



THE IMPACT OF HIGH-ENERGY BALL MILLING DURATION ON MORPHOLOGICAL, COLOUR, AND HYDRATION PROPERTIES OF PINEAPPLE POMACE POWDER

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ARTICLE INFO

Article History:

Received: 1 September 2025

Revised: 1 February 2026

Accepted: 6 April 2026

Published: 15 August 2026

Keywords:

Ball milling, pineapple, pomace, powder, SEM

ABSTRACT

The management of agricultural waste such as pineapple pomace plays a crucial role in promoting sustainability. Specifically, utilising pineapple pomace offers a practical solution for minimising waste generated during juice processing. This study investigates the impact of high-energy ball milling durations (0, 1, 3, and 5 h) on the morphological, colour, and hydration properties including Water-Holding Capacity (WHC), Oil-Holding Capacity (OHC), and solubility of pineapple pomace powder. The results demonstrated progressive changes in powder morphology, with prolonged milling durations leading to reduced particle size and altered surface characteristics. Prolonged milling also resulted in decreased yellowness, attributed to the degradation of natural pigments. Both WHC and OHC decreased from 1124.93% to 373.19% and 244.33% to 172.78%, respectively, due to structural disruptions. In contrast, solubility improved with extended milling durations up to 82.77%. These findings highlight the importance of optimising milling conditions to achieve a balance between functionality and stability, facilitating the use of pineapple pomace powder in diverse food and functional applications.

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Introduction

Agricultural food waste has emerged as a critical issue globally, driven by the vast quantities of byproducts and residues generated during farming and food processing activities. From crop residues to processing activities, this waste often goes underutilised, contributing to environmental challenges such as land, water, and air pollution (Shaaban & Nasr, 2020; Koul *et al.*, 2022). Minimising food waste is essential for food security as it enhances supply chain efficiency and optimises resource use to support sustainable agriculture. Effective waste management can significantly reduce strain on agricultural systems, lessen environmental impacts, and contribute to building resilient communities (Wani *et al.*, 2024).

Pineapple is a tropical fruit grown and consumed worldwide in both fresh and processed forms, including juices, canned slices, jams, and dried pineapple, which generate substantial amounts of by-products (Figure 1). This fruit is known for its vitamins and minerals, which are vital for human health (Kumar *et al.*, 2023). In 2022, world statistics on pineapple production reached approximately 29.36 million tonnes, showing an increasing trend since 2020, with Malaysia contributing about 287,799 tonnes to this total (FAOSTAT, 2024). Due to increasing production, the industrial processing of pineapples generates significant amounts of waste, particularly in the juice production sector. The pomace constitutes a major component at

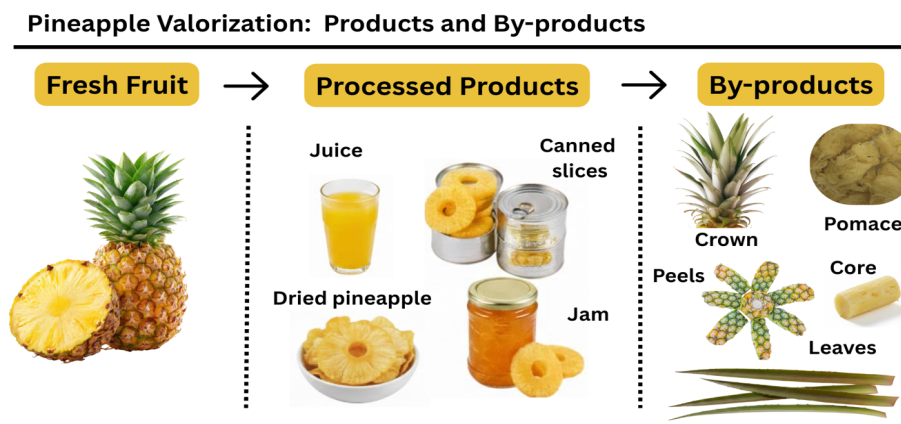


Figure 1: Pineapple valorisation pathway showing fresh fruit, processed products, and major by-products generated during pineapple processing

50% of these waste materials (Meena *et al.*, 2022).

Pineapple pomace, rich in dietary fibre, also contains bioactive compounds with significant nutritional and therapeutic value (Meena *et al.*, 2022; Kumar *et al.*, 2023). Converting this waste into value-added products is crucial for both environmental sustainability and economic development. However, the large particle size and fibrous structure of pineapple pomace present challenges for its direct application in food formulations, packaging materials, and other industrial uses (Nath *et al.*, 2023; Chaudhary & Singh, 2024).

To address these limitations, high-energy ball milling emerges as an effective mechanical process for reducing particle size and increasing surface area of plant-derived powders. The duration of milling plays a critical role in determining the extent of particle size reduction and surface modification, leading numerous studies to investigate its impact on the characteristics of milled materials (Jiang *et al.*, 2020; Nasrullah *et al.*, 2021; Telidi & Salim, 2023). Transformation from coarse powders into superfine particles significantly improves the functional properties. A previous study reported that particle reduction of tobacco leaves increased hydration capacity and had better colour values (Zhang *et al.*, 2022). The

decrease in the size of milled starch leads to a higher number of active sites, which enhances the solubility (Bangar *et al.*, 2023).

Moreover, the structural changes such as increased porosity and altered surface morphology through the milling process also contribute to these improved water and oil holding capacities, as reported on whole-grain mung bean flour (Yu *et al.*, 2023) and citrus fibre (Song *et al.*, 2021). Enhanced solubility, water-holding capacity, and oil-holding capacity make the resulting powders highly suitable for diverse applications such as food formulations, emulsions, and encapsulated systems (Bangar *et al.*, 2023; Ke *et al.*, 2024).

However, despite numerous studies on particle size reduction in plant-based materials, the influence of ball milling duration on pineapple pomace remains limited. Based on these insights, this study aims to examine the impacts of different ball-milling durations on the morphological, colour, and hydration properties of pineapple pomace powder.

Materials and Methods

Sample Preparation

Pineapples (MD2 variety) were obtained from the local market in Kuala Nerus, Terengganu, Malaysia. This variety was selected due to its

widespread cultivation and growing demand in industrial pineapple processing. The pineapples were chosen at a similar commercial maturity stage based on external shell colour and washed thoroughly with water to remove any surface contaminants. After rinsing, the pineapples were cut into smaller pieces to facilitate juice extraction. The pineapple pieces were fed into a juice extractor (SJ-6, Russell Taylors, Malaysia) to separate the juice from the pomace. Afterwards, the pomace was dried in a freeze dryer (Labconco, USA) for 24 hours. After freeze-drying, the dried pomace was coarsely ground into powder using a laboratory grinder (7010S, Waring Laboratory, USA). The coarse powder was then stored in an airtight container to prevent moisture absorption. Figure 2 illustrates the stages of pineapple pomace preparation for this study.

The coarse pineapple pomace powder was further processed using high-energy ball milling (BM40, POWTEQ, China) to achieve superfine powder form. For this process, 10 grams of the powder were placed in a stainless-steel jar with zirconium oxide (ZrO_3) with weight ratio of powder to balls maintained at 1:10 (Chao & Liao, 2023). Zirconium oxide beads were used as grinding media to ensure efficient particle size reduction with minimal contamination. The ball milling process was conducted at 300 rpm for varying durations (1, 3, and 5), and the temperature was kept at room conditions ($25^\circ\text{C} \pm 2^\circ\text{C}$).

Morphological Observations

The morphology of the pineapple pomace powder at different ball-milling durations was examined using a high-vacuum Scanning Electron Microscope (SEM) (Tescan VEGA, Tescan Orsay Holding, Brno, Czech Republic). Samples were mounted on metal holders and the images were captured under vacuum at an accelerating voltage of 20 kV.

Colour Measurement

The colour of the pineapple pomace powder was measured using a colorimeter (WR-18, FRU, China) to determine L^* , a^* , and b^* and values. L^* indicates lightness, a^* represents red to green, and b^* denotes yellow to blue. The total colour difference, ΔE was calculated using Equation 1 (Telidi & Salim, 2023).

$$\Delta E = \sqrt{(L^* - L_o^*)^2 + (a^* - a_o^*)^2 + (b^* - b_o^*)^2} \quad (1)$$

where L_o^* , a_o^* , and b_o^* were the colour parameters of the unmilled powder, used as a control for comparison.

Hydration Properties

Water-Holding Capacity (WHC)

The WHC was determined using methods described by Chen *et al.* (2015), with slight modifications in sample-to-solvent ratio and centrifugation conditions. Specifically, a sample mass of 0.5 grams was dispersed in 10 mL of distilled water and incubated at 60°C for 30

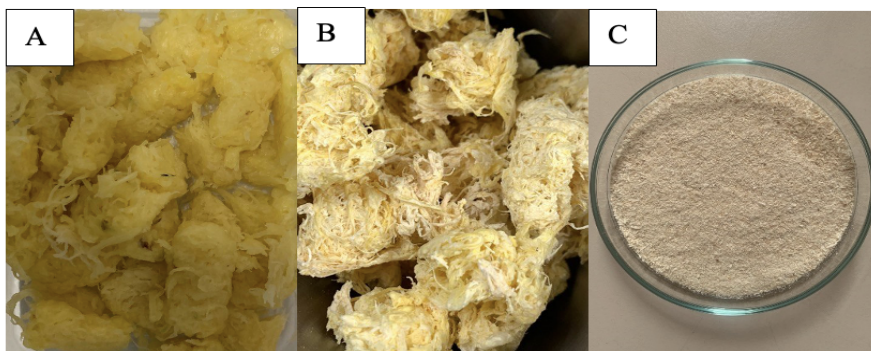


Figure 2: Visual representation of the pineapple pomace preparation stages: (A) pineapple pomace, (B) freeze-dried pomace, and (C) coarse pomace powder

minutes in a temperature-controlled water bath (Cole-Parmer, USA). After cooling to room temperature, the mixture was centrifuged (Hettich Universal 320R, Germany) at 3,000×g for 20 minutes. The supernatant was decanted and the remaining hydrated sediment (residual) representing the water retained by the powder was weighed. The WHC was calculated using Equation 2 (Jose *et al.*, 2022).

$$WHC(\%) = \frac{\text{Weight of residual}}{\text{Weight of sample}} \times 100 \quad (2)$$

Oil-Holding Capacity (OHC)

The OHC was determined according to the method used by Jose *et al.* (2022) with slight modifications in centrifugation conditions. Briefly, 1 gram sample was mixed with 10 mL of oil and allowed to stand for 30 minutes at ambient temperature. Then, the mixture was centrifuged (Hettich Universal 320R, Germany) at 2,000 rpm for 30 minutes. After centrifugation, the supernatant was decanted and the remaining oil-bound sediment (residual) was weighed. The OHC was subsequently calculated using Equation 3 (Jose *et al.*, 2022) as follows:

$$OHC (\%) = \frac{\text{Weight of residual}}{\text{Weight of sample}} \times 100 \quad (3)$$

Solubility

The solubility of the powder was determined as described by Badjona *et al.* (2019). A 1 g of sample was mixed with 10 mL of distilled water and heated at 80°C for 30 minutes. After centrifugation (Hettich Universal 320R, Germany) at 2,200 rpm for 15 minutes, the supernatant was dried in an oven (Mettler 100-800, Germany) at 105°C to measure the dried residue. Solubility was calculated using Equation 4 (Jose *et al.*, 2022).

$$\text{Solubility } (\%) = \frac{\text{Weight of dried sample in supernatant}}{\text{Weight of original sample}} \times 100 \quad (4)$$

Statistical Analysis

All experiments were performed in three replicates and the data were analysed using analysis of variance (ANOVA) to identify significant differences among treatments. Duncan's multiple range test was applied to compare the means, with a significance threshold set at $p < 0.05$.

Results and Discussion

Morphological Analysis

The progressive reduction in particle size of pineapple pomace powder influenced by varying milling durations is illustrated at 300x magnification in Figure 3. Unmilled powder has a large, irregularly shaped particle with rough and porous surfaces. These characteristics are typical of unmilled pineapple pomace, where the structural integrity of the fibrous material remains largely intact. This can be attributed to its natural composition, primarily cellulose, hemicellulose, and lignin, which are key components contributing to its dietary fiber properties (Kumar *et al.*, 2018).

Initial milling breaks down larger aggregates, resulting in a slight reduction in particle size and a decrease in irregularity. Milling for 3 hours produces finer particles with increased uniformity while 5 hours of milling achieves the finest particle size with minimal agglomeration, resulting in a powder with improved homogeneity and an enhanced surface area. This trend highlights the significant impact of the milling process in refining particle size and achieving a more homogeneous particle distribution, consistent with previous studies (Ramachandraiah & Chin, 2016; Nasrullah *et al.*, 2021; Telidi & Salim, 2023). The reduction in

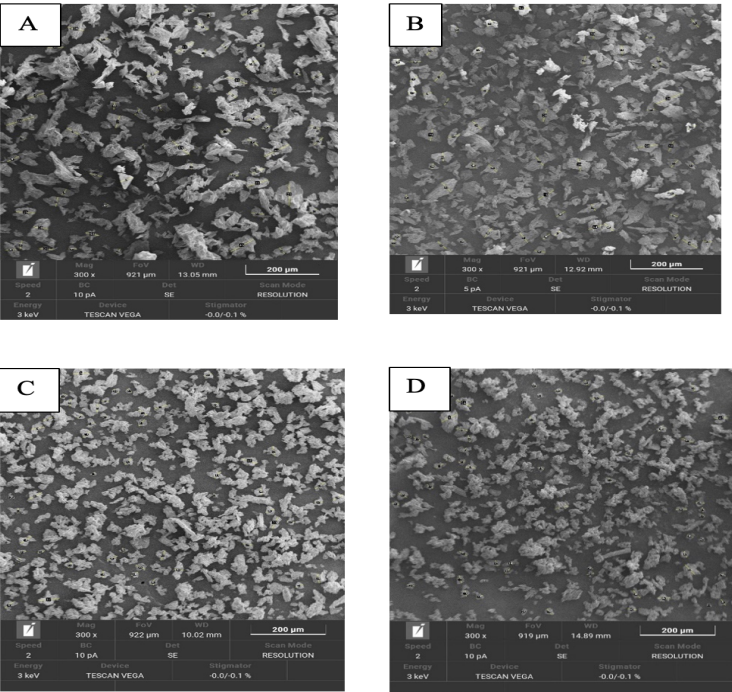


Figure 3: SEM images of different milling durations of pineapple pomace powder: (A) = 0 h, (B) = 1 h, (C) = 3 h, and (D) = 5 h

particle size can be attributed to the mechanical forces exerted during ball milling, where impact and attrition forces between the balls, jar, and powder particles facilitate the breakdown of the pineapple pomace powder particles.

Colour Measurement

The data in Table 1 shows the impact of milling duration on the colour parameters (L^* , a^* , b^* , and ΔE) of pineapple pomace powder. The results indicate that increasing the milling

duration significantly changes the powder’s colour attributes. The L^* values increase with milling duration indicates that the powder becomes lighter in colour with extended milling. The increased lightness can be attributed to the reduction in particle size, which enhances the reflection of light from the surface of the finer particles (Gueli *et al.*, 2017).

Similarly, the value of a^* and b^* decreases as milling progresses. A decrease in the yellowness of the powder may be caused by

Table 1: Colour parameters and total colour difference of pineapple pomace powder at different milling durations

Milling Duration	L^*	a^*	b^*	ΔE
0 h	88.70 ± 0.22^a	5.12 ± 0.05^a	17.13 ± 0.17^a	-
1 h	92.08 ± 0.10^b	4.12 ± 0.04^b	15.63 ± 0.08^b	3.78 ± 0.17^b
3 h	93.61 ± 0.18^c	3.54 ± 0.03^c	13.96 ± 0.10^c	5.86 ± 0.51^c
5 h	94.14 ± 0.15^d	3.34 ± 0.08^d	13.01 ± 0.07^d	6.74 ± 0.47^d

The superscript letters within the column indicate statistical differences between the means.

Table 2: Water-Holding Capacity (WHC), Oil-Holding Capacity (OHC), and solubility of pineapple pomace powder at different milling durations

Milling Duration	WHC (%)	WHC (%)	WHC (%)
0 h	1124.93 ± 3.04 ^a	244.33 ± 1.05 ^a	57.87 ± 0.15 ^a
1 h	996.67 ± 4.65 ^b	189.79 ± 1.88 ^b	66.55 ± 2.50 ^b
3 h	562.60 ± 2.82 ^c	181.07 ± 1.46 ^c	73.99 ± 1.76 ^c
5 h	373.19 ± 2.79 ^d	172.78 ± 0.20 ^d	82.77 ± 0.91 ^d

The superscript letters within the column indicate statistical differences between the means.

pigment degradation or structural changes to the pigment molecules during prolonged milling. This trend is consistent with findings in other food materials such as onions, where prolonged milling has been shown to cause significant degradation of valuable compounds (Jiang *et al.*, 2020). The ΔE increases steadily with milling duration when compared to unmilled powder, reflecting an overall colour change induced by prolonged mechanical processing. These results suggest that prolonged milling affects the visual characteristics of pineapple pomace powder, which may influence its visual appearance and potential applications.

Hydration Properties

The data in Table 2 present the effect of milling duration on the WHC, OHC, and solubility of pineapple pomace powder. The results indicate significant changes in these properties as milling duration increases. For WHC, it was observed that the unmilled powder exhibited the highest water-holding capacity when compared to the WHC of the milled powders. The WHC decreased by approximately two-thirds after 5 hours of milling. This result suggests the larger particle size in the unmilled powder retained its fibrous structure, providing more pore networks for water absorption (Jose *et al.*, 2022).

A similar trend is observed for OHC, where the powder's OHC decreases by approximately 29.28% after 5 hours of milling compared to the unmilled powder. The reduction in OHC can be attributed to the loss of internal pore networks and the disruption of the structural matrix that traps oil. Similar trends have been reported for

asparagus leaf during prolonged ball milling (Chitrakar *et al.*, 2020).

In contrast to WHC and OHC, the solubility of the powder improves significantly with milling duration. The solubility increased by 43.03% from the unmilled powder to the powder milled for 5 hours. The increase in solubility is likely due to the finer particle size and increased surface area, which facilitate the dissolution of soluble components. Furthermore, the mechanical forces during milling may enhance the breakdown of cell walls and release water-soluble compounds (Jiang *et al.*, 2024).

Conclusions

This study demonstrated the significant impact of ball milling on the morphology, colour, and hydration properties of pineapple pomace powder. The results show that the reduction in particle size contributes to the changes in colour and its hydration properties. Excessive milling duration degrades the natural pigments responsible for the powder's colour. While milling enhances solubility, which is beneficial for many functional food applications, it reduces both WHC and OHC. These changes reflect the structural modifications caused by milling. Therefore, optimising the milling duration is critical in achieving a balance between these properties, depending on the intended application of the powder.

Over time, the government and the local community have implemented various flood mitigation strategies. These efforts encompass both structural and non-structural measures.

Completed structural initiatives encompass the creation of watercourses, river embankments, pump houses, and water gates. Non-structural measures include the organisation of riverbank structures, the installation of early warning systems, and the establishment of community-based disaster forums. These combined flood mitigation endeavours in Solo City have effectively lessened the extent and severity of flooding; however, completely eradicating flood events has remained elusive. The factors that cause flooding in Solo City are the location, topography, and urban development of the city, as well as damage to the upstream area and a need for more public awareness of the importance of environmental conservation.

Acknowledgements

The authors would like to express their gratitude to Universiti Malaysia Terengganu (UMT) for the support provided through the Talent and Publications Enhancement-Research Grant [UMT/TAPE-RG-2023/Vot. 55464]. Special thanks are also extended to the Nanophysics lab staff for their invaluable assistance.

Conflict of Interest Statement

The authors agree that this research was conducted in the absence of any self-benefits, commercial, or financial conflicts and declare absence of conflicting interests with the funders.

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