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Fourier Transform Infrared (FT-IR) Spectroscopy as a Possible Rapid Tool to Evaluate Abiotic Stress Effects on Pineapple By-Products

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Abstract: Fourier transform infrared (FT-IR) spectroscopy is a physicochemical technique based on the vibrations of a molecule energized by infrared radiation at a specific wavelength range. Abiotic stresses can induce the production of secondary metabolites, increasing bioactivity. The objectives of the study were to evaluate the impact of heat treatments on the bioactivity of pineapple by-products, and whether FT-IR analysis allows understanding of the changes imparted by abiotic stress. The by-products were treated at 30, 40, and 50 °C for 15 min, followed by storage at 5 ± 1 °C for 8 and 24 h. Lyophilized samples were characterized for total phenolic content and antioxidant capacity and analyzed by FT-IR. Thermal treatments at 50 °C reduced the content of phenolic compounds (21–24%) and antioxidant capacity (20–55%). Longer storage time (24 h) was advantageous for the shell samples, although this effect was not demonstrated for the core samples. The principal components analysis (PCA) model developed with the spectra of the pineapple shell samples showed that the samples were grouped according to their total phenolic compounds content. These results allow the conclusion to be drawn that FT-IR spectroscopy is a promising alternative to the conventional chemical analytical methodologies for phenolic and antioxidant contents if there are significant differences among samples.

Keywords: FT-IR; pineapple by-products; postharvest abiotic stress; bioactive compounds

1. Introduction

Infrared spectroscopy is one of the most important analytical techniques in existence, able to analyze practically any sample in almost any state. Fourier transform infrared (FT-IR) spectroscopy is an instrumental method based on measurement of the vibration of a molecule excited by infrared radiation at a specific wavenumber range. Infrared spectroscopy has advantages over some conventional techniques. This technique is non-destructive, fast and simple to use, precise, mechanically simple, and can be used for routine quantitative and qualitative analysis. The spectroscopic techniques are inexpensive and they do not require time-consuming sample pre-treatment or the use of (environmentally harmful) chemical extracts [1,2]. In the near infrared and mid-infrared regions, an element normally absorbs at more than one wavelength, and the absorbance at a presented wavelength

may be influenced by more than one analyte in chemically complicated matrices [3]. Therefore, these spectroscopic analytical techniques are widely used in association with multivariate data analysis for the qualitative and quantitative analysis of complex matrices. Qualitative assessment of the spectra can be accomplished by principal components analysis (PCA), while partial least squares (PLS) regression permits the enhancement of calibration models between spectral and analytical data.

Fresh-cut fruit consumption is increasing because of the rising awareness of its benefits [4]. The fresh-cut fruit industries generate large amounts of by-products that are traditionally undervalued, and the removal of these materials is a problem exacerbated by legal restrictions [5]. The by-products normally correspond to more than 25–35% of the fruits. The shell is the predominant by-product and represents around 10% of the weight of the fruit [5,6]. Pineapple wastes have potential as raw materials or, after biological treatment, as a high-added-value product to be used as an ingredient in the food or feed industries [7,8]. It has been reported that pineapple by-products are an abundant source of antioxidant polyphenols, which could be recovered for food or cosmetic applications with advantages in terms of economic and environmental sustainability [9–11]. As these by-products are living tissues, still physiologically active and capable of synthetizing phenolic and carotenoid compounds, they can be used as biofactories of secondary metabolite compounds with pharmaceutical and nutraceutical applications.

Postharvest abiotic stresses can change the secondary metabolite synthesis in crop tissues, participating in the biosynthesis of the three main groups of secondary metabolites: terpenes, phenolics, and nitrogen-containing compounds [12]. Numerous biochemical pathways are susceptible to being implicated in responses to abiotic stresses, like the accumulation of phenolic compounds caused by a change in phenylalanine ammonia lyase activity (PAL) [13]. Increased PAL activity promotes the accumulation of different phenolic compounds (chlorogenic acid, dicaffeoyl tartaric acid, and isochlorogenic acid) and tissue browning [14]. The wound-induced accumulation of phenolic compounds can be influenced by the submission to an extra abiotic stress in the wounded tissue, involving changes in the total quantity and type of phenolic compounds amassed [15].

Bioactive compound synthesis can be increased by abiotic stresses like heat treatment, UV-C radiation, and wounding, increasing phenolic and carotenoid contents [16,17]. Heat treatments, in addition to controlling postharvest deterioration and enhancing the storage quality of fruits, have been investigated as abiotic stresses able to promote bioactive synthesis by living plant tissues. For instance, by-products of the minimally processed fruits industry are used to produce natural additives that can be used in the dietary supplements industry, enhancing the extractable yields of particular active compounds that have nutraceutical or additional functional properties [12].

As mentioned above, FT-IR has several advantages when compared to conventional chemical analytical methods. Due to these advantages, FT-IR has been used in the determination and quantification of functional compounds in fruit and vegetables [18,19], and also as a routine analysis of antioxidants in foods, plants, and agricultural products. In fact, Park and co-workers have determined the bioactive compounds that can be used as indicators of the quality of kiwifruits [20]. The total phenolic content and the antioxidant capacity has also been determined for garlics and elephant garlic [21], avocado, durian, and mango [22], and blueberry, grape, and blackberry [23] by chemical methods and using FT-IR spectroscopy. These studies showed that FT-IR is an appropriate analytical method for speedily measuring fruit extract antioxidant capacity.

The present work aimed to evaluate the use of mid-infrared spectroscopy to study the impact of abiotic stresses, wounding (cutting or slicing), and thermal treatments on the amounts of bioactive compounds in by-products of the pineapple processing industry.

2. Materials and Methods

2.1. Materials

Pineapple (*Ananas comosus* L.) by-products (core and shell) (Figure 1) were provided by the industrial partner Campotec, located in Torres Vedras, in the central west of Portugal.

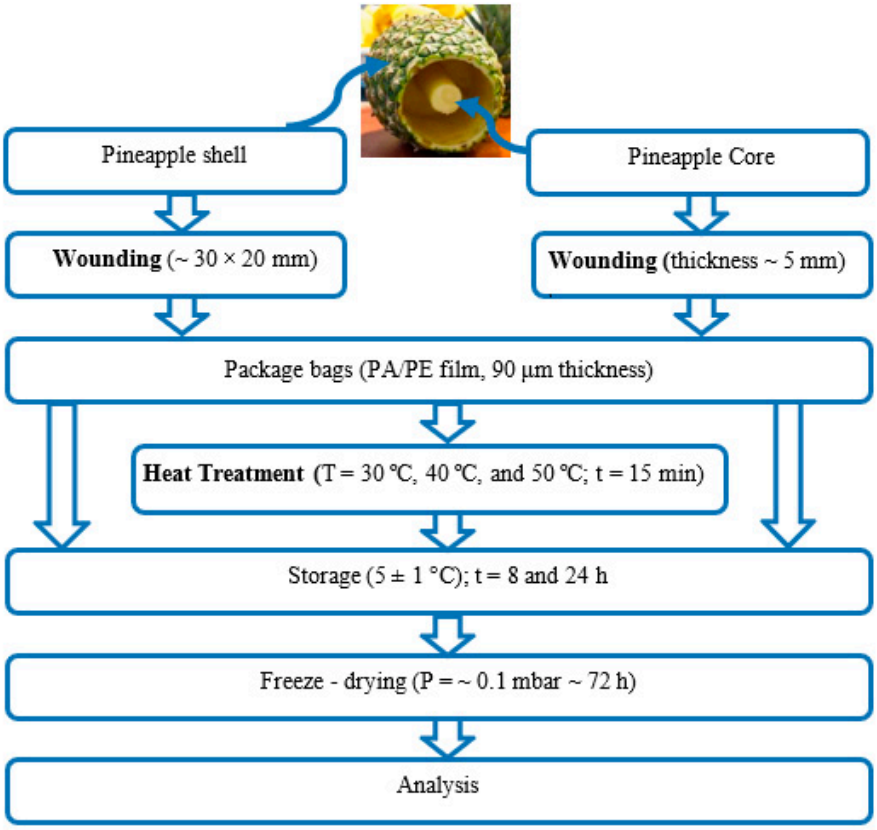


Figure 1. Schematic representation of a longitudinal section of a pineapple fruit and flow diagram of the abiotic stress treatments (wounding and thermal treatment).

2.2. Abiotic Stresses

Wounding: The core cylinders (~25 mm) were sliced (~5 mm thickness) and the shell was cut into rectangles (~30 × 20 mm) and packaged in polyamide/polyethylene (PA/PE-90, Alempack, Elvas, Portugal) bags with 90 µm film thickness (~120 × 200 mm). Samples were sealed under vacuum (Sealer ECOVAC, Model ECOVAC 40, Italy).

Thermal treatments: The bags with wounded by-products were submerged in a water bath (Selecta, Spain) at temperatures of 30, 40, and 50 °C for 15 min and stored at 5 ± 1 °C for 8 and 24 h: T30_8, T30_24, T40_8, T40_24, T50_8, T50_24.

The control samples were maintained in the same conditions, but the heat treatment was not applied: Ctr_0, Ctr_8, and Ctr_24.

After storage time, all samples were frozen at −80 °C and lyophilized (Telstar Lyo Quest, Telstar, Spain). Treatments were performed in duplicate in two independent composite samples.

2.3. Analytical Methods

2.3.1. Total Phenolic Content and Antioxidant Capacity

The extract preparation involved making a ratio of 1:10 (m:v) of sample and methanol (Sigma–Aldrich, Germany), followed by Ultra-Turrax homogenizer (IKA LABORTECHNIK T25 basic, Janke & Kunkel

GmbH &Co., Germany) at 8000 rpm for 2 min and incubation overnight at 4 °C. The extracts were obtained by centrifugation (HERMLE Z383K LABORTECHNIK, Germany) at 8000 rpm for 20 min (4 °C), and the supernatants were stored at 4 °C protected from light until needed for analysis.

The total phenolic content (TPC) was determined according to the method of Swain and Hills (1959) [24] and Heredia and Cisneros-Zevallos (2009) [25], with some alterations. Aliquots of the extract supernatant after centrifugation (150 µL) were diluted with 2400 µL nanopure water, followed by the addition of 150 µL of 0.25 M Folin–Ciocalteu (Panreac AppliChem, Germany), and the sample was incubated for 3 min at room temperature. The reaction was interrupted by adding 300 µL of 1M Na_2CO_3 (Panreac AppliChem, Germany) and the mixture was incubated for a further 2 h, protected from light. Afterwards, the absorbance of the solution was measured at 725 nm. The total phenolic content was defined using a standard curve developed with equivalent gallic acid (GAE) and expressed as mg GAE.g^{-1} dry weight. The average of three replicates was used for each condition.

The antioxidant capacity was evaluated by DPPH (2,2-diphenyl-1-picrylhydrazyl) method following the procedure of Brand-Williams et al. (1995) [26], with some modifications. In this case, the DPPH solution was formulated with methanol until it achieved 1.1 units of absorbance at 515 nm. The sample extracts were prepared as described above. Sample aliquots of 100 µL were taken from the supernatants and then added to 3900 µL DPPH solution. This mixture was homogenized, and the reaction took place for 40 min in the dark. The sample absorption was read at 515 nm. The blank was prepared with methanol and used as control and to calibrate the spectrophotometer for readings. The antioxidant capacity was determined using a standard curve developed with Trolox, and the results are expressed as Trolox equivalent antioxidant capacity (TEAC; µmol Trolox.g^{-1} dry weight). The average of three replicate samples was used for each condition.

Ferric-reducing antioxidant power (FRAP) was performed according to Benzie and Strain (1996) [27], with some modifications. Solutions of 300 mM acetate buffer (3.1 g sodium acetate ($C_2H_3NaO_2 \cdot 3H_2O$; Panreac AppliChem, Germany) and 16 mL acetic acid glacial ($C_2H_4O_2$; FisherChemical (United Kingdom)), pH 3.6, 10 mM TPTZ (2,4,6-tripyridyl-s-triazine; Sigma–Aldrich, Germany) in 40 mM hydrochloric acid (HCl; Sigma–Aldrich, Germany), and 20 mM iron (III) chloride ($FeCl_3$; Sigma–Aldrich, Germany) were prepared. The working solution was made by combining 35 mL acetate buffer 300 mM, 3.5 mL TPTZ solution, and 3.5 mL $FeCl_3$ solution. The procedure involved mixing 2.7 mL of the FRAP solution with 270 µL H_2O and 90 µL of the extract samples, which had been warmed in a water bath at 37 °C for 30 min. The coloured result (ferrous tripyridyltriazine complex) was then read at 595 nm using water as the blank. The antioxidant capacity was determined using a standard curve established with Trolox, and the results are expressed as Trolox equivalent antioxidant capacity (TEAC; µmol Trolox.g^{-1} dry weight). The average of three replicates was used for each condition.

The antioxidant capacity was also measured using the ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid)) method as described by Re et al. (1999) and Rufino et al. (2007) [28,29], with some modifications. Two stock solutions were prepared: ABTS solution (7 mM) and potassium persulfate solution (140 mM). The working solution was prepared by mixing 2 mL of ABTS solution with 35.2 µL of the potassium persulfate solution and keeping it in the dark at room temperature for 12–16 h. The ABTS solution was then diluted with methanol to reach an absorbance of 0.700 at 734 nm. The reaction was performed by mixing 2970 µL ABTS solution with 30 µL sample aliquots for 6 min and the absorbance at 734 nm was immeditately recorded. The absorbance of the reaction samples was related to the Trolox standard and the results are expressed in terms of Trolox equivalent antioxidant capacity (TEAC; µmol Trolox.g^{-1} dry weight). The average of three replicate samples was used for each condition.

2.3.2. Fourier Transform Infrared Spectroscopy

A FT-IR spectrometer Spectrum Two (Perkin-Elmer, USA) with a diamond ATR (attenuated total reflection) single reflection accessory was used. PerkinElmer Spectrum software was used to draw the spectra. The spectra (32 scans per spectrum) of the lyophilized pineapple by-products were collected in duplicate in the mid-infrared wavenumber range from 4000 to 400 cm^{-1}, with a spectral resolution of 4 cm^{-1}.

2.3.3. Statistical Analysis

The results obtained in the analytical assays were subjected to statistical analysis using Statistica™ v.8 Software (StatSoft Inc., USA). Statistically significant differences ($p < 0.05$) between samples were defined using Tukey's honestly significant difference test.

The results were also submitted to principal component analysis (PCA) using Statistica™ v.8 Software. In this case, all variables were mean-centered and standardized (scaled) to unit variance prior to analysis [30].

Matlab version 7.9 (MathWorks, Natick, MA, USA) and the PLS Toolbox version 4.0 (Eigenvector Research Inc.—USA) for Matlab were utilized to perform the qualitative analysis of the spectra using PCA, according to the description presented elsewhere [3,31]. To derive the PCA models, only the region of 600–4000 cm^{-1} was used, in order to exclude noise and the non-informative range of the spectra. To enhance the correlations concerning radiation absorption and the properties of each sample, different preprocessing methods were evaluated. The choice was made by analyzing the ones that allowed the highest data variance with the lowest number of PCs to be captured with the smaller errors, using cross-validation and leave-one-out method as the internal validation strategy. In this work, baseline correction followed by mean-centering were always used as spectrum pre-processing methods.

3. Results and Discussion

3.1. Total Phenolic Content and Antioxidant Capacity

3.1.1. In Pineapple Shell and Core

As shown in Table 1, pineapple shell samples (65.75 mg GAE.g^{-1} dry weight) presented significantly higher levels ($p < 0.05$) of phenolic compounds than core (59.89 mg GAE.g^{-1} dry weight). Antioxidant capacity, determined by the three tested methods (DPPH, FRAP, and ABTS) was also higher in the shell samples.

Table 1. Total phenolic content (TPC) and antioxidant capacity of the wounded shell and core pineapple samples submitted to thermal treatments evaluated by three different methods (DPPH—2,2-diphenyl-1-picrylhydrazyl; FRAP—ferric-reducing antioxidant power; ABTS—2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid).

Raw Material	Sample	TPC (mgGAE.g^{-1} dry weight)	Antioxidant Capacity (μmol Trolox.g^{-1} dry weight)		
			DPPH	FRAP	ABTS
Shell pineapple	Ctr_0	65.75 ± 1.40 ^{b}	21.56 ± 0.78 ^{ac}	41.37 ± 1.73 ^{ac}	26.10 ± 1.54 ^{ad}
	Ctr_8	79.72 ± 2.87 ^{e}	33.59 ± 3.81 ^{ef}	52.72 ± 3.05 ^{de}	33.54 ± 2.27 ^{ce}
	Ctr_24	84.83 ± 2.01 ^{f}	38.70 ± 3.38 ^{f}	63.71 ± 2.96 ^{f}	38.64 ± 2.24 ^{f}
	T30_8	69.08 ± 1.70 ^{bc}	26.08 ± 1.25 ^{ad}	46.45 ± 1.55 ^{b}	30.99 ± 1.65 ^{bc}
	T40_8	75.76 ± 1.75 ^{d}	25.02 ± 3.47 ^{a}	48.13 ± 3.05 ^{bd}	29.87 ± 2.54 ^{bc}
	T50_8	57.05 ± 2.39 ^{a}	16.85 ± 1.33 ^{bc}	38.15 ± 2.03 ^{a}	24.06 ± 1.12 ^{a}
	T30_24	78.80 ± 2.28 ^{de}	31.22 ± 3.81 ^{de}	55.26 ± 3.51 ^{e}	34.72 ± 2.56 ^{e}
	T40_24	70.28 ± 1.89 ^{c}	22.75 ± 1.98 ^{a}	45.56 ± 1.89 ^{bc}	28.25 ± 1.73 ^{bd}
	T50_24	57.01 ± 1.57 ^{a}	14.91 ± 2.78 ^{b}	39.65 ± 1.54 ^{a}	24.25 ± 1.58 ^{a}
Core pineapple	Ctr_0	59.89 ± 1.86 ^{D}	24.06 ± 1.24 ^{A}	31.18 ± 1.98 ^{C}	22.34 ± 1.19 ^{E}
	Ctr_8	67.14 ± 2.58 ^{A}	25.01 ± 1.48 ^{AB}	38.06 ± 1.17 ^{B}	26.64 ± 0.66 ^{ABC}
	Ctr_24	65.07 ± 1.83 ^{AB}	22.84 ± 1.07 ^{AD}	38.03 ± 1.12 ^{B}	25.67 ± 0.96 ^{ABD}
	T30_8	70.92 ± 2.09 ^{C}	28.60 ± 1.20 ^{C}	42.62 ± 1.90 ^{A}	31.94 ± 1.78 ^{F}
	T40_8	73.49 ± 1.91 ^{C}	26.99 ± 1.60 ^{BC}	45.85 ± 1.63 ^{D}	27.05 ± 1.11 ^{ABC}
	T50_8	71.94 ± 1.62 ^{C}	24.50 ± 0.99 ^{AB}	41.14 ± 1.54 ^{A}	25.17 ± 1.28 ^{AD}
	T30_24	65.05 ± 1.37 ^{AB}	23.87 ± 1.38 ^{A}	41.10 ± 1.08 ^{A}	23.68 ± 0.84 ^{DE}
	T40_24	62.01 ± 1.12 ^{BD}	29.48 ± 1.78 ^{C}	40.03 ± 1.03 ^{AB}	28.15 ± 1.09 ^{C}
	T50_24	65.99 ± 1.72 ^{A}	20.52 ± 1.98 ^{D}	41.42 ± 1.03 ^{A}	27.81 ± 1.06 ^{BC}

Legend: **Ctr_Y**—Control sample; **TX_Y**—Heat treated sample (X = treatment temperature: 30, 40, or 50 °C); **Y**—storage time at 4 °C after heat treatment (Y = 8 or 24 h). Different letters represent statistically significant differences in the same column (comparison of different samples for the same analytical method; Tukey's test $p < 0.05$). Small letters compare shell pineapple samples and capital letters compare core pineapple samples.

3.1.2. Wounding Effect in Pineapple Shell and Core

As the main objective of the current work was to evaluate the use of mid-infrared spectroscopy to study the impact of abiotic stresses on the amount of bioactive compounds, wounded samples without thermal treatment, control samples, were analyzed for total phenolic compounds and antioxidant capacity using conventional methods. As shown in Table 1, the control samples after 8h (Ctr_8) and 24 h (Ctr_24) of storage time presented significantly higher ($p < 0.05$) levels of phenolic compounds than the initial control samples (Ctr_0). This indicates that storage time after cutting improves the synthesis of phenolic compounds. The same behavior was observed for antioxidant capacity, determined by the three tested methods (DPPH, FRAP, and ABTS). The enhancement of bioactive compounds in fresh and treated fruits by applying abiotic stresses (wounding, heat shock, UV irradiation, among others) has been reported previously by Heredia and Cisneros-Zevallos (2009). The wound-induced increase of phenolic compounds has been suggested to be due to phenylpropanoid pathway activation [13].

3.1.3. Thermal Treatment Effect in Pineapple Shell and Core

As shown in Table 1, thermal treatment for 15 min affected the total phenol content and antioxidant capacity of the samples. The samples heat-treated at 30 or 40 °C for 15 min (T30_15 and T40_15) showed significantly higher ($p < 0.05$) phenolic content and antioxidant capacity than control samples. The thermal treatment at 40 °C during 15 min (T40_15) seemed to be the treatment that promoted the highest synthesis of phenolic compounds. Results are not consistent as to which storage time after thermal treatment, 8 or 24 h, was more effective. The storage time studied was short, to verify more evident differences.

On the other hand, the thermal treatments at 50 °C (T50) showed lower phenolic compounds and antioxidant capacity for all storage times tested. This results indicates that the temperature was excessive and decreased phenylalanine-ammonia lyase (PAL) activity, which is in accordance with other authors [32].

3.2. Fourier Transform Infrared Spectroscopy

Responding to the main objective of the research, to evaluate the use of mid-infrared spectroscopy to study the impact of abiotic stresses, samples were also analyzed using FT-IR.

The spectra of Ctr_8 and Ctr_24 are shown in Figure 2. The band presented at 3600–3000 cm^{-1}, with a maximum value close to 3300 cm^{-1} was associated with the stretching vibration of O–H groups (3600–3200 cm^{-1}), at 3400–3300 cm^{-1} with N–H stretching, and at 3100–3000 cm^{-1} with =C–H stretching [33,34]. The peak at 1719 cm^{-1} was associated with carbonyl group C=O stretching. Since the samples were lyophilized, the O–H stretching band should have been mainly associated with compounds such as carboxylic acids, alcohols, and phenols [34]. In fact, according to Table 1, the TPC values of the shell control samples, which had more intense bands in this region, were higher (6.4%). On the other hand, the band 3000–2800 cm^{-1}, with a maximum near 2930 cm^{-1}, corresponded to the C–H stretching of CH_2 groups. This band is usually associated with carbohydrates and fats [35]. The bands in the 1800–1500 cm^{-1} region corresponded to C=C and C=O stretching. In the pineapple sample, these bonds may have been related to proteins, amide I (1700–1600 cm^{-1}) and amide II (1565–1520 cm^{-1}), and fats (1745–1725 cm^{-1}) [34].

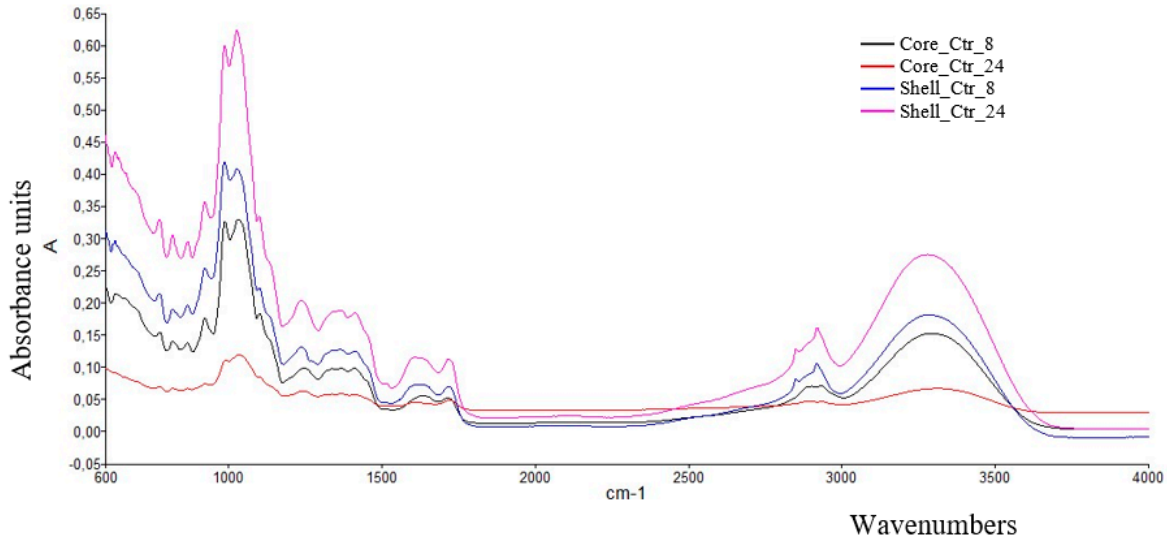
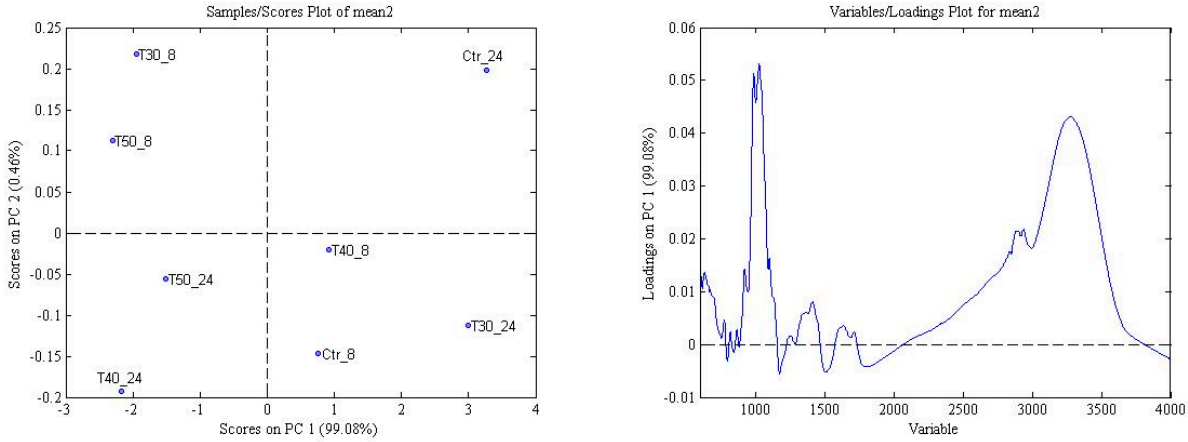


Figure 2. As-collected FT-IR spectra of the control samples of the wounded pineapple shell and core. Ctr_8: control stored at 5 ± 1 °C for 8 h; Ctr_24: control stored at 5 ± 1 °C for 24 h.

Several marker bands were identified in the spectral range of 1400–800 cm^{-1}. These bands may have been associated with the stretching and bending of carbohydrates. The bands in the region of 1150–900 cm^{-1} were assigned to C–O and C–C stretch modes, while those in the 1400–1200 cm^{-1} region were due to O–C–H, C–C–H, and C–O–H bending vibrational modes of the carbohydrates [36]. In addition to sugars, bands characteristic of organic acids (malic, citric, and ascorbic acids) might also have been observed in the absorption region 1500–900 cm^{-1} [37]. The bands between 900–600 cm^{-1} corresponded to a fingerprint region with vibrational C–H or CH_2 group deformation. The absorption region at 750–500 cm^{-1} may have been associated with =C–H bending or C–C bending vibrations that occur at low frequencies (below 500 cm^{-1}). The absorption region at 700–720 cm^{-1} was related to colour pigments (β-carotene) [34,38].

The score plots of the principal component analysis of the PCA model established with all the FT-IR spectra of the shell and core samples are shown in Figures 3 and 4, respectively. In Figure 3b, the loadings plot of the first principal component (PC1) of the shell samples is also presented. As shown, this plot presented the most significant peaks at around 3300 cm^{-1}, which related to the combination bands of O–H bonds, and at 1000 cm^{-1}, which were assigned to the O–C–H, C–C–H, and C–O–H vibrational modes of the carbohydrates. This result confirmed that PC1 captured the change of the TPC of the samples. The loadings plot with respect to core samples was similar to the one obtained for the shell samples, with two important peaks at around 3300 cm^{-1} and 1000 cm^{-1} (data not shown).

As shown in Figures 3 and 4, PC1 captured 99.08% and 98.75% of the spectra variance for shell and core samples, respectively. In shell samples (Figure 3a), it was observed that the model separated the samples with higher contents of phenolic compounds (Ctr_8, Ctr_24, T30_24, and T40_8), from the samples with lower contents (T30_8, T40_24, T50_8, and T50_24). Figure 3 also indicates that the FT-IR allowed detection of the differences between the samples in terms of the TPC and antioxidant capacity, and for results consistent with the conventional analyses to be obtained. The loadings plot of the model PC1, presented in Figure 3b, permitted the identification of the wavenumbers that were the most important to describe PC1. It is worth noting that FT-IR has also been applied to evaluate the quality of kiwi samples, and the results showed relatively high correlation between the values of the antioxidant capacity measured by the conventional methods and FT-IR spectroscopy [20].



(**a**) (**b**)

Figure 3. Principal component analysis score plot for pineapple shell FT-IR spectra of the control (Ctr) and heat-treated (T) wounded pineapple shell samples, stored at 5 ± 1 °C for 8 h (_8) and 24 h (_24). (**a**) PC1 versus scores on PC2; (**b**) loadings plot on PC1. T30_8: heat-treated at 30 °C for 15 min and stored at 5 ± 1 °C for 8 h; T30_24: heat-treated at 30 °C for 15 min and stored at 5 ± 1 °C for 24 h; T40_8: heat-treated at 40 °C for 15 min and stored at 5 ± 1 °C for 8 h; T40_24: heat-treated at 40 °C for 15 min and stored at 5 ± 1 °C for 24 h; T50_8: heat-treated at 50 °C for 15 min and stored at 5 ± 1 °C for 8 h; T50_24: heat-treated at 50 °C for 15 min and stored at 5 ± 1 °C for 24 h.

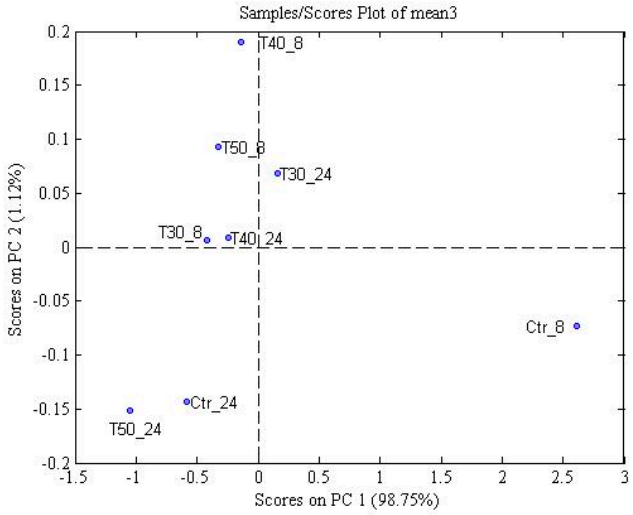


Figure 4. Principal component analysis score plot of pineapple core FT-IR spectra of the control (Ctr) and heat-treated (T) core pineapple wounded samples, stored at 5 ± 1 °C for 8 h (_8) and 24 h (_24). T30_8: heat-treated at 30 °C for 15 min and stored at 5 ± 1 °C for 8 h; T30_24: heat-treated at 30 °C for 15 min and stored at 5 ± 1 °C for 24 h; T40_8: heat-treated at 40 °C for 15 min and stored at 5 ± 1 °C for 8 h; T40_24: heat-treated at 40 °C for 15 min and stored at 5 ± 1 °C for 24 h; T50_8: heat-treated at 50 °C for 15 min and stored at 5 ± 1 °C for 8 h; T50_24: heat-treated at 50 °C dur for ing 15 min and stored at 5 ± 1 °C for 24 h.

On the other hand, as shown in Figure 4, most of the samples presented similar scores on PC1, between −0.5 and 0, whereas only the control sample with 8 h (8) of storage time was separated from the others. This result is a clear indication that the small differences in the phenolic compounds content and antioxidant capacity (Table 1) were not enough to lead to significant changes in the FT-IR spectra. Therefore, in the scores plot of the PCA model illustrated in Figure 4, the samples had similar scores on PC1 and, contrary to what was discussed above for the shell samples, it was not possible to find any distribution pattern.

However, the utilization of FT-IR spectroscopy to establish the polyphenols in durian, mango, and avocado samples has been described in the literature. The authors stated that these analytical methods might be applicable for phytochemical analysis in other samples [22].

4. Conclusions

The present study allowed the conclusion to be drawn that abiotic stresses, wounding, and thermal treatments applied to pineapple by-products (shell and core) samples induced the synthesis of phenolic compounds.

The use of FT-IR analysis based on the content of phenolic compounds and antioxidant capacity was only possible if there are significant differences among samples. It was possible for materials like the pineapple shell samples, which presented high phenol contents and reacted enough to the abiotic stress applied.

Author Contributions: D.I.S. made experimental part, analyzed and discussed the results, and wrote the manuscript. M.J.N.C analyzed data and discussed the results, wrote, editing and review the manuscript. M.M.M. planned and made the experimental part and analyzed the results. J.A.S. review the manuscript. A.A.V. review the manuscript. M.M. planned and organized the work, discussed the results, wrote and review the manuscript.

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