

EFFECTS OF DRYING METHODS ON THE NUTRITIONAL, ANTINUTRITIONAL, AND ANTIOXIDANT PROPERTIES OF PINEAPPLE (*Ananas comosus*) INDUSTRY WASTE AS A POTENTIAL FEED ADDITIVE

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ABSTRACT

A major challenge for many countries is managing agricultural waste. For instance, the pineapple processing industry in the Philippines generates tons of waste each year. Thus, this study aims to assess the impact of different drying methods (sun drying, oven drying, and dehydration) on the proximate composition, antinutritional, and antioxidant properties of pineapple industry waste, to determine its suitability for various applications. The results showed that sun-dried pineapple waste had a higher carbohydrate content compared to other treatments. Dehydrated pineapple samples had higher percentages of fiber (43.60 ± 0.43), ash (2.93 ± 0.03), protein (5.48 ± 0.08), and fat (1.00 ± 0.11) compared to sun-dried samples. Dehydrated pineapple industry waste had significantly higher tannin content *mg TAE/100g* (0.1388 ± 0.00) compared to oven-dried (0.1009 ± 0.00) and sun-dried (0.0877 ± 0.00), while oven-dried samples had the highest phytate content (0.077 ± 0.03), followed by dehydrating and sun drying. Non-significant differences in antioxidant activities (DPPH scavenging activity) and total phenolic content (TPC) were noted among the drying treatments. Oven-dried samples had a higher total flavonoid content (TFC) compared to dehydrated samples; however, the difference was not statistically significant when compared to sun-dried samples. This study suggests that sun drying and dehydration, due to their lower energy consumption and ability to maintain product nutrition, are viable options for mass-producing pineapple industry waste for other applications such as feed additive. Preserving the nutritional integrity of the waste could offer significant advantages for its utilization in the Philippine pineapple industry.

Keywords: Pineapple, Antioxidants, Proximate composition, Drying process, Antinutritional, Aquafeeds

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INTRODUCTION

Pineapple (*Ananas comosus*), part of the *Bromeliaceae* genus with approximately 2000 species, is a valuable source of essential nutrients, including sugars, minerals, fibers, organic acids, and vitamins, which contribute to its significance for human consumption (Assumi *et al.*, 2018; Chaudhary *et al.*, 2019). Pineapple offers health benefits such as it can improve digestion and enhance immunity. It is also rich in 131% vitamin C and 78% manganese along with antioxidants like flavonoids and phenolic acids (Mohsin *et al.*, 2020). In

2022, pineapple production in the Philippines reached about 2.91 million metric tons, with a production value of approximately 44.94 billion Philippine pesos, maintaining its status as the largest pineapple-producing country globally in 2021, followed by Costa Rica and Indonesia (Statista, 2023). The pineapple sector plays a crucial role in the agricultural economy and international trade of the Philippines, offering various canned products like juices, slices, and concentrate, as well as alternative products like sugar preserves; however, effectively utilizing pineapple by-products remains a challenge for the industry.

A primary challenge faced by many nations involves agricultural waste, encompassing pulp, crop residues, leaf litter, and by-products from fruits and vegetables, crucial for meeting global food supply demands. Notably, the pineapple industry is a major contributor to this waste stream, generating over 180,000 metric tons of organic waste, primarily pineapple peels, in 2018, as reported by Dole Philippines Inc. and Philippine statistical data. This figure was expected to increase in the following years (Beric, 2021). This waste is earmarked for various industrial applications such as processing and bioactive product production (Madhumeena *et al.*, 2021).

Pineapple processing industries generate processing waste rich in various bioactive compounds, often containing higher nutritional and therapeutic importance than the final product (Meena *et al.*, 2021). These wastes find applications in energy generation, antioxidant production, pharmaceuticals, food industry, citric and lactic acid production, ethanol and vinegar production, as well as fiber production (Abdullah & Mat, 2008; Hikal *et al.*, 2021). The significant nutritional content and rich chemical composition of pineapples have garnered attention from food industries seeking to integrate them into various food items. Additionally, both the production and consumption of pineapples generate substantial amounts of solid waste, which, in turn, present opportunities for the development of valuable waste-derived products through processing. Pineapple residues, which are rich in starch, vitamins, and growth factors due to their growth process, serve as a valuable substratum for extracting bromelain, organic acids, ethanol, and other substances. These residues consist of crop remnants, flesh, rind, and pomace, providing a diverse array of beneficial compounds including carotenoids, polyphenols, dietary fibers, vitamins, enzymes, and oils. The bioactive nature of these compounds highlights their significant potential value (Madhumeena *et al.*, 2021).

Drying serves as a crucial preservation technique to prolong shelf life and retain essential vitamins, minerals, fiber, carbohydrates, and antioxidant properties (Vidinamo *et al.*, 2021). Nevertheless, the drying process could induce nutrient depletion and unfavorable structural modifications, potentially undermining the quality and economic feasibility of the end product. Hence, this study aims to explore the antioxidant, nutritional and antinutritional properties of pineapple *Ananas comosus* (L.) Merr subjected to different drying methods (sun drying, oven drying, and dehydrating) to know its potential as an aquafeed ingredient.

MATERIALS AND METHODS

Sample Collection and Preparation: In this study, by-product (waste) of pineapple fruit processing industry

were collected in Bukidnon, Northern Mindanao, Philippines. The by-product includes pineapple pulp, peels and core. The collected samples were weighed, minced using a food processor, and stored at -4 °C until the drying process began. Different drying methods were employed on the wet samples, these are sun drying, oven drying, and dehydrating techniques.

Sun Drying: The sun-drying method was adapted from the previous study by Farahmandfar *et al.* (2020), with some modifications. A drying rack, constructed from a wooden frame and cloth, was positioned in an open area. Fresh samples were then placed on the cloth, exposed to sunlight. Drying occurred as solar radiation passed through the surface of the material. The samples were left to dry for 48 hours until they reached a moisture content of 10%. After drying, the samples were ground into fine powder and sieved using a 45-micron sieve for further analysis.

Dehydrating: The fresh samples were loaded onto a food dehydrator (Onetwofeet Food Dehydrator Machine 5-Layer, China) and dried at 45 °C for a period of 48 hours, allowing them to achieve a moisture content of 10%. Following the drying process, the samples were finely ground into powder form using a 45-micron sieve for subsequent analysis.

Oven Drying: A forced convection laboratory oven (Esco Isoterm® forced convection laboratory oven, Singapore) with interior dimensions of 41 × 42 × 32 cm was utilized during the process. The samples were evenly spread in a single layer tray and dried at 45 °C for 24-48 hours to reach a moisture content of 10% (Farahmandfar, *et al.*, 2020). After drying, the samples are then ground into fine powder using a 45-micron sieve and stored for further analysis.

Sample Analyses

Extraction of total polyphenols: A method used by Mahmood *et al.*, (2019) with modifications was used during the extraction process. Five grams of the pulverized samples were added with 100 mL of 80% ethanol and sonicated at the sonicator power of 20 kHz for 15 min. After sonication, the mixture was filtered and analyzed for its phytochemical composition and antioxidant activity.

Total Polyphenol Content (TPC): The estimation of the total phenolic content of ethanolic extract (0.50 mg mL⁻¹ in distilled water) is determined using the Folin-Ciocalteu reagent. A 40 µL is mixed with 200 µL of Folin-Ciocalteu's phenol reagent, 600 µL of 20% sodium carbonate, and diluted to 5 mL with distilled water. After 2 hours, the absorbance of the blue color solution is measured at 765 nm using a UV-VIS spectrophotometer (L7 Double-Beam spectrophotometer, China). The amount of TPC were calculated using linear regression

with gallic acid as the standard reference (0-1000 mg L⁻¹). The results are expressed as mg of gallic acid equivalents (GAE) per g of dry sample.

Total Flavonoid Content (TFC): The total flavonoid content was determined using the aluminum trichloride method, with catechin as the reference compound (as described by Zhishen *et al.*, 1999). A volume of 1000 µL of the extract was added to 300 µL of a 5% NaNO₂ solution. The mixture was allowed to stand for 6 minutes, after which 300 µL of 10% aluminum trichloride was added and incubated for 5 minutes. Subsequently, 2mL of 1M NaOH was added, and the final solution volume was adjusted to 5 mL with distilled water. After 15 minutes of incubation, the mixture turned pink, and the absorbance was measured at 510 nm. TFC concentration was evaluated from catechin standard ranging from 0 to 500mg L⁻¹. Values of the total flavonoid content was expressed as mg CE 100g⁻¹ DM.

Radical scavenging activity (DPPH): The radical scavenging activity of the ethanolic sample extract was assessed following the method outlined by Hossain and Rahman (2011) with slight modifications. About 1.6 mL ethanolic extract of the samples and 2.4 mL of 0.1 mM ethanolic solution of DPPH and were mixed thoroughly. The tubes were left at room temperature for 20 minutes. The changes in absorbance were measured using a UV-VIS spectrophotometer (L7 Double-Beam spectrophotometer, China) at 517 nm. A control was prepared following the same procedure as above but without the sample and methanol, serving as a baseline correction. The radical scavenging activity is then expressed as the inhibition percentage and calculated using the provided formula:

$$\% \text{ radical scavenging activity} = (\text{Control OD} - \text{sample OD} / \text{control OD}) \times 100$$

Proximate Analysis: The proximate analysis of pineapple industry waste involves assessing its moisture, ash, protein, fiber, and carbohydrate content, and this evaluation is carried out following the AOAC (2000) guidelines. This standardized method ensures accurate and reliable measurements, which are crucial for understanding the nutritional and compositional properties of the waste material.

Total Tannin Content (TTC): The tannins were determined by slightly modified Folin and Ciocalteu method, as referenced by Islam *et al.*, (2015). The standard (Tannic Acid) solution of six different concentrations (6.25, 12.5, 25, 50, 100 and 200 µg/mL) and the fruit extract (200µg/mL) of 0.1 mL were taken in different marked test tubes. Then 7.5mL of distilled water, 0.5 mL of Folin Phenol reagent, 1 mL of 35% sodium carbonate solution were added and the volume was finally adjusted up to 10 mL with distilled water. The

mixture was shaken well, kept at room temperature for 30 minutes and absorbance was measured using UV-Vis spectrophotometer (Shimadzu UV-Vis PC-1600, Kyoto, Japan) at 725 nm against a blank. Total Tannin content of the extracts was expressed as Tannic Acid Equivalent (mgTAE/100g sample).

Determination of Phytate Content: The procedure employed follows the Young and Greaves method from 1940, as referenced by Lucas Markakes in 1975. A sample of 0.2 g was measured and placed in a 250 mL conical flask, where it was immersed in 100 mL of 20% concentrated HCl for a duration of three hours. After filtration, 50 mL of the resulting filtrate was transferred to a 250 mL beaker, mixed with 100 mL of distilled water, and then treated with 10 mL of 0.3% ammonium thiocyanate as an indicator before being titrated using a standard iron (III) chloride solution containing 1.95 mg of iron per mL.

Statistical Design: The data were statistically analyzed and displayed as mean values with corresponding standard deviations using SPSS version 29 software. For measurement data with a normal distribution, the one-way analysis of variance (ANOVA) was performed; For data without a normal distribution or when homogeneity of variance was not met, the Kruskal-Wallis test was executed. Specifically, the Ash and DPPH data were analyzed using the Kruskal-Wallis test after verifying homogeneity of variance. The $\alpha \leq 0.05$ was chosen as the test's significance level.

RESULTS AND DISCUSSION

Proximate Analysis of Pineapple Industry Waste: Significant variations ($P \leq 0.05$) were observed in the proximate composition of pineapple industry waste utilizing different drying methods. Dehydrated pineapple industry waste exhibited higher moisture retention compared to the other treatments. Regarding ash content, no significant differences were observed among dehydrated and oven-dried samples. However, for fat and protein content, significant variations were noted among the treatments, with dehydrated samples showing higher retention than the other drying methods. On the other hand, sundried pineapple industry waste samples exhibited higher carbohydrate retention than the other treatments. Notably, there was no significant difference in fiber retention between dehydrated and sundried treated samples (Table 1).

Drying is a crucial food preservation method that removes moisture to reduce deterioration while maintaining quality and nutrient content (Babu *et al.*, 2018). However, challenges such as nutrient loss, particularly of bioactive compounds due to thermal degradation, necessitate continuous research to refine drying techniques (Natumanya *et al.*, 2021). Optimizing

drying methods is essential to preserving the nutritional value, sensory attributes, and overall quality of food products (Calín-Sánchez *et al.*, 2020). Sun drying is commonly viewed as a traditional technique utilized in many developing nations globally. The technique of open or direct sun drying entails exposing agricultural products to sunlight during the day. Typically, this method involves letting fruits dry naturally on the tree without harvesting or spreading harvested product on a surface to air dry. Sun drying is commonly practiced in tropical and subtropical regions with temperatures around 30°C, particularly prevalent in Southeast Asia for preserving medicinal herbs and spices (Desa *et al.*, 2019). The present study showed the efficiency of sun drying in preserving the carbohydrate content of the pineapple industry waste, yielding significantly higher values as compared to other drying processes. In the study of Mbah

et al. In 2012, it was concluded that sun-drying together with other treatments has improved the protein, fiber, carbohydrate, vitamin B1, vitamin A, calcium, and zinc levels of the *Moringa oleifera* leaf. The current research also showed that dehydrated pineapple samples had higher moisture ($8.79 \pm 0.36\%$), ash ($2.93 \pm 0.03\%$), protein ($5.48 \pm 0.08\%$), and fat ($1.00 \pm 0.11\%$) retention. Dehydrated pineapple samples also had higher fiber ($43.60 \pm 0.43\%$) contents but had no significant difference on sundried ($20.94 \pm 1.01\%$) treated samples. Food dehydration is the process of preserving and extending the shelf life of foods. Heat and air sources are used to remove the water content of foods which then prevents the growth and spread of bacteria. The goal of this research is to eliminate moisture using heat like electricity or solar to dehydrate foods (Sulaiman *et al.*, 2020).

Table 1. Proximate composition (Means \pm SD) of pineapple industry waste in different drying processes.

Proximate Composition (%)	Sun-Dried	Oven-Dried	Dehydrated
Moisture	7.41 ± 0.20^c	7.37 ± 0.04^b	8.79 ± 0.36^a
Ash	2.58 ± 0.01^b	2.82 ± 0.00^{ab}	2.93 ± 0.03^a
Protein	4.61 ± 0.02^c	5.14 ± 0.03^b	5.48 ± 0.08^a
Fat	0.58 ± 0.04^c	0.85 ± 0.01^b	1.00 ± 0.11^a
Carbohydrates	84.83 ± 0.23^a	83.83 ± 0.05^b	81.81 ± 0.20^c
Fiber	20.94 ± 1.01^{ab}	14.26 ± 0.25^b	43.60 ± 0.43^a

^{abc}Values in the same row with different superscripts are significantly different ($P \leq 0.05$)

Dehydration has been demonstrated to effectively preserve the majority of the proximate composition of samples, exhibiting notable retention of nutritional components. The food drying machine offers user-friendly operation with minimal energy consumption, facilitating time efficiency. This approach emerges as a feasible choice for mass drying of pineapple industry waste, suitable for subsequent applications such as medicinal use, agricultural feeds, and many more.

Antioxidant Properties of Pineapple Industry Waste:

No statistically significant variations ($P \leq 0.05$) were noted in the total phenolic content (TPC) and DPPH Scavenging Activity of pineapple industry waste when different drying methods were employed (Table 2). However, total flavonoid content (TFC) exhibited significant variations between oven-dried and dehydrated samples. Oven drying had higher TFC retention compared to dehydration but has no significant difference from sun drying.

Table 2. Effect of drying processes on antioxidant properties (Means \pm SD) of pineapple industry waste

	Sun-Dried	Oven-Dried	Dehydrated
TPC (mg GAE/g sample)	0.56 ± 0.30^a	2.70 ± 1.16^a	0.57 ± 0.17^a
DPPH (% Scavenging activity)	12.30 ± 2.45^a	22.97 ± 2.62^a	13.18 ± 3.52^a
TFC (mg CE/g sample)	3.03 ± 0.29^{ab}	7.38 ± 0.98^a	2.01 ± 0.58^b

^{abc}Values in the same row with different superscripts are significantly different $P \leq 0.05$.

Plant-derived phenolic compounds exhibit antioxidant properties, a trait extensively verified through both *in vivo* and *in vitro* investigations. Moreover, these compounds demonstrate various significant biological functions, rendering them viable substitutes for synthetic additives. The significance of the drying process in retaining antioxidants lies in its ability to preserve and protect the beneficial compounds present in a substance,

such as fruits, vegetables, or herbs. Drying process helps to remove moisture, inhibiting the growth of microorganisms and preventing enzymatic reactions that can degrade antioxidants (Maurya *et al.*, 2018). By minimizing these factors, the drying process contributes to the long-term stability and effectiveness of antioxidants, ensuring their retention and promoting the overall nutritional quality of the dried product.

The different drying processes have no significant difference in DPPH scavenging activity and total phenolic content (TPC) of pineapple industry waste. This indicated that regardless of the drying method used, whether it's sun drying, oven drying, or dehydrating, the TPC and DPPH scavenging activity of the pineapple waste remained relatively consistent. Significant differences were observed in the total flavonoid content (TFC) between oven-dried and dehydrated pineapple industry waste. Oven drying demonstrated higher levels of TFC compared to dehydration, although no statistically significant difference was found compared to sun drying. Sarkar *et al.* (2021) noted the positive effect of oven drying on retaining flavonoid content in ginger powder. In a similar vein, Shonte *et al.* (2020) noted an augmentation in both the total antioxidant activity and phenol content of nettle leaves when subjected to oven drying, surpassing the effects of freeze drying. Correspondingly, Nunes *et al.* (2016) documented a rise in antioxidant activity in guavas undergoing the oven-drying procedure. Additionally, research by Ghafoor *et al.* (2020) indicated that drying ginger rhizome using oven and freeze-drying methods could lead to improved contents of bioactive compounds and higher antioxidant activity. Tian *et al.* (2016) observed that the application of heat triggered various chemical reactions such as the Maillard reaction, Strecker degradation, and the

breakdown of esters and glycosides. These reactions resulted in the generation of new antioxidant compounds, consequently leading to an increase in the antioxidant activity of the heat-treated samples.

While oven drying may not be the most economical choice for mass production due to energy consumption and yield constraints, its controlled environment could ensure consistent preservation of antioxidant properties, which is crucial for maintaining product quality. Additionally, the choice between sun-drying and dehydration methods warrants consideration of environmental factors, such as weather conditions and available infrastructure, which may impact feasibility and efficiency. This implies that sun-drying or dehydration could be considered a suitable drying method for treating pineapple industry waste.

Antinutritional Properties of Pineapple Industry Waste: A significant differences were observed in phytate and tannin content under different drying processes (Table 3). Oven-dried pineapple industry waste had higher phytate content that was significantly higher compared to the dehydrated pineapple industry waste samples. However, dehydrated pineapple industry waste had significantly higher tannin content compared to oven-drying and sun-drying.

Table 3. Antinutritional Properties (Means \pm SD) of pineapple industry waste in different drying processes.

	<i>Sun-Dried</i>	<i>Oven-Dried</i>	<i>Dehydrated</i>
Phytate Amount (%)	0.073 \pm 0.03 ^a	0.077 \pm 0.03 ^a	0.062 \pm 0.03 ^b
Tannin (TTC, mg TAE/100g eks)	0.0877 \pm 0.00 ^c	0.1009 \pm 0.00 ^b	0.1388 \pm 0.00 ^a

^{abc}Values in the same row with different superscripts are significantly different $P \leq 0.05$.

Elevated temperatures can influence the concentration of anti-nutritional compounds, resulting in both increases and decreases that affect their biosynthesis, extraction, and the overall nutritional quality of food sources. Among these compounds, phytate and tannin were significant anti-nutritional factors present in various plant-based foods, known to impair the bioavailability of essential nutrients. These substances inhibited mineral absorption and protein digestibility, though they also offered potential health benefits.

The phytate content in pineapple had been reported to be approximately 0.09% of the fresh weight (Nititham, 2004). These findings suggested that while pineapples did contain phytates, their concentration was relatively lower compared to other fruits and vegetables. The present study demonstrated that dehydration results in a significantly lowered in phytate content (0.062%). Understanding the relationship between phytate levels, nutrient bioavailability, and fish growth were essential for optimizing aquaculture diets. A study by Kumar *et al.*

(2011) recommended maintaining a phytate level below 1.0% to ensure adequate growth and nutrient absorption in most aquaculture species. Therefore, this study indicated that the drying process effectively reduced the phytate level in pineapple industry waste (PIW), particularly through dehydration, which yielded significantly lower levels as compared to sun drying and oven drying methods.

Pineapple also contains tannins, which are phenolic compounds characterized by their astringent properties. Research has demonstrated that tannin content can vary significantly among different pineapple varieties. For example, the tannin levels in certain varieties, such as the Native Variety and Queens Pineapple, are reported to be approximately 4.97 mg/100g and 4.69 mg/100g, respectively, while the MD-2 Pineapple and Smooth Cayenne Pineapple exhibited higher concentrations of 14.07 mg/100g and 13.65 mg/100g (Terwanger Philip *et al.*, 2021). The current study indicated that the drying process significantly affected the tannin levels in pineapple, with sun-drying

yielding the lowest concentration at 0.0877 mg/g, followed by oven-drying at 0.1009 mg/g, and dehydrating resulting in the highest concentration at 0.1388 mg/g. Temperature played a crucial role in influencing tannin content in plants; for instance, certain studies had shown that increased growth temperatures could lead to reduced tannin levels in sorghum, where tannin content significantly decreased under high-temperature conditions (Wu *et al.*, 2016; Mamiro *et al.*, 2017). On the other hand, dehydration process could lead to a decrease in extractable tannins in plant materials, thereby improving the nutritional quality of the foods. This reduction was largely attributed to the alteration of tannins into forms that are less biologically active during the dehydration process (Julkunen-Tiitto *et al.*, 2021).

Tannins are regarded as moderately toxic to aquatic organisms, displaying both beneficial and detrimental effects depending on their concentration. In aquaculture, while tannins can offer protective roles against disease and enhance feed quality, excessive levels may impede digestion and growth in specific fish species (Tom Barr, 2006). Research on grass carp indicates that dietary tannin levels up to 1.75% can be tolerated, suggesting that while some tannin supplementation may be safe, higher concentrations could pose risks (Yao *et al.*, 2019).

Conclusion: Drying techniques have a notable impact on the antinutritional, nutritional, and antioxidant properties of pineapple industry byproducts. Dehydrated samples exhibited higher antinutritional composition, underscoring the need for further studies to explore the Maximum Tolerable Concentration of tannins in aquaculture commodities. Among the drying methods, both oven drying and sun-drying proved effective in preserving the nutritional and antioxidant properties of the waste. Oven-drying and sun-drying retained most of the total flavonoid content, while sun-drying resulted in notably lower tannin levels. These findings highlight the importance of selecting optimal drying methods to balance nutrient retention and minimize antinutritional compounds.

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Conflict of Interest: The authors declare that there is no conflict of interest. All findings, results, and

interpretations presented are solely based on objective scientific research, and no external financial or personal influences have affected the study's outcome.

Author's Contribution

Jhon Wary E. Repamonte - Lead author of the manuscript, responsible for coordinating sample collection and preparation.

Fiona L. Pedroso, Casiano H. Choresca, Jr., Fernand F. Fagutao and Mary Jane S. Apines-Amar - Provided supervision and oversight of the research design, as well as critical review and revision of the manuscript.

Gwen M. Anuevo, Fernie A. Catienza, and Mark Lloyd B. Bajalla - Assisted in laboratory analyses, including assessments of antinutritional factors and antioxidant properties.

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