

## EFFECTS OF THE THERMOSONICATION CLARIFICATION ON THE RHEOLOGICAL PROPERTIES OF APPLE JUICE

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In this study, the clarification of apple juice (AJ) was performed by using a bath type sonicator at different temperatures. The effects of thermosonication applied on the rheological properties, and some physico-chemical characteristics (total soluble solids: Brix, pH, titratable acidity, color, cloud value and total phenolics content) of these apple juices were studied. Using four different rheological models, namely Herschel Bulkley, Power-law, Bingham, and Newtonian, the flow behaviors of apple juices were assessed. The results showed higher lightness values for thermosonicated apple juices. The amounts of total soluble sugar and titratable acidity of control and thermosonicated apple juices weren't affected. The total phenolic contents of thermosonicated apple juices were significantly higher than control. The apple juices were found to be pseudoplastic fluids with a shear thinning nature. The thermosonication treatment was statistically significant in the change of rheological properties. The Herschel Bulkley model was found to be the best fit to the experimental data of all samples. Results revealed that the sonication technique could successfully be implemented on an industrial scale for the processing of apple juices. The obtained data could be taken into account in the industrial production of AJ with ultrasound treatment, particularly in the estimation of pumping requirement and the change of velocity profiles.

**Keywords:** apple juice, clarification, rheology, thermosonication, ultrasound treatment.

### Introduction

The apple (*Malus domestica*) is the most widely consumed fruit in the world. It is available on the market throughout the year. There has been growing interest in the consumption of apple juice (AJ), which is one of the most popular juices consumed around the world, due to its high nutritious and medicinal value (Abid *et al.*, 2014). The raw apple juice obtained by the mechanical pressing of apples is very viscous,

turbid or cloudy, brown colored and it has the tendency to settle down during storage (Dey *et al.*, 2014). Because of these problems, before its commercialization, the clarification of AJ is essential and it is normally achieved through microfiltration (Fukumoto *et al.*, 1998), enzymatic treatment with pectinase (Gainvors *et al.*, 1994) or using common clarifying aids such as gelatin bentonite, silica sol, polyvinyl pyrrolidone or a combination of these compounds (Benítez *et al.*, 2009). However, clarification agents applied to clarify AJ could be harmful to human health (Jose *et al.*, 2014).

Due to advancements in scientific knowledge, consumers are now more conscious about health and diet in addition to extended shelf life. Therefore, researchers are now searching for non-thermal food processing technologies that can not only protect the original properties of food but also improve its nutritional profile (Aadil *et al.*, 2013). Ultrasound treatment is one of the non-thermal food processing technologies based on the use of sound waves under frequencies from 20 kHz to 10 MHz. When ultrasound treatments are used in conjunction with pressure treatment, heat treatment or both, the treatments are called manosonication, thermosonication, and manothermosonication, respectively. The major effects of ultrasound in a liquid medium are attributed to the cavitation phenomena. The collapse of the cavitation bubble creates a transitory hot spot with elevated localized temperature and pressure. These phenomena are used in numerous applications in the food industry, such as extraction, emulsification, preservation, homogenization, etc. (Chemat *et al.*, 2011). The ultrasound treatment is known as a new sustainable “green and innovative” technique (Zhang *et al.*, 2013).

Rheology is the study of the deformation and flow behavior of matter, which plays an important role for food systems. Moreover, it defines food structure during manufacturing (in factory) or preparation (in kitchen) and physiologically in the mouth, stomach, and intestine where food structures are perceived and digested (Tabilo-Munizaga and Barbosa-Cánovas, 2005; Fischer and Windhab, 2011). The rheological analysis can be particularly used in the estimation of pumping requirement and the change of velocity profiles. Rheological measurements are considered as an analytical tool to provide fundamental insights into the structural organization of food and also play an important role in heat transfer to fluid foods. Fruit juices exhibit different rheological behaviors because of the sugar content of fruit. The rheological properties of food products are strongly influenced by temperature, concentration and physical state of dispersion. Therefore, it is interesting to study the rheological properties of food products. There are many publications regarding the temperature and concentration effects on the flow properties of juice concentrates (Telis-Romero *et al.*, 1999; Sopade *et al.*, 2008; Bi *et al.*, 2013; Šimunek *et al.*, 2014). However, these works didn't include the effects of clarification and ultrasound treatment.

The aim of this study was to evaluate the effects of thermosonication treatment applied for the clarification of raw apple juice, as an alternative to industrial treatment. The raw apple juice was characterized in terms of rheological properties and other quality parameters.

## Materials and Methods

### *Chemicals*

Fresh apples (*Malus domestica*) were purchased from a local fruit market in Izmir, Turkey. Pectolytic enzyme and gelatin were purchased from Novo and ASYA Fruit Juice and Food Ind. Inc. (Isparta, Turkey), respectively. Gallic acid was purchased from Sigma Chemical Co. (St. Louis, MO, USA). Folin Ciocalteu reagent was purchased from Fluka (Buchs, Switzerland). All other chemicals and reagents were of analytical grade.

### *Preparation of apple juice*

Apples were washed with running tap water, dried with paper towels and then cut into four pieces with a stainless-steel knife. The stems and seeds were removed. The apple juice was extracted using a household tabletop juice extractor (Arzum, Turkey) to 85 % yield and centrifugated to remove impurities and coarse particles at 2000 g for 10 min. The resulting juice was divided into six different parts as a control sample and working samples for thermosonication treatment.

*Control sample* The apple juices were depectinized by heating in a water bath at 90°C for 5 min, cooled to 50°C, and enzymatically treated with 1.0 mL/L of pectolytic enzyme solution (Novo, Pectinex 100 L) at 50°C for 45 min. The amounts of pectolytic enzyme solution were chosen after performing preliminary tests. Following enzymatic treatment, the juice was processed into clear apple juice using two different clarification agents: treatment with bentonite (A type, 80–100 Bloom strength) (2500 mg/l) and then with gelatin (A type, 80–100 Bloom strength) (500 mg /l) at 50 °C for 2 h by continuous mixing using a magnetic stirrer (Wise Stir, MSH-20A, Germany). The dosages of clarification agents were selected according to the clarification process applied in the fruit juice industry. The fruit juices were centrifuged at 2000 g for 10 min and the juices were filtered with a plate filter (Seitzenzinger noll, 6136 D-6800, Mannheim). The juice samples were then transferred into 100 ml hermetically capped glass bottles.

*Working samples* The thermosonication of working samples (60 mL in 100 mL jacketed vessel) for 20, 30, 40, 50, 60 °C was applied for 1h at 20 kHz in the ultrasonic bath (Selecta, Spain). The working samples were placed in the ultrasound bath. During the thermosonication treatment, the water temperature in the ultrasound bath was controlled with a thermocouple and when the temperature began to rise, the temperature was fixed with ice. Immediately after sonication treatment, all the juice samples were centrifugated at 2000 g for 10 min, filtered through a plate filter (Seitzenzinger noll, 6136 D-6800, Mannheim) and stored at 4 °C until further analyses were performed.

### *Determination of total soluble solids (°Brix), pH and titratable acidity*

Total soluble solids were estimated as Brix with a refractometer (Kruss DR-201, Germany) at room temperature (25±1 °C). The pH of apple juice was determined using a digital pH meter (WTW-Inolab). For determination of the acidity of the apple juices, samples were titrated with standardized 0.1N NaOH in the presence of

the phenolphthalein (5%). The volume of NaOH was converted to g citric acid/100 ml of juice and TA (titratable acidity) was calculated using equation 1.

$$\text{Acidity}(\%) = \frac{\text{ml base titrant} \times \text{Normality of base} \times \text{Acid factor} \times 100}{\text{Sample volume in ml}} \quad (1)$$

#### **Determination of color and cloud value**

The color of the apple juices was measured using a HunterLab Color Flex model Colorimeter (Management Company, USA) based on three color coordinates, namely L\*, a\*, b\* at room temperature. The color values were expressed as L\* (whiteness or brightness / darkness), a\* (redness / greenness) and b\* (yellowness / blueness).

The cloud value of the apple juices was determined by following the method outlined by Szalóki-Dorkó *et al.* (2016). The cloud value was determined as the supernatant absorbance at 660 nm using a spectrophotometer (Varian Cary 50 Bio UV-Vis Spectrophotometer, Australia) with distilled water serving as a blank. The path length of the cuvette was 10 mm.

#### **Total phenolics content**

The Folin-Ciocalteu method modified by Singleton *et al.* (1965) was used to estimate the total phenolic content of the apple juices. Gallic acid was used to calibrate the method. Briefly, the sample extract (50 µl) was mixed with Folin-Ciocalteu reagent (250 µl). This mixture was kept in the dark at room conditions for 5 min. Then, a 7% Na<sub>2</sub>CO<sub>3</sub> (750 µl) solution was added to the mixture. The aqueous solution was then diluted to 5 ml with bi-distilled water. The reaction was left to take place for 120 min at ambient temperature in the dark. Absorbance was measured at 765 nm by using a diode array spectrophotometer (Varian Cary 50 Bio UV-Vis Spectrophotometer, Australia). The path length of cuvette was 10 mm. The total phenolic content was expressed as mg of gallic acid /equivalent (mg GAE g-1 sample). Each determination was performed in triplicate and repeated at least three times.

#### **Rheological measurements**

The rheological measurements were carried out using a Brookfield viscosimeter (Model LVDV-II Pro, Brookfield Engineering Laboratories, U.S.A) at 20°C between 10 and 100% fullscale torques, by selecting the specific spindle (S-18). The rotational speed of the spindle was adjusted in the range of 0-200 rpm. The measurements were performed by increasing rotor speeds up to the maximum speed and then gradually reducing it. During the rheological measurements, shear stress (SS), shear rate (SR), and torque % (T) values were recorded for each rotational speed (rpm). The rheological behavior of the apple juices thermosonicated at different temperatures (20, 30, 40, 50 and 60 °C) was studied. All experiments were performed in triplicate.

Experimental data were fitted to selected rheological model equations. Four different rheological models were applied to describe the flow behavior of liquid food, Power-law with yield stress (Herschel Bulkley; Equation 2), Power-law

model (Ostwald-de-Waele; Equation 3) and linear (Bingham model; Equation 4 or Newtonian model; Equation 5).

$$\tau = \tau_0 + \kappa\gamma^n \quad (2)$$

$$\tau = \kappa\gamma^n \quad (3)$$

$$\tau = \tau_0 + \eta_P\dot{\gamma} \quad (4)$$

where  $K$  is the the flow consistency index ( $\text{Pa}\cdot\text{s}^n$ ),  $\tau_0$  is yield stress (Pa),  $n$  is the flow behavior index (dimensionless),  $\eta_P$  is Bingham viscosity.

$$\tau = \eta\dot{\gamma} \quad (5)$$

where  $\eta$  is Newtonian viscosity ( $\text{Pa}\cdot\text{s}$ ),  $\tau$  is shear stress (Pa) and  $\dot{\gamma}$  is the shear rate ( $\text{s}^{-1}$ ).

Time dependency of rheological properties of apple juices was also investigated. Thixotropic or rheopectic characters were determined by analyzing the hysteresis loop between upward and downward shear stress-shear rate relations (Bozkurt and Icier, 2009; Icier and Bozkurt, 2011). The area between the upward ( $A_1$ ) and downward ( $A_2$ ) flow curves of the hysteresis loop was calculated by Simpson's rule. This area was named as thixotropic energy ( $\text{J}/\text{m}^3$ ). The speeds of 60-200 rpm were used for the determination of rheological properties (Equation 6).

$$\int_a^b f(x)dx = \frac{h}{3}[(y_0 + y_n) + 4(y_1 + y_3 + \dots + y_{n-1}) + 2(y_2 + y_4 + \dots + y_{n-2})] \quad (6)$$

where  $b$  is maximum shear stress,  $a$  is the starting point for shear stress, and  $n$  is the number of experiments,  $h$  is  $\frac{b-a}{n}$ .

The thixotropic index is one of the time dependency characteristics of apple juices. The thixotropic index,  $\alpha$ , was calculated as percent (Equation 7).

$$\alpha = \frac{(A_1 - A_2)}{(A_1)} \times 100 \quad (7)$$

### Statistical analysis

Physico-chemical data obtained in the study were represented as mean values  $\pm$  standard deviation (SD). One-Way ANOVA was performed at a significance level ( $\alpha$ ) of 0.05. Significant differences between the mean values were evaluated by using the Duncan test. Statistical analyses were performed by using statistic software (SPSS Statistics 20.0 software; Analytical Software, Tallahassee, FL, USA). The compatibility of the rheological model used in this study to the experimental data was determined by nonlinear regression analysis, high regression coefficient ( $R^2$ ), low root means square error (RMSE) and low  $\chi^2$  (chi-square) values were explained as an index of compatibility.  $\chi^2$  and RMSE were calculated using experimental and expected shear stress values according to Equations 8 and 9 for each temperature, respectively. The statistical criteria for having highest  $R^2$ , lowest RMSE and lowest  $\chi^2$  (chi-square) were chosen for the selection of the best model for fitting (Bozkurt and Icier, 2009).

$$\chi^2 = \frac{\sum_{i=1}^n (KG_{\text{experimental},i} - KG_{\text{predicted},i})^2}{N-n} \quad (8)$$

$$\text{RMSE} = \left[ \frac{1}{N} \sum_{i=1}^N (KG_{\text{experimental},i} - KG_{\text{predicted},i})^2 \right]^{1/2} \quad (9)$$

where  $KG$  is rheological data (experimental and predicted),  $i$  is observation value at  $i$  experiment,  $N$  is observation number, and  $n$  is number of parameters in model.

## Results and Discussion

### ***Total soluble solids (°Brix), pH and titratable acidity, color and cloud value***

The results of the effect of thermosonication treatment on total soluble solids (Brix), pH, titratable acidity, and cloud value are shown in Table 1. Statistically significant changes ( $p < 0.05$ ) in pH and °Brix of apple juices were observed due to the sonication treatment, while titration acidity was not affected ( $p > 0.05$ ). The increment of total soluble solids might be attributed to the increase in extraction efficiency with thermosonication treatment. This treatment damaged the cell walls and tissues of fruits and thus, more soluble solids can be found in the samples (Zou and Hou, 2017). In this study, the results of °Brix, pH, and acidity of sonicated apple juices were found to be different from previous studies (Abid *et al.*, 2013; Gao and Rupasinghe, 2012). These differences can be attributed to the fruit composition resulting from climatic and geographical conditions.

The cloud value of the non-thermosonicated (control) sample was found to be 0.08. This study showed that the thermosonication treatment significantly affected ( $p < 0.05$ ) the cloud value of the apple juices. The mineral substances, vitamins, phenolic compounds, and soluble solids released with thermosonicated treatment are presented in Table 1. The increase in cloud value might be due to high-pressure gradient that occurred due to cavitations during the thermosonication treatment at 20°C. These colloidal disintegrations, dispersion and breakdown of macromolecules to smaller ones make the juice properly homogenized and more consistent (Abid *et al.*, 2013).

The results showed that the thermosonication treatment caused significant differences ( $p < 0.05$ ) among the color properties of the apple juices (Table 2). The apple juice thermosonicated at 60°C showed the lowest  $L^*$  and  $a^*$  value, while it had the highest  $b^*$  value. The results were found to be in agreement with the observations of Abid *et al.* (2013). The changes in the color of fruit juice could be attributed to the cavitations originating from thermosonication at 20 °C, although the changes might easily be observed with the naked eye (Tiwari *et al.*, 2008).

### ***Total phenolic content and free radical scavenging activity***

As shown in Table 1, the total phenolic contents of the thermosonicated apple juices were found to be higher than the control apple juice. After thermosonication, the total phenolic content of the apple juices ranged from 195 µg GAE/g to 242.1 µg GAE/g, while the total phenolic content of control apple juice was found to be 176.74 µg GAE/g. It was observed that there was a rising trend in the total phenolic content of thermosonicated apple juices with increasing ultrasound temperature (20-60°C). Generally, high temperatures can accelerate the softening and swelling of the raw materials as well as the solubility of compounds and so this could decrease the viscosity of the solvent (Tao *et al.*, 2014; Tao and Sun, 2015). As a result, the mass transfer of phenolic compounds from samples was improved when the temperature was increased gradually. Furthermore, the other possible reason for

the significant increases in total phenolic contents in thermosonicated apple juices could be attributed to the disruption of the cell wall due to cavitations. The cause of the cavitations is rapid change in pressure resulting from the shear forces that occurred during sonication and so some chemically bound phenolic compounds are released and ultimately, this increases the amount of total phenolic content in the juice. The formation of hydroxyl radicals from bubble implosion during sonication in the aromatic ring of phenolic compounds might also be a cause of the improvement in the apple juices (Abid *et al.*, 2013).

**Table 1.** Effect of thermosonication on °Brix, pH, acidity, total phenolic content and cloud values of control and apple juices thermosonicated at different temperatures.

Samples	Total soluble solids (°Brix)	pH	Titrateable acidity (%)	Total phenolic content (µg GAE/g)	Cloud value
Control	12.17±0.21 <sup>d</sup>	3.82±0.03 <sup>d</sup>	0.34±0.05 <sup>a</sup>	176.74±1.58 <sup>f</sup>	0.08±0.00 <sup>f</sup>
20 °C	12.97±0.15 <sup>c</sup>	3.96±0.02 <sup>b,c</sup>	0.34±0.05 <sup>a</sup>	194.90±0.53 <sup>a</sup>	0.99±0.00 <sup>c</sup>
30 °C	13.7±0.10 <sup>b</sup>	3.94±0.00 <sup>c</sup>	0.38±0.08 <sup>a</sup>	207.52±0.88 <sup>d</sup>	1.04±0.00 <sup>d</sup>
40 °C	13.79±0.15 <sup>b</sup>	3.97±0.01 <sup>b</sup>	0.38±0.10 <sup>a</sup>	218.86±0.77 <sup>c</sup>	1.06±0.00 <sup>c</sup>
50 °C	14.57±0.25 <sup>a</sup>	4.00±0.01 <sup>a</sup>	0.40±0.10 <sup>a</sup>	225.12±0.41 <sup>b</sup>	1.071±0.00 <sup>b</sup>
60 °C	14.80±0.00 <sup>a</sup>	4.02±0.01 <sup>a</sup>	0.47±0.06 <sup>a</sup>	242.10±3.07 <sup>a</sup>	1.32±0.00 <sup>a</sup>

Values with different letters in the same column (a–f) are significantly different ( $p < 0.05$ ) from each other.

**Table 2.** Effect of thermosonication on color parameters of apple juice samples thermosonicated at different temperatures.

Samples	Color		
	L*	a*	b*
Control	26.90±0.05 <sup>a</sup>	10.64±0.40 <sup>a</sup>	19.77±0.41 <sup>d</sup>
20 °C	22.83±0.03 <sup>b</sup>	10.58±0.09 <sup>a</sup>	20.20±0.20 <sup>c</sup>
30 °C	22.58±0.05 <sup>c</sup>	9.77±0.10 <sup>b</sup>	20.22±0.12 <sup>c</sup>
40 °C	22.31±0.01 <sup>d</sup>	9.33±0.08 <sup>c</sup>	20.62±0.15 <sup>b,c</sup>
50 °C	22.15±0.05 <sup>e</sup>	9.21±0.04 <sup>c</sup>	20.81±0.14 <sup>a,b</sup>
60 °C	21.60±0.01 <sup>f</sup>	9.12±0.04 <sup>c</sup>	21.08±0.28 <sup>a</sup>

Values with different letters in the same column (a–f) are significantly different ( $p < 0.05$ ) from each other.

### **Rheological measurement**

The effect of thermosonication on the rheological properties of apple juices was investigated at ambient temperature. The relationship between shear stress (SS) and shear rate (SR) for apple juices clarified with thermosonication at different temperatures is shown in Figure 1. As shown in Figure 1, the shear stress values decreased as the temperature increased for the same shear rate value in the range of



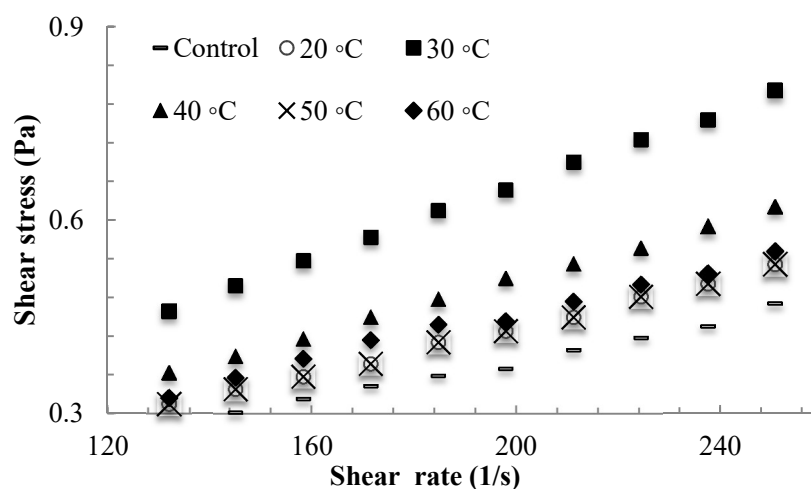
132-260 s<sup>-1</sup>. Rheological properties were predicted to describe this behavior by means of fitting different rheological models to the experimental data (Table 3).

**Table 3.** Statistical evaluation of rheological models applied to fit the experimental shear stress-shear rate data for control and apple juices thermosonicated at different temperatures

Samples	Statistical Criteria	Models			
		Herschel Bulkley	Power-law	Bingham	Newtonian
Control	X <sup>2</sup>	2.82x10 <sup>-5</sup>	1.086x10 <sup>-3</sup>	8.915x10 <sup>-3</sup>	68.2x10 <sup>-5</sup>
	RMSE	140.5x10 <sup>-5</sup>	932.1x10 <sup>-5</sup>	2670.6x10 <sup>-5</sup>	783.5x10 <sup>-5</sup>
	R <sup>2</sup>	0.995	0.987	0.989	0.989
20 °C	X <sup>2</sup>	3.8x10 <sup>-5</sup>	3.6x10 <sup>-5</sup>	5x10 <sup>-5</sup>	57.1x10 <sup>-5</sup>
	RMSE	150.4x10 <sup>-5</sup>	153.7x10 <sup>-5</sup>	180.4x10 <sup>-5</sup>	636.7 x10 <sup>-5</sup>
	R <sup>2</sup>	0.999	0.998	0.999	0.999
30 °C	X <sup>2</sup>	7.7x10 <sup>-5</sup>	144.9x10 <sup>-5</sup>	8.2x10 <sup>-5</sup>	361x10 <sup>-5</sup>
	RMSE	207.8x10 <sup>-5</sup>	942x10 <sup>-5</sup>	467.9x10 <sup>-5</sup>	1547.3x10 <sup>-5</sup>
	R <sup>2</sup>	0.999	0.999	0.999	0.999
40 °C	X <sup>2</sup>	44x10 <sup>-5</sup>	100.9x10 <sup>-5</sup>	238.4x10 <sup>-5</sup>	828.6x10 <sup>-5</sup>
	RMSE	510.3x10 <sup>-5</sup>	810.6x10 <sup>-5</sup>	1245.7x10 <sup>-5</sup>	2425.6x10 <sup>-5</sup>
	R <sup>2</sup>	0.999	0.998	0.999	0.999
50 °C	X <sup>2</sup>	376.52x10 <sup>-5</sup>	151.83x10 <sup>-5</sup>	189.92x10 <sup>-5</sup>	151.78x10 <sup>-5</sup>
	RMSE	1577.77x10 <sup>-5</sup>	1025.4x10 <sup>-5</sup>	1188.5426x10 <sup>-5</sup>	1120.01x10 <sup>-5</sup>
	R <sup>2</sup>	0.996	0.995	0.996	0.996
60 °C	X <sup>2</sup>	6.647x10 <sup>-5</sup>	6.1x10 <sup>-5</sup>	5.89x10 <sup>-5</sup>	41.8x10 <sup>-5</sup>
	RMSE	209.6x10 <sup>-5</sup>	213.6x10 <sup>-5</sup>	209.2x10 <sup>-5</sup>	588x10 <sup>-5</sup>
	R <sup>2</sup>	0.994	0.993	0.994	0.994

The experimental results on the variation of shear rate and shear stress were fitted to four different models, namely the Herschel Bulkley model (Equation 1), Power-law model (Equation 2), Bingham model (Equation 3) and Newtonian model (Equation 4). X<sup>2</sup> and RMSE values were used as statistical criteria to assess the suitability of the rheological models describing the rheological characteristics of the apple juices. The Herschel Bulkley model gave a very good fit (high R<sup>2</sup>) for all temperatures studied with the experimental data, since the X<sup>2</sup> and RMSE values obtained in this model were lower than those of the other models applied (p<0.05) (Table 3). For the control sample, the R<sup>2</sup> values of the Newtonian and Bingham models were lower than those of Herschel Bulkley and Power-law (p<0.05). Furthermore, the X<sup>2</sup> and RMSE values of the Herschel Bulkley model were lower than those of the Power-law model (p<0.05). Since the Herschel Bulkley model had higher R<sup>2</sup> and lowest error values (RMSE and X<sup>2</sup>), it was also selected as the best model fitting the experimental data of the control group.





**Figure 1.** Rheograms for shear stress versus shear rates plots of control and apple juices thermosonicated at different temperatures.

The control apple juices demonstrated non-Newtonian characteristics since the Herschel Bulkley model satisfactorily fitted the experimental shear stress and shear rate values. The same model was also the most suitable one describing the rheological properties of the apple juices thermosonicated at 20, 30, 40, 50 and 60 °C (Table 3).

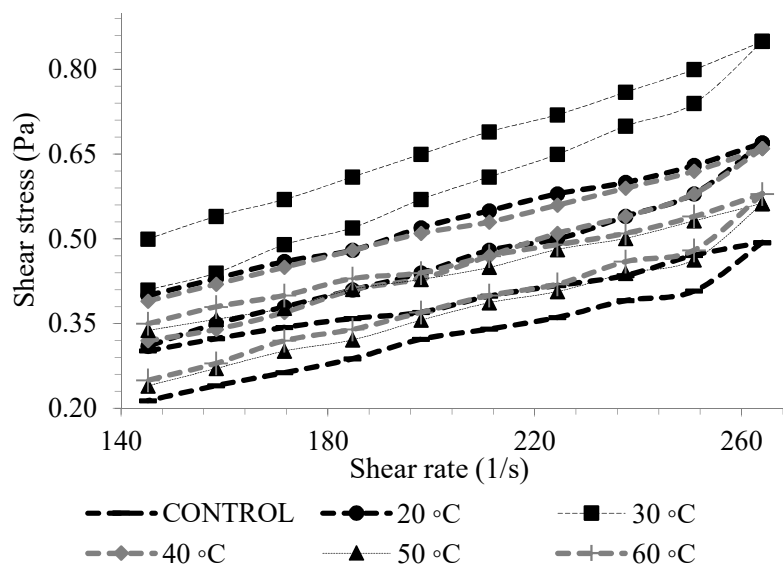
In our study, in the Herschel Bulkley model estimation, values of  $0.00003 \text{ Pa}\cdot\text{s}^n$  for consistency coefficient ( $k$ ), 1.65 for flow behavior index ( $n$ ) and  $0.019 \text{ Pa}\cdot\text{s}$  for yield stress ( $k_0$ ) were found for control. The represented values for the samples ultrasonicated at 20, 30, 40, 50 and 60 °C were given in Table 4. Since the flow consistency index ( $k$ ) decreased with the increasing temperature,  $k$  of the thermosonicated apple juices decreased as a result of the increasing temperature. The flow behavior index ( $n$ ) of a fluid is used to predict its behavior during industrial processing during different unit operations. Since the  $n$  value of the control apple juices was higher than 1, it can be deduced that it presented a dilatant behavior. On the other hand, the ultrasonicated AJs were referred to as pseudoplastic as they exhibited  $n$  values smaller than 1. There was no trend for the change of  $n$  values with temperature.

Genovese and Lozano (2006) investigated the viscosity of cloudy apple juice and correlated with Power-law model estimation. Bruijn and Borquez (2006) clarified the apple juice and removed the suspended solids with ultrafiltration and fitted the rheological parameters to a Herschel Bulkley model. The previous studies summarized that non-Newtonian fluid behavior is used to describe flow behavior of fruit juices, while the applied rheological model is dependent on the type of food and the temperature of application. Thus, it can be stated that the investigation of the rheological properties of apple juices processed by novel alternative technologies can be regarded as an essential tool to characterize the effects of the new applications on the consistency of the juices.

**Table 4.** Rheological parameters obtained from different model equations for control and apple juices thermosonicated at different temperatures.

Sample	Model	Newtonian viscosity (Pa×s)	Flow coefficient, <i>k</i> (Pa×s)	Yield stress, $\tau_0$ (Pa)	Flow behavior index, <i>n</i>
Control		-	$3 \times 10^{-5}$	0.190	1.655
20 °C	Herschel Bulkley	-	$16 \times 10^{-3}$	0.087	0.686
30 °C		-	$3 \times 10^{-3}$	0.071	0.993
40 °C		-	$3 \times 10^{-3}$	0.041	0.946
50 °C		-	$2.6 \times 10^{-3}$	0.050	0.945
60 °C		-	$3 \times 10^{-3}$	0.068	0.941
Control			-	$5 \times 10^{-3}$	-
20 °C	Power-law	-	$7 \times 10^{-3}$	-	0.813
30 °C		-	$7 \times 10^{-3}$	-	0.868
40 °C		-	$5 \times 10^{-3}$	-	0.862
50 °C		-	$6 \times 10^{-3}$	-	0.824
60 °C		-	$7 \times 10^{-3}$	-	0.796
Control			-	$2 \times 10^{-3}$	0.065
20 °C	Bingham	-	$2 \times 10^{-3}$	0.086	-
30 °C		-	$3 \times 10^{-3}$	0.074	-
40 °C		-	$2 \times 10^{-3}$	0.063	-
50 °C		-	$2 \times 10^{-3}$	0.075	-
60 °C		-	$2 \times 10^{-3}$	0,09	-
Control			-	$2 \times 10^{-3}$	-
20 °C	Newtonian	0.003	-	-	-
30 °C		0.003	-	-	-
40 °C		0.003	-	-	-
50 °C		0.002	-	-	-
60 °C		-	$2 \times 10^{-3}$	-	-

The time dependency tests were performed to characterize the effects of thermosonication on the time-dependent consistency properties of apple juices in detail (Figure 2). The area between the upward and downward flow curves is defined as thixotropic energy and corresponds to the power required to break down the thixotropic structure of the sample (Table 5). There was no significant ( $p > 0.05$ ) effect of the thermosonication on the thixotropic energy. Since the time dependency of the thermosonicated apple juices was similar to the control samples, the thermosonication clarification did not cause any different effect on the rheological properties of apple juices from control.



**Figure 2.** Thixotropic hysteresis curves for control and apple juices thermosonicated at different temperatures.

**Table 5.** The thixotropic characteristics of control and thermosonicated at different temperatures apple juices at different temperatures.

Sample	Thixotropic energy ( $\text{Pa s}^{-1}$ )	Thixotropic index
Control	10.13±0.09	17.87±8.95
20 °C	11.24±0.21	16.42±2.48
30 °C	14.85±1.64	11.70±3.10
40 °C	9.82±0.76	14.77±4.70
50 °C	12.75±1.09	18.57±4.47
60 °C	14.68±1.42	18.84±2.99

## Conclusions

This study showed that apple juices could be satisfactorily clarified by thermosonication treatment at different temperature gradients. The non-Newtonian characteristic of the clarified apple juices was determined by fitting the experimental shear stress-shear rate data by the Herschel Bulkley model. The study showed that the thermal treatments influenced the physico-chemical characteristics of the studied juices as a result of temperature treatment, which modified the intrinsic properties of some chemical components present in suspension. Furthermore, it was found that the thermosonication treatment significantly improved ( $p < 0.05$ ) the phenolic compounds and cloud value of apple juice. The results suggest that sonication technology could successfully be employed for the processing of apple juice with improved quality from the perspective of human health.

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