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Effect of ultrasound on isolation and properties of oat starch

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Abstract: The aim of the present study was to analyse the effect of ultrasound (US) on the isolation and properties of oat starch. Starch isolation was done by a water extraction method and with sonication for 30 (US30), 45 (US45) and 60 (US60) min. The highest starch yield was found in US45 as 49.04 g·100 g^{-1} of oat flour when the extraction time decreased from 6 h to 45 min. The functional properties of starches, such as bulk density, water solubility index, dispersibility and water absorption capacity, ranged from 0.65 to 0.80 g·mL^{-1}, 3.22 to 5.75%, 78 to 87.5% and 84.23 to 95.87%, respectively. US45 had lower bulk density and higher dispersibility than other starches. US treatments decreased the gelatinisation temperature ranges and increased the gelatinisation enthalpy values. The enthalpy of the gelatinisation value of oat starch was found as 8.45 J·g^{-1} and increased with sonication up to 13.65 J·g^{-1}. Retrogradation endotherms were observed after 6 days of storage, and enthalpies of retrogradation were lower than in the gelatinised starches. Fourier transform infrared spectroscopy (FTIR) spectra showed that US treatment did not affect the functional groups of oat starch. US application during oat starch isolation gave desired results, such as time reduction, higher yields and increased functional properties.

Keywords: colour; retrogradation; starch dispersibility; starch gelatinisation

Oat (*Avena sativa* L.) grain is a good source of starch, protein, phenolic compounds, unsaturated fatty acids, and dietary fibre (Ovando-Martínez et al. 2013). Unfortunately, it is little used in human nutrition and is used primarily as animal feed. Starch is the main component of oat grains, accounting for almost 60% of their weight. Oat starch exhibits specific properties such as granule size, morphology, crystallinity, lipid content and fine structure of amylose and amylopectin components (Berski et al. 2011; Kasturi and Bordenave 2013).

The high fat and β-glucan content of oats reduces the efficiency of starch isolation. To increase the effectiveness of starch isolation, chemicals are often used, and the extraction time is increased. Ultrasound (US) has been one of the non-thermal methods used for

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food production in recent years. The primary purpose of US technology is to decrease processing time, save energy, and improve food products' quality and shelf life (Ercan and Soysal 2013). US-assisted extraction has gained interest in the food industry as an efficient extraction method.

In this study, the effect of sonication on oat starch isolation was investigated. As far as we know, the use of US in oat starch isolation has yet to be described in the literature. Falsafi et al. (2019) investigated the effects of sonication to produce physically modified oat starch, but they did not use sonication for starch isolation. The application of US in the extraction of oat starch and examining its effect will be brought to the literature for the first time with this study.

MATERIAL AND METHODS

Commercially obtained dehulled oat was first milled by using a hammer mill to obtain oat flour. The obtained flour was sieved manually with a 0.425 mm sieve and used in this study. The chemicals used were all reagent grade and purchased from Merck (Merck SA, Merck KGaA, Germany) or Sigma-Aldrich (Germany).

Starch extraction

Oat starch was extracted with water by modification of the method of Lim et al. (1992). Oat flour was mixed with distilled water, kept for 6 h at 20 °C and then centrifuged (Benchtop Centrifuge 5810 R; Eppendorf, Germany) for 15 min at 3 200 g. Distilled water was added to the precipitate (starch fraction), and the mixture was filtrated through bolting cloth to separate the cell wall debris from starch. Obtained starch milk was centrifuged for 15 min at 3 200 g, and the precipitate was dried at 40 °C overnight to obtain oat starch. Starch obtained by this method is referred to as control throughout the manuscript.

Ultrasound-assisted starch extraction

Oat starch was extracted with the same method as explained above, but in this case, instead of waiting for 6 h, US was applied for 30, 45 and 60 min. US was applied using an Ultrasonic Cleaner (Branson 2200; Emerson, USA) with a working frequency of 47 kHz and an output power of 60 W. All extractions were performed at least in the duplicate run. The extraction yield of oat starch was calculated on a dry weight basis as the ratio of the dry weight of oat starch to the dry weight of oat flour and multiplied by 100.

Colour attributes

Colour measurements of starch powders (*L*, *a*, and *b* values) were made using a HunterLab ColorFlex (A60-1010-615 Model Colorimeter, HunterLab, USA). The Whiteness index (*WI*) was calculated using Hunter *L*, *a*, and *b* values as follows:

$$\mathrm{WI} = 100 - \left[\left(100 - L\right)^2 + a^2 + b^2\right]^{1/2} \quad (1)$$

where: *L* – lightness; *a* – greenness (–*a*) / redness (+*a*); *b* – blueness (–*b*) / yellowness (+*b*).

Functional properties of oat starch

The functional properties of oat starches were determined by using standard methods such as bulk density, water solubility index (*WSI*) (Nishad et al. 2017), dispersibility and water absorption capacity (*WAC*) (Onabanjo and Ighere Dickson 2014).

Bulk density (BD). Starch samples were filled into 5 mL, weighed and tared measuring cylinder tubes by constant tapping until there was no further change in volume. The measuring cylinders with samples were weighed, and the bulk density ($g \cdot mL^{-1}$) was calculated as the weight of the sample divided by the volume occupied by the sample.

Water solubility index (WSI). Starch samples (1 g) were suspended with 10 mL of distilled water in 45 mL weighted centrifuge tubes. Then they were stirred well for 30 min in a water bath at 30 °C. After stirring, the samples were centrifuged at 1 100 g for 15 min. The supernatants were poured into dry evaporator dishes of known weight and stored overnight at 120 °C for evaporation. *WSI* was calculated as the weight of the solid in a supernatant divided by the weight of the dry sample in the original sample, the result was multiplied by 100.

Starch dispersibility. 5 g of the starch sample was placed in a 50 mL measuring cylinder and completed with distilled water. Mixtures in the cylinder were stirred vigorously, followed by three-hour waiting to obtain sedimentation. The volume of the settled particles was determined. Starch dispersibility was calculated by multiplying this volume by 2 and subtracting the result from 100 to represent per cent dispersibility.

Water absorption capacity (WAC). 15 mL of distilled water was added to 1 g of the starch in a weighed 45 mL centrifuge tube. The tube was stirred for 2 min with a vortex. Then it was centrifuged for 20 min at 2 000 g. The clear supernatant was discarded, and the centrifuge tube was reweighed. The weight of wa-

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ter bound by 100 g of the dried sample is expressed as the water absorption capacity (Onabanjo and Ighere Dickson 2014).

Determination of thermal properties

The thermal properties of starches were determined using a differential scanning calorimeter (DSC Pyris 6; PerkinElmer, Netherlands). An amount of 3 mg of starch with 11 µL of water was closed hermetically in the DSC pan. After a 2 h waiting time at room temperature, the scanning was performed in the range of 20–120 °C with a speed of 10 °C·min^{-1}. An empty pan was used as a reference. The transition temperatures reported are the onset (*To*), peak (*Tp*) and endset (*Tc*) determined from the result of the thermogram. The gelatinised samples were stored at 4 °C for 6, 14, 20 and 28 days and heated again in the range of 20–120 °C to study the effect of US on the retrogradation of oat starches. The retrogradation ratio was calculated by dividing the retrogradation enthalpy (ΔH_2) in the second heating by the gelatinisation enthalpy (ΔH_1) in the first heating (Banchathanakij and Suphantharika 2009).

Attenuated total reflectance-Fourier transform infrared spectroscopy (FTIR-ATR) analysis

An attenuated total reflectance-Fourier transform infrared spectrometer (Spectrum 100 FTIR-ATR; PerkinElmer, USA) at scanning ranges of 400–4 000 cm^{-1} was used to examine the effect of US on the structure of starch samples.

Statistical analysis

Experimental results are expressed as mean ± SD. The SPSS 9.0 (SPSS Inc., USA) computer program analysed all experimental data using the ANOVA and Duncan's multiple range test. The $P < 0.05$ level was used as being significantly different.

RESULTS AND DISCUSSION

Extraction yield. Oat starch and protein are tightly bound molecules, and soaking oat flour in water weakens this bond and allows starch isolation. It was reported that a 6 h soaking of oat flour improved starch yield and purity (Lim et al. 1992). US-assisted extraction with different sonication times was performed to reduce extraction time and increase extraction efficiency. Yields (g starch·100 g^{-1} oat flour) were found as 43.07 ± 0.18a, 48.64 ± 0.24b, 49.04 ± 0.33b, and 47.91 ± 0.16c for control, 30, 45, and 60 min sonication, respectively. The extraction yields significantly increased ($P < 0.05$) with the application of the US. The highest extraction yield was obtained as 49.04 g·100 g^{-1} oat flour after a 45 min US application. This could be attributed to the weakening of starch-protein bonds with the US rather than the soaking application.

Effect of ultrasound on colour attributes. Increasing duration of US application caused significant ($P < 0.05$) changes in *L*, *a* and *b* values of starch samples (Table 1). The highest whiteness index was found in the control sample and the lowest after 45 min of US application. The presence of pigments in the starch, if carried over to the final product, reduces the quality, hence the acceptability of a starch product. A high value for lightness and a low value for chroma is desirable for starch to satisfy the consumer choice (Ali et al. 2016). It was found that the US affected the colour quality of oat starch slightly.

Table 1. Effect of ultrasound treatment on colour and functional properties of oat starches

Properties	Control	US 30 min	US 45 min	US 60 min
L	94.24 ± 0.04^{a}	92.04 ± 0.09^{b}	91.51 ± 0.33^{c}	92.24 ± 0.32^{b}
a	–0.04 ± 0.02^{a}	–0.24 ± 0.01^{b}	–0.14 ± 0.02^{c}	–0.15 ± 0.00^{cd}
b	3.39 ± 0.02^{a}	4.17 ± 0.05^{b}	4.31 ± 0.09^{c}	4.09 ± 0.08^{b}
WI	93.32 ± 0.03^{a}	91.01 ± 0.01^{b}	90.48 ± 0.06^{c}	91.44 ± 0.04^{d}
BD (g·ml^{-1})	0.80 ± 0.01^{a}	0.73 ± 0.03^{b}	0.65 ± 0.02^{c}	0.67 ± 0.04^{c}
WSI (%)	3.22 ± 0.01^{ab}	5.63 ± 0.24^{b}	4.37 ± 0.31^{ab}	5.75 ± 0.93^{b}
Dispersibility (%)	78.00 ± 4.24^{a}	84.50 ± 2.12^{ab}	87.50 ± 0.71^{b}	86.50 ± 6.36^{b}
WAC (%)	95.87 ± 3.13^{ab}	84.23 ± 3.42^{a}	86.83 ± 2.61^{ab}	90.31 ± 3.84^{ab}

$^{a–d}$values in the same line with different letters differ significantly ($P < 0.05$); US – ultrasound; *L* – lightness; *a* – greenness (–*a*) / redness (+*a*); *b* – blueness (–*b*) / yellowness (+*b*); *WI* – whiteness index; *BD* – bulk density; *WSI* – water solubility index; *WAC* – water absorption capacity

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Effect of ultrasound on functional properties of oat starch. The effect of the US on the functional properties of starch is given in Table 1. Relative bulk density ($g \cdot mL^{-1}$) of the starches was determined in the range from 0.59 ± 0.03 to 0.80 ± 0.01, in which these values are higher than the results from related articles (Eke-Ejiofor 2015; Ali et al. 2016). The results showed that all density values differed statistically except US45 and US60 values. It was determined that the bulk density decreased with the application of the US.

Sonication did not significantly change the *WSI* values of starches. Our dispersibility values are similar to the findings of Kulkarni et al. (1991), who found oat dispersibility in the range of 63–79%, similar to our control, 78.00 ± 4.24%. They specified that the higher the dispersibility, the better the starch reconstitutes in water to give a fine and consistent paste. It was observed that the US application increased the amount of dispersibility, but the effect of sonication time on dispersibility was not significant.

WAC shows the amount of water absorbed by starch in a limited water condition. It is important in bulking and consistency of products. Similarly to our results, the *WAC* of native and sonicated oat starch was reported as 77.20 to 99.11% by Falsafi et al. (2019). It was found that US treatment had no adverse effect on the water absorption capacity of oat starches.

Thermal properties. The gelatinisation transition temperatures (onset, *To*; peak, *Tp*; and endset, *Tc*), gelatinisation temperature ranges (*Tc*–*To*) and gelatinisation enthalpies (Δ*H*) are given in Table 2. The use of US and duration of the US treatment caused significant changes ($P < 0.05$) in *To*, *Tp*, and *Tc* values. The application of US for 30, 45, and 60 min during starch extraction significantly decreased the *To*, *Tp*, and *Tc* values. Gelatinisation temperature ranges (*Tc*–*To*) seemed to be affected by the US application. The enthalpy value of control starch is similar to the result given as 8.94 $J \cdot g^{-1}$ for oat starch (Vamadevan et al. 2013) and 7.7 $J \cdot g^{-1}$ for starch for whole oat flour (Moisio et al. 2015). US application increased the enthalpies of gelatinisation values of starches significantly. Cui and Zhu studied the effect of the US on sweet potato and wheat flour starches and found a decrease in gelatinisation enthalpies by US treatment (Cui and Zhu 2020). Different effects of the US on enthalpies of gelatinisation could be due to differences in US duration. They applied the US for 2–20 h, which were longer than our treatment times.

Changes in the transition temperatures (*To*, *Tp*, and *Tc*) during storage are given in Figures 1A, 1B, and 1C, respectively. Retrogradation transition temperatures *To*, *Tp*, and *Tc* were found to be lower than the gelatinisation temperatures. The application of the US resulted in a significant change in *To*, *Tc*, and *Tp* values. Retrogradation endotherms were observed after 6 days of storage at 4 °C. A similar trend was observed for the US-treated samples with that of the control for all transition temperatures. Increasing the storage time caused a decrease in retrogradation enthalpy. The ratio of retrogradation enthalpy to gelatinisation enthalpy decreased with the application of US, showing that sonication slows down the retrogradation of starch. A large increase in the ratios of ΔH_2 / ΔH_1 was observed during the first week, and after 14 days of storage, they remained nearly constant, meaning no further change in energy requirement for melting (Figure 2).

Fourier transform infrared spectroscopy analysis of starch. FTIR is a useful method for characterising starch molecules and analysing the molecular changes after processes are applied. The intensity of all peaks observed in spectra decreased with sonication, but their positions remained constant with no shifting (Figure 3). The peaks observed at 3 290 and 1 642 cm^{-1} were dependent on the O-H stretching and generally related toto the samples' water content. The intensity of the sonicated sample peaks at these wavenumbers was lower than in the control sample. It is reported that the 1 640 cm^{-1} band was due to the

Table 2. Thermal properties of oat starches extracted with ultrasound

Treatment	*To* (°C)	*Tp* (°C)	*Tc* (°C)	*Tc*–*To* (°C)	Δ*H* ($J \cdot g^{-1}$)
Control	60.16 ± 0.04^{a}	65.21 ± 0.00^{a}	71.28 ± 0.13^{a}	11.11 ± 0.18^{a}	8.4526 ± 0.51^{a}
US 30 min	59.09 ± 0.04^{b}	64.33 ± 0.12^{b}	71.22 ± 0.22^{a}	12.13 ± 0.18^{b}	13.0667 ± 0.11^{bc}
US 45 min	59.73 ± 0.09^{c}	64.28 ± 0.00^{b}	70.60 ± 0.15^{b}	10.87 ± 0.05^{a}	13.4178 ± 0.34^{c}
US 60 min	60.91 ± 0.04^{d}	64.70 ± 0.00^{c}	69.86 ± 0.03^{c}	8.96 ± 0.06^{c}	13.6450 ± 0.02^{c}

$^{a–d}$ values in the same column with different letters differ significantly ($P < 0.05$); *To* – onset temperature; *Tp* – peak temperature; *Tc* – conclusion temperature; *Tc*–*To* – gelatinisation temperature ranges; Δ*H* – enthalpy of gelatinisation

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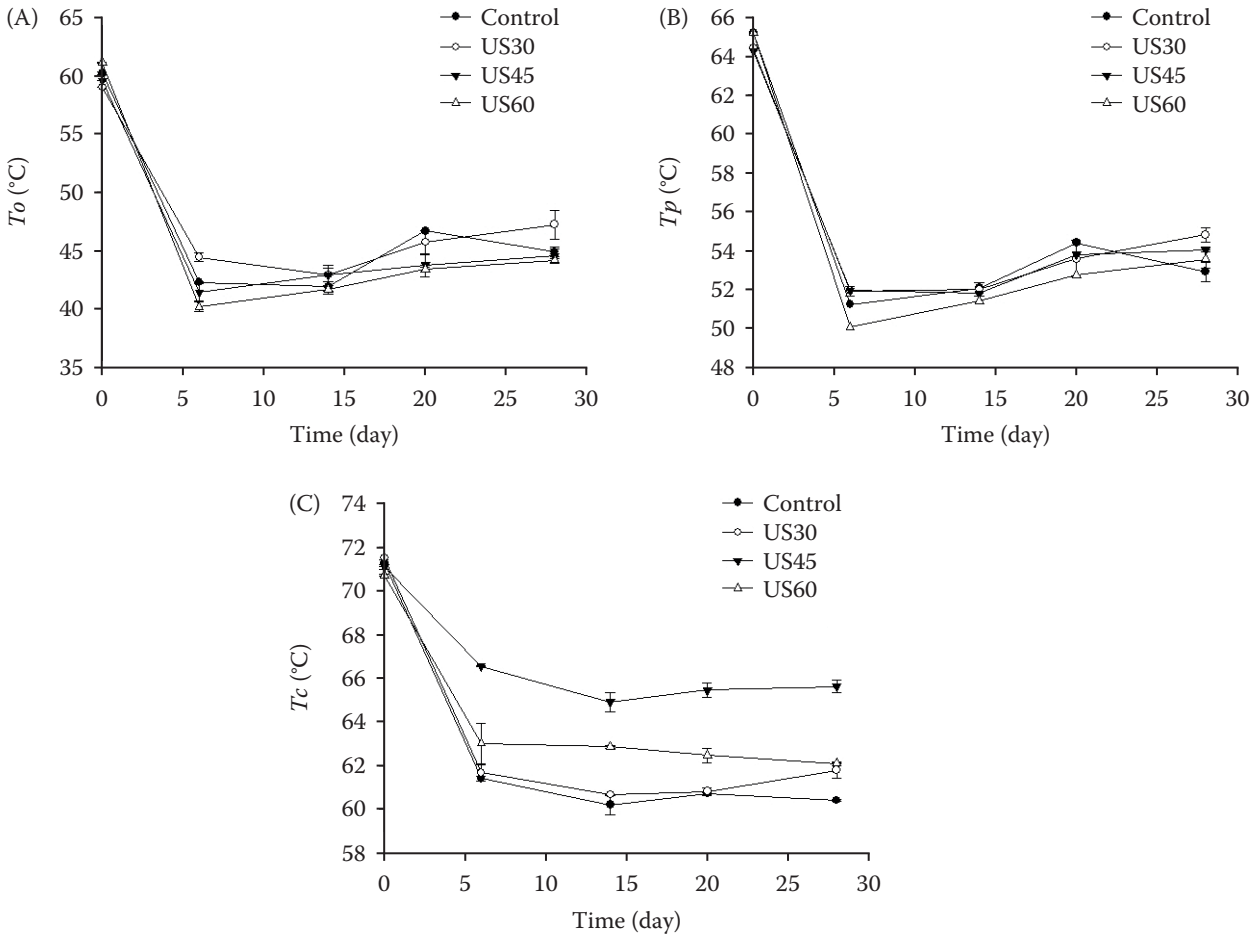

Figure 1. Changes in A) onset, B) peak, C) endset temperatures of oat starches extracted with and without sonication as a function of storage time

Error bars – SD; US – ultrasound; *To* – onset temperature; *Tp* – peak temperature; *Tc* – conclusion temperature

firmly bound water content of the starch granule (Fang et al. 2002). US application decreased the tightly bound water content of oat starch. A decrease in the intensity of the peaks at 1 336 cm^{-1} by sonication could be due to the vibrations of hydrogen and carbon atoms in the region of 1 300–1 500 cm^{-1} (Kizil et al. 2002). We observed a big difference in alteration in the intensity of the peak at 1 000 cm^{-1}. In the study by Warren et al. (2016), the authors indicated that the spectrum region between 1 000 and 1 022 cm^{-1} was substantially sensitive to water. Some researchers related the change of those bands with the destruction of the crys-

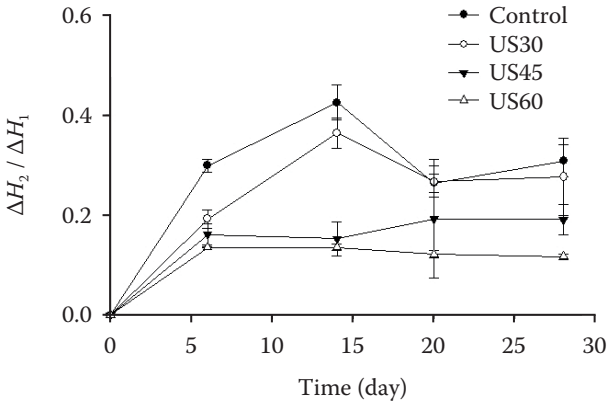

Figure 2. Changes in the retrogradation ratio (ΔH_2 / ΔH_1) of control and ultrasound-applied oat starches as a function of storage time

Error bars – SD; US – ultrasound; ΔH_2 – retrogradation enthalpy; ΔH_1 – gelatinisation enthalpy

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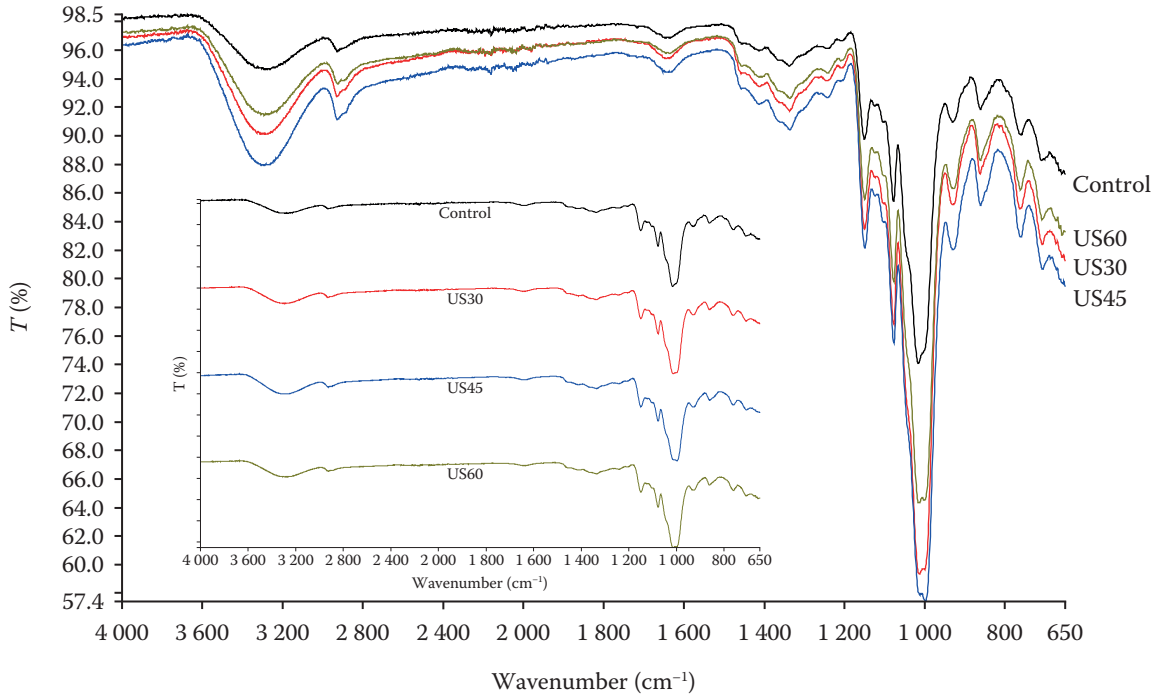

Figure 3. Fourier transform infrared spectrums (FTIR) of oat starches extracted with and without ultrasound treatments

US – ultrasound; *T* – transmittance

talline starch structure by high pressure, sonication and microfluidization (Wang et al. 2020). In our study, the change in the peak intensity may probably be due to the water release during US treatment. We conclude that using US in starch extraction did not cause a structural change, but it caused a change in the water contents of starch samples.

CONCLUSION

US application is a promising technique to isolate oat starch. It can potentially decrease the extraction time from 6 h to 45 min and increase the starch yield from 43.04 up to 49.04% without altering the functional properties of oat starch. It slightly but significantly affected starches' colour values, possibly due to the pigment extraction during sonication. The functional properties of starch were not adversely affected by the US application. Gelatinisation enthalpy values increased by sonication regardless of the time. The application of the US slows down starch retrogradation. The use of US in starch extraction did not result in a structural change, but a decrease in peak intensities was observed in FTIR spectra. Our results showed that using the US is a viable method for oat starch isolation. It is easy to use, feasible, and of no adverse effects, and it is an environmentally friendly technique.

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