Innovative Food Science and Emerging Technologies 85 (2023) 103313

Contents lists available at ScienceDirect

Innovative Food Science and Emerging Technologies

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

Bioprocessing of pineapple waste for sustainable production of bioactive compounds using solid-state fermentation

Sarah L. Paz-Arteaga [a], Juan A. Ascacio-Valdés [b], Cristóbal N. Aguilar [b], Edith Cadena-Chamorro [a], Liliana Serna-Cock [c], Miguel A. Aguilar-González [d], Nathiely Ramírez-Guzmán [e], Cristian Torres-León [f,*]

[a] Universidad Nacional de Colombia, Faculty of Sciences, Agricultural Engineering Research Group, Medellín, Antioquia 050034, Colombia
[b] School of Chemistry, Universidad Autonoma de Coahuila, Unidad Saltillo, Saltillo, Coahuila 25280, Mexico
[c] Universidad Nacional de Colombia, Faculty of Engineering and Administration, Palmira, Valle del Cauca 763533, Colombia
[d] Centro de Investigación y de Estudios Avanzados del Instituto Politécnico Nacional, Unidad Saltillo, Ramos Arizpe, Coahuila 25900, Mexico
[e] Center for Interdisciplinary Studies and Research, Universidad Autonoma de Coahuila, Unidad Saltillo, Arteaga, Coahuila 25280, Mexico
[f] Research Center and Ethnobiological Garden, Universidad Autonoma de Coahuila, Unidad Torreón, Viesca, Coahuila 27480, Mexico

ARTICLE INFO

Keywords:
Pineapple by-products
Bioactive compounds
Phenolic compounds
Biological properties
Antioxidant activity
Aspergillus Niger

ABSTRACT

The bioprocessing of pineapple wastes into value-added bioproducts is a sustainable solution. This study examined a novel alternative for producing bioactive compounds using *Aspergillus niger* GH1 for solid-state fermentation (SSF) of pineapple peel and core. The results revealed that the chemical composition of pineapple waste was suitable for use in SSF. During the first 32 h of the SSF, free phenols increased by 72.31% and were positively related to antioxidant activity as measured by DPPH (2,2-diphenyl-1-picrylhydrazyl). ß-Glucosidase and cellulase activities were increased by the SSF and were positively associated with free phenolic acids such as 5-caffeoylquinic acid. Scanning electron microscopy (SEM) confirmed mycelial invasion in pineapple waste. Analysis of free and bound phenols by high-performance liquid chromatography-mass spectrometry (HPLC-MS) showed more conjugated phenols in the unfermented than fermented waste. These results provided a broad overview of the chemical compounds with antioxidant capacity that are generated from the growth of *A. niger*.

Industrial relevance: The market for polyphenols and enzymes is expected to grow exponentially in the coming years. In response to this requirement and to the circular economy that seeks to take advantage of waste by generating new products, solid-state fermentation is applied, which produces in a short time polyphenols with antioxidant capacity and enzymes with possible application in the food industry from the fermentation of organic waste such as pineapple waste.

1. Introduction

Pineapple (*Ananas comosus* (L.) Merr.) is a consumed fruit worldwide due to its good flavor, consistency, and nutritional value. The largest producer of pineapple in the world is Costa Rica, followed by the Philippines and Brazil. The agro-industrialization of this fruit produced one ton of dry waste per hectare (Aruna, 2019). Pineapple waste consists mainly of peel (29–42%), core (9.4–20%), and crown (2.7–5.9%) (Rico, Gullón, Alonso, & Yáñez, 2020). This waste is a problem for industries due to the final disposal cost. In most developing countries, waste is buried or burned, thus generating environmental and health issues, and wasting the high nutritional and functional value present in this material (Qdais, Abdulla, & Qrenawi, 2010). Some of the value-added products that can be obtained from pineapple waste are protein (Aruna, 2019), polyhydroxyalkanoates (Vega-Castro et al., 2016), and polyphenols. Some of the polyphenols found in pineapple waste are compounds with high biological activity, such as gallic acid, catechin, epicatechin, and ferulic acid (Steingass, Glock, Schweiggert, & Carle, 2015).

* Corresponding author.
E-mail addresses: slpaza@unal.edu.co (S.L. Paz-Arteaga), alberto_ascaciovaldes@uadec.edu.mx (J.A. Ascacio-Valdés), cristobal.aguilar@uadec.edu.mx (C.N. Aguilar), emcadenac@unal.edu.co (E. Cadena-Chamorro), lserna@unal.edu.co (L. Serna-Cock), miguel.aguilar@cinvestav.edu.mx (M.A. Aguilar-González), nathiely.ramirez@uadec.edu.mx (N. Ramírez-Guzmán), ctorresleon@uadec.edu.mx (C. Torres-León).

https://doi.org/10.1016/j.ifset.2023.103313
Received 29 November 2022; Received in revised form 8 February 2023; Accepted 22 February 2023
Available online 25 February 2023

The main bioactive compounds that have gained great economic importance recently are polyphenolic (extracted from natural sources). These organic compounds significantly benefit human health and have multiple applications in the food industry. Phenolic compounds have biological functions, such as antimicrobial, antioxidant, anticancer, antidiabetic, and anti-inflammatory, activities. These natural compounds can be used in the food and pharmaceutical industries and replace the function of synthetic additives such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT). Two controversial substances are due to their adverse effects on human health (Abdel-Aty, Elsayed, Salah, Bassuiny, & Mohamed, 2021; Barakat, Bassuiny, & Saleh, 2020).

Polyphenols or phenols are secondary metabolites in plants. Therefore, the extraction of polyphenolic compounds can be an alternative for the recovery of pineapple waste. However, 24% of these compounds cannot be easily extracted (bound phenolic) (Acosta-Estrada, Gutiérrez-Uribe, & Serna-Saldívar, 2014). Conventional and unconventional extraction methods that use high temperatures for extraction are not very cost-effective or practical for this process. These methods can cause damage to phenolic compounds, toxicity, low specificity, and low yield in the release of bound phenolic compounds (Oroian & Escriche, 2015).

In this sense, Solid-State Fermentation (SSF) using agro-industrial waste as a substrate and support is increasingly common. The SSF is an economical and environmentally friendly technology used to recover bioactive compounds. One of the essential variables in SSF is the fermenting microorganism. Fungi are the most used microorganisms in SSF. They are better adapted to work in solid matrices where the mycelium can grow and expand (Lizardi-Jiménez & Hernández-Martínez, 2017). *Aspergillus niger* GH1 is a good fungus for this type of technology. It has a great capacity to produce a wide variety of enzymes and develop in a wide range of temperatures and pHs (Ascacio-Valdés et al., 2016).

Taking advantage of the production of enzymes by the microorganism involved in the fermentation, the SSF has recently been used for obtaining free and bound phenolic compounds from substrates such as avocado seed (Yepes-Betancur et al., 2021), mango seed (Torres-León et al., 2019) and orange peel (Xue et al., 2022). Despite these advances, the use of *A. niger* GH1 as a fermenting microorganism on pineapple waste and identifying the released bound phenols remain to be explored. Consequently, following the twelfth of the Sustainable Development Goals adopted in 2015 by the United Nations, which addresses "responsible production and consumption" and establishes the efficient management of waste, this work evaluates the use of agroindustrial pineapple waste as a substrate for the microorganism *A.niger* GH1 in a solid fermentation to obtain compounds of high commercial value, such as phenols and enzymes.

2. Materials and methods

2.1. Chemicals and reagents

Folin-Ciocalteu reagent, gallic acid (GA), DPPH (2,2-diphenyl-1-picrylhydrazyl), Trolox ((±)-6 Hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid), and BHT (butylated hydroxytoluene) were purchased from Sigma-Aldrich (St. Louis, MO, EE. UU.); Ethyl acetate ($C_4H_8O_2$) from FisherChemicals (New Jersey, USA); Ethanol (C_2H_5OH)) from CTR scientific (Monterrey, México). Sodium hydroxide (NaOH), hydrochloric acid (HCl), sodium carbonate (Na2CO3) from J.T. Baker® (Mexico City, Mexico); Potato dextrose agar (PDA) from Solviosa ® (Jalisco, Mexico) and distilled water from SumiNorte (Saltillo, México).

2.2. Microorganism

The microorganism *A. niger* GH1 strain was obtained from Departamento de Investigación en Alimentos / Universidad Autónoma de Coahuila (DIA-UAdeC), preserved at 4 °C in glycerin solution and skim milk. The fungus was reactivated by adding 1 mL of the frozen solution to PDA agar, then incubated for 7 days at 30 °C.

2.3. Plant material

MD2 pineapples (*Ananas comosus* (L.) Merr.) were obtained from the Cañaveral market in Palmira, Valle del Cauca, Colombia. The pineapples were washed with drinking water, disinfected with sodium hypochlorite at 200 pm for 10 min, manually peeled, and cut into pieces of approximately 1 × 2 cm. Subsequently, the residues (peel and core) were dried in a convective oven (70 °C) (Binder, Germany) until reaching a humidity of 5%. After drying, the waste was ground in a hammer mill (I.K. A., Germany) and sieved, and three fractions of particles <0.25 mm, 0.25–2.3 mm and < 0.25 mm- 2.3 mm were obtained.

Brix degrees were measured using a digital refractometer (Atago, Japan), and the pH was measured with a pH-meter (Ohaus STR3100, USA) acidity was measured by the titration method (AOAC 942.152000). Finally, the total fruit yield was measured in Eq. 1, and the pineapple peel and core percent with Eq. 2.

$$\%yield = \frac{Total\ weight\ of\ waste\ (g)}{Total\ fruit\ weight\ (g)} * 100 \quad (1)$$

$$\%peel\ or\ core\ participation = \frac{peel\ or\ core\ weight\ (g)}{peel\ weight\ (g) + core\ weight\ (g)} * 100 \quad (2)$$

2.3.1. The chemical composition of pineapple peel and core

Chemical analysis was measured independently for pineapple waste (peel and core). Ash content was measured at 550 °C for 8 h, and crude protein content using the Kjeldahl method with a conversion factor of 6.25. The ethereal extract was obtained with a soxhlet system according to AOAC 920.39 (1990) and the neutral detergent fiber using the Van Soest method (AOAC 973.18, 1990). The content of minerals was measured (Ca, P, Mg, and K) using X-ray fluorescence (Epsilon 1, UK). Total carbohydrates (TC) were calculated using the following equation:

$$TC = 100 - \%A + \%CPAG + \%L \quad (3)$$

where %A ash content, %CPAG crude protein content, and %L lipid content.

2.4. Tests of support in solid-state fermentation

A pineapple peel and core ratio of 70:30 was used with three different particle sizes to test their potential use as support (1: small <0.25 mm, 2: medium 0.25 mm–2.3 mm, and 3: all material <0.25 mm-2.3 mm). For the Water Absorption Index (WAI), 2.5 g of sample dissolved in 30 mL of distilled water was heated in a thermal bath (VWR, USA) at 70 °C for 30 min; the resulting paste was weighed and centrifuged at 3000*g* for 20 min, the supernatant was decanted, the sediment was weighed (Du, Jiang, Yu, & Jane, 2014), WAI calculated, and the results expressed as weight of sediment (g) per weight of dry solid (g):

$$WAI\ (g/g) = \frac{Weight\ of\ sediment\ (g)}{Weight\ of\ dry\ solid(g)} \quad (4)$$

The Critical Humidity Point (CHP) was measured using the sediment from the water absorption test. About 1 g of saturated sample was placed on the 60 °C thermobalance (Ohaus MB 23, USA), weighed every min for up to 10 min, and then every five minutes until it reached a constant weight value. The a_w was measured using an Aqualab at 25 ± 2 °C, and pH was measured using a pH meter; briefly, 1 g of sample with distilled water (1:2) was homogenized in a thermal bath at 30 °C for 10 min. Invasion capacity was evaluated by measuring the growth kinetics of *Aspergillus niger* GH1 in Petri dishes, with a humidity of 65% (above the critical humidity point and below the maximum moisture). An 8 mm fragment of mycelium was inoculated in the center of the Petri dish, previously activated in PDA agar. The fragment was obtained using a

sterile punch and measured its radial growth every 12 h. The value was reported as mm/h.

2.5. Solid-state fermentation

Petri dishes were used as reactors in the fermentation tests with the best conditions found in section 2.4. Conditions for the SSF included 2 * 10^7 spores per g and 8 g of dry material between pineapple peel and core (70:30) with a particle size between <0.25 mm–2.3 mm, the humidity of 65%, and fermentation process monitoring every 8 h at 30 °C to determine the time necessary for greater extraction of phenols.

2.6. Measurement of phenolic compounds

2.6.1. Free phenolic fraction

The extraction of the phenolic content was carried out according to the methodology described by Torres-León et al. (2019). Briefly, 1 g of fermented material dissolved in 90% ethanol (1:30 *w/v*) was incubated in a 45 °C water bath for 1 h; the extracts were filtered using Whatman No. 1 paper and a vacuum pump (Helch 2534B-01, USA), and stored at −18 °C. The sediment was saved for the determination of bound phenols (See section 2.10).

2.6.2. Determination of total phenolic content (TPC)

Phenolic content determination was measured using the methodology described by Makkar (2003). A volume of 20 μL of the extract was mixed with 20 μL of Folin-Ciocalteu reagent in one well. After 5 min of manual shaking, 20 μL of sodium carbonate (0.01 M) was added to each sample, the solution was stirred again for 5 min, diluted with 125 μL of distilled water, and the absorbance at 790 nm was read using a spectrophotometric microplate reader (Epoch, BioTek, Instruments, Inc.). The results were expressed as mg gallic acid/g dry matter. The calibration curve was made by measuring the absorbance of known concentrations of gallic acid to obtain the phenolic content of the extract of each sample. Then, the correlation coefficient was established (R^2 = 0,9969).

2.7. Antioxidant activity assay

The DPPH radical scavenging activity of the sample was analyzed using the method reported by Molyneux (2004). A volume of 7 μL of the phenolic extract mixed with 193 μL of DPPH radical solution (60 μM) was kept in the dark at room temperature for 30 min. Then, the absorbance at 517 nm was measured using a spectrophotometer microplate reader (Epoch, BioTek, Instruments, Inc.). The results were expressed in mg of Trolox equivalents per gyk of extract, using a standard Trolox calibration curve (R^2 = 0,9932).

2.8. Enzymatic activities assays

The enzyme extraction was performed according to the modified methodology described by Idris, Pandey, Rao, and Sukumaran (2017). The fermented biological material was immersed in citrate buffer (0.05 M, pH 4.9) in a 26:74 ratio, the solid material separated from the liquid by mechanical compression, and the extracts were filtered using Whatman No. 1 paper, a vacuum pump (Helch 2534B-01, USA) and stored at −18 °C.

2.9. β-glucosidase activity assay

The β-Glucosidase activity was measured with the modified method described by Leite, Silva, Salgado, and Belo (2019). β-Glucosidase activity was determined using *p*-nitrophenyl-β- d-glucopyranoside (PNG) as substrate. The reaction was carried out with 40 μL of PNG and 40 μL of the sample. The enzymatic reaction was carried out at 50 °C for 10 min. After 10 min, 200 μL sodium carbonate (0.1 M) was added. Finally, 200 μL of each sample was added to the microplate, and the absorbance at 400 nm was read.

2.9.1. Cellulose activity assay

Cellulose activity was measured with the modified method described by Leite et al. (2019). Cellulase activity was determined using Whatman No. 4 filter paper as the substrate. The reaction was carried out with 20 μL of the sample at 50 °C for 60 min. After 60 min, 120 μL of DNS will be added and heated to 100 °C. Finally, 36 μL of each sample after being cooled and 160 μL of distilled water were added to the microplate and read at 540 nm.

For the determination of the enzymatic activity of β-Glucosidase and cellulose, a calibration curve was made with p-nitrophenol and glucose with concentrations between 0 and 500 ppm, respectively. One international unit (IU) of enzyme activity was defined as the amount of enzyme required to release 1 μmol of product per minute under standard assay conditions. The values will be expressed in units per gram of dry substrate (U/g).

2.10. Microstructural analysis

The surface morphology of the fermented and unfermented material was visualized using an environmental scanning electron microscope (Philips XL30-ESEM). Briefly, the samples are placed in a container with liquid nitrogen and fractured. The samples were held in a sample holder with copper tape perpendicular to the microscope detectors. The films were coated with gold-palladium metals using an ion spray for 30 s at 2 μA, generating a layer of approximately 3 nm. In the electron microscope, three modes were used under the same conditions (working distance: 5.5 mm, accelerating voltage: 12 keV, gap size: 4 to 5 nm, magnification: 1000× and 2500×) (Saenz-Mendoza et al., 2020).

2.11. Analytical RP-HPLC-ESI-MS

The free fractions obtained in section 2.6.1 and the bound fractions obtained from the sediment of the free fraction were evaluated by RP-HPLC-ESI-MS for the identification of molecules. The bound fraction was obtained following the methodology described by Torres-León et al. (2019). The sediment was mixed with 50 mL of sodium hydroxide (2 M) at room temperature for four hours. Then, hydrochloric acid was added to the mixture until reaching pH 2.0, filtered using Whatman No. 1 paper, and liquid-liquid extraction was carried out in three parts with 70 mL of ethyl acetate. The fractions were combined and rotary evaporated (40 cm Hg, 60 °C, 150 RPM) until the ethyl acetate was eliminated. The bound phenols were constituted in 5 mL of water. The extracts (free fraction and bound fraction) were pre-purified with amberlite XAD 16, lyophilized, reconstituted in distilled water, and filtered through a 0.45 μm nylon filter. The bound fractions from times 0 and 32 h and the free fractions from times 0, 8, 32, and 80 h were analyzed by high-performance liquid chromatography/electrospray ionization/mass spectrometry (HPLC/ESI/MS). In the analytical RP-HPLC-ESI-MS, 1.5 mL of each filtered extract was taken and injected into the HPLC (C18 column: 150 mm × 2.1 mm, 3 μm, Grace, USA). The Varian HPLC system consists of an autosampler (Varian Pro Star 410, USA), a PDA detector (Varian Pro Star 330, USA) (eluents: formic acid and acetonitrile), and a ternary pump (Varian Pro Star 230I, USA) (Ascacio-Valdés et al., 2016).

2.12. Statistical analysis

All assays were performed in triplicate with independent samples. All analyzes were reported using means and standard deviations. The results were analyzed by ANOVA, Tukey test was used to analyze the significant difference. Pearson's correlation was performed to evaluate the correlations among variables. Statistical analysis was performed using the RStudio Version 1.4.1103 ® software (Boston, USA).

3. Results and discussion

The state of fruit ripening significantly influences the type and amount of phenols present in the fruit components (Briante et al., 2002). Three of the chemical characteristics that are most related to the level of pineapple ripening, soluble solids content, acidity, and pH were evaluated. The pineapples used in this study have 14.83 ± 0.15°Bx, a citric acid value of 0.92 ± 0.03%, and 3.79 ± 0.005 pH. These values relate to commercial maturity. These values are similar to those reported by Micanquer-Carlosama, Cortés-Rodríguez, and Serna-Cock (2020) for the same variety of pineapple.

Regarding the proportion of residues present in pineapple, this fruit has a percentage of 45 ± 0.05 residues (peel and heart). The peel within that percentage of biowaste has a participation of 73 ± 0.06% and the core of 27 ± 0.12%. These results are similar to those described by Rico et al. (2020) to the pineapple variety Morris. The percentage of waste from pineapple is high compared to other fruits such as melon (25%) and watermelon (30%) (Rico et al., 2020). The use of pineapple residues would allow a new source of income and a new products.

3.1. The chemical composition of pineapple peel and core

The chemical characterization on a dry basis of pineapple waste (peel and core) is shown in Table 1. The peel presented a higher amount of dry matter, ashes, and protein than the core, contrary to the total carbohydrate content. The total carbohydrates, NDF, and protein content show that the dry material is an elemental carbon and nitrogen source, elements necessary for fungal growth and enzyme production. Torres-León et al. (2019) y Yepes-Betancur et al. (2021) demonstrated that biomaterials with similar proximal content are suitable for SSF.

Aruna (2019), Dibanda Romelle, Ashwini, and Manohar (2016), and Kodagoda and Marapana (2017) obtained lower values of total carbohydrates for peel (55.52–74.08% *w*/w) and core (83.03% w/w). Regarding the protein content of the pineapple peel and core, Dibanda Romelle et al. (2016) and Kodagoda and Marapana (2017) reported similar values (5.11–3.67% w/w). The lipid content of pineapple peel and core is lower than that reported by Morais et al. (2015) and Kodagoda and Marapana (2017) (1.10–2.35% w/w). Variation in proximal content is attributed to factors including variety, stage of maturity, soil, agricultural practices, and climatic conditions (Difonzo et al., 2019).

The amount of ash in the peel is lower than that reported by Dibanda Romelle et al. (2016) (4.39 ± 0.14) and higher than that reported by Kodagoda and Marapana (2017) (4.32 ± 0.09). Ca and Mg values are lower than those reported by Dibanda Romelle et al. (2016) (8.30 ± 0.54 and 5.32 ± 0.49), equally to those reported for K by (Micanquer-Carlosama et al., 2020) (0.20). The minerals Ca, P, Mg, and K are part of the microelements necessary for the excellent development of the microorganism. Ca and Mg markedly promote enzyme activity. Particularly Ca favors the activity of cellulases (Zeng et al., 2016).

Table 1
Chemical characterization of pineapple waste.

Component	Peel	Core
Total dry matter (% *w*/w)	84.5 ± 0.14	83.1 ± 0.52
Total carbohydrates (%w/w)	88.4 ± 0.13	92.8 ± 0.12
Ashes (%w/w)	5.2 ± 0.03	3.4 ± 0.03
Ca (%w/w)	0.6 ± 0.08	0.3 ± 0.01
P (%w/w)	0.2 ± 0.01	0.2 ± 0.01
Mg (%w/w)	0.9 ± 0.03	1.9 ± 0.11
K (%w/w)	2.6 ± 0.24	2.3 ± 0.01
Protein (%w/w)	5.5 ± 3.87	3.3 ± 0.08
Ethereal extract (%w/w)	0.8 ± 0.01	0.5 ± 0.06
NDF (%w/w)	31.9 ± 0.42	15.9 ± 0.83

NDF: Neutral detergent fiber.

3.2. Tests of support in solid-state fermentation

The CHP represents the water bound to the plant matrix as the water that microorganisms cannot use for their biological functions, and the WAI is the amount of water the support can absorb. The WAI depends on the gel-forming ability of macromolecules and the availability of hydrophilic groups that bind water molecules together (Mussatto, Aguilar, Rodrigues, & Teixeira, 2009).

CHP and WAI values are shown in Table 2. Low CHP values benefit microbial growth. High WAI values benefit the fermentation process because the humidity could be changed to benefit the growth of the fungus. Although the values of CHP and WAI for the three sizes of fermentable material evaluated are not significantly different ($P > 0.05$), these values demonstrate that plant material is helpful for a solid fermentation process. WAI and CHP values are similar to those reported in other materials used for SSF. Buenrostro-Figueroa et al. (2017) reported WAI amounts of 3.74 ± 0.10 g/g of dry matter for agro-industrial waste of figs and CHP values of 58% for cork oak bark, and Torres-León et al. (2019), CHP values of 56.5% for mango seed.

The optimum pH and a_w for growth of *A. niger* are 5.0 and 0.96 at 30 °C, respectively (Aguilar et al., 2007). Although the pH of the 70:30 ratio of pineapple peel and core is 3.92 and the a_w is between 0.546 and 0.601, the radial growth rate shows that the microorganism grows under the conditions evaluated. The radial growth rate is not significantly different ($P > 0.05$) among the three particle sizes estimated. However, the growth was higher than that previously reported for *A. niger* GH1 in agro-industrial waste such as pomegranate seed (0.2 mm/h) and pomegranate peel (0.4 mm/h) (Robledo et al., 2008).

The particle size of the fermentable material is significant due to the surface area and porosity. These characteristics determine the transfer of heat, oxygen, and nutrients, determining how the microorganism will develop on the support. Very small particles will prevent the correct transfer of mass and energy. Although large particles allow proper aeration in the fermentation medium, these particles can make it more difficult for the microorganism to dispose of nutrients (Pandey, Soccol, & Mitchell, 2009). In the case of pineapple waste, a better growth rate was obtained when all the available material was used, the combination of large and small particles.

3.3. Solid-state fermentation (SSF)

The polyphenolic compounds increased as the *A. niger* fermentation time increased (Fig. 1). Although it increased at 8 h, the polyphenol content remained without significant difference from hour 32 (72.31% with respect to the value of time 0) until 80 h. This result is superior to the polyphenolic content of pineapple waste extracted by Soxhlet (30.2%) (de Oliveira et al., 2009) and SSF (72 h) with *Kluyveromyces marxianus* NRRL Y-8281 (7.14%) (Rashad, Mahmoud, Ali, Nooman, & Al-Kashef, 2015). The difference between conventional methods, such as Soxhlet extraction, and SSF for obtaining polyphenols, lies in the lack of specificity and the impossibility of breaking bonds that allow the release

Table 2
Pineapple peel and core (70:30) support tests.

Analysis	Particle size		
	<0.25 (mm)	0.25–2.3 (mm)	<0.25–2.3 (mm)
CHP (g/g)	55.601 ± 3.881	60.530 ± 3.287	61.950 ± 6.140
WAI (%)	5.023 ± 0.418	5.214 ± 0.081	4.706 ± 0.117
pH	3.92 ± 0.043	3.92 ± 0.032	3.92 ± 0.058
aw	0.601 ± 0.123	0.631 ± 0.112	0.546 ± 0.113
Moisture (%)	8.750 ± 1.484	3.450 ± 0.494	4.600 ± 0.565
Radial growth rate (mm/h)	0.651 ± 0.001	0.468 ± 0.019	0.688 ± 0.054

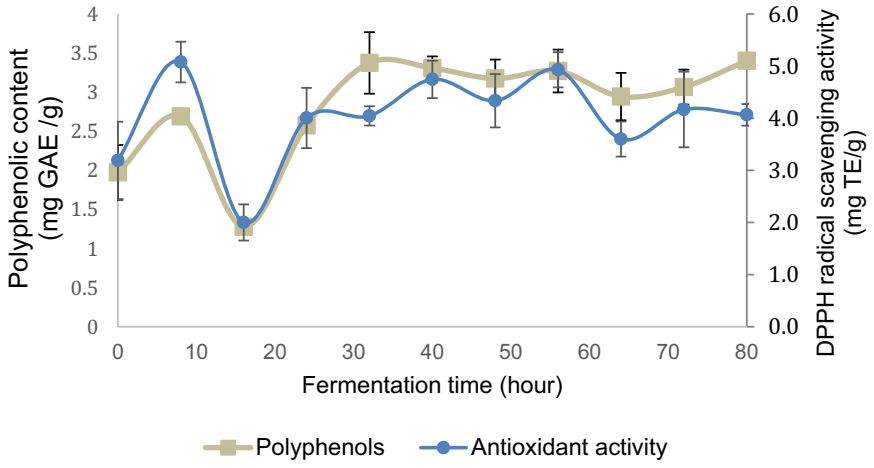

Fig. 1. Polyphenolic content and antioxidant activity during the fermentation of pineapple waste.

of compounds (Gligor et al., 2019). The increase in phenolic content at hour 8 may be due to the rise in compounds released. *A. niger* GH1 is a microorganism with the potential to obtain high phenolic content in a short fermentation time (Torres-León et al., 2019). The decrease of the compounds may be due to the decomposition or the consumption during the exponential phase. Phenolic compounds such as gallocatechin, protocatechial acid, syringic acid, and quercetin tend to decrease during fermentation after being released (Yeo, Tsao, Sun, & Shahidi, 2021). These alterations in phenolic profiles are prevalent during SSF. The discrepancy in the phenolic content is due to the biological matrix's composition and the microorganism's development. Yeo et al. (2021) and Romero, Brenes, García, García, and Garrido (2004) demonstrate similar behavior in lentil hull and Black Olives, respectively.

The polyphenolic content was positively correlated with the sequestering activity of DPPH ($r = 0{,}63$, p ⟨0,01), confirming that the increase in antioxidant activity is due to the increase in phenolic content during fermentation. DPPH value with respect to time at 32 h increased by 25% and behaved similarly to the study presented by Abdel-Aty, Barakat, Bassuiny, and Mohamed (2022b). and its correlation with the amount of phenolic compounds can be compared with that reported in the SSF of tamarind residues ($r = 0.7254$) (Jericó Santos et al., 2020). The increase in polyphenols is due to the synthesis and release of these compounds during the metabolism of *A. niger*. The microorganism breaks down the cell wall of the plant material through the production of cellulases, pectinases, and ligninases. These enzymes hydrolyze the ester bonds responsible for the union between the phenolic compounds and the cell wall matrix and are also responsible for the oxidative degradation of lignin.

3.4. Enzymes

Fig. 2 shows the behavior of β-glucosidase activity and cellulose during the first 56 h of FES of pineapple residues (peel and core) with *A. niger* GH1. The results showed that the activity of β-glucosidase is greater than the activity of cellulase throughout the fermentation time. It is observed that β-glucosidase and cellulase express the highest enzymatic activity between 32 and 40 h of fermentation, 29 U/g fermented material and 4 U/g fermented material, respectively; which coincides with the maximum peak of the amount of free phenols; in addition, it is observed that cellulase activity decreases at 24 h of fermentation and then, together with β-glucosidase, decreases again after 40 h of fermentation.

The development of fungi in SSF depends on enzymatic activity. Enzymes convert high molecular weight cellulosic material into low molecular weight compounds. These low molecular weight molecules are the nutrients that the mycelium will absorb. Cellulase initiates the

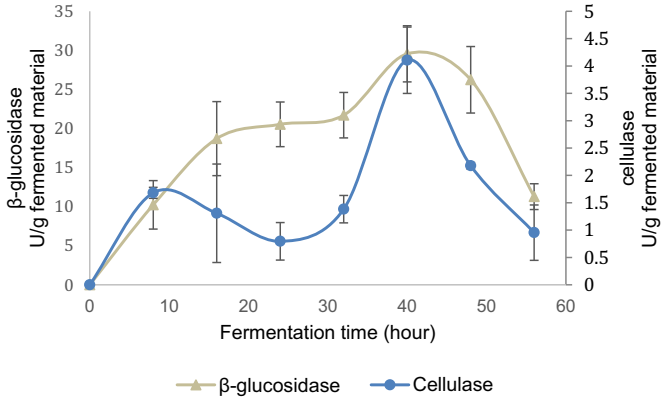

Fig. 2. The activity of β-glucosidase and cellulase during the fermentation time.

breakdown of cellulose and produces cellobiose. High cellobiose production inhibits cellulase activity (dos Santos, Ximenes, Kim, & Ladisch, 2019). β-glucosidase breaks down cellobiose and generates glucose monomers that are easy for the microorganism to use (Behera, Sethi, Mishra, Dutta, & Thatoi, 2017). In addition, the breaking of the long cellulose chain allows the release of compounds covalently linked to this polymer, allowing its easy extraction and use (Acosta-Estrada et al., 2014). The β-glucosidase and cellulase were measured because they have been related to the release of compounds from the biological matrix and because they are produced by *A. niger* (Ascacio-Valdés et al., 2014).

Total cellulase or filter papers (FPase) activity was similar to that reported by Prajapati, Kumar Suryawanshi, Agrawal, Ghosh, and Kango (2018) (3.8 FPU/g) using substrate copra meal and *A. tubingensis* NKBP-55. The β-glucosidase activity reported in this study is lower than in studies with *A. niger*. Manfrin Dias et al. (2016) reported production of 54.9 U/g using sorghum biomass as substrate and Aliyah, Alamsyah, Ramadhani, and Hermansyah (2017) reported 91.67 U/g using sugarcane bagasse as a substrate. According to Chakraborty, Gupta, Jain, and Kuhad (2016) difference in the expression of cellulases and β-glucosidase is due to the metabolism of each microorganism and the carbon and nitrogen source of a fermented substrate. Taherzadeh-Ghahfarokhi, Panahi, and Mokhtarani (2019) claim that a high amount of NDF positively affects cellulose production. In addition to β-glucosidase and cellulase, *A. niger* GH1 can produce other enzymes such as tannases, xylanases, ellagitannase, and polyphenol oxidase (Ascacio-Valdés et al., 2014). Cellulase enzyme activity is positively correlated with the amount of free phenols (Fig. 1) ($r = 0{,}46$, $p < 0{,}01$), like β-glucosidase (r

= 0,42, p < 0,01). Previous studies show that using agroindustrial waste to obtain free phenolic compounds is positively related to the enzymes generated in the same process. Abdel-Aty et al. (2022b) show a correlation of 0.84 between the number of phenols and β-glucosidase activity and Teles, Chávez, Santiago, Gottschalk, and Tonon (2021) and Correia, McCue, Magalhães, Macêdo, and Shetty (2004) show that during SSF using *A. niger* 3T5B8 and *Rhizopus oligosporus*, high cellulase and β-glucosidase activity is obtained. This activity is related to increasing phenolic compounds such as hydrolyzable tannins, protocatechuic, and p-coumaric acid.

3.5. Microstructural analysis

Electron microscopy was used to observe the morphological changes of fermented and unfermented pineapple waste. Fig. 3-a shows the structure of unfermented pineapple waste. A compact, organized, and rough structure is observed due to the presence of fiber. Fig. 3-b shows pineapple waste fermented with *A. niger* GH1. The rupture of the vegetable biological matrix and invasion of the mycelium of the fungus are observed. Changes in the structure of the vegetable matrix of pineapple designs after fermentation may be due to damage to the crystal structures of the cellulose chains during fermentation, in addition to the decomposition of other structures such as lignin, hemicellulose, and proteins during the metabolism of the fungus. These results are similar to those described by Selvam et al. (2014). These authors also show the modification of the morphology of organic waste due to fermentation.

3.6. Analytical RP-HPLC-ESI-MS

Table 3 shows the bound and free compounds identified at different fermentation times of pineapple waste. The first two samples correspond to the bound compounds at times 0 and 32 h of the fermentation. The remaining four samples correspond to the free compounds at times 0, 8, 32, and 80 h. The identification of these compounds was carried out with the help of the RP-HPLC-ESI-MS database used and the bibliography reported for pineapple waste by Steingass et al. (2015). Caffeoyl hexoside, p-Coumaroyl hexoside, p-Coumaryl alcohol hexoside, Feruloyl aldarate, and 5-Caffeoylquinic acid were identified as written by Steingass et al. (2015). Table 3 shows that the samples of bound compounds (0 h and 32 h) present a more significant variability of phenolic compounds than the samples of free phenols. However, the compounds bound at time 0 are more than those found at fermentation time 32. This difference may be because phenols soluble in organic solvents such as the one used in this study are found in free or conjugated, esterified forms. Therefore, during fermentation, these bonds are broken with the help of fungal enzymes, leading to a partial or complete release of the compounds (Roasa, De Villa, Mine, & Tsao, 2021), transforming many bound/insoluble phenols into soluble and free phenols, or generating new phenols (Abdel-Aty, Barakat, Bassuiny, & Mohamed, 2022a).

Caffeoyl hexoside is present in almost all samples. Caffeoyl hexoside and 5-caffeoylquinic acid are cinnamate esters and have antimicrobial activity. Caffeoil hexoside is also present in fruits such as ciricote (Pacheco et al., 2021). Gallagil-hexoside is a derivative of gallic acid. Galagyl-hexoside is also present in the free phenols at time 0 and not in the other samples. Benzoic acid is one of the bound compounds that is not released during the evaluated fermentation process. Benzoic acid is an essential substructure of various natural products and can be found in microorganisms and plants (Ye et al., 2021). p-Coumaroyl glycolic acid and p-coumaric acid ethyl ester are free at fermentation 8. These two compounds are derivatives of p-coumaric acid, plant metabolites that exhibit antioxidant, anti-inflammatory, and antimicrobial properties (Li et al., 2022). (+)-Gallocatechin is a catechin present in a bound manner at time 0, which is present in an accessible manner at times 8, 32, and 80 and is not found at time 32 of the bound fraction. The presence of this free compound demonstrates the efficacy of SSF.

4. Conclusions

This study demonstrated the ability of the fungus *A. niger* GH1 to develop and degrade compounds present in pineapple agro-industrial residues. The fermentation conditions, particle size between <0.25–2.3, 65% humidity, 30 °C, and 32 h were useful for total invasion of the fermentation support and maximum release of phenolic compounds. Phenolic compounds were positively related to enzyme production and antioxidant capacity. The presence of compounds such as (+)-Gallocatechin and Galagyl-hexoside demonstrate the release and production of phenolic compounds by the activity of the fermenting fungus. These results provide a cost-effective and environmentally friendly alternative for the pharmaceutical and food industry for the production of natural antioxidants.

CRediT authorship contribution statement

Sarah L. Paz-Arteaga: Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Writing – original draft, Writing – review & editing. **Juan A. Ascacio-Valdés:** Investigation, Methodology, Validation, Software. **Cristóbal N. Aguilar:** Formal analysis, Investigation, Validation, Writing – review & editing. **Edith Cadena-Chamorro:** Formal analysis, Investigation, Validation, Writing – review & editing. **Liliana Serna-Cock:** Formal analysis, Investigation,

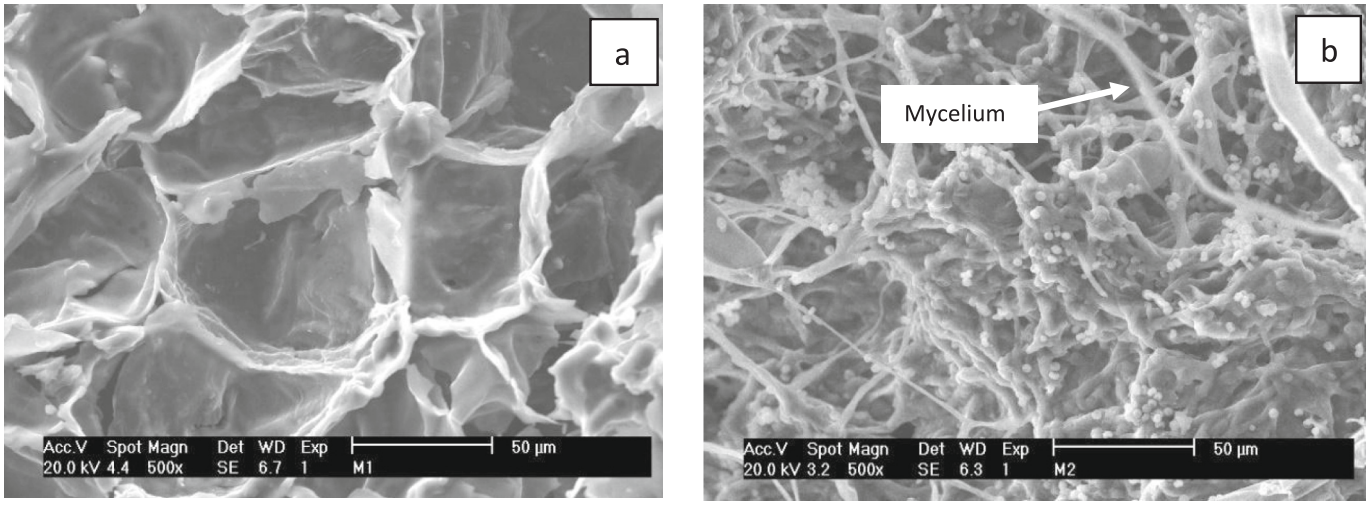

Fig. 3. Micrographs of unfermented pineapple peel and core (a). Pineapple peel and core at 32 h SSF (b). Image scale: 500× – 50 µm.

Table 3
Phenolic compounds identified from MD2 pineapple peel and core fermented with *A.niger* GH1 identified with the RP-HPLC-ESI-MS technique.

RT (min)	[M-H]-	Bioactive compounds	Molecular formula	Family	Bound compounds		Free compounds			
	m/z				0 h	32 h	0 h	8 h	32 h	80 h
5.989	340.9	Caffeoyl hexoside	$C_{15}H_{18}O_9$		X		X	X	X	X
6.162	353	5-Caffeoylquinic acid	$C_{16}H_{18}O_9$	Phenolic acids				X	X	
6.608	800.9	Spinacetin 3-O-glucosyl-(1- > 6)-[apiosyl(1- > 2)]-glucoside	$C_{34}H_{42}O_{22}$	Flavonoids			X			
7.965	191	p-Coumaric acid ethyl ester	$C_{11}H_{12}O_3$	Phenolic acids				X		
11.154	121	Benzoic acid	$C_7H_6O_2$	Phenolic acids	X					
27.731	327	p-Coumaroyl tyrosine	$C_{18}H_{17}NO_5$	Phenolic acids	X	X				
32.083	357	p-Coumaryl alcohol hexoside	$C_{15}H_{20}O_7$		X	X				
32.973	119	4-Vinylphenol	C_8H_8O	Alkylphenols	X	X				
33.633	222	p-Coumaroyl glycolic acid	$C_{11}H_{10}O_5$	Phenolic acids				X		
34.755	351	unidentified						X		X
38.814	385	Feruloyl aldarate			X	X				
39.877	796.7	unidentified							X	
40.752	185	Psoralen	$C_{11}H_6O_3$	Furanocoumarins				X		
41.387	341.1	Caffeic acid 4-O-glucoside	$C_{15}H_{18}O_9$	Phenolic acids		X				
44.056	325	p-Coumaroyl hexoside	$C_{15}H_{18}O_8$		X		X			
45.952	304.8	(+)-Gallocatechin	$C_{15}H_{14}O_7$	Flavonoids	X			X	X	X
46.124	781	Gallagyl-hexoside	$C_{34}H_{22}O_{22}$	Phenolic acids			X			

Validation, Writing – review & editing. **Miguel A. Aguilar-González:** Investigation, Methodology, Validation, Software. **Nathiely Ramírez-Guzmán:** Investigation, Methodology, Validation, Software. **Cristian Torres-León:** Funding acquisition, Investigation, Methodology, Project administration, Supervision, Writing – review & editing.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

Acknowledgments

Sarah L. Paz-Arteaga thanks the Universidad Nacional de Colombia for the scholarship granted to develop master's studies in biotechnological sciences (Resolution 012 of 2020) and the financial support to carry out the stay at the Universidad Autonoma de Coahuila (Resolution F·C 0349 of 2022). Authors are thankful to Ph.D. Monica L. Chavez Gonzalez, MSc Orlando Simon Ruiz Villadiego and Ph.D. Claudia M. Lopez Badillo for their valuable assistance during this study.

References

Abdel-Aty, A. M., Barakat, A. Z., Bassuiny, R. I., & Mohamed, S. A. M. (2022a). Antioxidant - polyphenols of saw palmetto seeds : Statistical optimized production and improved functional properties under solid - state fermentation by Trichoderma reesei. *Journal of Food Measurement and Characterization, 0123456789*. https://doi.org/10.1007/s11694-022-01675-w

Abdel-Aty, A. M., Barakat, A. Z., Bassuiny, R. I., & Mohamed, S. A. M. (2022b). *Improved production of antioxidant-phenolic compounds and certain fungal phenolic-associated enzymes under solid-state fermentation of chia seeds with Trichoderma reesei: Response surface methodology- based optimization* (pp. 3488–3500).

Abdel-Aty, A. M., Elsayed, A. M., Salah, H. A., Bassuiny, R. I., & Mohamed, S. A. (2021). Egyptian chia seeds (Salvia hispanica L .) during germination : Upgrading of phenolic profile, antioxidant , antibacterial properties and relevant enzymes activities. *Food Science and Biotechnology, 30*(5), 723–734. https://doi.org/10.1007/s10068-021-00902-2.

Acosta-Estrada, B. A., Gutiérrez-Uribe, J. A., & Serna-Saldívar, S. O. (2014). Bound phenolics in foods, a review. *Food Chemistry, 152*, 46–55. https://doi.org/10.1016/j.foodchem.2013.11.093

Aguilar, C. N., Rodríguez, R., Gutiérrez-Sánchez, G., Augur, C., Favela-Torres, E., Prado-Barragan, L. A., … Contreras-Esquivel, J. C. (2007). Microbial tannases: Advances and perspectives. *Applied Microbiology and Biotechnology, 76*(1), 47–59. https://doi.org/10.1007/s00253-007-1000-2

Aliyah, A., Alamsyah, G., Ramadhani, R., & Hermansyah, H. (2017). Production of α-amylase and β-glucosidase from aspergillus Niger by solid state fermentation method on biomass waste substrates from rice husk, bagasse and corn cob. *Energy Procedia, 136*, 418–423. https://doi.org/10.1016/j.egypro.2017.10.269

Aruna, T. E. (2019). Production of value-added product from pineapple peels using solid state fermentation. *Innovative Food Science and Emerging Technologies, 57*(July), Article 102193. https://doi.org/10.1016/j.ifset.2019.102193

Ascacio-Valdés, J. A., Aguilera-Carbó, A. F., Buenrostro, J. J., Prado-Barragán, A., Rodríguez-Herrera, R., & Aguilar, C. N. (2016). The complete biodegradation pathway of ellagitannins by Aspergillus niger in solid-state fermentation. *Journal of Basic Microbiology, 56*(4), 329–336. https://doi.org/10.1002/jobm.201500557.

Ascacio-Valdés, J. A., Buenrostro, J. J., De la Cruz, R., Sepúlveda, L., Aguilera, A. F., Prado, A., … Aguilar, C. N. (2014). Fungal biodegradation of pomegranate ellagitannins. *Journal of Basic Microbiology, 54*(1), 28–34. https://doi.org/10.1002/jobm.201200278

Barakat, A. Z., Bassuiny, R. I., & Saleh, A. M. A. (2020). *Diabetic complications and oxidative stress: The role of phenolic-rich extracts of saw palmetto and date palm seeds. June, 1–13*. https://doi.org/10.1111/jfbc.13416

Behera, B. C., Sethi, B. K., Mishra, R. R., Dutta, S. K., & Thatoi, H. N. (2017). Microbial cellulases – Diversity & biotechnology with reference to mangrove environment: A review. *Journal, Genetic Engineering & Biotechnology, 15*(1), 197–210. https://doi.org/10.1016/j.jgeb.2016.12.001

Briante, R., Patumi, M., Limongelli, S., Febbraio, F., Vaccaro, C., Di, A., La, F., & Nucci, R. (2002). *Changes in phenolic and enzymatic acti v ities content during fruit ripening in two Italian culti v ars of Olea europaea L. 162* pp. 791–798). https://doi-org.ezproxy.unal.edu.co/10.1016/S0168-9452(02)00022-5.

Buenrostro-Figueroa, J. J., Velázquez, M., Flores-Ortega, O., Ascacio-Valdés, J. A., Huerta-Ochoa, S., Aguilar, C. N., & Prado-Barragán, L. A. (2017). Solid state fermentation of fig (Ficus carica L.) by-products using fungi to obtain phenolic compounds with antioxidant activity and qualitative evaluation of phenolics obtained. *Process Biochemistry, 62*, 16–23. https://doi.org/10.1016/j.procbio.2017.07.016

Chakraborty, S., Gupta, R., Jain, K. K., & Kuhad, R. C. (2016). Cost-effective production of cellulose hydrolysing enzymes from Trichoderma sp. RCK65 under SSF and its evaluation in saccharification of cellulosic substrates. *Bioprocess and Biosystems Engineering, 39*(11), 1659–1670. https://doi.org/10.1007/s00449-016-1641-6

Correia, R. T. P., McCue, P., Magalhães, M. M. A., Macêdo, G. R., & Shetty, K. (2004). Production of phenolic antioxidants by the solid-state bioconversion of pineapple waste mixed with soy flour using Rhizopus oligosporus. *Process Biochemistry, 39*(12), 2167–2172. https://doi.org/10.1016/j.procbio.2003.11.034

Dibanda Romelle, F., Ashwini, R. P., & Manohar, R. S. (2016). Chemical composition of some selected fruit peels. *European. Journal of Food Science and Technology, 4*(4), 12–21. www.eajournals.org.

Difonzo, G., Vollmer, K., Caponio, F., Pasqualone, A., Carle, R., & Steingass, C. B. (2019). Characterisation and classification of pineapple (Ananas comosus [L.] Merr.) juice from pulp and peel. *Food Control, 96*(July 2018), 260–270. https://doi.org/10.1016/j.foodcont.2018.09.015

Du, S. K., Jiang, H., Yu, X., & Jane, J. L. (2014). Physicochemical and functional properties of whole legume flour. *LWT - Food Science and Technology, 55*(1), 308–313. https://doi.org/10.1016/j.lwt.2013.06.001

Gligor, O., Mocan, A., Moldovan, C., Locatelli, M., Crișan, G., & Ferreira, I. C. F. R. (2019). Enzyme-assisted extractions of polyphenols – A comprehensive review. *Trends in Food Science and Technology, 88*(September 2018), 302–315. https://doi.org/10.1016/j.tifs.2019.03.029

Idris, A. S. O., Pandey, A., Rao, S. S., & Sukumaran, R. K. (2017). Cellulase production through solid-state tray fermentation, and its use for bioethanol from sorghum Stover. *Bioresource Technology, 242*, 265–271. https://doi.org/10.1016/j.biortech.2017.03.092

Jericó Santos, T. R., Santos Vasconcelos, A. G., de Aquino, L., Santana, L. C., Gualberto, N. C., Buarque Feitosa, P. R., & Pires de Siqueira, A. C. (2020). Solid-state fermentation as a tool to enhance the polyphenolic compound contents of acidic Tamarindus indica by-products. *Biocatalysis and Agricultural Biotechnology, 30* (November). https://doi.org/10.1016/j.bcab.2020.101851

Kodagoda, K., & Marapana, R. (2017). Development of non-alcoholic wines from the wastes of Mauritius pineapple variety and its physicochemical properties KHGK Kodagoda and RAUJ Marapana. *Journal of Pharmacognosy and Phytochemistry, 6*(3), 492–497. https://pdfs.semanticscholar.org/eb27/1c2ab90a5564bbb01aadfb140858942a8acb.pdf.

Leite, P., Silva, C., Salgado, J. M., & Belo, I. (2019). Simultaneous production of lignocellulolytic enzymes and extraction of antioxidant compounds by solid-state fermentation of agro-industrial wastes. *Industrial Crops and Products, 137*(April), 315–322. https://doi.org/10.1016/j.indcrop.2019.04.044

Li, Y., Saravana Kumar, P., Qiu, J., Ran, Y., Tan, X., Zhao, R., Ai, L., Yuan, M., Zhu, J., & He, M. (2022). Production of bioactive compounds from callus of Pueraria thomsonii Benth with promising cytotoxic and antibacterial activities. *Arabian Journal of Chemistry, 15*(6), Article 103854. https://doi.org/10.1016/j.arabjc.2022.103854

Lizardi-Jiménez, M. A., & Hernández-Martínez, R. (2017). Solid state fermentation (SSF): Diversity of applications to valorize waste and biomass. *3. Biotech, 7*(1). https://doi.org/10.1007/s13205-017-0692-y

Makkar, H. P. S. (2003). Measurement of total phenolics and tannins using Folin-Ciocalteu method. Quantification of tannins in tree and shrub foliage, 49-51. 2, 49–51. doi: https://doi.org/10.1007/978-94-017-0273-7_3.

Manfrin Dias, L., dos Santos, V., Beatriz Brant Albuquerque, C. J., Bruno, E., Pasquini, D., & Alves Baffi, M. (2016). Biomass sorghum as a novel substrate in solid state fermentation for the production of hemicellulases and cellulases by aspergillus Niger and A. fumigatus. *Journal of Applied Microbiology, 38*, 42–49. https://doi.org/10.1111/jam.13672

Micanquer-Carlosama, A., Cortés-Rodríguez, M., & Serna-Cock, L. (2020). Formulation of a fermentation substrate from pineapple and sacha inchi wastes to grow Weissella cibaria. *Heliyon, 6*(4), Article e03790. https://doi.org/10.1016/j.heliyon.2020.e03790, 0–7.

Molyneux, P. (2004). The use of the stable free radical Diphenylpicryl-Hydrazyl (DPPH) for estimating antioxidant activity. *Songklanakarin Journal of Science and Technology, 26*(May), 211–219.

Morais, D. R., Rotta, E. M., Sargi, S. C., Schmidt, E. M., Bonafe, E. G., Eberlin, M. N., … Visentainer, J. V. (2015). Antioxidant activity, phenolics and UPLC-ESI(−)-MS of extracts from different tropical fruits parts and processed peels. *Food Research International, 77*, 392–399. https://doi.org/10.1016/j.foodres.2015.08.036

Mussatto, S. I., Aguilar, C. N., Rodrigues, L. R., & Teixeira, J. A. (2009). Colonization of aspergillus japonicus on synthetic materials and application to the production of fructooligosaccharides. *Carbohydrate Research, 344*(6), 795–800. https://doi.org/10.1016/j.carres.2009.01.025

de Oliveira, A. C., Valentim, I. B., Silva, C. A., Bechara, E. J. H., Barros, M. P.d., Mano, C. M., & Goulart, M. O. F. (2009). Total phenolic content and free radical scavenging activities of methanolic extract powders of tropical fruit residues. *Food Chemistry, 115*(2), 469–475. https://doi.org/10.1016/j.foodchem.2008.12.045

Oroian, M., & Escriche, I. (2015). Antioxidants: Characterization, natural sources, extraction and analysis. *Food Research International, 74*, 10–36. https://doi.org/10.1016/j.foodres.2015.04.018

Pacheco, N., Méndez-Campos, G. K., Herrera-Pool, I. E., Alvarado-López, C. J., Ramos-Díaz, A., Ayora-Talavera, T., … Cuevas-Bernardino, J. C. (2021). Physicochemical composition, phytochemical analysis and biological activity of ciricote (Cordia dodecandra A. D.C.) fruit from Yucatán. *Natural Product Research, 36*(1), 440–444. https://doi.org/10.1080/14786419.2020.1774763

Pandey, A., Soccol, C. R., & Mitchell, D. (2009). Pre-treatment of agro-industrial residues. In *, 35. Biotechnology for agro-industrial residues utilisation: Utilisation of agro-residues* (pp. 13–33). https://doi.org/10.1007/978-1-4020-9942-7_2

Prajapati, B. P., Kumar Suryawanshi, R., Agrawal, S., Ghosh, M., & Kango, N. (2018). Characterization of cellulase from aspergillus tubingensis NKBP-55 for generation of fermentable sugars from agricultural residues. *Bioresource Technology, 250*, 733–740. https://doi.org/10.1016/j.biortech.2017.11.099

Qdais, A. H., Abdulla, F., & Qrenawi, L. (2010). Solid waste landfills as a source of green energy: Case study of Al Akeeder landfill. *Jordan Journal of Mechanical and Industrial Engineering, 4*(1), 69–74.

Rashad, M. M., Mahmoud, A. E., Ali, M. M., Nooman, M. U., & Al-Kashef, A. S. (2015). Antioxidant and anticancer agents produced from pineapple waste by solid state fermentation. *International Journal of Toxicological and Pharmacological Research, 7*(6), 287–296.

Rico, X., Gullón, B., Alonso, J. L., & Yáñez, R. (2020). Recovery of high value-added compounds from pineapple, melon, watermelon and pumpkin processing by-products: An overview. *Food Research International, 132*(January), Article 109086. https://doi.org/10.1016/j.foodres.2020.109086

Roasa, J., De Villa, R., Mine, Y., & Tsao, R. (2021). Phenolics of cereal, pulse and oilseed processing by-products and potential effects of solid-state fermentation on their bioaccessibility, bioavailability and health benefits: A review. *Trends in Food Science and Technology, 116*(March), 954–974. https://doi.org/10.1016/j.tifs.2021.08.027

Robledo, A., Aguilera-Carbó, A., Rodriguez, R., Martinez, J. L., Garza, Y., & Aguilar, C. N. (2008). Ellagic acid production by aspergillus Niger in solid state fermentation of pomegranate residues. *Journal of Industrial Microbiology and Biotechnology, 35*(6), 507–513. https://doi.org/10.1007/s10295-008-0309-x

Romero, C., Brenes, M., García, P., García, A., & Garrido, A. (2004). Polyphenol changes during fermentation of naturally black olives. *Journal of Agricultural and Food Chemistry, 52*(7), 1973–1979. https://doi.org/10.1021/jf030726p

Saenz-Mendoza, A. I., Zamudio-Flores, P. B., García-Anaya, M. C., Velasco, C. R., Acosta-Muñiz, C. H., de Jesús Ornelas-Paz, J., … Salgado-Delgado, R. (2020). Characterization of insect chitosan films from Tenebrio molitor and Brachystola magna and its comparison with commercial chitosan of different molecular weights. *International Journal of Biological Macromolecules, 160*, 953–963. https://doi.org/10.1016/j.ijbiomac.2020.05.255

dos Santos, A. C., Ximenes, E., Kim, Y., & Ladisch, M. R. (2019). Lignin–Enzyme Interactions in the Hydrolysis of Lignocellulosic Biomass. *Trends in Biotechnology, 37*(5), 518–531. https://doi.org/10.1016/j.tibtech.2018.10.010

Selvam, K., Govarthanan, M., Kamala-Kannan, S., Govindharaju, M., Senthilkumar, B., Selvankumar, T., & Sengottaiyan, A. (2014). Process optimization of cellulase production from alkali-treated coffee pulp and pineapple waste using Acinetobacter sp. TSK-MASC. *RSC Advances, 4*(25), 13045–13051. https://doi.org/10.1039/c4ra00066h

Steingass, C. B., Glock, M. P., Schweiggert, R. M., & Carle, R. (2015). Studies into the phenolic patterns of different tissues of pineapple (Ananas comosus [L.] Merr.) infructescence by HPLC-DAD-ESI-MSn and GC-MS analysis. *Analytical and Bioanalytical Chemistry, 407*(21), 6463–6479. https://doi.org/10.1007/s00216-015-8811-2

Taherzadeh-Ghahfarokhi, M., Panahi, R., & Mokhtarani, B. (2019). Optimizing the combination of conventional carbonaceous additives of culture media to produce lignocellulose-degrading enzymes by Trichoderma reesei in solid state fermentation of agricultural residues. *Renewable Energy, 131*, 946–955. https://doi.org/10.1016/j.renene.2018.07.130

Teles, A. S. C., Chávez, D. W. H., Santiago, M. C. P.d. A., Gottschalk, L. M. F., & Tonon, R. V. (2021). Composition of different media for enzyme production and its effect on the recovery of phenolic compounds from grape pomace. *Biocatalysis and Agricultural Biotechnology, 35*(January). https://doi.org/10.1016/j.bcab.2021.102067

Torres-León, C., Ramírez-Guzmán, N., Ascacio-Valdés, J., Serna-Cock, L., dos Santos Correia, M. T., Contreras-Esquivel, J. C., & Aguilar, C. N. (2019). Solid-state fermentation with aspergillus Niger to enhance the phenolic contents and antioxidative activity of Mexican mango seed: A promising source of natural antioxidants. *Lwt, 112*(December 2018), Article 108236. https://doi.org/10.1016/j.lwt.2019.06.003

Vega-Castro, O., Contreras-Calderon, J., León, E., Segura, A., Arias, M., Pérez, L., & Sobral, P. J. A. (2016). Characterization of a polyhydroxyalkanoate obtained from pineapple peel waste using Ralsthonia eutropha. *Journal of Biotechnology, 231*, 232–238. https://doi.org/10.1016/j.jbiotec.2016.06.018

Xue, P., Liao, W., Chen, Y., Xie, J., Chang, X., & Peng, G. (2022). Release characteristic and mechanism of bound polyphenols from insoluble dietary fiber of navel orange peel via mixed solid-state fermentation with Trichoderma reesei and aspergillus Niger. *LWT - Food Science and Technology, 113387*. https://doi.org/10.1016/j.lwt.2022.113387

Ye, Y., Chen, Y., Hou, Y., Yu, H., Zhu, L., Sun, Y., … Dong, M. (2021). Two new benzoic acid derivatives from endophytic fungus aspergillus versicolor. *Natural Product Research, 36*(1), 223–228. https://doi.org/10.1080/14786419.2020.1777121

Yeo, J. D., Tsao, R., Sun, Y., & Shahidi, F. (2021). Liberation of insoluble-bound phenolics from lentil hull matrices as affected by Rhizopus oryzae fermentation: Alteration in phenolic profiles and their inhibitory capacities against low-density lipoprotein (LDL) and DNA oxidation. *Food Chemistry, 363*(January), Article 130275. https://doi.org/10.1016/j.foodchem.2021.130275

Yepes-Betancur, D. P., Márquez-Cardozo, C. J., Cadena-Chamorro, E. M., Martinez-Saldarriaga, J., Torres-León, C., Ascacio-Valdes, A., & Aguilar, C. N. (2021). Solid-state fermentation – Assisted extraction of bioactive compounds from hass avocado seeds. *Food and Bioproducts Processing, 126*, 155–163. https://doi.org/10.1016/j.fbp.2020.10.012

Zeng, R., Yin, X. Y., Ruan, T., Hu, Q., Hou, Y. L., Zuo, Z. Y., … Yang, Z. H. (2016). A novel cellulase produced by a newly isolated trichoderma virens. *Bioengineering, 3*(2), 1–9. https://doi.org/10.3390/bioengineering3020013