsequent analysis.

### 2.3. Texture profile analysis (TPA)

The texture profile analysis (TPA) was carried out by a TA-XT plus Texture Analyzer (Stable Microsystems Ltd., Godalming, UK) equipped with a load cell of 5 kg and a P50 cylindrical probe which was supplied with Texture Exponent Programs. Two flesh cylinders (5 cm of diameter, 2 cm of thickness) were removed from two opposite sides of each fruit around the equator. The flesh cylinders were compressed twice by a cylindrical probe under a controlled force of 5 g and the speed of 1 mm/s, while the deformation distance was determined to be 6 mm. Parameters including hardness (N), adhesiveness (N s), springiness (mm), cohesiveness, chewiness (N mm), and resilience were analyzed, wherein they were calculated on the record curves (Fig. 1) by the method described by Costa et al. (2011). These two recorded curves taken on opposing sides of each fruit were averaged to obtain the values of six parameters of each fruit. Data was converted into Newtons force.

### 2.4. Chemical measurements

The fruit soluble solids content (SSC) was determined by a handheld

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