

Original Research Article

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Effect of Ultrasound Treatment on Physicochemical and Functional Properties of Cassava Starch

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ABSTRACT

Ultrasound is a non-thermal processing technique that offers the opportunity to modify the functionality of starch in terms of physicochemical and functional properties. The present work was aimed to study the effect of ultrasound treatment on physicochemical and functional properties of cassava tuber starch. The cassava starch extracted using conventional extraction technique was used in the present study. A 3 × 2 factorial design with three factors and two level, i.e., sonication temperature (40 and 50°C), sonication time (10 and 20 min) and solid-liquid ratio (1:1 and 1:2) was adopted in this study. A significant increase in solubility, swelling power, textural, pasting and rheological properties of the ultrasonicated starch was observed. A slight decrease in clarity of the sonicated cassava starch paste was observed compared to the control starch, but the differences were not much significant statistically. The whiteness of the sonicated cassava starch powder was lower compared to control native starch, but the differences were not statistically significant. Freeze-thaw stability of the treated cassava starches was found to be better compared to the control native cassava starch.

Keywords

Ultrasound, Cassava,
Modification of starch,
Functional properties, Pasting
properties, Rheological
properties, Textural properties

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Introduction

Tropical tuber crops are rich sources of starch. Cassava (*Manihot esculenta* Crantz) is one of the major tuber crops locally called as Tapioca and cultivated in tropical and subtropical regions of the world. It is the third largest source of carbohydrates after rice and wheat for people all over the world and the starch content of cassava tubers varies according to varieties. In India, it is cultivated about 0.20 Million hectares with a total production of 8.13 Million Tonnes and a productivity of 22.3 Metric Tonnes per hectare (India

Agristat, 2014). In India, cassava starch is a major industrial starch and a large portion of this used for sago production.

The native and different modified forms of cassava starch is used as a base material for an array of processed products viz., sago, dextrin, glucose, core binder, stabilizer, adhesives, sizing yarns and as thickener for printing clothes (Sheriff *et al.*, 2005). Processing of cassava starch is an easy process compared to that of cereal starches because of the presence of relatively small amount of non-starchy substances in the cassava tubers.

Cassava starch is immiscible in cold water and some of its natural properties limit its usefulness in many commercial applications (Jyothi *et al.*, 2011). The presence of amorphous and crystalline regions in the starch granules offers wide opportunity for modification of cassava starch (Moorthy, 2002). In order to make tailor-made starches suitable for specific end uses, modification is highly required. Modified starches are widely used in food, textile, paper and adhesive industries. Physical modification of starches are gaining more attention in recent years due to less amount of byproducts and chemical agents and makes this approach more sustainable and environment-friendly (Carmona *et al.*, 2016; Krishnakumar and Sajeev, 2017).

Ultrasound (US) is a sound waves having frequency above 20 kHz (beyond human hearing range) that passes in liquid medium creates cavitation. This mechanical action of cavitation with high velocity and shear force lead to high penetration in to cell membranes that causes cell disruption (Ahmad *et al.*, 2015). Ultrasound treatment (UT) is a physical method for modification of starch that has many advantages such as less usage of chemicals and processing time and environment - friendly processing (Krishnakumar and Sajeev, 2017; Monroy *et al.*, 2018). Ultrasound treatment (UT) is useful to modify the functionality of starch in terms of physico-chemical and functional properties (Luo *et al.*, 2008; Chan *et al.*, 2010; Jambrak *et al.*, 2010; Manchun *et al.*, 2012; Zheng *et al.*, 2013; Carmona *et al.*, 2016). To the best of our knowledge, only very limited studies have been reported on the effect of ultrasound treatment on the modification of functional properties of native cassava starch. Thus, the objectives of the present work were to study the influence of ultrasound on the physico-chemical and functional properties and correlation between the different properties of

native cassava starches under different sonication temperature, solid-liquid ratio and time.

Materials and Methods

Raw material

Matured cassava (*Manihot esculenta* Crantz) variety of Sree Pavithra collected from the CTCRI Research farm was used as raw material for the extraction of starch.

Extraction of starch

Cassava tuber starch was extracted from the freshly harvested cassava tubers using the standard procedure (Krishnakumar and Sajeev, 2017). The method used in the study was very similar to extraction of starch from sweet potato and arrowroot. The fresh tubers were thoroughly rinsed, manually peeled and were cut into small pieces using a motor operated chipping machine, then crushed in a mobile type starch extraction unit with supply of adequate water. The crushed starch milk was passed through a 150-mesh sieve and the resultant cassava starch milk was allowed to settle in a sedimentation tank for 12 hr. The settled cassava starch removed from sedimentation tank was washed with excess water for three times to obtain bright white colour. The starch was then dried in tray drier at 50°C for 12 h and stored under airtight condition with moisture content of 12 % (dry basis) for further ultrasound treatment.

Ultrasound treatment of cassava starch

Ultrasound treatment (US) of cassava starch was conducted as per the method reported by ying *et al.*, (2011), using a probe ultrasonicator (Sonic, Model: VCX750) operating frequency of 30 ± 3 kHz, input voltage of 230 V and heating strength of 750 W, attached with digital timer. The schematic

diagram of the probe type ultrasound treatment system used in the study is shown in Figure 1.

The aqueous cassava starch suspension obtained from the isolated cassava starch were treated with a constant ultrasound power of 750 W and 50 % amplitude during different sonication temperature (40, 50°C), sonication time (10, 20 min), solid-liquid ratio (1:1, 1:2 %). The temperature during the extraction was maintained by circulating water through the double-jacketed glass beaker. Ultrasonic probe of 19 mm diameter was directly placed in the suspension (cassava mash + distilled water) at a depth of 28 mm from the suspension surface and the desired amplitude (%) and extraction time (min) were maintained by means of digital amplitude and time controller. After the treatment, the pure starch was dried, powdered using pestle and mortar, sieved through standard BSS 100 mesh sieve and then stored in airtight container for further analysis.

Solubility and swelling power

Solubility index (%) of the cassava starch was determined using Ding *et al.*, (2006). The 2.5 g of cassava starch was weighed into 50 ml centrifuge tube and heated in 30 ml distilled water in a water bath at 60°C for 30 min without mixing and then centrifuged at 3000 rpm for 10 min. The supernatant was dried at 105°C to constant weight and the weight of the dry solids was measured. All the experiments were made in triplicate. The following equation used to calculate the solubility index.

$$\text{Solubility index (\%)} = \frac{m_s}{m_d} \times 100 \quad (1)$$

Where,

m_s - Weight of soluble starch (g)

m_d - Weight of starch sample on dry basis (g)

Swelling power (g/g) was determined by modified method of Betancur *et al.*, (2001). 2.5 g of the ultrasonicated cassava starch sample was weighed into 50 ml centrifuge tube. Then 30 ml of distilled water was added and mixed gently. The sample was heated in a water bath at 60°C for 30 min and centrifuged at 3000 rpm for 10 min. The supernatant was decanted immediately after centrifuging. The weight of the sediment was taken and recorded.

$$\text{Swelling power (g/g)} = \frac{\text{Weight of sedimented starch paste (g)}}{\text{Weight of starch sample on dry basis} \times (100 - \% \text{ solubility})} \times 100 \quad (2)$$

Paste clarity

Paste clarity of the ultrasonicated (US) cassava starch was measured according to the procedure described by Sandhu and Singh (2007). Aqueous starch suspension containing 1% starch was prepared by heating 0.2 g starch in 20 ml water in a shaking water bath at 90°C for 1 h. The starch paste was cooled to room temperature, and the transmittance was measured at 640 nm in a spectrophotometer (Spectra scan uv-2600, Thermo fisher scientific, India).

Colour of starch

The colour of the US starch sample was analyzed using a colorimeter (Hunter Lab, Virginia). The primary colour parameters 'L', 'a', 'b' were measured by placing samples in the sample holder. The 'L' parameter represent light dark spectrum with a range from 0 (black) to 100 (white), 'a' represents green red spectrum ranging from -60 (green) to +60 (red) and 'b' represents blue yellow spectrum with a range from -60 (blue) to +60 (yellow) dimensions respectively.

Freeze-Thaw stability

Freeze-thaw stability was determined according to the method of Singhal and

kulkarni (1990). Ultrasonicated (US) starch at a concentration of 5% (w/v) was heated in distilled water at 95°C for 30 min with constant stirring. Ten milliliters of paste was transferred into the weighed centrifuge tube. This was subjected to alternate freezing and thawing cycles (22h freezing at -20°C followed by 2 h thawing at 30°C) for 3 days and centrifuged at 5000×g for 10 min after each cycles. The percentage (%) of syneresis was then calculated as the ratio of the weight of the liquid decanted and the total weight of the gel before centrifugation multiplied by 100. Totally three freeze-thaw cycles were conducted for each sample.

Pasting properties of starch

The viscosity of control and ultrasound treated samples was determined using a rapid visco analyzer (RVA-4, Newport Scientific, and Warriewood, Australia). The powdered sago sample (2.5 g dry weight) was accurately weighed out into the aluminum canister and distilled water (25 g) was added and mixed well. The canister was placed in the RVA unit and the heating/cooling cycle was performed according to Standard I profile. The slurry was heated from 50 to 95°C at 12°C/min and held at 95°C for 2 min. The paste was cooled to 50°C at 12°C/min and finally maintained at 50°C for 2 min. The parameters such as peak viscosity, breakdown viscosity, setback viscosity in terms of centipoises (cP) and pasting temperature (°C) were measured. The breakdown ratio was calculated as the ratio of breakdown to peak viscosity.

Dynamic rheological properties of starch

In dynamic oscillation measurements, the potential energy and the energy that is dissipated as heat may be separated into storage modulus and loss modulus, respectively. Storage dynamic modulus (G') is a measure of the energy stored in the material

and recovered from it per cycle while the loss modulus (G'') is a measure of the energy dissipated or lost per cycle of sinusoidal deformation (Ferry, 1980). The ratio of the energy lost to the energy stored for each cycle may be defined by $\tan \delta$, which is another parameter indicating the physical behaviour of a system. In dynamic oscillation measurements, the frequency sweep of the two moduli (G' and G''), may be used to distinguish between the elastic and viscous properties of a material over a spectrum of times. When the viscous properties dominate, G'' exceeds G' , and *vice versa* ($G' > G''$) when the elastic properties prevail.

The dynamic rheological properties (storage modulus, loss modulus and phase angle) for the 10% (w/v) gel suspension of the cassava control and ultrasonicated starch samples obtained from RVA studies were determined using Rheoplus MCR 51 Rheometer (M/s Anton Paar GmbH, Germany) at 30°C, using a parallel plate geometry system (PP20-SN5912, 1mm diameter) at 1 mm gap. The following experimental conditions were selected: frequency 1 to 10 Hz; strain of 1 per cent (%). Fifteen measuring points were recorded for each experiment. For each sample, storage modulus (G'), loss modulus (G'') and phase angle (δ) were recorded and the measurements were conducted at least in duplicates.

Textural analysis

Textural properties (cohesiveness, consistency, firmness) of the cassava starch were determined by using Texture Analyser (TA.XT plus, Stable Micro Systems, UK) equipped with a 50 kg load cell. A probe adapter was used to connect the compression plate to the movable bar. Stainless steel cylindrical probe (P/35) attached in the movable bar was used. The following experimental conditions were adopted for the

analysis, Option-return to start, Pre-test speed: 2mm/s, Test speed: 2mm/s, Post-test speed: 2mm/s, Distance: 8 mm, Force: 10 g, Time: 5s, Data acquisition rate: 200 pps. Starch paste sample was prepared at a concentration of 5% (w/v) for measuring the textural properties. The sample was compressed to 80 % of their original height, a cross head with speed of 2 mm/s twice in two cycles. The aqueous cassava starch suspension at a concentration of 10 % (w/v) was prepared.

Experimental design and statistical analysis

A three factor two level full factorial design was employed to study the effect of ultrasound treatment on the physico-chemical and functional properties of cassava starch. The factors selected were sonication temperature (40, 50°C), sonication time (10, 20 min) and solid-liquid ratio (1:1, 1:2). A total of nine experiments with three replications were conducted including control (Table 1). Data analysis was performed using R software. Factorial completely randomized design (FCRD) was applied for all the statistical analysis. Analysis of variance (ANOVA) and pair-wise mean comparison were performed to determine the significant effect of the independent variables on the response variables. The treatments and their interactions were compared at $p < 0.05$ level using least significant difference (LSD) method.

Results and Discussion

Effect of sonication on solubility and swelling power of cassava starch

The solubility and swelling power of the ultrasonicated cassava starch samples increased significantly compared to control sample (Table 2). The treatments were found to be statistically significant except for solubility at Sonication time for 10 min (T₂) and swelling power at T₂, T₄, and T₆, which

might be due to lower sonication time and solid-liquid ratio, which resulted in a lesser degradation of starch molecules. This is due to the role of ultrasound makes starch molecules to increase solubility, particle content easy expansion, thereby weakening the reflection and refraction of light (Singh *et al.*, 2003; Lida *et al.*, 2008; Luo *et al.*, 2008; Jambrak *et al.*, 2010; Manchun *et al.*, 2012). Similar increase in starch swelling and solubility due to ultrasonication has been reported by Zheng *et al.*, (2013) for sweet potato starch and by Yu *et al.*, (2013) for non-waxy rice starch. The solubility and swelling power increased when temperature and solid-liquid ratio of ultrasound treatment was less. This might be due to the fact that with increase in temperature, the degradation of starch was more, and some degraded soluble starch might have been lost during recovery. Similar trend was also observed with solid liquid ratio and evinced that some soluble starch might have been lost during treatment with high solid liquid ratio.

Effect of sonication on paste clarity of cassava starch

The major factors affecting the clarity of starch paste includes distribution of different starch size particles and the proportion of amylose and amylopectin (Sitohy *et al.*, 2000). The clarity of the pastes prepared using ultrasonicated starch samples was observed to be lower compared to control but was not much statistically different (Table 2). The result showed that the clarity of the natural cassava starch was 21.42 %, but the clarity of ultrasonicated starch was decreased with increase in sonication temperature, time and solid-liquid ratio. The results were not in agreement with the results obtained by Jambrak *et al.*, (2010) for corn starch, Sujka and Jamroz (2013) for potato starch and Zheng *et al.*, (2013) for sweet potato starch. It can be seen that clarity decreased with

increase in time and temperature as well as solid-liquid ratio. This might be due to highest disintegration of the starch. The starch granules with more extraction time, solid-liquid ratio and higher temperature which allowed more water to be absorbed and thereby led to more viscous starch paste, thus decreasing the transmittance. It could also be due to disintegration of starch molecules by ultrasound made some bonds to be weaken which might allowed some impurities with the starch molecules, thus decreasing the clarity of starch paste.

Effect of sonication on colour of the starch powder

Colour is an important parameter to estimate starch quality. The variation of colour “L” value of ultrasonically treated cassava starch with respect to different sonication time, temperature and solid liquid ratio is presented in Table 3. The colour variation in L* value was found to be non-significant with control. The colour values increased with increase in the ultrasound treatment time and temperature. This might be due to the long exposure time leading to rapid hydration action hence leading to change in colour. This result was not in agreement with the study conducted by Nadir *et al.*, (2015) that potato starch samples modified by ultrasonication methods had lower values of lightness (L) value when compared with native starch. The “L” value of ultrasonically extracted starch ranged from 92.57 to 95.82, while those of conventionally extracted starch were 94.36. The lightness value greater than 90 confirms the purity of starch (Perez Sira and Amaz, 2004). Ultrasonically extracted starches also exhibited more redness and yellowness compared to control starch. This might be attributed to the disintegration of starch molecules during ultrasonication which might have allowed some impurities to bond with the starch molecules, thereby reducing the

whiteness and increasing the redness and yellowness.

Effects of sonication on freeze-thaw stability of the cassava starch paste

Syneresis (%) is used to find out the freeze-thaw stability of starches to withstand the undesirable physical changes during freezing (Adebowale *et al.*, 2005). Starch paste after freeze and thawing occur syneresis phenomenon, which is due to the freezing of starch paste. The percent syneresis of the starch gels measured by ultrasound treatments was found to be substantially lower than that of the control starch gel during all the three freeze-thaw cycles (Table 4). Moreover, ultrasonically extracted starches were observed to be more stable under repeated freeze-thaw cycles as less difference was noticed in percent syneresis between the freeze-thaw cycles. Because of the ultrasonic treatment, gel structure is destroyed, resulting in precipitation of free water, breakage of starch chains in the amorphous region caused extensive reordering of the chain segments. Therefore, amount of water expelled during thawing was comparatively less in ultrasonicated starches compared to that in control sample as due to breakage and reordering, a greater number of hydrophilic bonds were exposed which could hold more water during thawing, thereby reducing syneresis. This result was in agreement with the study conducted by Luo *et al.*, (2008) on ultrasonically treated maize starch. A similar observation on freeze-thaw behavior of ultrasonically treated maize starch was made by Hu *et al.*, (2014). Native starch retrogradation is considered to be unacceptable for many food applications (Nwokocha *et al.*, 2012; Collar and Rosell 2013; Wang *et al.*, 2015). Better freeze-thaw stability of the sonicated cassava starch is more acceptable for food application, meant for refrigeration.

Table.1 Treatment combinations at different ultrasound conditions

S. No	Treatment	Temperature, °C	Time, min	Solid-Liquid ratio, g/mL
1.	Control	0	0	0
2.	T ₁	40	10	1:1
3.	T ₂	50	10	1:1
4.	T ₃	40	20	1:1
5.	T ₄	50	20	1:1
6.	T ₅	40	10	1:2
7.	T ₆	50	10	1:2
8.	T ₇	40	20	1:2
9.	T ₈	50	20	1:2

Table.2 Effect of different ultrasonic treatments on solubility and Swelling power of the cassava starch

Treatments	Solubility (%)	Swelling power (g/g)	Clarity (% T)
Control	28.20±0.18 ^g	7.52±0.12 ^f	21.42±1.10 ^a
T ₁	30.36±0.23 ^b	8.67±0.15 ^c	21.27±1.11 ^a
T ₂	28.45±0.31 ^g	7.85±0.22 ^f	21.14±0.95 ^b
T ₃	31.26±0.35 ^a	9.62±0.05 ^a	21.38±0.82 ^a
T ₄	28.96±0.28 ^d	7.69±0.14 ^f	19.56±0.15 ^c
T ₅	29.56±0.16 ^c	7.42±0.12 ^e	20.05±0.64 ^d
T ₆	28.98±0.14 ^e	7.33±0.13 ^f	19.45±0.23 ^c
T ₇	30.44±0.18 ^b	7.75±0.12 ^d	20.15±0.32 ^d
T ₈	28.66±0.20 ^f	7.94±0.18 ^b	19.35±0.56 ^c

Values are mean ± standard deviation of three replications. Means followed by same letters in the superscript were found not significantly different at p>0.05.

Table.3 Effect of different ultrasonic treatments on colour of the cassava starch

Treatments	L value	a value	b value
Control	94.36±1.24 ^a	0.23±0.04 ^f	5.94±0.05 ⁱ
T ₁	92.57±1.56 ^a	0.89±0.03 ^b	7.17±0.04 ^b
T ₂	93.39±0.98 ^a	0.56±0.02 ^d	6.46±0.03 ^e
T ₃	93.27±1.57 ^a	1.24±0.05 ^a	7.34±0.04 ^a
T ₄	95.82±2.12 ^a	0.59±0.03 ^d	6.89±0.01 ^c
T ₅	95.26±1.32 ^a	0.81±0.04 ^c	6.21±0.02 ^g
T ₆	95.32±2.03 ^a	0.52±0.02 ^e	5.91±0.04 ^h
T ₇	95.18±1.26 ^a	0.84±0.01 ^c	6.36±0.03 ^f
T ₈	94.97±1.05 ^a	0.75±0.02 ^d	6.85±0.02 ^d

Values are mean ± standard deviation of three replications. Means followed by same letters in the superscript were found not significantly different at p>0.05.

Table.4 Freeze-thaw stability of cassava starch samples under different treatments

Treatments	First cycle	Second cycle	Third cycle
Control	29.15±1.12 ^l	27.21±1.37 ^l	24.36±1.55 ^k
T ₁	17.24±1.32 ^{hj}	16.37±1.22 ^{gh}	15.27±1.35 ^{df}
T ₂	19.36±1.57 ^j	18.34±1.26 ^{ij}	15.32±1.62 ^{eh}
T ₃	13.37±2.01 ^{bc}	13.89±1.53 ^{bc}	12.45±1.95 ^{ac}
T ₄	15.28±1.68 ^{eh}	14.67±1.84 ^{ce}	14.83±1.54 ^{ce}
T ₅	16.88±1.39 ^{hj}	11.32±1.67 ^{ab}	10.57±2.16 ^a
T ₆	14.86±1.83 ^{ce}	14.83±1.52 ^{ce}	13.38±2.17 ^{bc}
T ₇	15.94±2.04 ^{eh}	13.27±1.68 ^{ad}	12.53±2.14 ^{ac}
T ₈	16.19±2.11 th	15.53±2.16 ^{eh}	14.88±2.18 ^{ce}

Values are mean ± standard deviation of three replications. Means followed by same letters in the superscript were found not significantly different at p>0.05.

Table.5 Textural Properties of sonicated cassava starch gel

Treatments	Cohesiveness (g)	Consistency (g/s)	Firmness (g)
Control	8.92±0.03 ^c	145.36±1.12 ^d	10.54±0.06 ^e
T ₁	8.97±0.05 ^b	153.91±0.15 ^c	12.31±0.02 ^c
T ₂	8.80±0.15 ^d	156.86±1.32 ^a	12.53±0.15 ^b
T ₃	8.34±0.12 ^f	152.59±1.42 ^c	12.01±0.04 ^d
T ₄	9.06±0.09 ^a	153.79±0.83 ^c	12.64±0.08 ^b
T ₅	8.57±0.05 ^e	152.10±1.13 ^c	12.11±0.14 ^d
T ₆	8.91±0.13 ^c	153.03±0.92 ^c	12.52±0.06 ^b
T ₇	8.65±0.05 ^e	158.39±1.16 ^a	13.01±0.05 ^a
T ₈	8.96±0.02 ^b	154.32±1.45 ^b	12.52±0.11 ^b

Values are mean ± standard deviation of three replications. Means followed by same letters in the superscript were found not significantly different at p>0.05.

Table.6 Pasting profile of starch pastes under different ultrasonic treatment conditions

Treatment	Peak viscosity (cP)	Holding viscosity (cP)	Breakdown viscosity (cP)	Final viscosity (cP)	Setback viscosity (cP)	Pasting Temp (°C)
Control	2938±45 ^f	1273±28 ^f	1665±14 ^b	2190±19 ^g	917±18 ^f	71.15±0.06 ^f
T ₁	3363±40 ^c	1785±19 ^b	1578±11 ^d	2878±36 ^c	1093±15 ^d	72.35±0.03 ^b
T ₂	3232±41 ^e	1731±18 ^c	1501±16 ^e	2915±30 ^b	1184±12 ^b	72.35±0.01 ^b
T ₃	3729±38 ^a	1865±17 ^a	1864±19 ^a	3113±42 ^a	1248±09 ^a	71.85±0.04 ^d
T ₄	3584±24 ^b	1743±11 ^c	1843±12 ^a	2888±32 ^b	1145±08 ^c	71.95±0.05 ^c
T ₅	3339±21 ^d	1736±31 ^c	1605±14 ^c	2833±38 ^c	1097±14 ^d	72.75±0.02 ^a
T ₆	3276±24 ^e	1718±23 ^c	1560±17 ^d	2737±19 ^d	1019±17 ^e	71.55±0.03 ^e
T ₇	2741±38 ^h	1486±20 ^d	1257±20 ^f	2386±24 ^e	900±11 ^g	70.75±0.01 ^h
T ₈	2861±37 ^g	1371±18 ^e	1492±12 ^e	2261±36 ^f	891±15 ^g	71.05±0.02 ^g

Values are mean ± standard deviation of three replications. Means followed by same letters in the superscript were found not significantly different at p>0.05.

Fig.1 Schematic diagram of the probe ultrasound treatment system

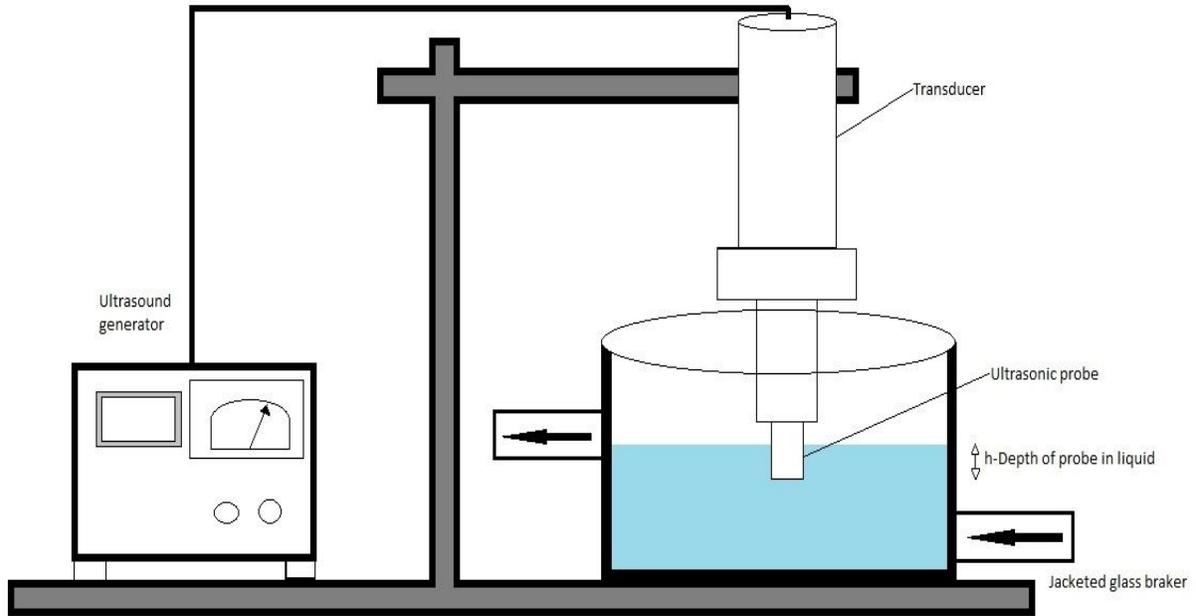


Fig.2 Pasting profiles of starch paste under different ultrasound treatments

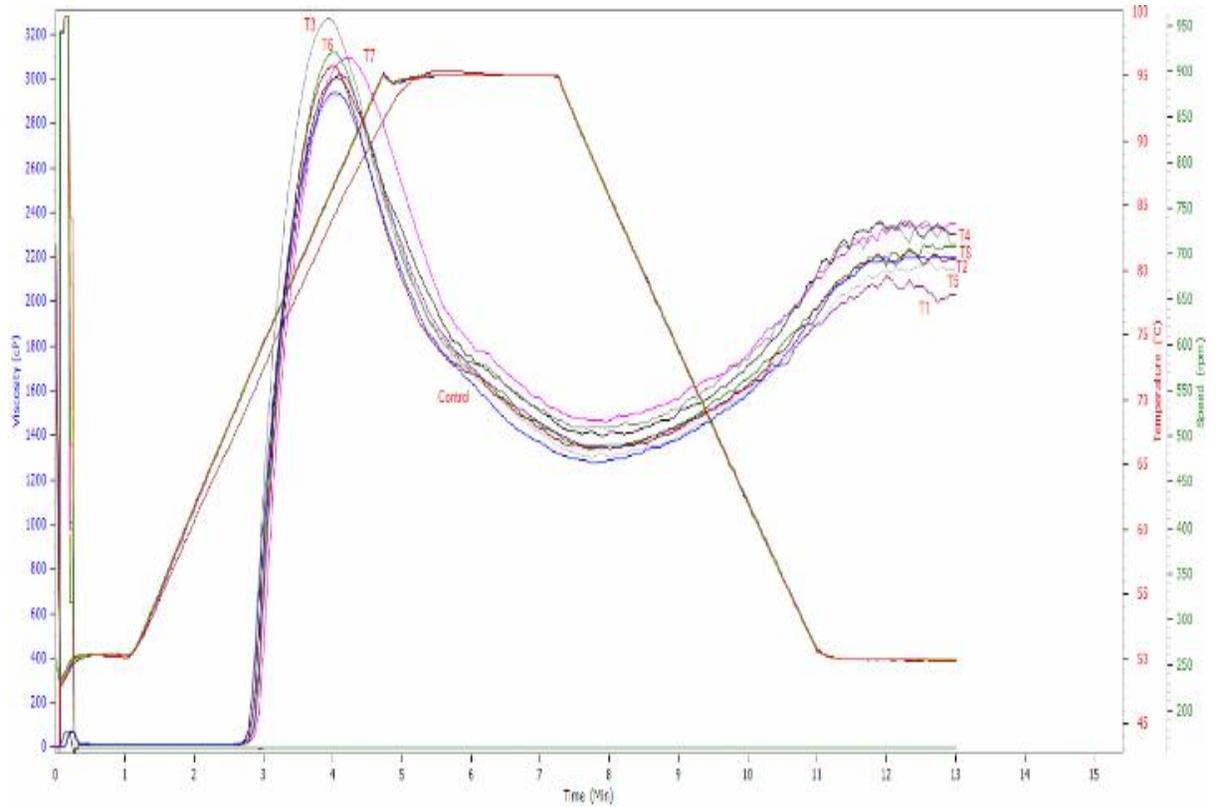
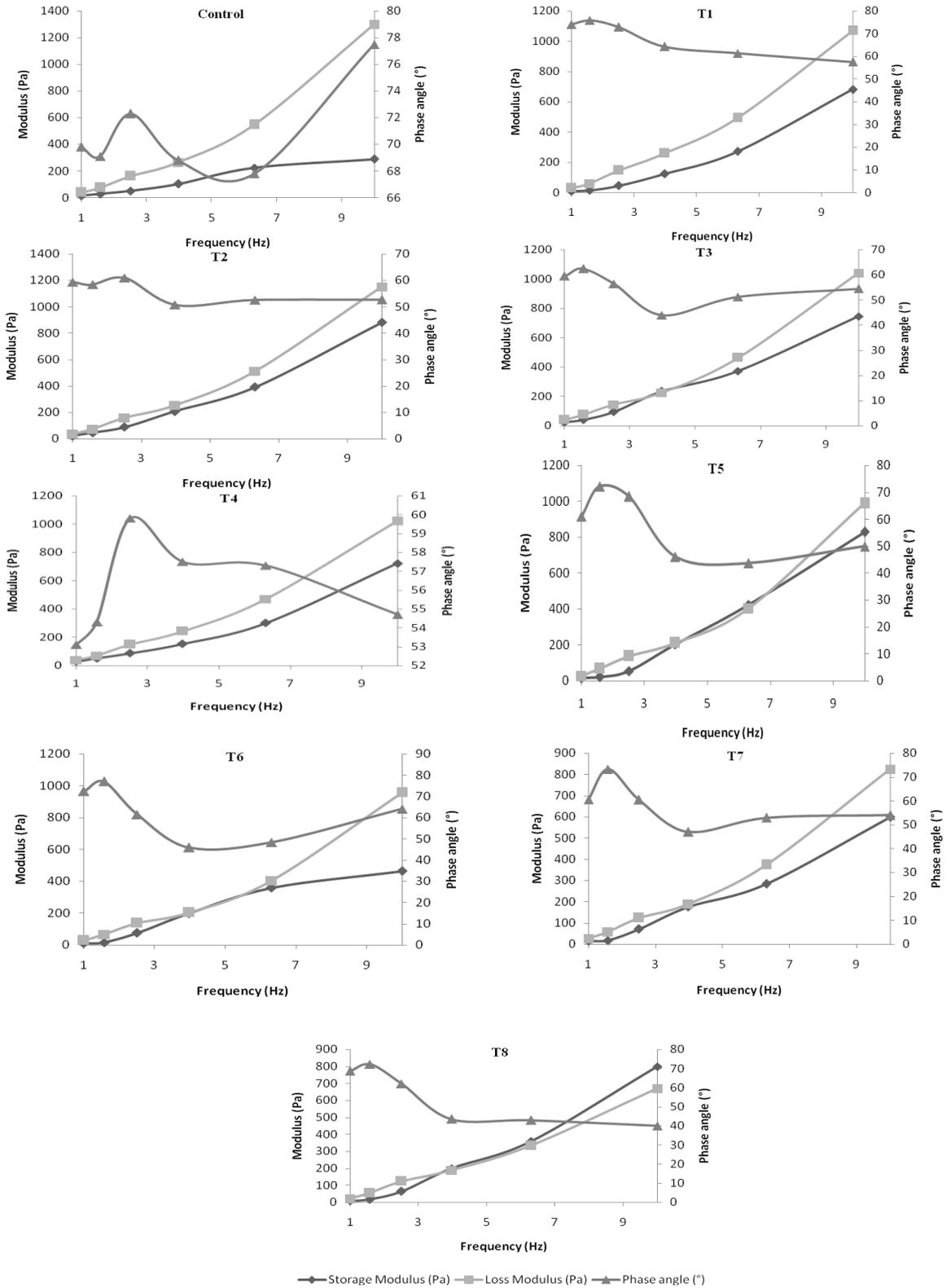


Fig.3 Dynamic moduli of the pastes (10%) of native and treated cassava starch



Effect of sonication on textural properties of cassava starch paste

The textural properties of the cassava starch paste were determined using a texture analyzer and the results are presented in Table 5. The consistency and firmness of the sonicated cassava pastes varied from 152.10 to 156.86 g/s and from 12.01 to 13.01 g, respectively and were significantly higher compared to the control sample with consistency of 145.36 g/s and firmness of 10.54 g. The increase of consistency and firmness of the sonicated cassava starch pastes might be ascribed to maximum swelling of the starch granules due to partial disruption of the starch structure. Additionally, this might be due to mechanical vibration, thermal and ultrasonic cavitation effects, the intramolecular hydrogen bonding of cassava starch can be broken; thus its molecular structure becomes loose and molecular winding nodes are reduced (Luo *et al.*, 2003). This result was in agreement with the study conducted by Herceg *et al.*, (2010) and Nie *et al.*, (2017). The increase in consistency and firmness of cassava starch pastes might be beneficial for developing certain starchy foods.

Effect of sonication on pasting properties of cassava starch paste

The pasting profile of the native and treated starches is shown in Table 6. The peak viscosity, holding viscosity, final viscosity, setback viscosity and pasting temperatures of the ultrasonicated cassava starch were significantly higher compare to that of control, while the breakdown viscosity was found to be lower. This result was not in agreement with the results obtained by Wang and Wang (2004) and Zuo *et al.*, (2009) for rice starch and Luo *et al.*, (2008) for maize starches where the viscosities decreased. Moreover this result was in accordance with

those obtained by Zhang *et al.*, (2005) for corn starch. The increase in pasting temperature indicates that starch granules after ultrasound treatment continue to swell at higher temperatures compared to control before further disruption of the granular structure. Increased values of peak viscosity, holding viscosity, set back viscosity and final viscosity implicated that the starch paste obtained after ultrasonic treatment could be more resistant to shear and formed rigid gel after cooling. The functionality of starch granules was significantly influenced by sonication time and temperature. It was observed that the peak, holding, final viscosities were more when ultrasound treatment time was more as disruption of starch structure could be more which increased swelling and thus viscosity also increased (Zuo *et al.*, 2009). A maximum viscosity of 3113 cP was reported for treatment T₃ (40°C, 1:1 solid-liquid (SL) ratio and 20 min) (Fig. 2). Peak viscosity was found to increase with increase in sonication time but decreased with increase in temperature. This might be attributed to a loss of some soluble portion of starch during extraction at high temperature, contributed for change in viscosity. On cooling, the final viscosity of starch pastes decreased due to retrogradation. A strong correlation was obtained between the pasting profile and swelling power and solubility of cassava starches.

Effect of sonication on rheological properties of cassava starch paste

The changes in viscoelastic properties of starch paste were determined using aforementioned rheometer equipped with parallel-plate geometry (PP-20). The dynamic rheological properties viz. Storage modulus (G') and Loss modulus (G'') and Phase angle (δ) of ultrasound treated cassava starch samples are presented in Figure 3. The

storage modulus (G') and loss modulus (G'') of the treated starch pastes varied from 8.35 to 881 Pa and from 21.2 to 1150 Pa, respectively and storage modulus was significantly higher and loss modulus was significantly lower compared to the control sample with observed maximum storage modulus of 289 Pa and loss modulus of 1300 Pa (Fig. 3). The storage and loss modulus of both treated and control starch samples increased with increase in frequency for all samples. The starch paste of control sample behaved as dilute solution compared with treated samples, which was evident from the comparatively higher loss modulus than storage modulus ($G' < G''$) as varied with frequencies. This is also confirmed from the higher phase angle (δ) values. Finally, the treated produced cassava starch pastes with reduced elastic properties. This result was in accordance with the study conducted by Bustillos *et al.*, (2018) for corn starches.

In the present study, treatment of native cassava tuber starch using power ultrasound significantly affected the physicochemical and functional properties compared to non-treated control cassava starch. A higher increase in solubility and swelling power was found in cassava starch treated with ultrasound. Lower temperature, higher time and lower solid-liquid ratio was found to be better for altering the functional properties of starch, whereas higher temperature, lower time yielded comparatively less changes in the functional properties of cassava starch. The clarity of the treated cassava starch was found to be slightly lower compared to the control. The increase in freeze-thaw stability and change in textural, pasting and rheological properties of the starch paste, suggest that starch treated with ultrasound can be used in products subjected to refrigeration, which indeed requires high viscosity. Therefore, the overall results in this study showed that ultrasound treatment could be

effectively used to modify starch granules, although the changes produced are dependent on time and temperature.

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