The role of hydrothermal treatment (steaming and tempering) parameters on oat groat, flake and flour properties

Sammalisto, Saara

2022-12-15

Sammalisto , S , Laitinen , M L , Sontag-Strohm , T & Katina , K 2022 , ' The role of hydrothermal treatment (steaming and tempering) parameters on oat groat, flake and flour properties ' , LWT-Food Science and Technology , vol. 171 . https://doi.org/10.1016/j.lwt.2022.114137

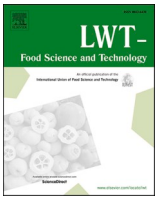
http://hdl.handle.net/10138/352968
https://doi.org/10.1016/j.lwt.2022.114137

LWT - Food Science and Technology 171 (2022) 114137

Contents lists available at ScienceDirect

LWT

journal homepage: www.elsevier.com/locate/lwt



The role of hydrothermal treatment (steaming and tempering) parameters on oat groat, flake and flour properties

Saara Sammalisto *, Miikka Laitinen, Kati Katina, Tuula Sontag-Strohm

Department of Food and Nutrition, Faculty of Agriculture and Forestry, University of Helsinki, Agnes Sjöbergin katu 2, P.O. Box 66, FI-00014, Helsinki, Finland

ARTICLE INFO

Keywords:
Oat milling
Hydrothermal treatment
Groat hardness
Oat flour particle size

ABSTRACT

The role of hydrothermal treatment (steaming and tempering) parameters on oat groat, flake, and flour properties was studied by using experimental design and response surface modelling approach. The modelled properties were oat groat hardness, oat flake hardness, and oat flour particle size milled from oat groats and oat flakes. In the design of experiments, the studied factors were moisture content, temperature, and duration of the hydrothermal treatment using the central composite face-centred design (CCF). Three untreated oat groat cultivar samples with varying native groat hardness were studied. Among the studied factors, tempering temperature influenced both groat hardness and flour particle size in all samples. High temperature led to fragile groats and fine flours, while low temperature resulted in hard groats and coarse flours. The treatment parameters affected groat and flour properties in a generally similar way, but sample-specific differences remained in groat hardness and flour particle size after identical treatments. In the sample with the softest native groats, also the moisture content influenced groat hardness and flour particle size. Our results suggest that with a proper selection of the native groats and by adjusting the hydrothermal treatment parameters, the milling properties of oat could be optimised for specific uses.

1. Introduction

Oat flour has gained popularity as a food ingredient, but the milling technology of oat is not known well. The oat milling process includes a hydrothermal treatment, which aims to inactivate lipolytic enzymes in oat that may cause rancidity during storage. Traditionally, the hydrothermal treatment has been performed in two sequential stages (Ganssmann & Vorwerck, 1995). According to Ganssmann and Vorwerck (1995), the groats are first given a longer heat treatment (kiln drying, kilning) to stabilize the enzymatic activity, where the groats are steamed and heated rapidly to reach about 18% moisture content and 100 °C temperature. Sufficient moisture content is crucial for enzyme inactivation. Additional heating is followed as the groats flow pass the heating radiators. At the end of the 90–120 min heat treatment, the groats are dried and cooled. Additionally, before flaking, oat groats usually undergo a second, shorter steam treatment to assist flaking that includes steaming and tempering. The groats are steamed for 1–2 min to increase the moisture content and to reach the temperature of the groats to around 95–104 °C (Ganssmann & Vorwerck, 1995). After steaming, a tempering step allows moisture equalization before flaking. Steaming softens the groats and reduces the tendency to fracture (Gates, 2007, p. 69).

The second treatment step alone, steaming and tempering before flaking, has been reported to be sufficient for enzyme inactivation and to assist flaking, which questions the need for a traditional two-step treatment. Gates, Sontag-Strohm, et al. (2008) reported that commercial kilning alone did not totally inactivate the lipolytic enzymes, but the moisture content of the groats during the commercial kiln was not specified. According to Gates, Dobraszczyk, et al. (2008), steaming and tempering treatment parameters influenced the mechanical properties of oat groat, while commercial kilning did not affect these properties. Gates et al. (2004, 2008b) reported that kilning was not necessary for the storage stability of oat flakes if the samples were sufficiently steam-treated. Kilning had only little effect on oat flake properties, while steam treatment before flaking was critical for the oat flake texture (Gates et a. 2004, 2008b). According to Hutchinson et al. (1951) and Deane and Commers (1986), as the groats are heated during kilning, their moisture content decreases and thus, lipase activity remains in the

* Corresponding author.
E-mail addresses: saara.sammalisto@helsinki.fi (S. Sammalisto), miikka.laitinen@helsinki.fi (M. Laitinen), kati.katina@helsinki.fi (K. Katina), tuula.sontag-strohm@helsinki.fi (T. Sontag-Strohm).

https://doi.org/10.1016/j.lwt.2022.114137
Received 17 June 2022; Received in revised form 25 October 2022; Accepted 31 October 2022
Available online 8 November 2022

kilned groats and the steam treatment prior to flaking eliminates the lipase activity. In the previous studies (Table 1), the groat moisture content during kilning was not always specified and thus, probably not controlled. Thus, a separate kilning step is not necessary in oat processing if the groats are steam-treated at sufficient moisture content. These days, at industrial scale, a one-step process is common where the flaking is done directly after the hydrothermal treatment. However, there is no standard for the treatment and thus, several variations of the hydrothermal treatment in the oat milling process exist.

The use of oat has increased in several types of food products, and with further knowledge about the milling behaviour of oat, the oat flour properties could be optimised for particular food processes. Recently, large variation in oat cultivar sample properties has been reported in oat grain and oat groat properties, and chemical composition (Jokinen et al., 2021). As the physicochemical properties differ between oat cultivar samples, presumably the milling properties vary as well. A further study showed that in the set of 20 oat cultivar samples, oat flour particle size parameters (average particle size $D_{4,3}$ and median particle size D_{50}) were related to the baking quality of whole grain oat flours (Sammalisto et al., 2021). Regarding oat flakes, it has been reported that oat cultivar samples varied in the oat flake properties (Jokinen et al., 2022; Lapveteläinen et al., 2001), oat flake granulation and oatmeal texture (Rhymer et al., 2005). In addition to cultivar-related differences, hydrothermal treatment conditions have been reported to influence oat groat strength and oat flake properties (Gates, Dobraszczyk, et al., 2008; 2008b). According to our knowledge, no studies about the relations between the oat cultivar samples, hydrothermal treatment, and oat flour properties have been published. Based on previous studies, it could be assumed that both the oat cultivar sample and the hydrothermal treatment parameters influence oat groat, oat flake, and oat flour properties, and these properties could be altered by the treatment parameters. Further knowledge about the relations between the hydrothermal treatment and oat groat and oat flour properties could provide tools to adjust the oat flours to be most suitable for specific types of food products.

The aim of this study was to investigate whether oat groat hardness, oat flake hardness, and oat flour particle size could be controlled by the hydrothermal treatment that is included in oat processing. Three oat cultivar samples of differing native groat hardness were selected in this study to observe the possible variation between the cultivar samples.

Table 1
The conditions of two- and one-step kilning and steaming treatments of oat groats according to the literature.

Reference	Kilning conditions	Steaming and tempering conditions
Jokinen et al. (2021)		Steaming at 155 °C for 40 s, 15–20 min stabilization
Duque et al. (2020)	Kilning at 115 °C for 30 min	Steaming at 100–104 °C for 18 min
Girardet and Webster (2011)	Reference to Ganssmann and Vorwerck (1995)	Steaming to increase the moisture content 3–5%, tempering at 95–102 °C for 20–30 min
Head et al. (2010)	Steaming groats to 17% moisture, then kilning at 88–98 °C for 100 min	
Gates, Dobraszczyk, et al. (2008)	Industrial kiln of 90 min	Steaming at 101 °C for 30 s, tempering at 80–110 °C for 30–90 min
Gates (2007)	Steaming groats to 17–19% moisture at 80–102 °C for over 90 min	Steaming to 12–19% moisture at 95–105 °C for 10–30 min
Rhymer et al. (2005)	Steaming groats to 17% moisture, then at 100 °C for 10 min with a lid on and 45 min without a lid	Conditioning the groats to 16% moisture content overnight at room temperature
Gates et al. (2004)	At 85 °C for 2.5 h (Bühler kiln)	Tempering at 95–100 °C for 45 min
Ames and Rhymer (2003)		Steaming for 30 s, tempering at 100 °C for 15 min
Bryngelsson et al. (2002)	Steaming at 100 °C for 60 min	Steaming at 100 °C for 20 min
Zhou et al. (2000)	Steaming for 9 min, kilning at 100 °C for 45 min, at 65 °C for 15 min	Resteaming for 5 min
Sontag-Strohm et al. (1996)	Conditioning groats to 12–20% moisture and heated at 95 °C for 1–2 h	
Ganssmann and Vorwerck (1995)	Steaming groats to 17–18% moisture and to 102 °C, 90–120 min in total	Steaming for 1–2 min to increase groat moisture content 3–5% and temperature to 95–104 °C, tempering for 15–25 min
Molteberg et al. (1995)	Groats soaked in water for 2 min, then steaming at 100 °C for 10 min and drying at 100 °C for 3.5–4 h	
Salovaara (1993)	Steaming groats to 16–17% moisture for 2–3 min, heating at >95 °C for over 70 min, and drying for 30 min	
Mahnke-Plesker (1991)	Steaming for 0.5–2 min, kilning at 100–120 °C for 1–3 h	1–2 min steaming at 100 °C to increase the groat moisture to 17%, 15 min tempering
Deane and Commers (1986)	Kilning groats at 88–93 °C for at least 1 h	12–15 min steaming at 99–104 °C

2. Materials and methods

2.1. Materials

The materials of this study were oat groat samples from three cultivar-pure oat batches grown in Finland in 2019. The unkilned oat groats were dehulled by Vääksy Mill Ltd. (Asikkala, Finland). The oat groats were stored in the freezer before the experiments (in sealed bags at −19 °C). The chemical compositions of these oat cultivar samples (coded with the same sample numbers G24, G28, G29) were published earlier by Jokinen et al. (2021), and the main chemical data is presented in section 2.2.1, Table 2.

2.2. Methods

2.2.1. Analyses of the native oat groats

The basic chemical compositions of the oat cultivar samples G24, G28, and G29 were reprinted with a permission of Jokinen et al. (2021) (Table 2). The oat groat samples were selected to the study according to the hardness of the untreated groats in preliminary experiments: one sample of low groat hardness (G24), intermediate groat hardness (G28), and high groat hardness (G29) (Table 2). The method for groat hardness measurement is described in detail in section 2.2.5 but in the preliminary experiments, the groats were analysed at room temperature. From the untreated native groats, the amount of broken groats was analysed with a sieve shaker (CISA RP-08-S, Cedacería Industrial, Spain) from three replicate samples of 10 g that were sieved for 3 min at an amplitude of 0.8 mm. The amount of small groat material (% of the original sample weight) that passed a 1.8 mm screen represented the amount of broken groats. Groat thickness (mm) was measured with a micrometer (Mitutoyo, Japan) from thirty randomly selected, intact oat groats.

2.2.2. Design of experiments

Response surface methodology (RSM) was applied to investigate the influence of the steaming and tempering parameters (groat moisture content, tempering temperature, tempering time) on oat groat hardness, oat flake hardness, and oat flour particle size. In RSM, the values of response variables are predicted by regression analysis, based on the controlled experimental design. MODDE Pro software (MODDE 13,

Table 2
The basic chemical composition of the oat cultivar flour samples (analysed from the heat-treated flours), and the physical properties of the oat groat samples (analysed from the untreated groats). The hardness of untreated oat groats was measured by a compression test at room temperature. The hardness values are presented as average values of fifteen measurements, amount of broken groats as average values of three measurements, and groat thickness as average of thirty samples. Error values represent standard errors of the means (SEM).

Sample	Starch[a]	Protein[a]	Fat[a]	TDF[a,b]	Beta-glucan[a]	Groat hardness (N)[c]	The amount of broken groats (%)[c]	Groat thickness (mm)[c]
G24	63.6	14.2	6.9	11.0	3.3	46 ± 2	41 ± 2	2.12 ± 0.03
G28	60.4	16.4	7.6	11.9	4.2	51 ± 3	26 ± 1	2.11 ± 0.03
G29	62.7	16.1	8.0	11.0	3.9	57 ± 3	17 ± 1	2.15 ± 0.04

[a] Percentage by dry mass basis, results reprinted with a permission of Jokinen et al. (2021).
[b] Total dietary fibre.
[c] Analysed from the untreated oat groats.

Sartorius Stedim Data Analytics AB, Umeå, Sweden) was used to design the experiments of central composite face-centred design CCF (Table 3), calculate the regression analysis, and plot the response surfaces. In the central composite design, each process variable (groat moisture content, tempering temperature, and tempering time) had three levels, −1, 0, and +1, and twenty experiments were conducted in randomized order with each oat cultivar sample. The levels of −1 (15%, 85 °C, 30 min), centre point (17.5%, 100 °C, 60 min), and +1 (20%, 115 °C, 90 min) were determined based on the literature, and the centre point was repeated six times (experiments N15–N20). Peroxidase test was conducted to measure the possible residual enzymatic activity present after the hydrothermal treatment. The samples after the mildest treatment conditions (experiment N1, 15%, 85 °C, 30 min) were tested negative, so it was assumed that the rest of the samples did not have enzymatic activity. The measured response variables were oat groat hardness, oat flake hardness, and average particle size ($D_{4,3}$) and median particle sizes (D_{50}) of oat groat flours and oat flake flours.

2.2.3. Moistening the oat groats

Untreated oat groats (100 g per sample) were weighed into aluminium containers (volume of 0.85 L), and distilled water was added to reach the desired moisture content of the groats (15%, 17.5%, or 20%) to simulate the steaming of the groats. The groats and the water were mixed carefully to form a thin layer, and the sealed containers were stored at 5 °C overnight (18 h) to allow the moisture content to equalize in the groats before the hydrothermal treatment. The required amounts of added water to reach the desired moisture contents were studied in preliminary experiments.

Table 3
Design of experiments for the hydrothermal treatments for each oat cultivar sample.

Experiment Number	Moisture content (%)	Tempering temperature (°C)	Tempering time (min)
N1	15	85	30
N2	20	85	30
N3	15	115	30
N4	20	115	30
N5	15	85	90
N6	20	85	90
N7	15	115	90
N8	20	115	90
N9	15	100	60
N10	20	100	60
N11	17.5	85	60
N12	17.5	115	60
N13	17.5	100	30
N14	17.5	100	90
N15	17.5	100	60
N16	17.5	100	60
N17	17.5	100	60
N18	17.5	100	60
N19	17.5	100	60
N20	17.5	100	60

2.2.4. Hydrothermal treatment, flaking and milling

Hydrothermal treatment, flaking and milling were performed on a laboratory scale. The moistened groats were equilibrated to room temperature in the sealed aluminium containers before the treatments. The groats were kept first at 170 °C in a convection oven for 3 min (Sveba Dahlen, Fristad, Sweden) to simulate the intensity of steaming and to ensure that the groats enter the second oven already hot. After the first oven, the groats were rapidly transferred to the second oven (Memmert GmbH + Co.KG, Germany) and tempered for 30, 60, or 90 min at 85, 100, or 115 °C, according to the experimental design.

After the tempering treatment, groat hardness was analysed immediately. The groats were flaked directly after the treatments as well. The laboratory-scale flaking machine was similar to the one previously used by Gates, Sontag-Strohm, et al. (2008). A speed of 70 rpm and a roll gap of 0.4 mm were used in flaking. After the flaking and groat hardness measurements, oat flakes and oat groats were placed in a fume hood for 1 h to stabilize the moisture content and the temperature (25 °C, 16% RH). After that, the samples were packed in plastic bags for storage.

After the treatments, oat groats and oat flakes were milled with a centrifugal mill (ZM 200, Retsch GmbH, Germany) using a 0.5 mm screen, a 11.2 cm diameter rotor and a speed of 8000 rpm (400 *g*). Both oat groat flours and oat flake flours were milled with the same procedure.

2.2.5. Oat groat and oat flake hardness

Oat groat hardness was studied with a Texture Analyser (TA XT2i Texture Analyser, Stable Micro Systems, Godalming, UK) using a method adapted from Gates, Dobraszczyk, et al. (2008). The groats were placed crease-down and compressed at 1 mm/s speed with a 36 mm diameter probe into 50% deformation. The deformation was held for 5 s, and the peak force of the compression represented groat hardness. The groats were analysed immediately after the heat treatments and a heating cabinet was connected to the equipment and set to 65 °C to simulate the industrial oat flaking conditions. Twenty replicate samples were measured, and a 10 kg load cell was used in the analyses.

Oat flake hardness was studied with a pin-deformation test by Texture Analyser (TA XT2i Texture Analyser, Stable Micro Systems, Godalming, UK), with a method reported by Gates et al. (2004). A blunt steel pin (diameter 2 mm) was driven through the intact oat flake at a speed of 10 mm/s. The peak force of the compression represented oat flake hardness. Twenty replicate samples were measured, and a 5 kg load cell was used in the analyses.

2.2.6. Particle size analyses of the oat flours

Particle sizes of the oat groat flours and oat flake flours were analysed by laser diffraction using a dry feed unit (Mastersizer 3000 Aero S, Malvern Instruments Ltd., Malvern, UK). A refractive index of 1.47, input pressure of 3 bar and input power 30% were used in the analyses. Values of D_{50} (median particle diameter, μm) and $D_{4,3}$ (mean particle diameter, μm) were calculated from three replicate measurements.

2.2.7. Statistical analyses

MODDE Pro software was used to create the design of experiments and to process the data. The response surface models were created in CCF design by using partial least squares (PLS), which describes the effects of factors in polynomial equations. The models were verified with an additional experiment to estimate the predictive capacity of the models. Only the models with high reproducibility and with no significant lack of fit are reported in this paper. The percent of the variation of the response predicted by the model according to cross validation (Q^2) indicated how well the model predicts the data in a new experiment. A measure of fit (R^2) is the percent of the variation of the response explained by the model and explains how well the model fits the data. A useful and good model should have the values of $Q^2 > 0.5$ and of $R^2 > 0.5$. Lack of fit >0.25 is considered a good, valid model.

3. Results

3.1. Particle size distributions of the oat flour samples

After the hydrothermal treatments, whole grain oat flour milled from groats yielded generally coarser flours compared to flake flours, and the particle size distributions differed between the flours milled from groats and flakes (Fig. 1A–C). In Fig. 1A–C, the average particle size distributions are presented after each treatment (N1–N20). Groat flours had a more bimodal particle size distribution and fewer particle size classes in the flour, while flake flours had generally smaller particle size and higher amount of particle size classes in the flour. In the mathematical models, groat flours were modelled generally better than flake flours (section 3.2.1).

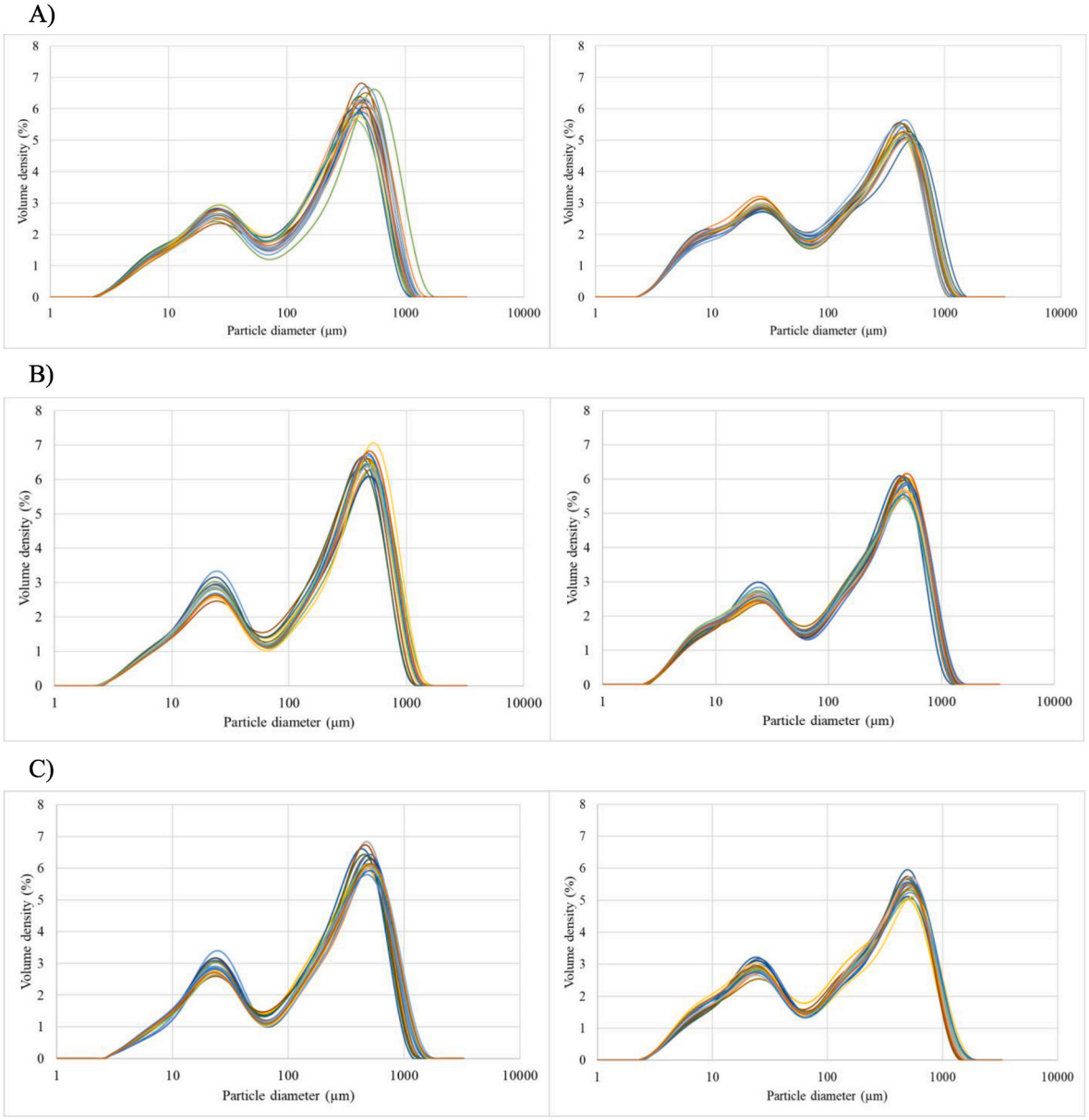


Fig. 1A–C. Particle size distribution of the whole grain oat flours milled from oat groats (left) and oat flakes (right) after experiments N1–N20, sample G24 (A). Particle size distribution of the whole grain oat flours milled from oat groats (left) and oat flakes (right) after experiments N1–N20, sample G28 (B). Particle size distribution of the whole grain oat flours milled from oat groats (left) and oat flakes (right) after experiments N1–N20, sample G29 (C).

3.2. Response surface methodology

3.2.1. Optimisation of the hydrothermal treatment

PLS model was used to predict the influence of moisture content, temperature, and duration of the treatment on the oat groat, oat flake, and oat flour properties for all oat cultivar samples. The mathematical models were validated with additional, verifying experiments from the optimal area, and only the acceptable models are presented where the measured value fitted to the range of predicted value (Table 4). Average particle size ($D_{4,3}$) was modelled generally better than median particle size (D_{50}) and thus, only the average particle size values are presented here.

The relations between the treatment conditions and the response factors were fitted by PLS (Table 5) and with these mathematical models, oat groat hardness and oat flour particle size could be predicted. For instance, groat hardness could be predicted for these samples within this experimental area by the following equations:

Groat hardness (sample G24) = 49.78 + 3.63Moist - 2.54Temp

Groat hardness (sample G28) = 57.21–3.6Temp + 3.34Moist*Temp

Groat hardness (sample G29) = 59.52 + 2.84Moist – 5.298Temp

Oat groat hardness and average particle size of groat and flake flours could be modelled from all samples, while oat flake hardness was modelled only in sample G29 (data not shown), which was the sample with the greatest native groat hardness. The most influential variable on groat hardness and flour particle sizes was the tempering temperature. Increased tempering temperature generally decreased groat hardness and yielded finer flours while at lower temperature, groat hardness increased and coarser flours were obtained. In the softest and hardest native groats (G24, G29), additionally, moisture content influenced groat hardness, as higher moisture content increased groat hardness. Moisture content also influenced the particle size of the groat and flake flours of sample G24 (softest native groat hardness), particle size of the groat flours of sample G28 (intermediate native groat hardness), and neither of the particle sizes of sample G29 (hardest native groats). The average particle size of the groat flours of sample G29 was influenced only by the tempering temperature and regarding the average particle size of the flake flours, only the quadratic effect of tempering temperature had an influence. Tempering time did not significantly influence any measured property, except the average particle size of groat flour in sample G24 (softest native groats). Thus, the sample with the softest native groats (G24) was most influenced by the treatment parameters.

3.2.2. Response surface plots

Response surface plots were drawn from the groat hardness of the samples G24 and G29, which were influenced both by the groat moisture content and the tempering temperature (Fig. 2A and B). The influence of the moisture content and tempering temperature on the groat hardness was similar in both samples, although groat hardness remained higher in sample G29, initially hardest sample, compared to sample G24, initially softest sample. Thus, groat hardness could be altered by the hydrothermal treatment parameters but after the treatments, sample-specific differences still existed.

Response surface plots of average flour particle size ($D_{4,3}$) of the samples G24 and G28 indicated that the treatment parameters had rather similar effects on flour particle size in these samples (Fig. 3A–C), as the maximum and minimum values were located in similar areas in the surface plots. When comparing the average particle size plots (Fig. 3A–C) to the groat hardness plots (Fig. 2A and B), it is clear that softer groats resulted in fine flours and harder groats in coarse flours. In the softest sample (G24), the average particle sizes of oat groat and oat flake flours were modelled very similarly (Fig. 3A and C).

3.3. Oat groat hardness and fracture

Increasing tempering temperature decreased the measured groat hardness in all cultivar samples. After the experiment N11 (17.5%, 85 °C, 60 min), the groat hardness curves were generally uniform whereas after the temperature was increased to 115 °C, experiment N12 (17.5%, 115 °C, 60 min), the oat groat samples were softer as they fractured during the compression to a greater extent (Fig. 4). Thus, increasing tempering temperature increased the fracture of oat groats. Additionally, the samples behaved in different ways in the compression test, as the sample of the softest native groats and the greatest amount of broken groats (G24) showed greater tendency to fracture also at lower temperature (experiment N11) compared to the other samples (G28, G29).

4. Discussion

In our study, both oat cultivar sample properties and hydrothermal treatment parameters influenced the oat groat and oat flour properties. Additionally, oat groats and oat flakes showed different milling behaviour, as they differed in particle size distribution and as groat flours were generally modelled better. High temperature during the hydrothermal treatment led to more fragile oat groats and fine flours, whereas low temperature led to harder groats and coarse flours. In the sample of the softest native groats, also the moisture content during the treatment influenced groat hardness and flour particle size. In the sample of the softest groats, high moisture content resulted in harder groats and coarser flours, while low moisture content led to softer groats and finer flours. These results suggest that oat groat hardness and oat flour particle size could be controlled by adjusting the hydrothermal treatment parameters, and softer native groats could be controlled better than harder groats.

In this study, harder groats resulted in coarser flours and softer groats resulted in finer flours after milling. In wheat, as well, harder groats generally yield coarser flours (Dobraszczyk et al., 2002). However, oat differs greatly from wheat regarding the chemical composition and endosperm structure and thus, their milling properties also differ. The wheat milling process includes several steps, where the objective is the efficient separation of the endosperm from the rest of the grain to produce mainly white wheat flour, but also other milling fractions of varying composition and particle size can be separated (Campbell et al., 2012). Compared to wheat, oat bran does not separate cleanly from the endosperm, and contains also aleurone and subaleurone layers (Girardet

Table 4
Measured and predicted values (with the range) for groat hardness and average particle sizes of the groat and flake flours of three oat cultivar samples after the treatment 15%, 115 °C, 60 min, at the optimal area. [a]

Sample	Groat hardness (N)		$D_{4,3}$ of groat flours (μm)		$D_{4,3}$ of flake flours (μm)	
	Measured value	Predicted value	Measured value	Predicted value	Measured value	Predicted value
G24	36	41 (34–47)	224	225 (212–240)	212	207 (197–217)
G28	40	39 (33–44)	248	238 (227–249)	257	257 (250–265)
G29	40	39 (34–45)	256	265 (254–276)	255	266 (251–280)

$D_{4,3}$ Average particle size of the flour.
[a] The predicted values were calculated by response surface modelling with a software MODDE 13 (Sartorius Stedim, Sweden).

Table 5
The effects of treatment factors with coefficients in validated models for oat groat, oat flake, and oat flour properties. [a]

Factor[b]	Sample G24			Sample G28			Sample G29		
	Groat hardness	Groat flour, $D_{4,3}$*	Flake flour, $D_{4,3}$	Groat hardness	Groat flour, $D_{4,3}$	Flake flour, $D_{4,3}$	Groat hardness	Groat flour, $D_{4,3}$*	Flake flour, $D_{4,3}$
Constant	49.78	2.39	227.63	57.21	277.63	2.4	59.52	2.48	28.49
Moist	3.63	0.017	6.44	–	7.43	–	2.84	–	–
Temp	−2.54	−0.0280	−9.59	−3.6	−10.89	−0.0088	−5.298	−0.015	–
Time	–	−0.0027	–	–	–	–	–	–	–
Moist*Moist	–	0.014	–	–	–	–	–	–	–
Temp*Temp	–	–	–	–	−9.04	0.013	–	−0.011	−9.34
Time*Time	–	–	–	–	–	–	–	–	–
Moist*Temp	–	–	–	3.34	–	–	–	–	–
Moist*Time	–	0.018	–	–	–	–	–	–	–
Temp*Time	–	–	–	–	−4.43	−0.0084	–	–	–
R^2	0.776	0.815	0.779	0.893	0.884	0.723	0.807	0.732	0.787
Q^2	0.619	0.624	0.519	0.664	0.598	0.514	0.595	0.565	0.617
Lack of fit	0.88	0.88	0.71	0.65	0.93	0.81	0.26	0.34	0.48

$D_{4,3}$ average particle size of the oat flours; * logarithm of the response.
[a] Only the values are presented with validated, significant coefficients (95% confidence level); -, not significant ($p > 0.05$).
[b] Moist, moisture content of the groats; Temp, tempering temperature; Time, tempering time; Moist*Moist, quadratic effect of moisture content; Moist*Temp, interaction between moisture content and temperature; R^2 measure of the fit of the model; Q^2 predictive power of the model.

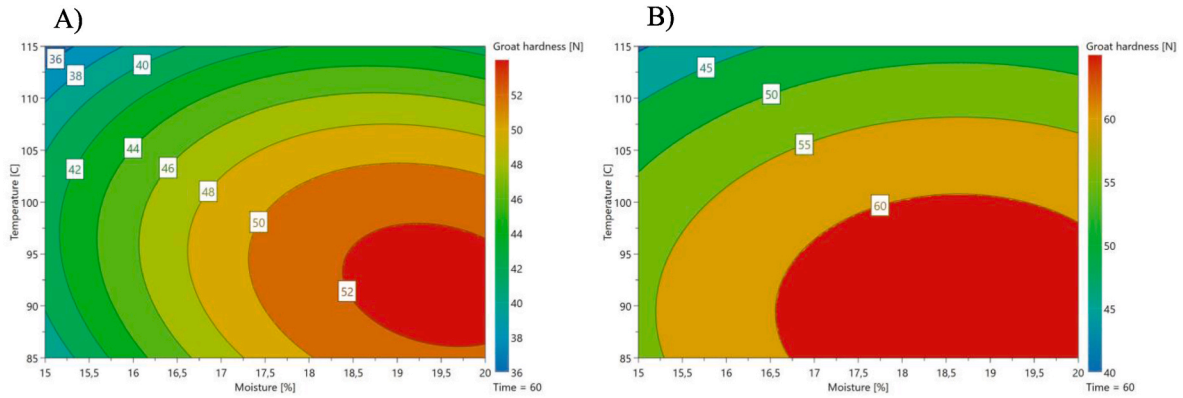


Fig. 2A–B. Response surface plots of groat hardness of sample G24 (A) and sample G29 (B), treatment time 60 min.

& Webster, 2011). The oat milling process is simpler, and the main milling fractions are either whole grain oat flour or oat bran and endosperm oat flour. The high fat content of oat groat and groat softness make milling and separation processes challenging (Fulcher, 1986). It has been shown that fat removal enhanced the separation of oat β-glucan, starch, and protein to distinct fractions (Sibakov et al., 2011). In our study, the sample with the softest groats was the lowest in fat, and the sample with the hardest groats was the richest in fat. The higher amount of fat could possibly interfere with the milling process and make modelling more complicated, but the differences in fat content were not prominent in this study. Instead, the amount of broken groats (analysed by sieving) seemed to be related to native oat groat hardness and thus, to the particle size of the milled flours. Possibly, the amount of broken groats could predict the milling behaviour of oat.

Oat flake hardness was fitted into a model only in the sample of the hardest native groats, and not in other samples. Also previously, Gates, Sontag-Strohm, et al. (2008) did not identify a relation between processing parameters and flake strength. Gates, Sontag-Strohm, et al. (2008) discussed that the sample variation in oat flakes caused small differences between the treatments that were difficult to detect. Oat flaking causes disruptions in the cell wall, starch granule and protein body organization in the oat endosperm (Lookhart et al., 1986). In our study, flake flours were more difficult to model than groat flours. In the sample with the hardest native groats, flour particle size could not be modelled as well as in the samples of softer native groats. Possibly, the harder groats broke less regularly and were more complex to model compared to the samples of softer native groats.

In our study, higher tempering temperatures led to softer groats and finer particle size of the milled flours, as the groats fractured more easily and resulted in finer particles from more fragile groats. Gates (2008a) also reported an increased number of cracks in oat groats as the temperature of the hydrothermal treatment increased. At increasing temperatures, it is possible that protein denaturation could have been responsible for the increased groat fracture. In the oat endosperm, protein bodies are located on the surfaces of the starch granules and cell walls in discrete structures (Lookhart et al., 1986). Globulins, the storage proteins of oat, are rather resistant to denaturation, with a denaturation temperature of around 112 °C (Marcone et al., 1998). Sontag-Strohm et al. (1996) studied the denaturation of oat protein fractions at 95 °C and reported a dramatic solubility decrease in albumin and avenin fractions during the heat treatment. Enzymes belong to the albumin fraction, which is mainly located in the outer endosperm, and subaleurone and aleurone layers, as their role is to hydrolyse storage components (starch, proteins, lipids) during germination to obtain nutrients for the embryo (Fulcher, 1986). As the enzymes are found in the surface structures, the solubility decrease or denaturation of this fraction could have led to a weakened groat structure, because the connecting points in the endosperm have been lost, resulting in softer groats. In addition, Gates (2007, p. 69) suggested that the bonds between starch granules are responsible for oat groat hardness.

Oat lipids are located in discrete oil bodies in the aleurone layer and germ while in the endosperm, the bodies often fuse with each other and

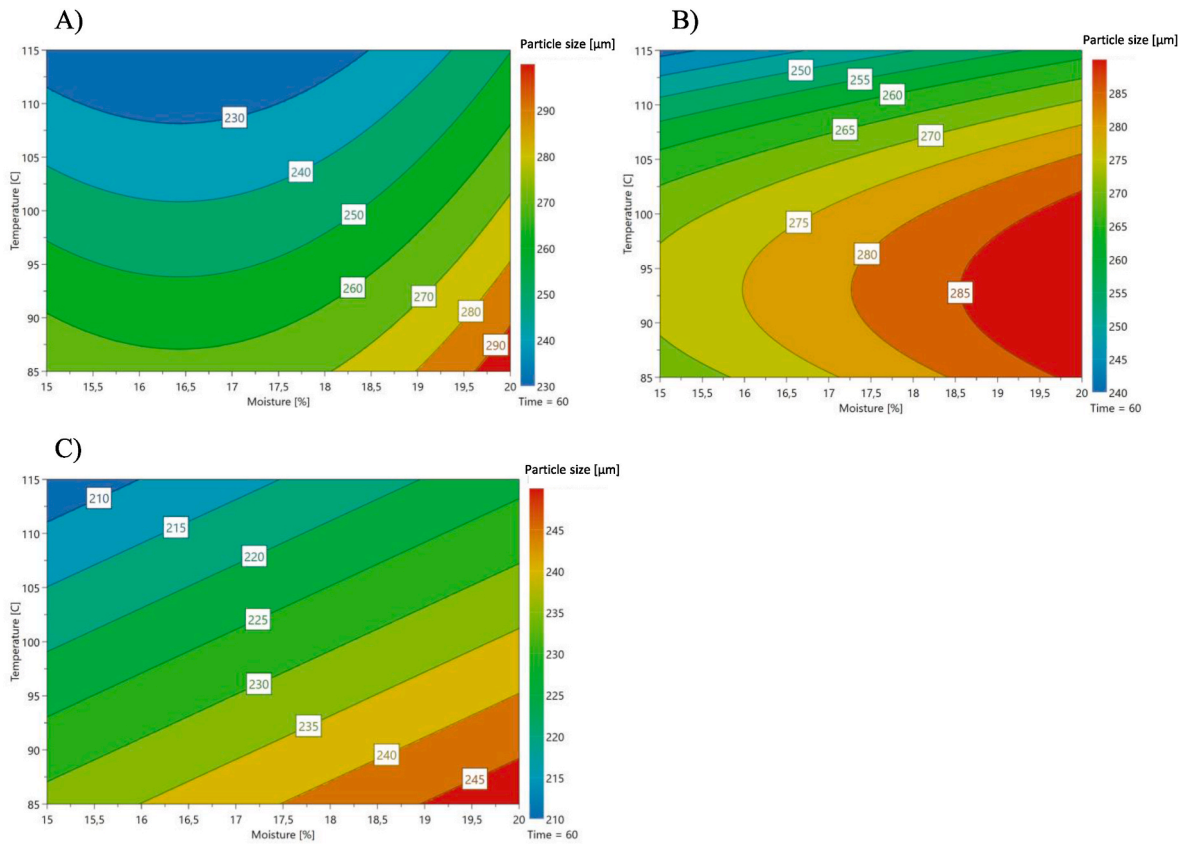


Fig. 3A–C. Response surface plots of average particle sizes of G24 groat flour (A) and G28 groat flour (B), treatment time 60 min. Response surface plot of average particle size of G24 flake flour (C), treatment time 60 min.

form a continuous matrix with protein and starch (Heneen et al., 2008). Part of the lipids of oat are adhered to the starch granule surface (Morrison, 1981; Paton, 1987). During the treatments of increasing temperatures, lipids or amylose-lipid complexes could have melted and their organization might have changed. The melting temperature of amylose-lipid complexes has been reported to be between 89 and 111 °C studied by differential scanning calorimetry (Li et al., 2021; Paton, 1987; Zhou et al., 2000), which is within the temperature range that was used in our study (85–115 °C). In our study, melting of the complexes could have resulted in softer groats and smaller particle size of flours in milling. Previously, Gates and Talja (2004) suggested that the breakage of oil bodies in oat endosperm could allow the oil to plasticize the structure.

Moisture content during the treatment influenced the groat hardness in the samples of the lowest and highest native groat hardness. At the higher moisture contents, harder groats were obtained. Water-induced stiffening during the hydrothermal treatment of oat has been previously reported by Gates and Talja (2004), which could have occurred in our study, as well. Gondek and Lewicki (2006) also reported that at certain water activities, water-induced stiffening was observed as the structure of corn flakes swelled and became more compact. The moisture content during the treatment influenced the particle size of both groat and flake flours of the softest sample while in the other samples, the role of moisture content on the particle size was significant only in groat flours of the sample with intermediate hardness. In the intermediate and harder native groats, which also had less broken groats, the penetration of water could have been less complete or uniform during the treatments due to the denser structure and thus, mathematical models could not be formed from the particle sizes of the flours and moisture content. In conditioning of wheat, grain hardness affects the time required to reach the desired moisture content of the grains, and harder grains absorb the water at a slower rate (Campbell et al., 2012).

Regarding the other structural elements of oat groat, oat β-glucan is considered moderately stable in heat treatments. Notable starch pasting should not occur during the hydrothermal treatment, as the moisture content of oat groats remained between 15 and 20%, which is not enough for starch gelatinization (Ganssmann & Vorwerck, 1995; Mahnke-Plesker, 1991, p. 209). However, Lookhart et al. (1986) reported that oat kilning induced compound starch granules to break into individual granules. The hydrothermal treatment might weaken the groat structure by influencing the physical starch integrity and thus, increase the facture and yield finer flours in milling.

Oat cultivar samples of this study differed in groat hardness both as native groats and after the hydrothermal treatments. Among the native groat properties, the softest groat sample had notably higher amount of broken groats compared to the other samples, and the groats had a greater tendency to fracture during compression test. Regarding the basic chemical properties, some variation was observed between the samples mainly in the β-glucan and protein contents (Jokinen et al., 2021). In the oat groat, β-glucan is located in the cell wall and according to Engleson and Fulcher (2002), related to groat hardness by strengthening the structure. In our study, β-glucan content was the lowest in the softest native sample and rather higher in harder native samples (Jokinen et al., 2021). In wheat, grain hardness has been shown to be related to the presence of endosperm-specific proteins (Morris, 2002). However, wheat and oat endosperm structures differ from each other regarding the protein composition and distribution (Zhou et al., 1998). In oat endosperm, protein bodies are located on the surfaces of the starch granules and cell walls in discrete structures (Lookhart et al., 1986), and not in a continuous matrix, as in wheat (Zhou et al., 1998). Still, increasing protein content has been reported to increase oat groat hardness (Engleson & Fulcher, 2002). In our study, the sample of the softest native groats had somewhat lower protein content compared to the other samples that had harder native groats (Jokinen et al., 2021).

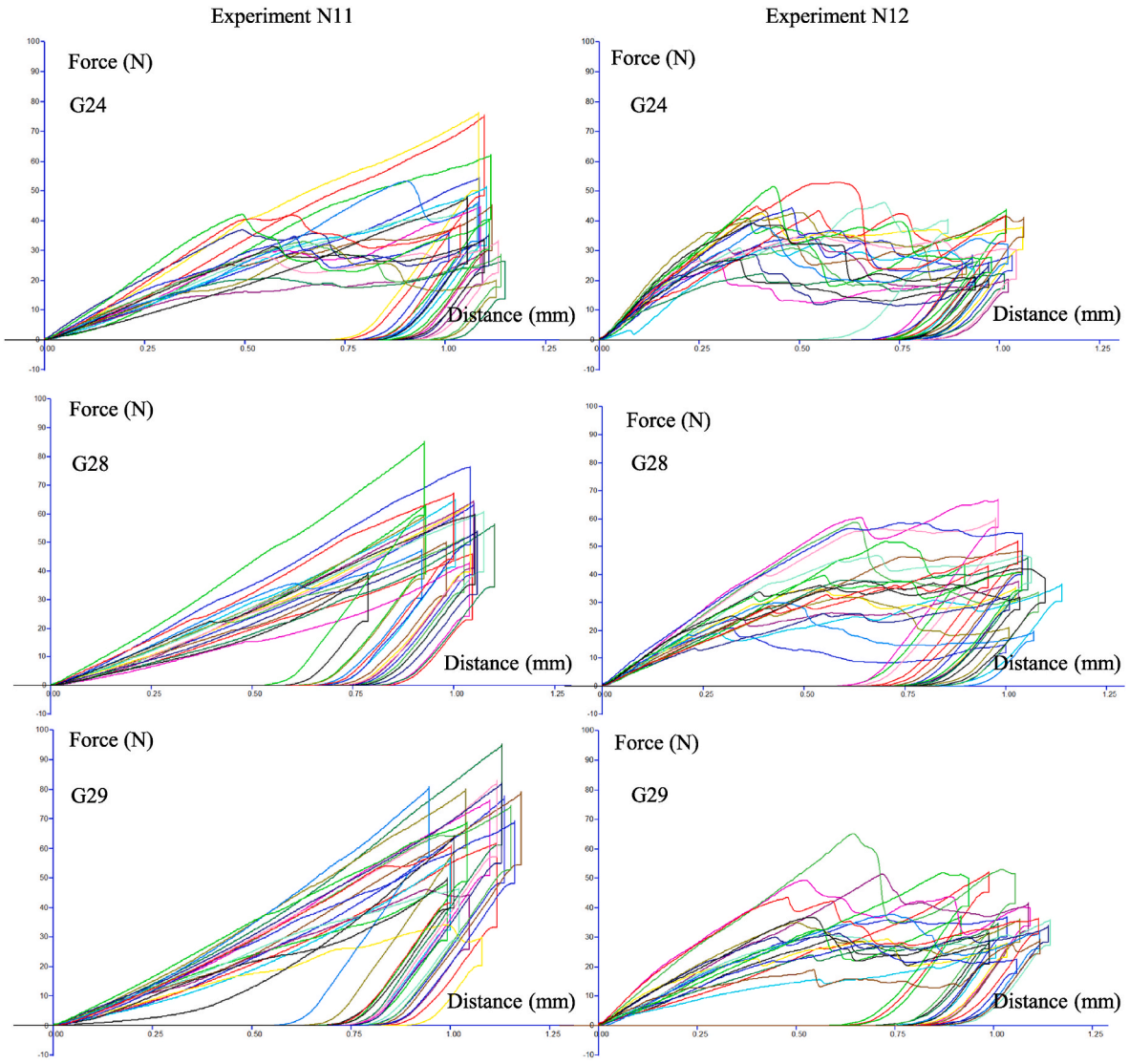


Fig. 4. Groat hardness of samples G24, G28, and G29 after experiment N11 (17.5%, 85 °C, 60 min, on the left side) and after experiment N12 (17.5%, 115 °C, 60 min, on the right side) from 20 replicate measurements.

5. Conclusions

It is crucial to understand the role of hydrothermal treatment parameters on the groat and flour properties in the oat milling process, so that the oat flour properties could be optimised for specific food uses. In this study, the tempering temperature influenced oat groat hardness and oat flour particle size in all samples while in the sample of the softest native groats, also the moisture content of the groats influenced these properties. Sample-specific differences of oat cultivar samples remained after the treatments, as the sample with the softest native groats remained the softest after the treatments and was controlled by the treatment parameters. Also, oat samples showed different tendency for fracture at the compression test. Our results strongly suggest that with a proper selection of the native groats and by adjusting the hydrothermal treatment parameters, the milling properties of oat could be controlled and optimised for specific uses. This study provides tools for millers for optimisation of their oat milling processes and could arouse interest to conduct more studies about oat milling, so that the scientific knowledge could be deepened.

Funding

This research was funded by Business Finland [grant number 6960/31/2018] as a part of the project "Novel indicators and technologies for oat quality and applicability (OatHow)" cofunded by University of Helsinki, the University of Turku, Luke Natural Resources Institute Finland and VTT Technical Research Centre of Finland Ltd.

CRediT authorship contribution statement

Saara Sammalisto: Conceptualization, Methodology, Investigation, Formal analysis, Data curation, Writing – original draft, editing, Visualization. **Miikka Laitinen:** Investigation, Writing – review & editing. **Kati Katina:** Methodology, Validation, Writing – review &. **Tuula Sontag-Strohm:** Conceptualization, Methodology, Writing – review & editing, Validation, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgments

We thank Vääksy Mill Ltd. for providing us the untreated oat groat samples for this study as a part of OatHow project (Novel indicators and technologies for oat quality and applicability, grant number 6960/31/2018, Business Finland).

References

Ames, N. P., & Rhymer, C. R. (2003). Development of a laboratory-scale flaking machine for oat end product testing. *Cereal Chemistry, 80*, 699–702.

Bryngelsson, S., Dimberg, L. H., & Kamal-Eldin, A. (2002). Effects of commercial processing on levels of antioxidants in oats (Avena sativa L.). *Journal of Agricultural and Food Chemistry, 50*(7), 1890–1896. https://doi.org/10.1021/jf011222z

Campbell, G. M., Webb, C., Owens, G. W., & Scanlon, M. G. (2012). Milling and flour quality. In S. P. Cauvain (Ed.), *Breadmaking: Improving quality* (pp. 188–215). Elsevier Science & Technology.

Deane, D., & Commers, E. (1986). Oat cleaning and processing. Oats. In F. H. Webster (Ed.), *Chemistry and technology* (pp. 371–412). MN, USA: AACC Inc.

Dobraszczyk, B. J., Whitworth, M. B., Vincent, J. F. V., & Khan, A. A. (2002). Single kernel wheat hardness and fracture properties in relation to density and the modelling of fracture in wheat endosperm. *Journal of Cereal Science, 35*(3), 245–263.

Duque, S. M. M., Leong, S. Y., Agyei, D., Singh, J., Larsen, N., & Oey, I. (2020). Understanding the impact of Pulsed Electric Fields treatment on the thermal and pasting properties of raw and thermally processed oat flours. *Food Research International, 129*, Article 108839. https://doi.org/10.1016/j.foodres.2019.108839

Engleson, J. A., & Fulcher, R. G. (2002). Mechanical behavior of oats: Specific groat characteristics and relation to groat damage during impact dehulling. *Cereal Chemistry, 79*(6), 790–797.

Fulcher, R. G. (1986). Morphological and chemical organization of the oat kernel. Oats. In F. H. Webster (Ed.). *Chemistry and technology* (Vol. 1986, pp. 47–74). MN, USA: AACC Inc.

Ganssmann, W., & Vorwerck, K. (1995). Oat milling, processing and storage. In R. W. Welch (Ed.), *The oat crop. Production and utilization* (1st ed., pp. 369–405). Chapman & Hall. Ganssmann and Vorwerck (1995) reported the most detailed information about the traditional hydrothermal treatment of oat that we were able to find (kilning & tempering treatments). This reference gave us a broad overview of the treatment.

Gates, F. (2007). *Role of heat treatment in the processing and quality of oat flakes. EKT series 1380*. University of Helsinki, Department of Food Technology. Gates (2007) also provided a broad view of the hydrothermal treatments of oat, and discussed that the 2-step treatment is not necessary for the enzyme inactivation and end-product quality. In the reference, the treatments of several countries (Germany, Finland, Australia) were discussed.

Gates, F. K., Dobraszczyk, B. J., & Salovaara, H. (2004). Influence of some processing and storage conditions on the mechanical properties of oat flakes. *ASAE, 47*(1), 223–226.

Gates, F. K., Dobraszczyk, B. J., Stoddard, F. L., Sontag-Strohm, T., & Salovaara, H. (2008). Interaction of heat-moisture conditions and physical properties in oat processing: I. Mechanical properties of steamed oat groats. *Journal of Cereal Science, 47*, 239–244. https://doi.org/10.1016/j.jcs.2007.04.003Gates et al. (2008a) studied the hydrothermal treatment parameters on oat groat properties. It was observed that the tempering treatment parameters had influence on oat groat properties, while kilning did not affect groat hardness. This reference supported our hypothesis that with the tempering treatment parameters, oat groat hardness could be controlled, and separate kiln treatment is not necessary.

Gates, F. K., Sontag-Strohm, T., Stoddard, F. L., Dobraszczyk, B. J., & Salovaara, H. (2008b). Interaction of heat-moisture conditions and physical properties in oat processing: II. Flake quality. *Journal of Cereal Science, 48*, 288–293. https://doi.org/10.1016/j.jcs.2007.09.009Gates et al. (2008b) studied the role of the hydrothermal treatment parameters on oat flake properties and observed, that the tempering parameters had an effect on oat flake quality. Instead, kilning had very little effect. This reference supported our hypothesis that oat flake hardness could be altered with the hydrothermal treatment parameters, and separate kiln is not necessary.

Gates, F. K., & Talja, R. (2004). Effect of temperature and moisture on the mechanical properties of oat. *ASAE, 47*(4), 1223–1227.

Girardet, N., & Webster, F. H. (2011). Oat milling: Specifications, storage, and processing. In F. H. Webster, & P. J. Wood (Eds.), *Oats: Chemistry and technology* (Vol. 2, pp. 301–319).

Gondek, E., & Lewicki, P. P. (2006). Antiplasticization of cereal-based products by water. Part II: Breakfast cereals. *Journal of Food Engineering, 77*(3), 644–652. https://doi.org/10.1016/j.jfoodeng.2005.07.023

Head, D. S., Cenkowski, S., Arntfield, S., & Henderson, K. (2010). Superheated steam processing of oat groats. *Lebensmittel-Wissenschaft und -Technologie, 43*(4), 690–694.

Heneen, W. K., Karlsson, G., Brismar, K., Gummeson, P. O., Marttila, S., Leonova, S., Carlsson, A. S., Bafor, M., Banas, A., Mattsson, B., Debski, H., & Stymne, S. (2008). Fusion of oil bodies in endosperm of oat grains. *Planta, 228*(4), 589–599. https://doi.org/10.1007/s00425-008-0761-x

Hutchinson, J. B., Martin, H. F., & Moran, T. (1951). Location and destruction of lipase in oats. *Nature, 167*(4254), 758–759.

Jokinen, I., Pihlava, J. M., Puganen, A., Sontag-Strohm, T., Linderborg, K. M., Holopainen-Mantila, U., Hietaniemi, V., & Nordlund, E. (2021). Predicting the properties of industrially produced oat flours by the characteristics of native oat grains or non-heat-treated groats. *Foods, 10*(7), 1552. https://doi.org/10.3390/foods10071552

Jokinen, I., Sammalisto, S., Silventoinen-Veijalainen, P., Sontag-Strohm, T., Nordlund, E., & Holopainen-Mantila, U. (2022). Variation in the physical properties of oat groats, flakes and oat flake flour–Processability of thirty pure cultivar oat batches from Finland. *Lebensmittel-Wissenschaft und -Technologie.* , Article 113595. https://doi.org/10.1016/j.lwt.2022.113595

Lapveteläinen, A., Alho-Lehto, P., Sinn, L., Laukkanen, T., Lindman, T., Kallio, H., Kaitaranta, J., & Katajisto, J. (2001). Relationships of selected physical, chemical, and sensory parameters in oat grain, rolled oats, and cooked oatmeal—a three-year study with eight cultivars. *Cereal Chemistry, 78*, 322–329.Lapveteläinen et al. (2001) reported the variation in oat flake properties between oat cultivars after similar kiln treatment. This was one of the references supporting our hypothesis, that the oat cultivar samples may perform differently at the similar hydrothermal treatments.

Li, Y., Obadi, M., Shi, J., Xu, B., & Shi, Y. C. (2021). Rheological and thermal properties of oat flours and starch affected by oat lipids. *Journal of Cereal Science, 102*, Article 103337. https://doi.org/10.1016/j.jcs.2021.103337

Lookhart, G., Albers, L., & Pomeranz, Y. (1986). The effect of commercial processing on some chemical and physical properties of oat groats. *Cereal Chemistry, 63*(3), 280–282.

Mahnke-Plesker, S. (1991). *Veränderungen der Inhaltsstoffe bei der hydrothermischen Behandlung von Hafer under Berücksichtigung ernährungsphysiologischer Gesichtspunkte. Dissertation. Detmold.* der Justus-Liebig-Universitet Giesen.

Marcone, M. F., Kakuda, Y., & Yada, R. Y. (1998). Salt-soluble seed globulins of dicotyledonous and monocotyledonous plants II. Structural characterization. *Food Chemistry, 63*(2), 265–274.

Molteberg, E. L., Vogt, G., Nilsson, A., & Frolich, W. (1995). Effects of storage and heat processing on the content and composition of free fatty acids in oats. *Cereal Chemistry, 72*(1), 88–93.

Morris, C. F. (2002). Puroindolines: The molecular genetic basis of wheat grain hardness. *Plant Molecular Biology, 48*(5), 633–647.

Morrison, W. (1981). Starch lipids: A reappraisal. *Starch Staerke, 33*(12), 408–410.

Paton, D. (1987). Differential scanning calorimetry of oat starch pastes. *Cereal Chemistry, 64*(6), 394–399.

Rhymer, C., Ames, N., Malcolmson, L., Brown, D., & Duguid, S. (2005). Effects of genotype and environment on the starch properties and end-product quality of oats. *Cereal Chemistry, 82*(2), 197–203.

Salovaara, H. (1993). Oats in food processes. In T. Aalto-Kaarlehto, & H. Salovaara (Eds.), *The nordic cereal industry in an integrating europe. Proceedings from the 25th nordic cereal congress* (pp. 133–138). Helsinki: University Press.

Sammalisto, S., Laitinen, M., & Sontag-Strohm, T. (2021). Baking quality assessment of twenty whole grain oat cultivar samples. *Foods, 10*(10), 2461. https://doi.org/10.3390/foods10102461

Sibakov, J., Myllymäki, O., Holopainen, U., Kaukovirta-Norja, A., Hietaniemi, V., Pihlava, J. M., Poutanen, K., & Lehtinen, P. (2011). Lipid removal enhances separation of oat grain cell wall material from starch and protein. *Journal of Cereal Science, 54*(1), 104–109. https://doi.org/10.1016/j.jcs.2011.04.003

Sontag-Strohm, T., Aalto-Kaarlehto, T., Horn, E., & Salovaara, H. (1996). Effect of heat treatment on solubility of oat proteins. In *Poster. Seventh international symposium on coeliac disease*. Tampere.

Zhou, M., Robards, K., Glennie-Holmes, M., & Helliwell, S. (1998). Structure and pasting properties of oat starch. *Cereal Chemistry, 75*(3), 273–281.

Zhou, M., Robards, K., Glennie-Holmes, M., & Helliwell, S. (2000). Effects of enzyme treatment and processing on pasting and thermal properties of oats. *Journal of the Science of Food and Agriculture, 80*(10), 1486–1494.