Future Foods 13 (2026) 100918

Contents lists available at ScienceDirect

Future Foods

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

Unlocking the functional potential of asparagus fibre via a novel wet ball milling strategy

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ARTICLE INFO

Keywords:
Ball milling
Asparagus pomace
Water condition
Fibre functionalities
Fibre morphology

ABSTRACT

This study investigated ball milling as a method to transform white asparagus pomace into functionalised fibre. The effects of ball milling conditions (i.e., temperature: 0 °C, −20 °C or −40 °C; water content: pre-dried 9 % or wet 74 %) on the properties of obtained asparagus fibre were evaluated. Ball milling of wet pomace at 0 °C significantly improved water-holding capacity, viscosity, emulsifying stability, and enzymatic digestibility of asparagus fibre, despite not producing the smallest particles. In contrast, ball milling of pre-dried pomace led to the greatest size reduction but resulted in poor functionality of asparagus fibre due to the collapse of the dry fibrous structure that is in the glassy state. Fibre morphological analyses using confocal laser scanning microscopy and optical imaging indicated that changes in particle morphology/microstructure contributed more to the improved functionality than particle size reduction. Nevertheless, milling of wet pomace resulted in asparagus fibre with a dark colour which is likely to be attributed to polyphenol–iron interactions. Pre-treating asparagus pomace using ethylenediaminetetraacetic acid (EDTA) solution effectively reduced the discoloration after milling. These findings highlight that careful control of water content and temperature during ball milling, combined with appropriate pre-treatment, enables an energy-efficient strategy to produce high-functionalized fibre ingredients from fibre-rich plant by-products like asparagus pomace.

1. Introduction

Mechanical processing is widely applied to convert fibre-rich plant materials into dietary fibre ingredients for food applications. Common unit operations include chopping, washing, blanching, drying, milling and sieving (Garcia-Amezquita et al., 2018). Among these, milling is a critical step for reducing particle size and enhancing functional properties. However, traditional dry milling techniques, such as pin milling and knife milling, face several limitations, including inconsistent particle size distribution, heat-induced quality degradation, high energy requirements, and limited improvements in fibre functionality (Aradwad et al., 2021; Jones et al., 2015). Compared to conventional dry milling techniques, wet processing methods such as colloid milling, microfluidization and ultrasound treatment can further reduce particle size and enhance the techno-functional properties of the plant fibres (Chen et al., 2013; Lu et al., 2022; Siddiqui et al., 2023). The presence of water is critical to the success of these wet processing methods (Lu et al., 2022; Ullah et al., 2017). Nevertheless, these wet processing methods typically involve additional steps, including pre-milling, dilution into suspensions and post-drying, which are energy- and cost-intensive and pose challenges for large-scale production (Zimmermann et al., 2016).

Ball milling is a promising alternative mechanical processing method. It operates by rapidly agitating grinding media, such as steel balls, within a rotating or oscillating chamber to apply repeated impact and shear forces to the material. This technique has been shown to significantly reduce particle size and improve the techno-functional properties of various plant fibres, including citrus fibre, hemp fibre and asparagus leaf (Chitrakar et al., 2020; Jiang et al., 2022; Viscusi et al., 2020). Due to its strong ability in particle size reduction, ball milling has also been investigated to produce nanocellulose or nanofibrils from plant fibres (Cebreiros et al., 2024; Zhang et al., 2015). Compared to the aforementioned wet processing techniques, ball milling can directly process coarse fibrous material without diluting it into a suspension, eliminating the need for drying and pre-milling as the pre-treatment steps. Hence, a limited or no drying step is required after ball-milling, which benefits the overall energy efficiency and simplifies the process enormously. For fibre-rich materials such as asparagus pomace, this direct processing route not only simplifies production but

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https://doi.org/10.1016/j.fufo.2026.100918
Received 28 May 2025; Received in revised form 5 December 2025; Accepted 23 January 2026
Available online 26 January 2026

also provides a practical means to valorise an otherwise low-value side stream. By avoiding dilution steps and energy-intensive drying, ball milling contributes to a more circular and efficient utilisation of asparagus residues.

Despite these advantages, ball milling also exhibits several well-documented limitations when applied to plant fibre materials. Mechanical impact and friction can lead to heat generation, which may alter temperature-sensitive compounds or accelerate oxidation and browning reactions. In addition, milling performance is highly dependent on operational variables such as ball size, mass, filling ratio and rotational speed, which may limit reproducibility across different setups (Stolle et al., 2011). Excessive milling can further induce mechanochemical activation or structural degradation, potentially reducing the functional performance of certain fibres (Sitotaw et al., 2023). These considerations highlight the need for controlled milling conditions.

Ball milling can be conducted under ambient, low (0–10 °C), or sub-zero (below 0 °C) temperature conditions and can accommodate both wet and dry materials. While many studies have focused on the effect of milling time (Chitrakar et al., 2020; Viscusi et al., 2020), milling intensity (Ramadhan and Foster, 2018), or combined treatments (Jiang et al., 2022; Zakaria et al., 2014; Zhang et al., 2015), few studies have investigated the effects of water conditions on the ball milling performance of fibres (Sitotaw et al., 2023).

This knowledge gap is noteworthy, as the mechanical forces involved differ between dry and wet ball milling. Dry milling relies mainly on impact and attrition, with milling efficiency enhanced when materials are cooled below their glass transition temperature (Tg), increasing brittleness (Hemery et al., 2011; Paes et al., 2010). In contrast, wet ball milling primarily applies tearing forces (Sitotaw et al., 2023), and the presence of liquid water can disrupt hydrogen bonding between nanofibrils, promoting fibre structural disruption (Lu et al., 2022). However, such studies remain limited, possibly due to the specific equipment requirements. Maintaining stable temperatures at either ambient or below 0 °C, requires continuous cooling to compensate the heat generated during ball milling.

In this study, asparagus pomace from the juiced bottom cut-off of white asparagus is used as the raw material. Approximately one-third of the total length of each white asparagus spear is cut off and discarded due to its firm texture and undesirable mouthfeel (Zhang et al., 2014). Moreover, asparagus fibre produced through conventional drying and milling also exhibit poor techno-functional performance (Lu et al., 2022). This study aims to assess ball milling as a more sustainable technique to functionalize asparagus fibre. We hypothesise that under sub-zero milling conditions, pre-drying raises the glass transition temperature (Tg) of asparagus fibres, thereby increasing brittleness and enhancing milling efficiency. In contrast, under wet milling conditions, the presence of liquid water weakens hydrogen bonding within the fibre network, promoting microstructural disintegration. These distinct water conditions are expected to result in different particle size distributions and techno-functional properties of the milled fibres. To test this hypothesis, asparagus pomace was ball-milled with three water states: sub-zero condition (−20 °C or −40 °C), wet condition (above 0 °C), and low-moisture (pre-dried, −20 °C). The resulting fibres were analysed for their microstructure and techno-functional properties to evaluate milling performance. FTIR was used to examine potential changes in the chemical structure of the fibres. Additionally, ball milling was explored as a pre-treatment to enhance enzymatic hydrolysis, aiming to valorise asparagus pomace as a substrate for fermentable sugar production (Siccama et al., 2022). Fibre colour and visual appearance were also evaluated. Finally, a brief comparison was made between ball milling and colloid milling, focusing on process simplification and energy consumption.

2. Materials and methods

2.1. Sample preparation

2.1.1. Preparation of asparagus pomace

Fresh asparagus bottom cut-offs (typically 4–8 cm in length and 1.5–2.5 cm in diameter) were kindly provided by a local asparagus producer (Teboza BV, Helden, The Netherlands). No additional size-selection or separation of peel and inner tissues was performed, as the mixed cut-offs inherently reflect the natural heterogeneity of the asparagus waste stream. The cut-offs were processed using a juicer (Angel Juicer, Busan, South Korea) to obtain wet pomace, which had a moisture content of approximately 74 g/100 g (wet basis). A portion of the wet pomace was further dried in a fluidized-bed dryer (TG200, Retsch, Haan, Germany) at 50 °C with an airflow of 50 m^3/h for 2 h, yielding dried pomace with a moisture content of 9 g/100 g (wet basis).

2.1.2. Ball milling setting

Ball milling was performed using a laboratory-scale ball mill with two chambers (Mixer Mill MM 500 Control, Retsch, Germany). For each milling run, 15 g of wet pomace or 5 g of dried pomace (equivalent in dry matter) was placed into a 100 mL milling chamber along with 6 stainless steel beads (10 mm in diameter). The equipment was cooled with liquid nitrogen. A pre-cooling step was conducted with the shaking frequency at 3 Hz for 15 min. The milling process was carried out at a shaking frequency of 30 Hz under different conditions, with milling times of 10 min or 30 min and temperature settings of 0 °C, −20 °C, and −40 °C.

Table 1 summarizes the milling conditions, including treatment time, setting temperature, and pomace state. The actual temperature inside the milling chamber after processing was immediately measured using an infrared thermal camera, and the corresponding images are provided in Figure A1 (Appendix). As shown in Figure A1, when the milling temperature was set to 0 °C, the actual chamber temperature reached 5 °C due to heat generation during milling, preventing the water in the pomace from freezing. Treatments conducted at −20 °C and −40 °C were classified as sub-zero ball milling. All milling experiments were performed in triplicate, and the samples were stored at 4 °C for further analysis.

2.2. Techno-functional properties of milled fibre

Water holding capacity (WHC), swelling capacity (SWC) and emulsifying activity (EA) were measured following the methods from previous study (Lu et al., 2022). The moisture content of the ball-milled samples was determined by oven drying, and the amount of water added was adjusted accordingly to prepare suspensions.

For WHC, a 1 g fibre/100 g suspension was prepared, centrifuged (3000×g, 20 min, 20 °C), and the pellet was weighed before and after drying at 105 °C. WHC (%) was calculated as:

Table 1
Ball milled samples prepared with different moisture content, setting temperatures and milling time.

Sample state	Sample name	Temperature (°C)	Moisture content (Wet basis)	Time (minutes)
Wet	T0t10	0	74 %	10
	T0t30	0	74 %	30
Sub-zero (Frozen)	T−20t10	−20	74 %	10
	T−20t30	−20	74 %	30
	T−40t10	−40	74 %	10
	T−40t30	−40	74 %	30
Dried	T−20t10-D	−20	9 %	10
	T−20t30-D	−20	9 %	30

Note: Capital T refers to setting temperature, Lowercase t refers to milling time. D refers the dried pomace.

$$WHC = \frac{M_{sf} - M_{sd}}{M_{sd}} * 100\% \quad (1)$$

Where M_{sf} (g) is the mass of the fresh pellet and M_{sd} (g) is the mass of the dried pellet.

For SWC, a 10 mL 1 g fibre/100 g suspension was equilibrated for 24 h, and the SWC was calculated:

$$SWC = \frac{V_s}{M_s} \quad (2)$$

Where V_s(mL) is the volume of the particle sediment layer and M_s (g) is the mass of the powder in the suspension.

For EA, a 1 g fibre/100 g suspension (30 mL) was mixed with sunflower oil (7.5 mL), and subsequently homogenized with a T18 digital Ultra Turrax (IKA, Staufen, Germany) at 10,000 rpm for 1 min. After 14 days, the EA was measured:

$$EA = \frac{V_{EL}}{V} * 100\% \quad (3)$$

V_{EL} is the volume of the emulsified layer (mL) and V is the total volume of the mixture (mL).

2.3. Microstructure observation and analysis

2.3.1. Particle size and elongation analysis

Particle size was measured using a Mastersizer 3000 (Malvern Instruments, UK) with a Hydro-MV setup, using a refractive index of 1.468. A 1 gfibre/100 gsuspension was prepared in Milli-Q water for the measurements. The volume mean diameter (D[4,3]), which represents the average particle size weighted by volume, was used to characterize the distribution.

The particle elongation analysis was performed according to the method described in our previous study (Lu et al., 2023). Elongation is defined as 1 minus the ratio of particle width to length, describing how stretched or fibre-like a particle is. A 30 μL of diluted suspension was placed on a microscope slide and scanned at 10× magnification using Malvern Morphologi 4 (Malvern Instruments, Worcestershire, UK). The morphological parameters Elongation was calculated by the Morphologi software v10.34. Particles smaller than 2 μm or an elongation less than 0.2 were excluded from the analysis to exclude air bubbles.

2.3.2. Confocal laser scanning microscopy

Confocal laser scanning microscopy (CLSM; Stellaris 5, Leica, Germany) was used to examine the microstructure of milled asparagus pomace. The pomace was diluted to a 1 g fibre/100 g suspension, and a 30 mL suspension was stained with 25 μL Calcofluor white (0.1 g/L, excitation at 405 nm). Images were captured with a 20× lens at 100 Hz (512 × 512 pixels) and processed using LAS X Small software.

2.4. Fourier transform infrared spectroscopy (FT-IR) analysis

FTIR spectra were collected from diluted aqueous suspensions of ball-milled asparagus fibre using a FTIR spectrometer (INVENIO FT-IR Spectrometer, Bruker, USA). Wavenumbers ranged from 4000 cm−1 to 900 cm−1.

2.5. Cellulase efficiency to hydrolyse milled fibre

Enzymatic hydrolysis was performed using an earlier reported method (Siccama et al., 2022). Briefly, 0.0375 g of asparagus fibre (calculated with dry weight) was suspended in 1.5 mL of 0.5 M citrate buffer (pH 5.0) to achieve a solid loading of 25 g/L. Cellulase (Celluclast®, kindly provided by Novozymes, Bagsværd, Denmark) was added at 700 nkat /g of substrate, and the reaction was carried out with Eppendorf thermomixer (Thermo Fisher Scientific, Waltham, USA) at 50 °C, 900 rpm for 6 h. The cellulase hydrolysed fibre solution were analysed by using high-performance liquid chromatography (HPLC) (Thermo Ultimate 3000 HPLC, ThermoFisher Scientific, Waltham, USA) with a Shodex KS-802 8.0 × 300 mm column (Showa Denko K·K., Tokyo, Japan). Cellobiose was used as the target compound to assess the efficiency of enzymatic hydrolysis.

2.6. Colour analysis of milled asparagus fibres

The colour of the milled samples was measured by the Chroma Meter CR-400 (Konica Minolta, Japan). For each measurement, 2 *g* of a sample was placed into a transparent measuring cup. The average value of L* represents lightness which evaluates the whiteness of the sample.

2.6.1. Pre-treatment of asparagus pomace to investigate colour change

To investigate strategies for mitigating colour changes during wet ball milling, asparagus pomace was subjected to the following pre-treatments: 1) water washing, 2) acid treatment (pH adjusted to 3 with hydrochloric acid), 3) thermal treatment (heating at 95 °C for 10 min in a water bath), 4) antioxidant soaking (1 mmol/L-Ascorbic acid), 5) EDTA soaking (1 mmol/L ethylenediaminetetraacetic acid). All reagents used were of reagent grade. L-Ascorbic acid (≥98 %), EDTA disodium salt dihydrate (≥99 %), citric acid (≥99 %), and hydrochloric acid (37 %) were purchased from Sigma-Aldrich, Netherlands. For acid, antioxidant, and EDTA treatments, 20 g of wet pomace was immersed in 200 mL of the corresponding solution and stirred for 2 h. The pomace was then squeezed to adjust the moisture content to approximately 74 g/100 g, matching that of the untreated sample. For the washing treatment, the soaking and squeezing process was repeated three times to ensure thorough rinsing. The corresponding ball milling conditions are described in Section 2.1.2.

2.7. Statistical analysis

All samples were prepared and analysed in triplicate, and the experimental results were expressed as the mean ± standard deviation (Mean ± SD). The techno-functionalities, elongation and cellobiose yield of fibre samples were assessed on significance with SPSS Statistics.25. One-way analysis of variance (ANOVA) and Tukey's HSD post hoc test were performed and the difference between samples was determined statistically significant if $p \leq 0.05$. Unpaired *t*-tests were conducted to compare 10 min and 30 min treatments within each group for the techno-functional properties.

3. Results and discussion

The techno-functional properties of dietary fibres are indicative for potential applications and can be improved upon processing. This section starts with a report on the evaluation of the impact of different ball milling conditions on functional properties. Subsequently, these results are linked to particle size and morphology, as assessed by laser diffraction, microscopy, and morphology analysis. Then, changes in chemical structure are examined using FTIR, followed by an evaluation of enzymatic hydrolysis efficiency. Finally, we report on the visual appearance and colour stability of the milled fibres, along with potential strategies to mitigate discoloration.

3.1. Techno-functional properties of milled fibre

The original untreated asparagus pomace consisted of fibrous strands (1~2 cm in length), which could not be reliably assessed for swelling capacity, water holding capacity, or emulsifying activity due to their coarse and non-dispersible nature. Therefore, only ball-milled samples were evaluated for these properties. The results demonstrate that water content and milling temperature are the dominant factors influencing the techno-functional properties of ball-milled asparagus pomace

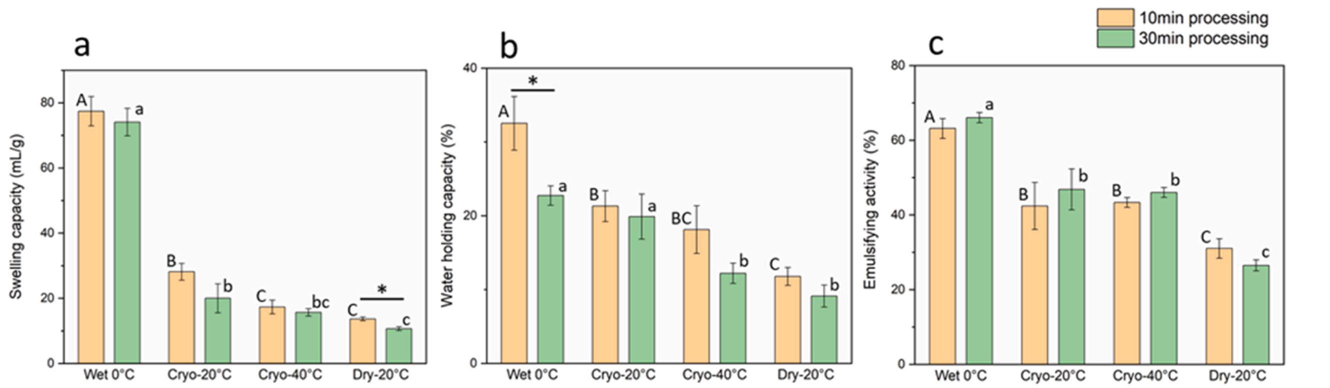

Fig. 1. Techno-functionalities of asparagus pomace with various ball-milling conditions: (a) swelling capacity, (b) water holding capacity, and (c) emulsifying activity. Different capital letters indicate significant differences among treatments at 10 min, and lowercase letters indicate differences at 30 min, based on Tukey's HSD test ($p < 0.05$). Asterisk (*) labels significant differences between 10 min and 30 min within the same treatment group, based on unpaired *t*-tests ($p < 0.05$).

(Fig. 1). Milling wet pomace at 0 °C consistently resulted in the highest swelling capacity, water-holding capacity, and emulsifying activity across treatments. In contrast, pre-drying and milling resulted in the lowest techno-functional properties, while sub-zero milling at −20 °C or −40 °C showed minimal differences. For most milling conditions, extending the milling time from 10 to 30 min did not significantly enhance techno-functional properties, and even led to slight decreases in swelling and water-holding capacity.

Previous studies suggested that the presence of water (e.g. during colloid milling) enhances the techno-functional properties of dry-milled plant fibres, likely due to water's role in fibre hydration and structural modification (Lu et al., 2022). In this study, ball milling conditions were precisely controlled, and processing wet fibres at temperatures above 0 °C consistently yielded the highest techno-functional properties, compared to dry or frozen samples. Notably, asparagus pomace processed with wet ball milling showed better techno-functional properties than those obtained through colloid milling in a previous study (Lu et al., 2022), highlighting the potential of this method.

The impact of milling time on techno-functional properties remains inconclusive. Some studies report that prolonged milling reduces fibre functionality, likely due to excessive fragmentation and structural degradation (Chitrakar et al., 2020; Ramachandraiah and Chin, 2016). Conversely, other studies suggest that longer milling times enhance fibre functionality (Jiang et al., 2022). Given the importance of techno-functional properties for fibre applications in foods, optimizing these factors could enhance fibre utilization while reducing energy consumption. However, a deeper understanding requires further microstructural analysis of asparagus pomace.

3.2. Microstructure observation and analysis

3.2.1. Particle size distribution

The original, untreated asparagus pomace consisted of coarse fibrous strands with approximately 1~2 cm in length (raw material in Fig. 7). Particle size decreased significantly with prolonged milling time (Fig. 2). For samples in frozen (−20 °C, −40 °C) or wet (0 °C) conditions, the particle size decreased from approximately 500 μm to 100 μm after 30 min of milling. However, the pre-dried sample exhibited the most obvious size reduction, the peak of size distribution reached around 20 μm after 30 min. These results indicate that extending ball milling time effectively reduces particle size, with the more brittle pre-dried pomace achieving the smallest size.

Techno-functional properties are essential for dietary fibre applications, and milling is a key step in fibre processing. Some studies have shown that reducing the particle size of insoluble dietary fibres (carrot and grape pomace) can enhance their functionality (Ma et al., 2016; Yang et al., 2019), while others have reported opposite results (Cadden,

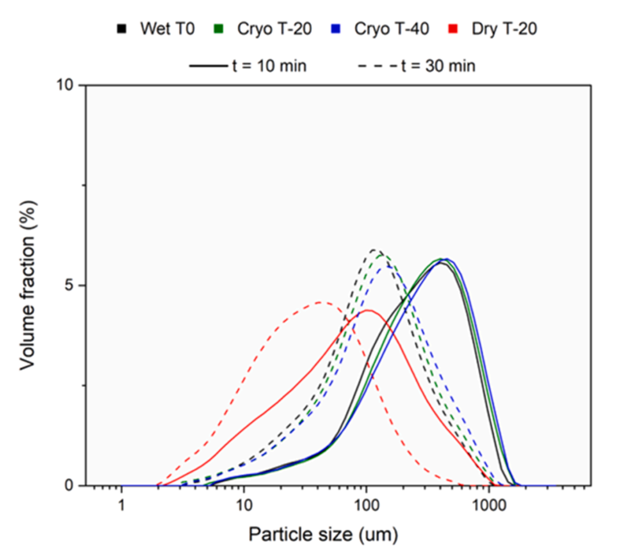

Fig. 2. Particle size distribution of ball milled white asparagus pomace with various ball-milling conditions.

1987; Gidley and Yakubov, 2019). One study suggested that optimal functionality requires a specific particle size range (Ma and Mu, 2016), as both excessively fine particles (which may be loose fibril structures) and coarse particles (which have low surface area) can reduce the techno-functional performance. In this study, the pre-dried sample with the finest particle size showed the lowest techno-functional properties. In contrast, the wet ball-milled sample (0 °C) had the highest functionality, despite having a particle size similar to the sub-zero ball milled samples (−20 °C, −40 °C). These results suggest that particle size alone cannot explain the differences in functionality. According to our previous study (Lu et al., 2023), particle morphology can be more closely linked to fibre functionality than size. To further investigate, microscopy images and morphological analyses of ball milled fibres were conducted to explain their differences in techno-functionalities.

3.2.2. Confocal laser scanning (CLSM) microscopy

Compared to the particle size distribution results, the CLSM images in Fig. 3 provide more detailed insights into the morphology of the ball-milled fibres. In the sub-zero milled samples (−20 °C and −40 °C), the size reduction was limited, and many fibre particles retained their original elongated structure. In the wet ball-milled sample (0 °C), the asparagus pomace was fragmented into smaller pieces while maintaining a long, fibrous shape. These elongated fragments appeared to entangle and aggregate, forming a loosely connected network. This

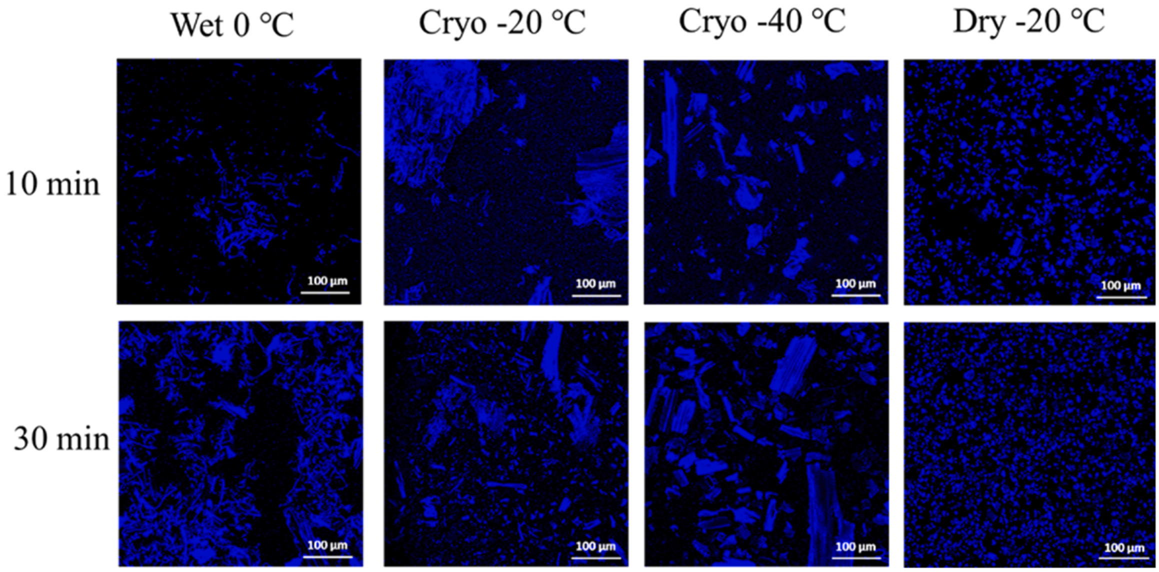

Fig. 3. Confocal Laser Scanning Microscopy (CLSM) images of ball-milled white asparagus pomace under various milling conditions. Fibres were stained with Calcofluor White and appear in blue.

elongated morphology and entangled structure may explain why the visual particle size appears smaller than that measured by the Mastersizer in Fig. 2. In contrast, the dry ball-milled samples exhibited the most pronounced size reduction. However, the resulting particles lost their fibrous structure entirely, and transformed into short, solid, and non-entangled particles.

In terms of milling efficiency, as indicated by particle size reduction, dry ball milling at −20 °C achieved the greatest effect, while sub-zero milling of non-dried samples (−20 °C or −40 °C) was much less effective. This observation aligns with the proposed hypothesis regarding the role of glass transition temperature (T_g) in increasing fibre brittleness. Asparagus fibre is predominantly composed of cellulose, a polymer whose T_g is highly dependent on moisture content (Paes et al., 2010; Siccama et al., 2022). Water acts as a plasticizer and thereby lowering the T_g of cellulosic materials. The glass transition temperatures of water and dry cellulose are approximately 136 K and 493 K, respectively (Salmen and Back, 1977). The same study also showed that changing the water content from 11 to 30 g /100 g changed the T_g from 240 K (−33 °C) to 203 K (−70 °C). Therefore, the pre-dried asparagus pomace (9 % moisture) likely exhibited a T_g sufficiently above −20 °C, which indicates that this pomace was in the glassy state and thus underwent efficient fracture upon impact with milling media. In contrast, the high-moisture asparagus pomace had a T_g well below −40 °C, and therefore remained in the rubbery state throughout sub-zero milling and thus resisted fragmentation by impact.

During wet ball milling at 0 °C, a different milling mechanism is expected. The dominant mechanical forces shift from impact to shear and tearing forces (Sitotaw et al., 2023). The presence of liquid water can facilitate the disruption of hydrogen bonding within the fibre matrix, enabling fibre fragmentation while preserving their elongated morphology. Although sub-zero dry milling exhibited the highest milling efficiency in terms of particle size reduction, the resulting fibres showed the lowest techno-functional properties. This suggests that milling performance should not be evaluated solely based on particle size, but also on how the microstructural changes contribute to functional performance.

The capillary structure formed by pores and cavities in between and in the plant fibres plays a key role in their water-holding capacity (Teixidó et al., 2022). A previous study demonstrated that reducing the particle size of wheat bran led to a decrease in water-holding capacity, primarily due to the collapse of its fibre matrix (Cadden, 1987). Collapse of the fibre matrix refers to the loss of internal porosity and fibrillar architecture that facilitate capillary water retention. This helps explaining why sub-zero milled samples, retained more of the fibre structure, exhibiting higher techno-functionalities, while dry ball-milled samples that lost their fibrillar texture, showed the lowest. Interestingly, the wet ball-milled samples displayed the highest techno-functional properties despite undergoing size reduction. As shown in our previous study (Lu et al., 2022), wet processing methods can better preserve the native fibre structure compared to conventional dry milling. The resulting long, branched fibres tend to form entangled networks, which enhance properties such as water-holding capacity, viscosity, and emulsifying stability. Furthermore, our recent study (Lu et al., 2023) introduced 'elongation' as a morphological parameter to quantify the degree of fibre fibrillation during processing.

3.2.3. *Elongation morphology analysis of milled particles*

The elongation of asparagus fibre particles varied markedly across water conditions (Fig. 4). Elongation is defined as ‘1 − (width/length)’, indicating the degree of particle stretch. A value of 0 corresponds to a spherical particle, while values approaching 1 represent highly elongated, fibrous structures. Consistent with observations from CLSM images, fibres subjected to wet and sub-zero ball milling exhibited similarly high elongation values, while those from dried pomace showed significantly lower elongation.

After screening of fibres from various plant sources it was shown that fibre particles with higher elongation tend to exhibit better techno-functional properties (Lu et al., 2023). This aligns with our findings: although dry ball milling resulted in the greatest particle size reduction, it produced fibres with the lowest elongation and the poorest techno-functionalities. This suggests that, beyond the raw material source, processing conditions can affect fibre microstructure and, consequently, the functionality. Overall, among all the moisture conditions tested, 10 min of wet ball milling (0 °C) did not only effectively reduce the particle size of asparagus pomace but better preserved the elongated fibre structure, resulting in improved techno-functional properties. Achieving such results during a short milling treatment highlights the potential of wet ball milling for improving both product properties and energy efficiency.

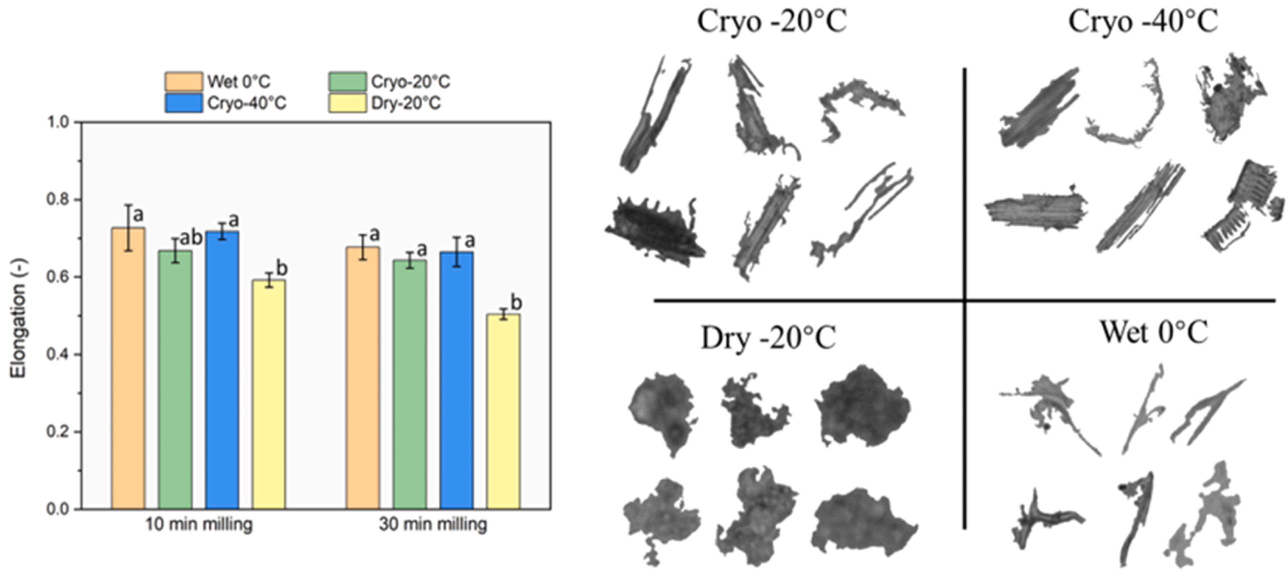

Fig. 4. Elongation values (left) and representative particle morphologies (right) of ball-milled white asparagus pomace under different water conditions after 30 min of treatment. Images on the right illustrate the observed particle morphologies for the different conditions.

3.3. Fourier transform infrared spectroscopy (FT-IR) analysis

As milling time had limited effect on asparagus fibre properties, only samples milled for 30 min were analysed with FTIR (Fig. 5). The most notable difference among the samples was observed in the region between 950 and 1150 cm^{-1} (highlight within the vertical zone), where the band intensity was lowest in the wet ball-milled sample (0 °C). Previous studies have attributed the peak near 1040 cm^{-1} to O–H stretching in lignin or C–OH bending vibrations in hemicellulose (Khan et al., 2018; Ramadoss and Muthukumar, 2016). The diminished intensity of this peak may indicate disruption of intermolecular hydrogen bonding within the polysaccharide matrix during wet milling. Similar changes have been reported in corn stalk fibres subjected to superfine milling, where disruption of hydrogen bonding led to the formation of amorphous cellulose and soluble saccharides (Zhao et al., 2013). In contrast, other spectral regions remained relatively unchanged. In particular, the peaks near 2920 cm^{-1} and 2850 cm^{-1}, which correspond to C–H stretching vibrations in –CH_2– groups and are characteristic of the cellulose backbone (Baeza and Freer, 2000), showed consistent position and intensity across all samples. This suggests that while the hydrogen bonding network may have been disrupted, the overall chemical structure of cellulose remained intact during ball milling. These results support previous findings that wet processing promotes the breakdown of hydrogen bonds, exposing more hydrophilic groups, which may explain the improved techno-functional properties like higher water-holding capacity (Lu et al., 2023).

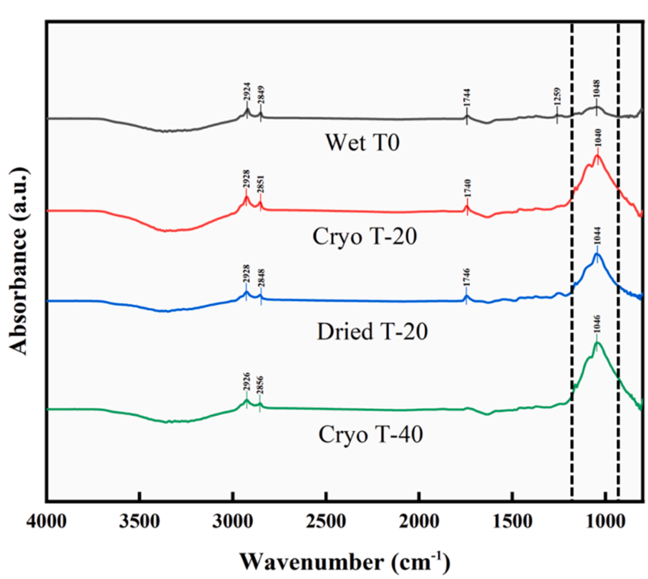

Fig. 5. The FTIR spectra of the white asparagus pomace treated with different ball milling settings.

3.4. Cellulase efficiency to hydrolyse milled fibre

To explore how ball milling affects the enzymatic digestibility of asparagus fibre, all samples were subjected to enzymatic hydrolysis, and the resulting cellobiose yield was measured (Fig. 6). All ball-milled samples produced higher cellobiose yields than the untreated control. The wet ball-milled sample (0 °C) and dry ball-milled sample achieved the highest yield, significantly greater than the untreated fibre ($p \leq 0.05$). Sub-zero but non-drying samples also showed improved hydrolysis performance, though the differences were less pronounced.

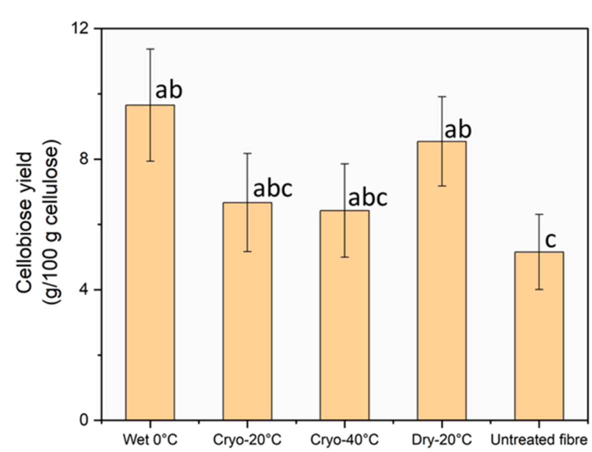

Fig. 6. Cellobiose yield of untreated and ball-milled asparagus pomace under various milling conditions.
Note: The different superscript letters on column label significant differences ($p \leq 0.05$).

Interestingly, despite having the smallest particle size, the dry ball-milled sample did not result in the highest enzymatic yield.

Previous studies have reported a positive correlation between the specific surface area of milled wood powders and their enzymatic saccharification efficiency (Zhu et al., 2009). This relationship can explain the high hydrolysis efficiency observed for the dry ball-milled sample, as extensive size reduction increases the accessible surface area for enzyme binding. However, in this study, wet ball milling also generated a favourable microstructure for enzymatic hydrolysis. As discussed earlier, wet ball milling loosened the fibre matrix of asparagus pomace, producing long, branched, and porous fragments. The presence of water during milling likely facilitated the disruption of internal hydrogen bonds, thereby exposing additional binding sites for enzymatic action. This may explain why the wet ball-milled fibres, despite having a larger apparent particle size than the dry-milled ones, exhibited higher hydrolysis efficiency—attributable to their more open and enzyme-accessible structure. This interpretation is consistent with previous reports showing that cellulose samples with higher water sorption capacity exhibit higher enzymatic hydrolysis efficiency, as increased pore volume and fibre swelling improve cellulase accessibility (Ogeda et al., 2012). This finding supports the potential of wet ball milling as an effective mechanical pre-treatment to enhance the enzymatic digestibility of fibre-rich by-products such as asparagus pomace.

Beyond the improvements in hydration behaviour, emulsifying performance, and enzymatic digestibility, these functional changes also hold practical relevance for food applications. The increased WHC and viscosity suggest that wet ball-milled fibres can act as structuring agents in plant-based or fat-reduced products (Stanišić et al., 2025). The enhanced emulsifying stability further indicates their potential as clean-label stabilisers in oil-in-water systems (Zhang et al., 2024). In addition, the higher enzymatic digestibility suggests greater accessibility for microbial fermentation, indicating potential prebiotic applications (Gill et al., 2021). These attributes highlight wet ball-milled asparagus fibre as a versatile ingredient for emerging food innovations.

3.5. Colour change of milled asparagus fibre

Although wet ball milling (0 °C) can effectively reduce particle size and enhance techno-functional properties, a major drawback is the noticeable darkening of the fibre (Fig. 7A). This discoloration is likely due to the oxidation of phenolic compounds and their interactions with proteins during processing (Kroll et al., 2003; Zhang et al., 2021). Mechanical disruption of the cell wall can release bound phenolics, which may undergo enzymatic browning catalysed by polyphenol oxidase (PPO), similar to the browning observed in apples and potatoes (Arnold and Gramza-Michałowska, 2022; Cantos et al., 2002; Singh et al., 2018). Alternatively, non-enzymatic browning pathways such as Maillard reactions and caramelization may contribute (Wang et al., 2023). However, the low-temperature milling conditions (< 10 °C) used in this study do not favour the occurrence of Maillard reactions or caramelisation, suggesting that these pathways play a minimal role in the observed colour changes. Chelation of phenolics with metal ions, especially iron, has also been reported as a cause of dark pigment formation in plant tissues (Chen et al., 2024; Nath et al., 2022). White asparagus contains about 0.3 mg Fe per 100 g fresh weight (Wichrowska et al., 2018), providing sufficient metal ions for such complexation reactions. This colour change is undesirable for applications where a neutral appearance is preferred, highlighting the need for strategies to control

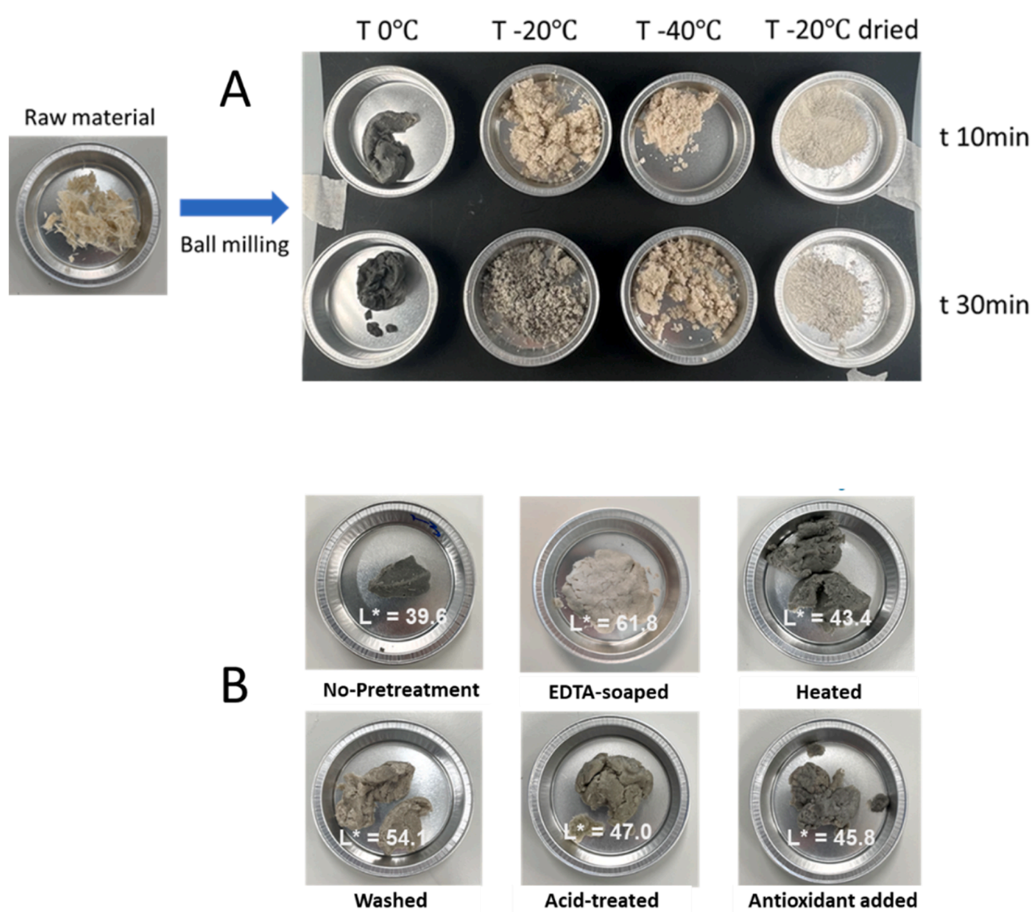

Fig. 7. Visual appearance of asparagus pomace under different ball milling conditions (A) and after 10 min wet ball milling with various pre-treatments (B). The L* value in the pictures indicates the measured lightness.

discolouration during fibre processing.

In contrast, the dried sample exhibited the lightest colour, likely because pre-drying at elevated temperatures denatured PPO and other enzymes involved in enzymatic browning. Differences in water activity among treatments can alter PPO activity and further explain the observed colour patterns (Korbel et al., 2013). Wet-milled samples, with high moisture content, provide favourable conditions for PPO activity, whereas freezing at sub-zero temperatures reduces water activity and limits enzymatic browning. The dried fibres, characterised by very low water activity in addition to enzyme denaturation, therefore showed minimal oxidative discolouration.

To prevent fibre darkening during wet ball milling and identify its main contributing factors, several pre-treatment strategies were applied: washing, acid treatment, heat treatment, and the addition of antioxidants or EDTA. Washing aimed to remove interacting compounds such as phenolics, proteins. Acid treatment was used to suppress protein–-phenol interactions and reduce enzymatic activity. Heat treatment served to denature enzymes in the pomace, thereby limiting enzymatic oxidation. Antioxidants were added to block oxidation reactions during milling. Finally, EDTA was used to prevent possible chelation of polyphenols with metal ions.

All pre-treatments had some effect in reducing the colour change, with EDTA soaking showing the most significant improvement (Fig. 7B). These results suggest that the darkening of white asparagus pomace during wet ball milling could be due to reactions between polyphenols and iron ions, leading to the formation of dark-coloured metal–phenol complexes (Chen et al., 2024; Mellican et al., 2003). This hypothesis is supported by previous studies that reported the high content of both polyphenols and mineral elements (e.g., iron, zinc, and copper) in asparagus (López et al., 1999; Pegiou et al., 2020; Yu and Fan, 2021). Simple washing was ineffective in removing phenolics, as total phenolic content has been shown to increase with prolonged ball milling due to cell rupture (Chitrakar et al., 2020). Other studies have proposed strategies to mitigate undesired colour changes caused by polyphenol interactions, including the use of ferrous sulphate, citrate, and micronutrients such as calcium and zinc (Habeych et al., 2016). This finding could help optimize processing conditions for producing fibre with a more neutral appearance.

3.6. Energy and process flow advantages of wet ball milling

In addition to the functional benefits, wet ball milling offers important advantages in terms of process efficiency and energy use. Unlike dry ball milling, wet ball milling can process fresh asparagus pomace directly without prior drying. Drying is typically one of the most energy-intensive steps in fibre processing, with reported energy efficiencies often below 30 % (Barrios et al., 2023; Menon et al., 2020). Using an energy efficiency of 30 %, approximately 16 MJ of energy is required to reduce the moisture content of asparagus pomace from 70 to 8 g/100 g in weight, yielding 1 kg of dry pomace (latent heat value 2.3 MJ/kg). Based on a standard calorific value (31.65 MJ/m^3) of natural gas in the Netherlands, 0.492 m^3 of natural gas would then be consumed to produce 1 kg of dried asparagus pomace, (www.rvo.nl). Alternatively, sub-zero ball milling would also consume a lot of energy, as it requires freezing of the product. This leads to significantly higher energy use and equipment costs. If long-term preservation is required, an additional drying step would still be necessary for the wet-milled fibres, resulting in additional energy consumption. Nevertheless, the increased evaporation surface area generated during milling may enhance drying efficiency and partially offset the corresponding energy demand.

An interesting comparison can be made between wet ball milling and other wet mechanical fibre processing methods, such as colloid milling which we previously studied (Lu et al., 2022). Colloid milling of asparagus pomace requires a pre-drying and dry milling step, followed by rehydration to create a dilute fibre suspension with a solids content below 5 g/100 g. The low fibre content of the resulting suspension often requires additional dewatering steps to facilitate storage and transportation. In contrast, wet ball milling can directly process fresh asparagus pomace, producing milled fibres with a significantly higher solids content (~30 %). Therefore, compared to colloid milling, wet ball milling not only greatly simplifies the processing chain, but also reduces energy input required for pre-drying, initial milling, and post-processing dewatering.

4. Conclusion

This study showed that the presence of water during ball milling dramatically influenced the structure and functionality of milled asparagus fibre. Dry ball milling reduced particle size most efficiently but led to poor functionality due to structural collapse. This can be related to reaching the glass state of fibres upon drying, which promotes brittleness but compromises morphology. Wet ball milling at 0 °C for 10 min produced fibres with improved techno-functional properties due to better preservation of the fibrous structure. Fibre morphology, especially elongation, was more important than size alone in determining functionality. Darkening during wet milling was likely due to polyphenol–iron interactions, and EDTA pre-treatment was the most effective in reducing this effect. These results suggest that wet ball milling, combined with proper pre-treatment, is a promising method for producing high-quality fibre ingredients from asparagus pomace. Future research could focus on scaling up the process and assessing its energy efficiency and economic feasibility at pilot-scale settings. In addition, other fibre sources could be tested to evaluate whether similar effects can be achieved. The applicability of wet ball-milled fibres in food products and their sensory impact also warrant further investigation.

CRediT authorship contribution statement

Yifeng Lu: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Jingwei Liu:** Methodology, Investigation, Formal analysis. **Lu Zhang:** Writing – review & editing, Methodology, Conceptualization. **Maarten A.I. Schutyser:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization.

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.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.fufo.2026.100918.

Data availability

Data will be made available on request.

References

Aradwad, P.P., T v, A.K., Sahoo, P.K., Mani, I., 2021. Key issues and challenges in spice grinding. Clean. Eng. Technol. 5, 100347. https://doi.org/10.1016/j.clet.2021.100347.

Arnold, M., Gramza-Michałowska, A., 2022. Enzymatic browning in apple products and its inhibition treatments: a comprehensive review. Compr. Rev. Food Sci. Food Saf. 21, 5038–5076. https://doi.org/10.1111/1541-4337.13059.

Baeza, J., Freer, J., 2000. Chemical Characterization of Wood and Its Components, in: Wood and Cellulosic Chemistry, Revised, and Expanded. Marcel Dekker Inc., New York, pp. 275–384.

Barrios, N., Marquez, R., McDonald, J.D., Hubbe, M.A., Venditti, R.A., Pal, L., 2023. Innovation in lignocellulosics dewatering and drying for energy sustainability and enhanced utilization of forestry, agriculture, and marine resources - a review. Adv. Colloid Interface Sci. 318, 102936. https://doi.org/10.1016/j.cis.2023.102936.

Cadden, A.-M., 1987. Comparative effects of particle size reduction on physical structure and water binding properties of several plant fibers. J. Food Sci. 52, 1595–1599. https://doi.org/10.1111/j.1365-2621.1987.tb05886.x.

Cantos, E., Tudela, J.A., Gil, M.I., Espín, J.C., 2002. Phenolic compounds and related enzymes are not rate-limiting in browning development of fresh-cut potatoes. J. Agric. Food Chem. 50, 3015–3023. https://doi.org/10.1021/jf0116350.

Cebreiros, F., Seiler, S., Sánchez, G., Lareo, C., 2024. Sequential ball milling as a promising method for the isolation of cellulose nanofibers (CNF) from enzyme-treated eucalyptus kraft pulp. Ind. Crops Prod. 220, 119293. https://doi.org/10.1016/j.indcrop.2024.119293.

Chen, J., Gao, D., Yang, L., Gao, Y., 2013. Effect of microfluidization process on the functional properties of insoluble dietary fiber. Food Res. Int. 54, 1821–1827. https://doi.org/10.1016/j.foodres.2013.09.025.

Chen, X., Chen, Z., Duan, R., Yan, S., Li, J., 2024. Formation mechanism of blue pigment in boiled lotus rhizome discs: insight into the chelation of polyphenols and iron. Food Res. Int. 192, 114818. https://doi.org/10.1016/j.foodres.2024.114818.

Chitrakar, B., Zhang, M., Zhang, X., Devahastin, S., 2020. Bioactive dietary Fiber powder from asparagus leaf by-product: effect of low-temperature ball milling on physico-chemical, functional and microstructural characteristics. Powder Technol. 366, 275–282. https://doi.org/10.1016/j.powtec.2020.02.068.

Garcia-Amezquita, L.E., Tejada-Ortigoza, V., Serna-Saldivar, S.O., Welti-Chanes, J., 2018. Dietary Fiber concentrates from fruit and vegetable by-products: processing, modification, and application as functional ingredients. Food Bioprocess Technol. 11, 1439–1463. https://doi.org/10.1007/s11947-018-2117-2.

Gidley, M.J., Yakubov, G.E., 2019. Functional categorisation of dietary fibre in foods: beyond 'soluble' vs 'insoluble. Trends Food Sci. Technol. 86, 563–568. https://doi.org/10.1016/j.tifs.2018.12.006.

Gill, S.K., Rossi, M., Bajka, B., Whelan, K., 2021. Dietary fibre in gastrointestinal health and disease. Nat. Rev. Gastroenterol. Hepatol. 18, 101–116. https://doi.org/10.1038/s41575-020-00375-4.

Habeych, E., van Kogelenberg, V., Sagalowicz, L., Michel, M., Galaffu, N., 2016. Strategies to limit colour changes when fortifying food products with iron. Food Res. Int. 88, 122–128. https://doi.org/10.1016/j.foodres.2016.05.017.

Hemery, Y., Chaurand, M., Holopainen, U., Lampi, A.-M., Lehtinen, P., Piironen, V., Sadoudi, A., Rouau, X., 2011. Potential of dry fractionation of wheat bran for the development of food ingredients, part I: influence of ultra-fine grinding. J. Cereal Sci. 53, 1–8. https://doi.org/10.1016/j.jcs.2010.09.005.

Jiang, Z., Mu, S., Ma, C., Liu, Y., Ma, Y., Zhang, M., Li, H., Liu, X., Hou, J., Tian, B., 2022a. Consequences of ball milling combined with high-pressure homogenization on structure, physicochemical and rheological properties of citrus fiber. Food Hydrocoll. 127, 107515. https://doi.org/10.1016/j.foodhyd.2022.107515.

Jiang, Z., Zhang, M., Huang, Y., Ma, C., Mu, S., Li, H., Liu, X., Ma, Y., Liu, Y., Hou, J., 2022. Comparison and characterization of the structure and physicochemical properties of three citrus fibers: effect of ball milling treatment. Foods 11, 2665. https://doi.org/10.3390/foods11172665.

Jones, J.M., Adams, J., Harriman, C., Miller, C., Van Der Kamp, J.W., 2015. Nutritional impacts of different whole grain milling techniques: a review of milling practices and existing data. Cereal Foods World 60, 130–139. https://doi.org/10.1094/CFW-60-3-0130.

Khan, G.M., Khan, N.M., Khan, Z.U., Ali, F., Jan, A.K., Muhammad, N., Elahi, R., 2018. Effect of extraction methods on structural, physiochemical and functional properties of dietary fiber from defatted walnut flour. Food Sci. Biotechnol. 27, 1015–1022. https://doi.org/10.1007/s10068-018-0338-9.

Korbel, E., Attal, E.-H., Grabulos, J., Lluberas, E., Durand, N., Morel, G., Goli, T., Brat, P., 2013. Impact of temperature and water activity on enzymatic and non-enzymatic reactions in reconstituted dried mango model system. Eur. Food Res. Technol. 237, 39–46. https://doi.org/10.1007/s00217-013-2026-6.

Kroll, J., Rawel, H.M., Rohn, S., 2003. Reactions of plant phenolics with food proteins and enzymes under special consideration of covalent bonds. Food Sci. Technol. Res. 9, 205–218. https://doi.org/10.3136/fstr.9.205.

López, M.A.A., Rojas, R.M., Cosano, G.Z., Segarra, P.J.S., 1999. Nutritional changes in the essential trace elements content of asparagus during industrial processing. Food Res. Int. 32, 479–486. https://doi.org/10.1016/S0963-9969(99)00111-8.

Lu, Y., Kokje, T., Schutyser, M.A.I, Zhang, L., 2022. The effect of colloid milling on the microstructure and functional properties of asparagus dietary fibre concentrates. Lwt 169, 114013. https://doi.org/10.1016/j.lwt.2022.114013.

Lu, Y., Zhang, L., Schutyser, M.A.I., 2023. Linking particle morphology and functionality of colloid-milled dietary fibre concentrates from various plant sources. LWT 186, 115206. https://doi.org/10.1016/j.lwt.2023.115206.

Ma, M., Mu, T., 2016. Effects of extraction methods and particle size distribution on the structural, physicochemical, and functional properties of dietary fiber from deoiled cumin. Food Chem. 194, 237–246. https://doi.org/10.1016/j.foodchem.2015.07.095.

Ma, S., Ren, B., Diao, Z., Chen, Y., Qiao, Q., Liu, X., 2016. Physicochemical properties and intestinal protective effect of ultra-micro ground insoluble dietary fibre from carrot pomace. Food Funct. 7, 3902–3909. https://doi.org/10.1039/C6FO00665E.

Mellican, R.I., Li, J., Mehansho, H., Nielsen, S.S., 2003. The role of iron and the factors affecting off-color development of polyphenols. J. Agric. Food Chem. 51, 2304–2316. https://doi.org/10.1021/jf020681c.

Menon, A., Stojceska, V., Tassou, S.A., 2020. A systematic review on the recent advances of the energy efficiency improvements in non-conventional food drying technologies. Trends Food Sci. Technol. 100, 67–76. https://doi.org/10.1016/j.tifs.2020.03.014.

Nath, P., Pandey, N., Samota, M., Sharma, K., Kale, S., Kannaujia, P., Sethi, S., Chauhan, O.P., 2022. Browning reactions in foods. In: Chauhan, O.P. (Ed.), Advances in Food Chemistry: Food Components, Processing and Preservation. Springer Nature, Singapore, pp. 117–159. https://doi.org/10.1007/978-981-19-4796-4_4.

Ogeda, T.L., Silva, I.B., Fidale, L.C., El Seoud, O.A., Petri, D.F.S., 2012. Effect of cellulose physical characteristics, especially the water sorption value, on the efficiency of its hydrolysis catalyzed by free or immobilized cellulase. J. Biotechnol. 157, 246–252. https://doi.org/10.1016/j.jbiotec.2011.11.018.

Paes, S.S., Sun, S., MacNaughtan, W., Ibbett, R., Ganster, J., Foster, T.J., Mitchell, J.R., 2010. The glass transition and crystallization of ball milled cellulose. Cellulose 17, 693–709. https://doi.org/10.1007/s10570-010-9425-7.

Pegiou, E., Mumm, R., Acharya, P., de Vos, R.C.H., Hall, R.D., 2020. Green and white asparagus (Asparagus officinalis): a source of developmental, chemical and urinary intrigue. Metabolites 10, 17. https://doi.org/10.3390/metabo10010017.

Ramachandraiah, K., Chin, K.B., 2016. Evaluation of ball-milling time on the physicochemical and antioxidant properties of persimmon by-products powder. Innov. Food Sci. Emerg. Technol. 37, 115–124. https://doi.org/10.1016/j.ifset.2016.08.005.

Ramadhan, K., Foster, T.J., 2018. Effects of ball milling on the structural, thermal, and rheological properties of oat bran protein flour. J. Food Eng. 229, 50–56. https://doi.org/10.1016/j.jfoodeng.2017.10.024.

Ramadoss, G., Muthukumar, K., 2016. Ultrasound assisted metal chloride treatment of sugarcane bagasse for bioethanol production. Renew. Energy 99, 1092–1102. https://doi.org/10.1016/j.renene.2016.08.003.

Salmen, N.L., Back, E.L., 1977. The influence of water on the glass transition temperature of cellulose. Tappi [Technical Association of the Pulp and Paper Industry] 60.

Siccama, J.W., Oudejans, R., Zhang, L., Kabel, M.A., Schutyser, M.A.I., 2022. Steering the formation of cellobiose and oligosaccharides during enzymatic hydrolysis of asparagus fibre. LWT 160, 113273. https://doi.org/10.1016/j.lwt.2022.113273.

Siddiqui, H., Sultan, Z., Yousuf, O., Malik, M., Younis, K., 2023. A review of the health benefits, functional properties, and ultrasound-assisted dietary fiber extraction. Bioact. Carbohydr. Diet. Fibre 30, 100356. https://doi.org/10.1016/j.bcdf.2023.100356.

Singh, B., Suri, K., Shevkani, K., Kaur, A., Kaur, A., Singh, N., 2018. Enzymatic browning of fruit and vegetables: a review. In: Kuddus, M. (Ed.), Enzymes in Food Technology: Improvements and Innovations. Springer, Singapore, pp. 63–78. https://doi.org/10.1007/978-981-13-1933-4_4.

Sitotaw, Y.W., Habtu, N.G., Gebreyohannes, A.Y., Nunes, S.P., Van Gerven, T., 2023. Ball milling as an important pretreatment technique in lignocellulose biorefineries: a review. Biomass Conv. Bioref. 13, 15593–15616. https://doi.org/10.1007/s13399-021-01800-7.

Stanišić, N., Kurćubić, V.S., Stajić, S.B., Tomasevic, I.D., Tomasevic, I., Stanišić, N., Kurćubić, V.S., Stajić, S.B., Tomasevic, I.D., Tomasevic, I., 2025. Integration of dietary fibre for health benefits, improved structure, and nutritional value of meat products and plant-based meat alternatives. Foods 14. https://doi.org/10.3390/foods14122090.

Stolle, A., Szuppa, T., Leonhardt, S.E.S., Ondruschka, B., 2011. Ball milling in organic synthesis: solutions and challenges. Chem. Soc. Rev. 40, 2317–2329. https://doi.org/10.1039/C0CS00195C.

Teixidó, H., Staal, J., Caglar, B., Michaud, V., 2022. Capillary effects in Fiber reinforced polymer composite processing: a review. Front. Mater. 9. https://doi.org/10.3389/fmats.2022.809226.

Ullah, I., Yin, T., Xiong, S., Zhang, J., Din, Z., Zhang, M., 2017. Structural characteristics and physicochemical properties of okara (soybean residue) insoluble dietary fiber modified by high-energy wet media milling. LWT - Food Sci. Technol. 82, 15–22. https://doi.org/10.1016/j.lwt.2017.04.014.

Viscusi, G., Barra, G., Gorrasi, G., 2020. Modification of hemp fibers through alkaline attack assisted by mechanical milling: effect of processing time on the morphology of the system. Cellulose 27, 8653–8665. https://doi.org/10.1007/s10570-020-03406-0.

Wang, H., Iqbal, A., Murtaza, A., Xu, X., Pan, S., Hu, W., 2023. A review of discoloration in fruits and vegetables: formation mechanisms and inhibition. Food Rev. Int. 39, 6478–6499. https://doi.org/10.1080/87559129.2022.2119997.

Wichrowska, D., Rolbiecki, R., Rolbiecki, S., Jagosz, B., Ptach, W., Kazula, M., Figas, A., 2018. Concentrations of some chemical components in white asparagus spears depending on the cultivar and post-harvest irrigation treatments. Folia Hortic. 30. https://doi.org/10.2478/fhort-2018-0015.

Yang, B., Wu, Q., Song, X., Yang, Q., Kan, J., 2019. Physicochemical properties and bioactive function of Japanese grape (Hovenia dulcis) pomace insoluble dietary fibre modified by ball milling and complex enzyme treatment. Int. J. Food Sci. Technol. 54, 2363–2373. https://doi.org/10.1111/ijfs.14134.

Yu, Q., Fan, L., 2021. Improving the bioactive ingredients and functions of asparagus from efficient to emerging processing technologies: a review. Food Chem. 358, 129903. https://doi.org/10.1016/j.foodchem.2021.129903.

Zakaria, M.R., Hirata, S., Hassan, M.A., 2014. Combined pretreatment using alkaline hydrothermal and ball milling to enhance enzymatic hydrolysis of oil palm mesocarp fiber. Bioresour. Technol. 169, 236–243. https://doi.org/10.1016/j.biortech.2014.06.095.

Zhang, C., Gong, T., Qiang, S., Wang, X.Y., Wang, X.R., Zhang, S., Meng, Y.H., 2024. A clean label O/W pickering emulsion stabilized by pectin-containing lignocellulosic nanofibrils isolated from apple pomace. Food Hydrocoll. 150, 109583. https://doi.org/10.1016/j.foodhyd.2023.109583.

Zhang, L., Tsuzuki, T., Wang, X., 2015. Preparation of cellulose nanofiber from softwood pulp by ball milling. Cellulose 22, 1729–1741. https://doi.org/10.1007/s10570-015-0582-6.

Zhang, Q., Cheng, Z., Wang, Y., Fu, L., 2021. Dietary protein-phenolic interactions: characterization, biochemical-physiological consequences, and potential food

applications. Crit. Rev. Food Sci. Nutr. 61, 3589–3615. https://doi.org/10.1080/10408398.2020.1803199.
Zhang, W., Wu, W., Wang, Q., Chen, Y., Yue, G., 2014. The juice of asparagus by-product exerts hypoglycemic activity in streptozotocin-induced diabetic rats. J. Food Biochem. 38, 509–517.
Zhao, X., Chen, J., Chen, F., Wang, X., Zhu, Q., Ao, Q., 2013. Surface characterization of corn stalk superfine powder studied by FTIR and XRD. Colloids Surf. B Biointerfaces 104, 207–212. https://doi.org/10.1016/j.colsurfb.2012.12.003.
Zhu, J.Y., Wang, G.S., Pan, X.J., Gleisner, R., 2009. Specific surface to evaluate the efficiencies of milling and pretreatment of wood for enzymatic saccharification. Chem. Eng. Sci. 64, 474–485. https://doi.org/10.1016/j.ces.2008.09.026.
Zimmermann, M.V., Borsoi, C., Lavoratti, A., Zanini, M., Zattera, A.J., Santana, R.M., 2016. Drying techniques applied to cellulose nanofibers. J. Reinf. Plast. Compos. 35, 682–697. https://doi.org/10.1177/0731684415626286.