

Texture and Pasting Properties of Ultrasonically Treated Corn Starch

IVANA LJUBIĆ HERCEG¹, ANET REŽEK JAMBRAK², DRAGO ŠUBARIĆ¹,
MLADEN BRNČIĆ², SUZANA RIMAC BRNČIĆ², MARIJA BADANJAK², BRANKO TRIPALO²,
DAMIR JEŽEK², DUBRAVKA NOVOTNI² and ZORAN HERCEG²

¹Faculty of Food Technology, University of Osijek, Osijek, Croatia; ²Faculty of Food Technology and Biotechnology, University of Zagreb, Zagreb, Croatia

Abstract

HERCEG I.L., JAMBRAK A.R., ŠUBARIĆ D., BRNČIĆ M., BRNČIĆ S.R., BADANJAK M., TRIPALO B., JEŽEK D., NOVOTNI D., HERCEG Z. (2010): **Texture and pasting properties of ultrasonically treated corn starch.** Czech J. Food Sci., **28**: 83–93.

The effects of high power ultrasound of 24 kHz and ultrasound bath of 24 kHz frequency on the textural and pasting properties of corn starch suspensions was examined. Suspensions were treated with different intensities and treatment times (15 min and 30 min) using an ultrasound probe set and bath. The treatments with high power ultrasound probes caused a significant lowering of the starting gelatinisation temperatures of corn starch. The ultrasound treatment caused disruption of starch granules by cavitation forces and made the granules more permeable to water. The highest viscosity was observed for the treatment with 300 W probe. Also, a statistically significant increase in solubility in water (20°C) was observed, being caused by the disruption of starch granules and molecules by ultrasound treatment. When applying more powerful ultrasound, starch granules, specifically in the amorphous region, are much more mechanically damaged. The texture profile analyses of the starch gel prepared from the suspensions that had been treated with ultrasound probe presented higher hardness and higher values of adhesiveness and cohesiveness when compared with untreated suspensions or those treated with ultrasound bath. Micrography showed an obvious impact of ultrasound on the structure of starch granules. Ultrasound treatment ruptures and mechanically damages the starch granules causing collapse of cavitation bubbles which induces high pressure gradients and high local velocities of the liquid layers in their vicinity.

Keywords: corn starch; ultrasound; texture; solubility; pasting

Starch is the most common used polysaccharide in human nutrition. It is the most abundant reserve carbohydrate stored in plants. It is found in many different plant organs including seeds, fruits, tubers, and roots, where it is used as a source of energy. Starch is a useful polymer not only because it is a cheap, natural material but also because of the possibility to alter its physico-chemical properties through mechanical, chemi-

cal, or enzymatical modifications and/or physical treatment. The functional characteristics that are imparted by the starches to aqueous systems and their application in various foods vary with their biological origin (SVEGMARK & HERMANSSON 1993). Starch is used in many foods because it contributes greatly to their textural properties, and finds many industrial applications as thickener, colloidal stabiliser, gelling agent, bulking agent,

water retention agent, and adhesive. The great potential of starch in food industry has resulted in many studies having been carried out on the morphological, rheological, thermal, and textural properties of corn and potato starches (KIM *et al.* 1995; LI *et al.* 1996).

Ultrasound is the sound that is above the threshold of human ear hearing range (above 18 kHz). Ultrasound is typically divided into three regions of frequency. Power ultrasound lies in the region of 16–100 kHz (1 Hertz is 1 cycle/s), high-frequency ultrasound ranges between 100 kHz to 1 MHz, and diagnostic ultrasound between 1–10 MHz. Ultrasound is generated with either piezoelectric or magnetostrictive transducers that create high-energy vibrations. These vibrations are amplified and transferred to a sonotrode or probe, which is in the direct contact with the liquid. Some known applications of high-power ultrasound in food processing include the following: extraction, emulsification, homogenisation, crystallisation, filtration, separation, viscosity alteration, defoaming, and extrusion. Ultrasound also inactivates enzymes and bacteria by breaking cell membranes due to the violence of cavitation and due to the formation of free radicals. Cavitation is the formation of cavities filled with gas or vapour as the pressure decreases, and they collapse as soon as the pressure increases again (FREUDIG *et al.* 2003).

Some researches have been conducted on starch molecule in connection with ultrasound and high pressure processing in order to examine its properties and possible changes affected by new treatments. DOUZALS *et al.* (1996) showed that water molecules linked with starch occupy a smaller volume than do pure water molecules. Because the events that result in the volume reduction are favoured under pressure, the hydration of starch granules can be induced by pressure instead of heating. Whether induced by heat or pressure, starch gelatinisation is a swelling phenomenon which starts as water from the suspension enters starch granules. The ultrasonic process proved to be applicable for many kinds of starches (corn, potato, tapioca, and sweet potato) and polysaccharides (IIDA *et al.* 2008).

The viscosity of the starch solution of moderate concentration (5–10%) can be reduced by about two orders of magnitude to 100 mPa·s by ultrasonic irradiation for 30 minutes. The effects of ultrasound on aqueous solutions of chitosan and starch were also studied. This treatment is an efficient pro-

cedure for the reduction of molecular weights of both polysaccharides. It was demonstrated that, at the ultrasound frequency applied (360 kHz), degradation is caused both by OH radicals and mechanochemical effects (CZECHOWSKA-BISKUP *et al.* 2005).

The aim of this research was to examine the effect of high power ultrasound of 24 kHz frequency on the textural properties using Texture analyzer HD+ (Stable Micro System, Godalming, UK), pasting properties by Micro Visco-Amylo-Graph (Brabender GmbH & Co. KG, Duisburg, Germany), and solubility properties of corn starch suspensions. Different ultrasound treated suspensions were prepared, using ultrasound probe sets with different intensities (34, 55, 73 W/cm²) and treatment times (15 min and 30 min), and by applying ultrasound bath of 2–3 W/cm² intensity and different treatment times (15 min and 30 min).

MATERIALS AND METHODS

Sample preparation

A powdered corn starch sample (commercial name Maisita 21 000) was used whose composition had been declared by manufacturer (Agrana Starke GmbH, Wien, Austria) as follows: water 11.11%, starch 88.89%.

Suspensions were prepared by stirring the appropriate amounts of corn starch powder and distilled water as stated in Table 1 so as to contain 10% of dry matter (w/w). The suspensions thus prepared were homogenised using a magnetic stirrer and labelled as described in Table 2. The suspensions labelled K1, K2, K3, K4, K5, K6, K7, K8, and K9 were then treated with ultrasound as described in Table 2. The treated and untreated (K1) suspensions were used for determination of rheological parameters, gel preparation, and also gelatinisation and the determination of retrogradation properties using a differential scanning calorimeter.

Table 1. Composition of corn starch suspensions

Sample	Corn starch (g)	Water (g)
10% suspension (w/w) ¹	11.25	88.75
2% suspension (w/w) ²	2.25	97.75

¹used for pasting and textural properties of corn starch; ²used for solubility measurements

Table 2. Labels of samples

Sample	Treatment	Intensity (W/cm ²)	Time (min)
K1	untreated	–	–
K2	ultrasound bath	2	15
K3	ultrasound bath	2	30
K4	ultrasound probe	34	15
K5	ultrasound probe	34	30
K6	ultrasound probe	55	15
K7	ultrasound probe	55	30
K8	ultrasound probe	73	15
K9	ultrasound probe	73	30

Ultrasound treatment of corn starch model systems

Ultrasound treatment with 24 kHz probe. The samples prepared for ultrasound treatment with probe (24 kHz) of 500 ml volume were placed in flat bottom conical flasks. The samples were treated for 15 min and 30 min with power ultrasound of 100 W, 300 W, and 400 W of nominal power with constant amplitude of 100%. High intensity and low frequency 24 kHz probe (UP 400S, Dr Hielscher GmbH, Teltow, Germany) was attached to the transducer. The probe had a vibrating titanium tip of 7 mm in diameter and was immersed in the liquid and the liquid was irradiated with an ultrasonic wave directly from the horn tip.

Ultrasound treatment with 24 kHz bath. The prepared samples of 500 ml volume were placed directly in the ultrasound bath and were treated with ultrasound of 24 kHz frequency. The samples were treated for 15 and 30 minutes (the Model had been designed at the Faculty of Mechanical Engineering and Naval Architecture, Zagreb, Croatia: HF-Pk-power 300 W – overall dimensions: 370 × 175 × 250 mm; internal dimensions: 300 × 150 × 150 mm). Ultrasonic transducers were attached to the bottom of the outer surface of the liquid container and the liquid was irradiated with ultrasonic waves from the surface of the liquid container.

Determination of ultrasound power and intensity. Ultrasonic power, which is considered as mechanical energy, will be partly lost in the form of heat when ultrasound passes through the medium (THOMPSON & DORAISWAMY 1999). Since the ultrasonic irradiation of a liquid produces heat, recording the temperature as a function of

time leads to the acoustic power estimation (in W) by the equation (MARGULIS & MALT'SEV 1969; MARGULIS & MARGULIS 2003).

$$P = m c_p (dT/dt) \quad (1)$$

where:

- m – is the mass of the sonicated liquid (g)
- c_p – specific heat of medium at a constant pressure dependent on the composition and volume of the medium (J/g/K)
- dT/dt – slope at the origin of the curve

Ultrasound intensity is expressed in watts per unit area of the emitting surface (W/cm²), or in watts per unit volume of the sonicated solution (W/cm³).

Ultrasonic intensity was measured calorimetrically by thermocouple (model HI 9063, Hanna Instruments Ltd., Leighton Buzzard, UK) and expressed in W/cm².

Pasting properties determination of corn starch model systems

Pasting properties of corn starch suspensions were determined according to the method of HAGENIMANA and DING (2005). Corn starch suspension of 10% (w/w) in 100 ml volume was directly placed into a stainless steel measuring bowl of a Micro Visco Amylo-Graph (Brabender GmbH & Co. KG, Duisburg, Germany). The system operating at 250 rpm was then heated from 30°C to 93°C, with the heating rate of 7.5°C. The samples were held for 1 min at 52°C and for 5 min at maximal temperature of 93°C. The following parameters were derived: maximum viscosity, the decrease in viscosity during cooling,

increase in viscosity during heating, and beginning of gelatinisation (°C). Viscosity was measured in arbitrary Brabender units (BU) and was determined at least in triplicates.

Solubility index (IS) determination of corn starch model systems

Solubility of corn starch model systems was determined in triplicates according to the method of LEACH *et al.* (1959). Aqueous suspensions of 2% starch (w/w) were heated in a water bath at constant temperatures and shaken for 30 minutes. Each suspension was cooled and centrifuged at 3000 g for 15 min; the sediment was weighed and the supernatant was placed in a vacuum oven at 120°C for 4 hours. The data obtained were used to calculate the solubility of the starch granules.

Gel preparation and determination of gel hardness of corn starch model systems

The prepared corn starch model systems of 10% (w/w) after the ultrasound treatment were used for the gel preparation. The gel preparation was carried out by heating the suspensions placed in 150 ml glass beaker at 80°C for 15 min in water bath with constant stirring. After heating, the samples were rapidly cooled to room temperature by immersing the beaker in icy water and were then kept at 4°C for further analysis. The textural properties were determined 24 h after the gel preparation.

Textural properties of corn starch gels. Gel hardness was determined with a texture analyser (Texture analyser HD+, Stable Micro System, Godalming, UK). The speed of the measuring probe was 1 mm/s. The measurement depth was 10 mm. According to the measuring curves, the following properties were derived during two measuring cycles: hardness, cohesiveness, adhesiveness, springiness, and gumminess. Gel hardness was defined as maximal force applied (N).

Micrography of corn starch model systems

The previously prepared and treated 10% (w/w) corn starch suspensions were photographed using a digital camera (Olympus DP 12, Japan) that was attached onto a microscope (Olympus BX 51, Japan).

Magnification was 1000×. The pictures were computerised with computer software program Analysis Image Processing Olympus (Olympus, Japan).

Statistical analyses

The whole study was repeated. Each value represents the mean of three measurements from three independent ultrasound treatments. The effect of ultrasound treatment on the parameters tested was determined by analysis of variance, using statistical analyses with SPSS for Windows Version 13.0 (SPSS Inc., Chicago, USA). Analysis of variance (One-Way ANOVA), significant level used being 5% ($\alpha = 0.05$), was carried out to assess whether the different treatments led to statistically different results for the variables evaluated.

RESULTS AND DISCUSSION

Pasting properties determination of corn starch model systems

The treatment with high power ultrasound probes (K4–K9) caused a significant lowering of the beginning of the gelatinisation temperature, which indicated an earlier state of the granule swelling during heating (Table 3). The onset temperature reflects the first measurable swelling of the starch granules as reflected by the appearance of an increasing viscosity, and the gelatinisation temperature reflects the beginning of the granule distortion or disruption after the granular structure can no longer support the continuing swelling. Ultrasound treatment causes disruption of starch granules by cavitation forces and makes the granule more permeable to water during the heating step. The temperature differences between the ultrasound treatments are very small indicating that different ultrasound powers and intensities have similar effects on the pasting properties. From Table 4 can be seen a significant increase in the temperature of the suspension with increasing ultrasound power, the highest increase having been with K5 sample (treatment with 100 W probe for 30 min), i.e. from 18.1°C to 50.0°C, and for ultrasound treatment with 400 W probe for 30 min with the increase up to 56.4°C. From Figures 1 and 2, the graphical overview can be seen of the viscosities of starch suspensions treated with ultrasound

Table 3. Viscosity of corn starch suspensions

Sample	Maximum viscosity (BU)	Decrease in viscosity during cooling (BU)	Increase in viscosity during heating (BU)	Beginning of gelatinisation (°C)
K1	657 ± 1.1	203 ± 1.1	429 ± 1.1	73.0 ± 0.1
K2	679 ± 1.1	219 ± 1.1	441 ± 1.1	72.7 ± 0.2
K3	851 ± 1.2	318 ± 1.2	450 ± 1.2	72.4 ± 0.2
K4	1129 ± 1.1	527 ± 1.1	475 ± 1.1	71.1 ± 0.2
K5	1158 ± 1.1	499 ± 1.2	409 ± 1.1	71.3 ± 0.1
K6	1452 ± 1.2	738 ± 1.1	570 ± 1.2	71.1 ± 0.3
K7	1187 ± 1.3	529 ± 1.1	535 ± 1.1	71.1 ± 0.1
K8	1080 ± 1.2	428 ± 1.2	400 ± 1.1	71.2 ± 0.1
K9	1346 ± 1.2	693 ± 1.2	592 ± 1.2	71.0 ± 0.2

K1 – untreated; K2 – ultrasound bath – 15 min; K3 – ultrasound bath – 30 min; K4 – ultrasound probe 100 W – 15 min; K5 – ultrasound probe 100 W – 30 min; K6 – ultrasound probe 300 W – 15 min; K7 – ultrasound probe 300 W – 30 min; K8 – ultrasound probe 400 W – 15 min; K9 – ultrasound probe 400 W – 30 min

bath and probes. Obviously, viscosity increases from K1 to K5 sample indicating that, with increasing ultrasound power starch granules are much more disrupted and the crystalline region of the molecule is more weakened, thus causing more water being entrapped within the molecule which leads to higher viscosity. The mechanical treatment of starch suspensions caused by the

ultrasound treatment decreases the gelatinisation temperature. Such behaviour is consistent with mechanical activation increasing the amorphous regions of the starch granules while weakening and decreasing the crystalline regions of the starch (HUANG *et al.* 2007). However, in Figure 2 the highest viscosity can be observed with K6 sample for the treatment with 300 W probe that is afterwards decreasing with the increasing ultrasound power. This can be explained by that the ultrasound treatment caused severe damage to starch granules under the shear action and amylose molecules are forced to straighten out, which reduces the shear force between the fluid layers, thereby resulting in the decrease of viscosity. As the temperature is increasing, the molecules absorb translational energy and gradually lose their hydration, which results in the lowering of viscosity (CAMINO *et al.* 2009). The crystalline structure and hydrogen bonds of corn starch molecule are damaged during the mechanical treatment, its structure becomes loose and degradation takes place.

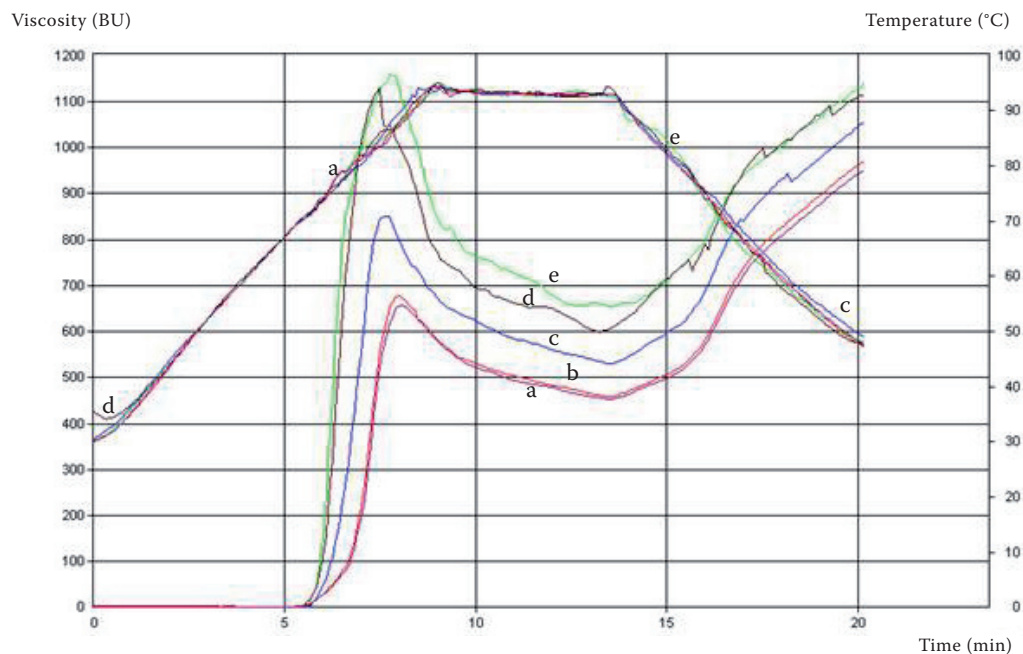
Table 4. Temperature of corn starch suspensions before and after ultrasound treatment

Sample	Initial sample temperature (°C)	Temperature of sample after treatment (°C)
K1	18.1 ± 0.1	18.1 ± 0.2
K2	18.2 ± 0.1	20.3 ± 0.2
K3	18.2 ± 0.1	21.4 ± 0.2
K4	18.2 ± 0.1	40.2 ± 0.2
K5	18.2 ± 0.1	50.0 ± 0.2
K6	19.6 ± 0.1	37.2 ± 0.2
K7	19.6 ± 0.1	42.2 ± 0.2
K8	19.1 ± 0.1	49.4 ± 0.2
K9	19.1 ± 0.1	56.4 ± 0.2

K1 – untreated; K2 – ultrasound bath – 15 min; K3 – ultrasound bath – 30 min; K4 – ultrasound probe 100 W – 15 min; K5 – ultrasound probe 100 W – 30 min; K6 – ultrasound probe 300 W – 15 min; K7 – ultrasound probe 300 W – 30 min; K8 – ultrasound probe 400 W – 15 min; K9 – ultrasound probe 400 W – 30 min

Solubility index (IS) determination of corn starch model systems

The results of solubility indices are shown in Table 5. One can observe a statistically significant increase in solubility in cold water that is caused by the disruption of starch granules and molecules by the ultrasound treatment. When applying more

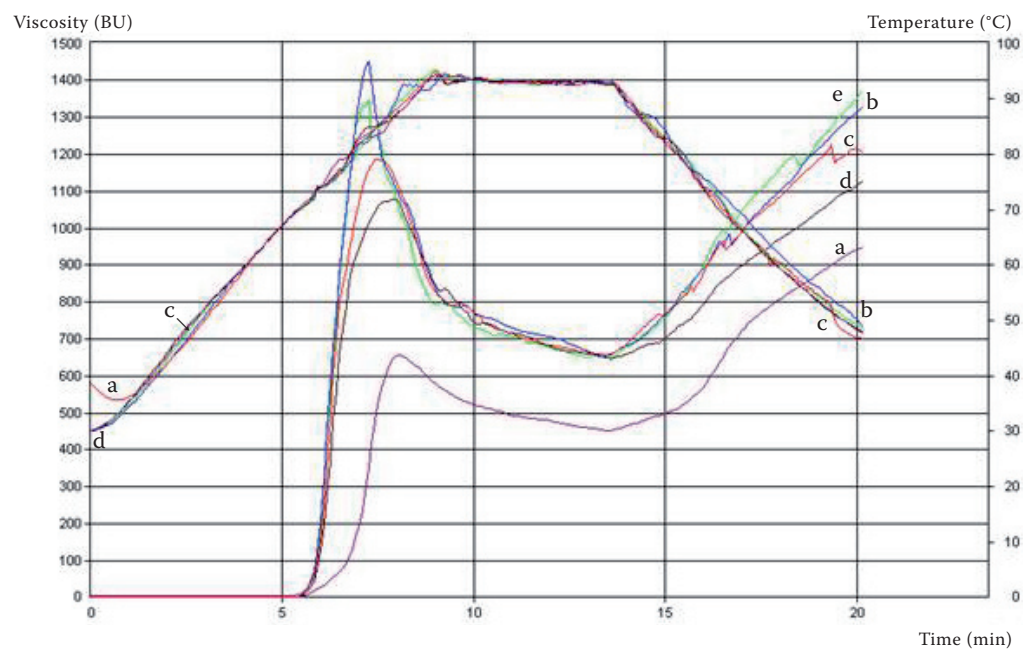


K1 – untreated (a); K2 – ultrasound bath – 15 min (b); K3 – ultrasound bath – 30 min (c); K4 – ultrasound probe 100 W – 15 min (d); K5 – ultrasound probe 100 W – 30 min (e)

Figure 1. Viscosity of corn starch suspensions for K1, K2, K3, K4, and K5 samples

powerful ultrasound, starch granules, specifically in amorphous region, become much more mechanically damaged. Also, the significant increase

in starch hydrophilicity after the treatment facilitates water percolation into the starch granules. The increase in water intake enhances leaching



K1 – untreated (a); K6 – ultrasound probe 300 W – 15 min (b); K7 – ultrasound probe 300 W – 30 min (c); K8 – ultrasound probe 400 W – 15 min (d); K9 – ultrasound probe 400 W – 30 min (e)

Figure 2. Viscosity of corn starch suspensions for K1, K6, K7, K8, and K9 samples

Table 5. Solubility indices (%) of corn starch suspensions

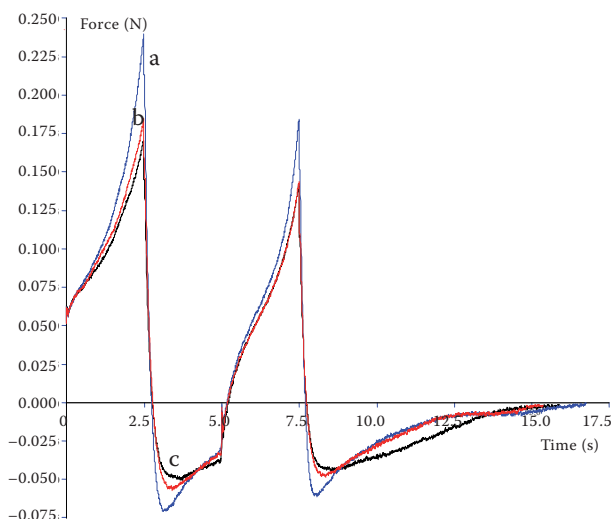
Sample	Preparation	Solubility index (%)
K1	20°C/5 min	0.18 ± 0.01
	20°C/15 min	0.04 ± 0.01
	70°C/5 min	4.52 ± 0.10
	70°C/15 min	4.43 ± 0.14
K2	20°C/5 min	3.22 ± 0.13
	20°C/15 min	3.01 ± 0.12
	70°C/5 min	5.27 ± 0.11
K3	70°C/15 min	5.82 ± 0.11
	20°C/5 min	3.28 ± 0.10
	20°C/15 min	3.29 ± 0.04
	70°C/5 min	5.29 ± 0.06
K4	70°C/15 min	5.85 ± 0.07
	20°C/5 min	3.85 ± 0.08
	20°C/15 min	3.86 ± 0.10
	70°C/5 min	5.95 ± 0.11
K5	70°C/15 min	5.92 ± 0.12
	20°C/5 min	4.12 ± 0.13
	20°C/15 min	4.02 ± 0.07
	70°C/5 min	6.23 ± 0.08
K6	70°C/15 min	6.94 ± 0.10
	20°C/5 min	5.25 ± 0.13
	20°C/15 min	5.07 ± 0.12
	70°C/5 min	7.05 ± 0.11
K7	70vC/15 min	7.81 ± 0.09
	20°C/5 min	5.39 ± 0.18
	20°C/15 min	5.28 ± 0.14
	70°C/5 min	7.07 ± 0.12
K8	70°C/15 min	7.27 ± 0.09
	20°C/5 min	5.66 ± 0.11
	20°C/15 min	5.85 ± 0.10
	70°C/5 min	7.87 ± 0.08
K9	70°C/15 min	7.30 ± 0.12
	20°C/5 min	6.53 ± 0.20
	20°C/15 min	6.45 ± 0.22
	70°C/5 min	8.18 ± 0.18
K9	70°C/15 min	8.08 ± 0.16

K1 – untreated; K2 – ultrasound bath – 15 min; K3 – ultrasound bath – 30 min; K4 – ultrasound probe 100 W – 15 min; K5 – ultrasound probe 100 W – 30 min; K6 – ultrasound probe 300 W – 15 min; K7 – ultrasound probe 300 W – 30 min; K8 – ultrasound probe 400 W – 15 min; K9 – ultrasound probe 400 W – 30 min

of amylose granules largely from the amorphous region of starch. Inter and intra molecular hydrogen bonds in the starch chains are disrupted and the motional freedom of starch chains increases as the temperature increases, thus improving solubility. It is well known that polysaccharides are degraded faster in dilute solutions than in concentrated solutions, and faster at lower temperatures than at higher ones (LIU *et al.* 2006).

Texture properties of corn starch gels

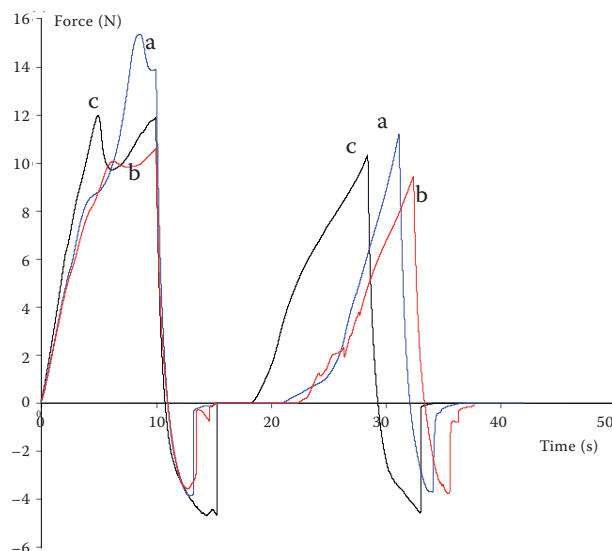
The texture properties of corn starch gels were determined using a texture analyser and are shown in Table 6 and Figures 3 and 4. The texture profile analyses of the starch gel prepared from the suspension that had been treated with ultrasound probe presented higher hardness and higher values of adhesiveness and cohesiveness when compared with the untreated suspensions or those treated with ultrasound bath (Table 6). The gel firmness is mainly caused by the starch gel retrograding, which is associated with water syneresis and amylopectin crystallisation loss (MILES *et al.* 1985). The starch gels presenting high stiffness tend to have a high amylose content and long amylopectin chains (MUA & JACKSON 1997). The results demonstrate that by applying high power ultrasound, gel hardness increases and is the highest for K6 treatment, that is the ultrasound treatment with 300 W probe for 15 minutes. When applying more power or higher intensity ultrasound, hardness decreases. It can be explained that starch molecule is degraded and amylose and amylopectin chains are disrupted and reorganised, while with the application of much more power (ultrasound treatment with 400 W probes – K8 and K9) starch granules become further disrupted and damaged causing less potential for gel organisation. In a potato starch variety, SINGH *et al.* (2006) found high fracturability and hardness, attributing this property to the presence of a high percentage of wide granules and low amylose content. Cohesiveness, adhesiveness, springiness and gumminess are statistically much greater than in untreated gels or those made from ultrasound treated suspensions with ultrasound bath. However, for ultrasound treatments at treatments for 15 min, adhesive and cohesive properties did not match the trends in hardness/gumminess and springiness. For 15 min treatment K4, K6, and K8, the values of adhesiveness and cohesiveness were



K1 – untreated (a); K2 – ultrasound bath – 15 min (b); K3 – ultrasound bath – 30 min (c)

Figure 3. Texture profile analysis of corn starch gel K1–K3

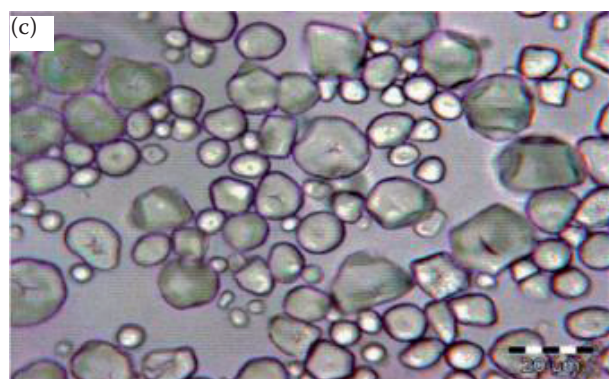
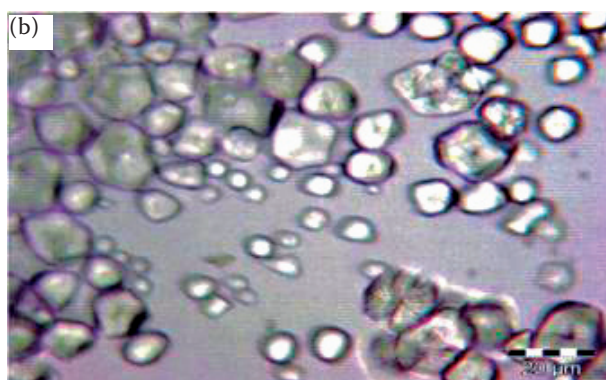
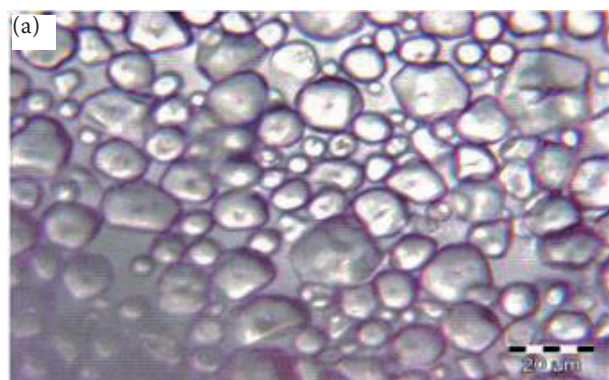
higher than for the untreated (K1) and ultrasound treated but smaller than after 30 min treatment for the same ultrasound power. For the ultrasound treatments with bath, the values of adhesiveness and cohesiveness were lower than the value for K1. The highest values of the texture gel proper-



K1 – untreated (a); K8 – ultrasound probe 400 W – 15 min (b); K9 – ultrasound probe 400 W – 30 min (c)

Figure 4. Texture profile analysis of corn starch gel K1 and K8–K9

ties mentioned could be observed for ultrasound treated suspensions with 300 W probes (K6 and K7). There are no statistically significant changes in the texture properties of gels prepared from ultrasound treated corn starch suspensions as compared to the untreated ones.



K1 – untreated; K2 – ultrasound bath – 15 min; K3 – ultrasound bath – 30 min

Figure 5. Microphotographs of samples K1 (a), K2 (b), and K3 (c)

Table 6. Texture parameters of native and ultrasound modified corn starches

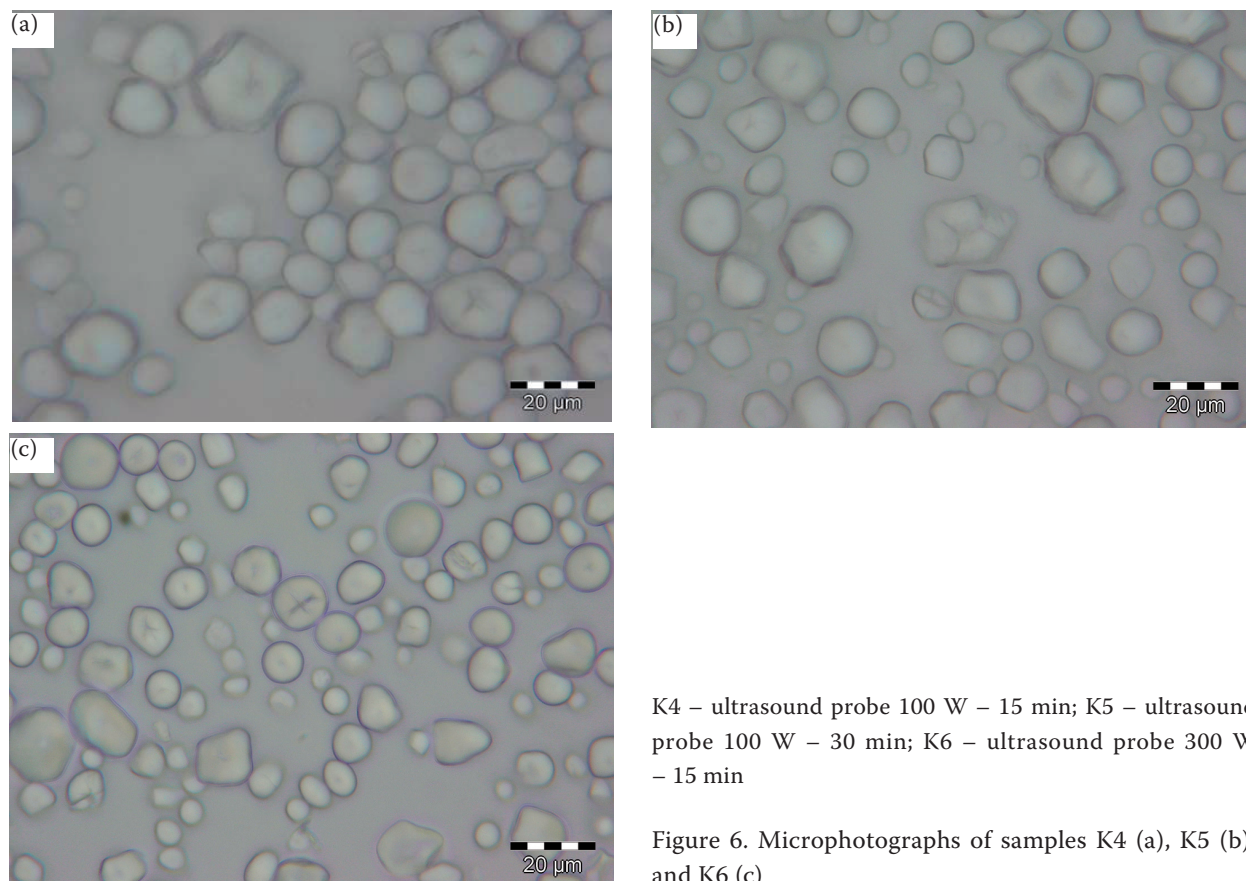
Sample	Hardness (N)	Adhesiveness (N/mm)	Cohesiveness	Springiness	Gumminess
K1	0.24 ± 0.12	0.113 ± 0.112	0.577 ± 0.101	0.917 ± 0.104	0.138 ± 0.104
K2	0.185 ± 0.15	0.099 ± 0.101	0.556 ± 0.103	0.917 ± 0.106	0.102 ± 0.103
K3	0.171 ± 0.14	0.096 ± 0.120	0.581 ± 0.108	0.913 ± 0.107	0.099 ± 0.107
K4	4.88 ± 0.18	0.173 ± 0.115	0.331 ± 0.105	1.019 ± 0.107	1.23 ± 0.101
K5	4.60 ± 0.14	0.562 ± 0.104	0.455 ± 0.110	1.032 ± 0.109	2.071 ± 0.101
K6	5.61 ± 0.13	0.183 ± 0.116	0.351 ± 0.111	1.250 ± 0.110	1.969 ± 0.111
K7	4.93 ± 0.20	0.861 ± 0.103	0.687 ± 0.109	1.005 ± 0.110	3.386 ± 0.112
K8	3.90 ± 0.11	0.256 ± 0.105	0.155 ± 0.106	1.002 ± 0.113	1.604 ± 0.106
K9	3.33 ± 0.15	0.632 ± 0.106	0.585 ± 0.105	1.002 ± 0.108	1.948 ± 0.103

K1 – untreated; K2 – ultrasound bath – 15 min; K3 – ultrasound bath – 30 min; K4 – ultrasound probe 100 W – 15 min; K5 – ultrasound probe 100 W – 30 min; K6 – ultrasound probe 300 W – 15 min; K7 – ultrasound probe 300 W – 30 min; K8 – ultrasound probe 400 W – 15 min; K9 – ultrasound probe 400 W – 30 min

Micrography of corn starch model systems

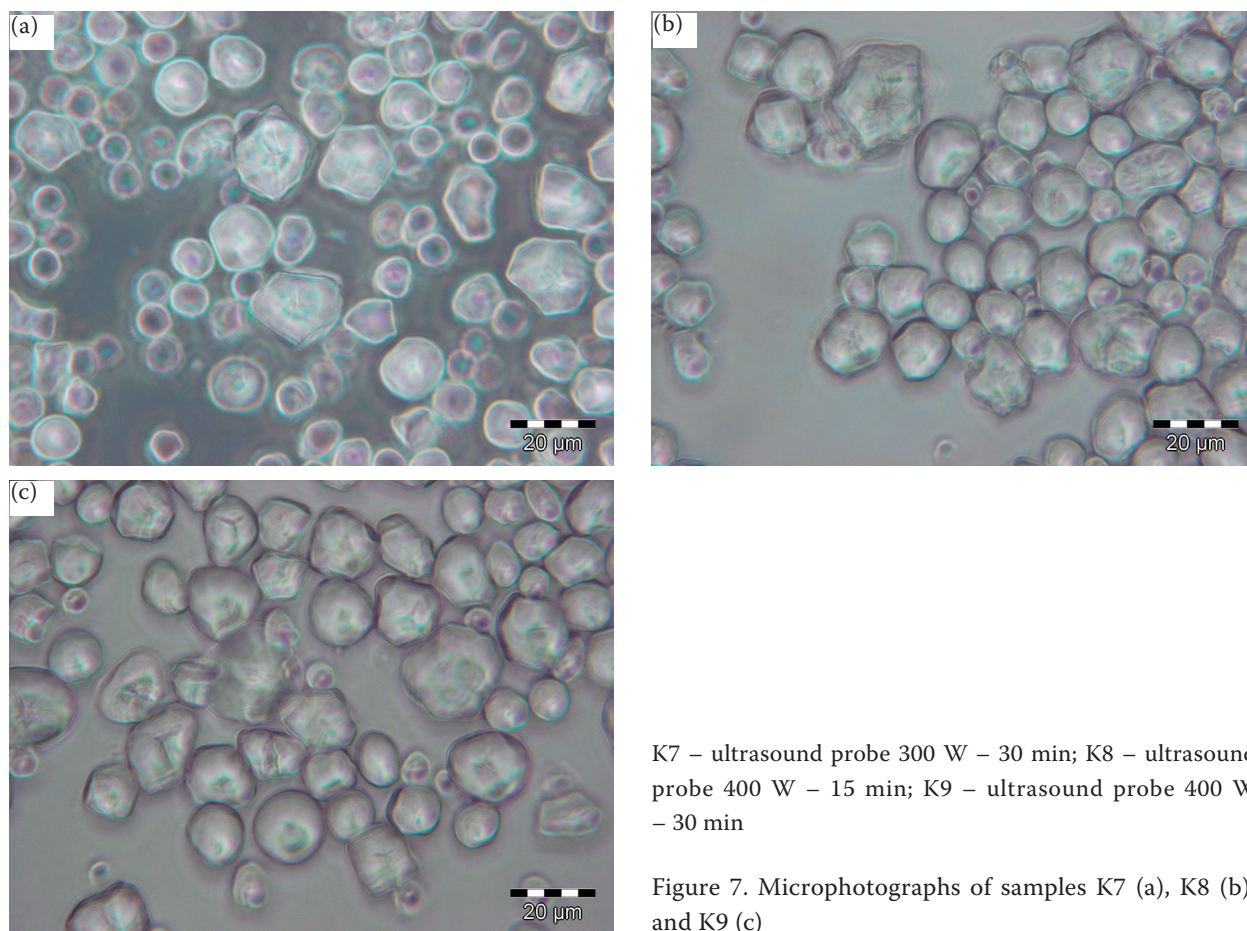
The micrography pictures are shown in Figures 5 to 7. The pictures reveal the impact of ultrasound on the structure and size of starch granules. The ultrasound treatment ruptures and mechanically damages the starch granules causing collapse of

cavitation bubbles which induces high pressure gradients and high local velocities of the liquid layers in their vicinity. In some treatments, when treated with ultrasound bath, the grains tend to agglomerate (K2 and K3). But when applying ultrasound probe with much higher powers, some smaller grains can be observed in the pictures.



K4 – ultrasound probe 100 W – 15 min; K5 – ultrasound probe 100 W – 30 min; K6 – ultrasound probe 300 W – 15 min

Figure 6. Microphotographs of samples K4 (a), K5 (b), and K6 (c)



K7 – ultrasound probe 300 W – 30 min; K8 – ultrasound probe 400 W – 15 min; K9 – ultrasound probe 400 W – 30 min

Figure 7. Microphotographs of samples K7 (a), K8 (b), and K9 (c)

This is caused by shear forces that are capable of breaking the chains of polymers and damaging the granules. Water is also partially decomposed into OH radicals and H atoms in the collapsing cavitation bubbles. Some of these radicals diffuse out of the cavities to the surrounding liquid and then react with the solute molecules causing the polymer degradation (CZECHOWSKA-BISKUP *et al.* 2005). It is widely known that the shape and size of starch granules vary (TESTER & KARKALAS 2002) with botanical source. From the pictures it is obvious that no significant changes occurred in the size of granules when the suspension was treated with ultrasound bath, but the size of granules slightly decreased and there were fewer smaller grains on applying ultrasound probes. The highest impact on the granules size occurred in the ultrasound process with 100 W and 300 W probes (K4, K5, and K6). With applying further power, starch granules tend to agglomerate acquiring larger shape and size due to the liberated bonds, providing the opportunity of connecting linkages between the polymers. Ultrasound results in changes in the starch granule size and in the slight decrease in

size observed and, consequently, in changes in the starch granules physical-chemical properties.

CONCLUSION

Ultrasound presents a novel technique that gives the possibility to change and improve some physical properties of food compounds. It can be effectively used in food industry in combination with elevated heat or pressure to obtain the desired food products. Starch is a component of the highest use in different products. Therefore, ultrasound can be applied in order to improve the physical properties and stability of products. In this research, ultrasound treatment caused disrupting of starch granules by cavitation forces and made the granule more permeable to water during the heating step. The highest viscosity was observed for K6 sample, for the treatment with 300 W probe that subsequently decreased with the increasing ultrasound power. Also, a statistically significant increase in the solubility in cold water was observed, caused by the disruption of starch

granules and molecules by ultrasound treatment. When applying more powerful ultrasound, starch granules become much more mechanically damaged specifically in the amorphous region. The profile of the starch gel texture prepared from the suspensions that had been treated with ultrasound probe presented a higher hardness and higher values of adhesiveness and cohesiveness when compared with the untreated or those treated suspension with ultrasound bath. Adhesiveness, cohesiveness, springiness, and gumminess are statistically much higher in what than in the untreated gels or those made from ultrasound treated suspensions with ultrasound bath. The highest values of the texture gel properties mentioned could be observed with ultrasound treated suspensions with 300 W probes (K6 and K7). Micrography showed an obvious impact of ultrasound on the structure and size of starch granules. The ultrasound treatment ruptures and mechanically damages the starch granules through collapse of cavitation bubbles which induces high pressure gradients and high local velocities of the liquid layers in their vicinity.

References

- CAMINO N.A., PEREZ O.E., PILOSOFF A.M.R. (2009): Molecular and functional modification of hydroxypropylmethylcellulose by high-intensity ultrasound. *Food Hydrocolloids*, **23**: 1089–1095.
- CZECHOWSKA-BISKUP R., ROKITA B., LOTFY S., ULANSKI P., ROSIAK J.M. (2005): Degradation of chitosan and starch by 360-kHz ultrasound. *Carbohydrate Polymers*, **60**: 175–184.
- DOUZALS J.P., MARECHAL P.A., COQUILLE J.C., GERVAIS P. (1996): Microscopic study of starch gelatinization under high hydrostatic pressure. *Journal of Agricultural and Food Chemistry*, **44**: 1403–1408.
- FREUDIG B., TESCH S., SCHUBERT H. (2003): Production of emulsions in highpressure homogenizers – Part II: Influence of cavitation on droplet breakup. *Engineering in Life Sciences*, **3**: 266–270.
- HAGENIMANA A., DING X. (2005): A comparative study on pasting and hydration properties of native rice starches and their mixtures. *Cereal Chemistry*, **82**: 72–76.
- HUANG Z.Q., LU J.P., LI X.H., TONG Z.F. (2007): Effect of mechanical activation on physico-chemical properties and structure of cassava starch. *Carbohydrate Polymers*, **68**: 128–135.
- IDA Y., TUZUUTI T., YASUI K., TOWATA A., KOZUKA T. (2008): Control of viscosity in starch and polysaccharide solutions with ultrasound after gelatinization. *Innovative Food Science and Emerging Technologies*, **9**: 140–146.
- KIM S.Y., WIESENBOEN D.P., ORR P.H., GRANT L.A. (1995): Screening potato starch for novel properties using differential scanning calorimetry. *Journal of Food Science*, **60**: 1060–1065.
- LEACH H.W., MCCOWEN L.D., SCHOCH T.J. (1959): Structure of the starch granule. Swelling and solubility patterns of various starches. *Cereal Chemistry*, **36**: 534–544.
- LIU H., BAO J., DU Y., ZHOU X., KENNEDY J.F. (2006): Effect of ultrasonic treatment on the biochemophysical properties of chitosan. *Carbohydrate Polymers*, **64**: 553–559.
- LIU C.Y., TSAI M.L., TSENG K.H. (1996): Effect of amylose content on the rheological property of rice starch. *Cereal Chemistry*, **73**: 415–420.
- MARGULIS M.A., MALT'SEV A.N. (1969): *Russian Journal of Physical Chemistry*, **43**: 055. (Translation of *Zhurnal Fizicheskoi Khimii*)
- MARGULIS M.A., MARGULIS I.M. (2003): Calorimetric method for measurement of acoustic power absorbed in a volume of a liquid. *Ultrasonic Sonochemistry*, **10**: 343–345.
- MILES J., MORRIS V.J., RING S.G. (1985): Gelation of amylose. *Carbohydrate Research*, **135**: 257–269.
- MUA J.P., JACKSON D.S. (1997): Relationships between functional attributes and molecular structures of amylose and amylopectin fractions from corn starch. *Journal of Agricultural and Food Chemistry*, **45**: 3848–3854.
- SINGH N., KAUR L., SINGH SANDHU K., KAUR J., NISHINARI K. (2006): Relationships between physicochemical, morphological, thermal, rheological properties of rice starches. *Food Hydrocolloids*, **20**: 532–542.
- SVEGMARK K., HERMANSSON A.-M. (1993): Microstructure and rheological properties of composites of potato starch granules and amylose: a comparison of observed and predicted structures. *Food Structure*, **12**: 181.
- TESTER R.E., KARKALAS J. (1996): Swelling and gelatinisation of oat starches. *Cereal Chemistry*, **73**: 271–277.
- THOMPSON L.H., DORAISWAMY L.K. (1999): Sonochemistry: science and engineering. *Industry Engineering and Chemistry Research*, **38**: 1215–1249.

Received for publication March 6, 2009

Accepted after corrections January 7, 2010

Corresponding author:

PhD ANET REŽEK JAMBRAK, University of Zagreb, Faculty of Food Technology and Biotechnology, Zagreb, Pierottijeva 6, Croatia
tel.: + 385 146 050 35, e-mail: arezek@pbf.hr