

1 Pineapple by-products as a source of bioactive compounds with
2 potential for industrial food application

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8 Tables and Figures

Tables

Table 1: Retention time (Rt), wavelengths of maximum absorption in the visible region ($\lambda_{\max}$), mass spectral data, and identification and quantification of phenolic compounds in pineapple peel and crown extracts.

Peak	Rt (min)	$\lambda_{\max}$ (nm)	[M-H] ⁻ (m/z)	MS ² (m/z)	Identification	Content (mg/g extract)	
						Peel extract	Crown extract
1	4.48	275	341	179(100)	Caffeic acid- <i>O</i> -hexoside ¹	0.0009±0.0001	nd
2	4.93	275	341	179(100)	Caffeic acid- <i>O</i> -hexoside ¹	0.0034±0.0002	nd
3	5.30	277	341	179(100)	Caffeic acid- <i>O</i> -hexoside ¹	0.0294±0.0002	nd
4	5.90	292	451	253(100), 179(3), 161(20), 135(11)	Dicaffeoylglycerol ¹	nd	0.011±0.001
5	6.79	277	341	179(100)	Caffeic acid- <i>O</i> -hexoside ¹	0.006±0.0002	nd
6	6.86	321	517	473(100), 269(18)	Apigenin-malonyl-hexoside ²	nd	1.1±0.1
7	7.86	326	385	209(8), 191(100), 147(5), 129(11)	Feruloyl aldarate ³	nd	0.035±0.001
8	8.03	332	461	347(100), 299(25)	Diosmetin 8- <i>C</i> -glucoside ⁴	0.1175±0.0002	nd
9	8.45	328	593	473(100)	Apigenin 6,8- <i>C</i> -diglucoside ⁵	0.46±0.01	0.56±0.01
11	9.29	274, 332	597	359(17), 295(20), 197(62), 179(56), 135(95)	Yunnaneic acid F ⁶	0.41±0.005	nd
12	10.15	275, 327	399	253(100), 235(23), 163(17), 145(9)	<i>p</i> -Coumaroyl-caffeoylglycerol ⁷	0.25±0.01	nd
13	10.70	326	385	209(6), 191(100), 147(7), 129(15)	Feruloyl aldarate ³	nd	0.031±0.001
14	12.19	324	593	431(100)	Apigenin 6,8- <i>C</i> -diglucoside ⁵	nd	0.483±0.005
15	13.05	365	771	609(95), 301(32), 179(12)	3,5-di- <i>O</i> -Caffeoyl-4- <i>O</i> -(3-hydroxy, 3-methyl) glutaroylquinic acid ⁷	0.18±0.01	nd
16	13.05	315	757	595(21), 449(46), 287(90)	Eriodictyol-4'- <i>O</i> -neohesperidoside-7- <i>O</i> -glucoside ⁸	nd	0.058±0.003
17	13.29	277, 301	337	191(85), 173(100), 155(4)	<i>p</i> -Coumaroylisocitrate ⁷	0.132±0.005	nd
18	14.06	225, 277	305	261(12), 221(47), 219(56), 179(100), 165(28), 137(30), 125(40)	(Epi)galocatechin ⁹	0.59±0.02	nd
19	14.40	315	537	491(72), 329(100), 209(2), 167(24), 152(4)	Vanilloyl dihexoside ¹⁰	0.118±0.001	nd
20	14.63	322	563	473(100), 443(69), 383(22), 353(28)	Apigenin-6- <i>C</i> -hexoside-8- <i>C</i> -pentoside ⁵	nd	0.462±0.002
21	15.42	225, 274	441	249(100)	N-L- γ -Glutamyl-S-sinapyl-L-cysteine ¹¹	0.17±0.02	nd
22	15.60	327	537	375 (100)	Anaflavoside B ¹²	nd	0.468±0.001
23	16.51	350	609	301(100)	Quercetin-3- <i>O</i> -rutinoside ¹²	nd	0.471±0.001
24	17.09	327	533	311(100)	Apigenin-di- <i>C</i> -pentoside ⁵	nd	0.467±0.002

25	20.55	327	623	315(100)	Isorhametin rhamosyl-hexoside ¹²	nd	0.469±0.001
Σ Phenolic compounds						2.48±0.03	4.7±0.1

Calibration curves used in the quantification: 1. caffeic acid ($y = 388345x + 406369$; $R^2 = 0.994$; LOD = 0.78 µg/mL; LOQ = 1.97 µg/mL); 2. apigenin-7-*O*-glucoside ($y = 10683x - 45794$; $R^2 = 0.999$; LOD = 0.10 µg/mL; LOQ = 0.53 µg/mL); 3. ferulic acid ($y = 633,126x - 185,462$; $R^2 = 0.999$; LOD = 0.20 µg/mL; LOQ = 1.01 µg/mL); 4. taxifolin ($y = 478.06x + 657.33$; $R^2 = 0.999$; LOD = 0.67 µg/mL; LOQ = 2.01 µg/mL); 5. apigenin 6-*C*-glucoside ($y = 107025x - 61531$; $R^2 = 0.9989$; LOD = 0.19 µg/mL; LOQ = 0.63 µg/mL); 6. rosmarinic acid ($y = 191291x - 652903$; $R^2 = 0.999$; LOD = 0.15 µg/mL; LOQ = 0.68 µg/mL); 7. chlorogenic acid ($y = 168823x - 161172$; $R^2 = 0.9999$; LOD = 0.20 µg/mL; LOQ = 0.68 µg/mL); 8. naringenin ($y = 18433x + 78903$; $R^2 = 0.9998$; LOD = 0.20 µg/mL; LOQ = 0.64 µg/mL); 9. epicatechin ($y = 10314x + 147331$; $R^2 = 0.9994$; LOD = 0.15 µg/mL; LOQ = 0.78 µg/mL); 10. vanillic acid ($y = 197,337x + 30036$; $R^2 = 0.999$; LOD = 0.17 µg/mL; LOQ = 1.22 µg/mL); 11. sinapic acid ($y = 197,337x + 30036$; $R^2 = 0.999$; LOD = 0.17 µg/mL; LOQ = 1.22 µg/mL); 12. quercetin-3-*O*-glucoside ($y = 34843x - 160173$; $R^2 = 0.9998$; LOD = 0.21 µg/mL; LOQ = 0.71 µg/mL). nd: not detected.

Table 2: Antioxidant and cytotoxic activities of pineapple peel and leaf crown extracts.

		Peel extract	Crown extract	Positive control
Antioxidant activity (IC ₅₀ , µg/mL)				Trolox
TBARS formation inhibition		4.3±0.1*	6.6±0.3	5.4±0.3
OxHLIA	Δt 60 min	190±7*	395±19	21.8±0.3
	Δt 120 min	333±9*	714±33	43.5±0.8
Cytotoxic activity (GI ₅₀ , µg/mL)				Ellipticine
Tumour cell lines	AGS	>400	>400	0.9±0.1
	CaCo-2	378±7*	>400	0.8±0.1
	MCF-7	322±3*	>400	1.020 ± 0.004
	NCI-H460	>400	>400	1.01±0.01
Non-tumour cell line	VERO	>400	>400	0.6±0.1

GI₅₀ values correspond to the concentration that causes 50% inhibition of cell proliferation; AGS - human gastric adenocarcinoma; CaCo-2 - human colon adenocarcinoma MCF-7 - human breast adenocarcinoma; NCI-H460 - human lung carcinoma. An * in each line corresponds to a significant statistical difference with a *p*-value of 0.05 using a Student's T test.

Table 3: Antimicrobial activity of pineapple peel and crown extracts against foodborne bacterial and fungal strains.

Antibacterial	Peel extract		Crown extract		E211		E224	
	MIC	MBC	MIC	MBC	MIC	MBC	MIC	MBC
<i>S. aureus</i>	1.00	2.00	1.00	1.00	4.00	4.00	1.00	1.00
<i>B. cereus</i>	1.00	2.00	1.00	2.00	0.50	0.50	2.00	4.00
<i>L. monocytogenes</i>	2.00	2.00	2.00	4.00	1.00	2.00	0.50	1.00
<i>E. coli</i>	0.50	1.00	0.50	1.00	1.00	2.00	0.50	1.00
<i>S. Typhimurium</i>	1.00	2.00	1.00	2.00	1.00	2.00	1.00	1.00
<i>E. cloacae</i>	1.00	1.00	1.00	1.00	2.00	4.00	0.50	0.50
Antifungal	MIC	MFC	MIC	MFC	MIC	MFC	MIC	MFC
<i>A. fumigatus</i>	0.50	1.00	0.50	0.50	1.00	2.00	1.00	1.00
<i>A. niger</i>	0.25	0.50	0.25	0.50	1.00	2.00	1.00	1.00
<i>A. versicolor</i>	1.00	2.00	0.50	1.00	2.00	2.00	1.00	1.00
<i>P. funiculosum</i>	0.25	0.50	0.25	0.50	1.00	2.00	0.50	0.50
<i>P. aurantiogriseum</i>	2.00	4.00	1.00	2.00	2.00	4.00	1.00	1.00
<i>T. viride</i>	0.25	0.50	0.25	0.50	1.00	2.00	0.50	0.50

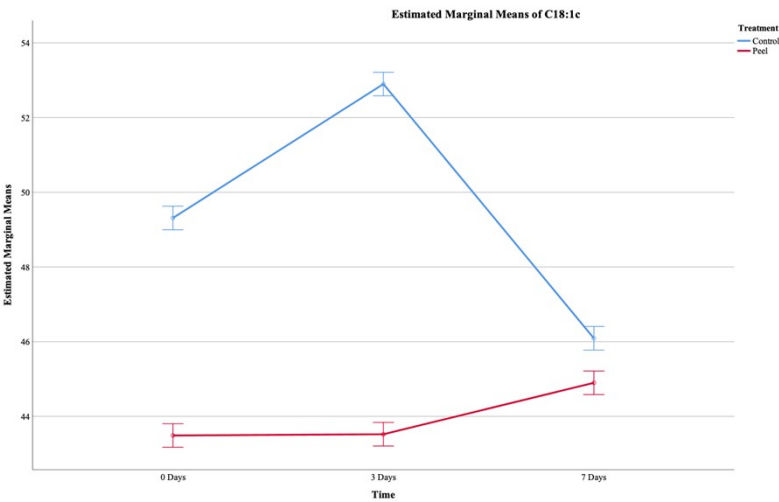
MIC: Minimum inhibitory concentration; MIC: Minimum inhibitory concentration (mg/mL); MBC: Minimum bactericidal concentration (mg/mL); MFC: Minimum fungicidal concentration (mg/mL). Sodium sulphite (E221) and potassium metabisulphite (E224) were used as positive controls.

Table 4: Nutritional composition, colour parameters, and antioxidant activity of control and pineapple peel extract functionalized “súplicas”. The effects of extract incorporation (I) and storage time (ST) on these quality parameters are demonstrated.

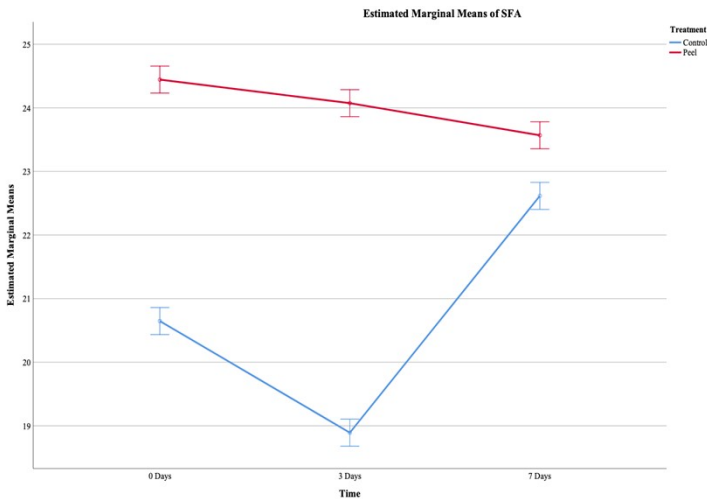
	Incorporation (I)		Student's t-test <i>p</i> -Value (n = 9)	Storage time (ST)			Tukey's test <i>p</i> -Value (n = 18)	ST × I <i>p</i> -Value (n = 27)
	Control	Functionalized		0 Days	3 Days	7 Days		
Moisture (g/100 g fw)	6.5±0.2*	7.4±0.2	<0.001	7.0±0.2	6.9±0.5	7.0±0.7	0.089	0.154
Fat (g/100 g fw)	4.13±0.06	3.56±0.04	<0.001	3.8±0.3	3.9±0.3	3.8±0.3	0.004	0.029
Proteins (g/100 g fw)	7.80±0.07*	7.90±0.01	0.005	7.92±0.07	8.0±0.1	7.92±0.07	0.050	0.152
Ash (g/100 g fw)	0.34±0.01	0.36±0.01	<0.001	0.37±0.01	0.35±0.01	0.35±0.01	<0.001	<0.001
Carbohydrates (g/100 g fw)	80.9±0.2	80.7±0.2	<0.001	80.8±0.1	80.8±0.3	80.8±0.4	0.897	<0.001
Energy (kcal)	393±1	386±1	<0.001	389±2	390±3	390±4	<0.001	<0.001
Energy (kJ)	1645±4	1618±4	<0.001	1630±9	1633±14	1631±19	<0.001	<0.001
Sucrose (g/100 g fw)	86±13	85±3	0.708	91±2 ^b	82±3 ^a	82±3 ^a	0.003	0.102
C16:0 (relative %)	20±2	23.6±0.4	<0.001	22±2	21±3	22.7±0.6	<0.001	<0.001
C16:1 (relative %)	2.5±0.2	2.96±0.08	<0.001	2.7±0.3	2.6±0.3	2.8±0.2	<0.001	<0.001
C18:1t (relative %)	5.8±0.3	6.3±0.1	<0.001	6.0±0.2	5.9±0.6	6.22±0.08	<0.001	<0.001
C18:1c (relative %)	49±3	44.0±0.7	<0.001	46±3	48±4	45.5±0.9	<0.001	<0.001
C18:2 (relative %)	19.3±0.5	20.0±0.2	<0.001	19.8±0.4	19.4±0.8	19.8±0.2	<0.001	<0.001
SFA (relative %)	21±2	24.0±0.4	<0.001	22±2	21±3	23.1±0.7	<0.001	<0.001
MUFA (relative %)	58±2	53.5±0.7	<0.001	55±2	57±4	54.8±0.7	<0.001	<0.001
PUFA (relative %)	21.3±0.8	22.4±0.3	<0.001	22.0±0.6	21±1	22.1±0.2	<0.001	<0.001
<i>L</i> * (lightness)	80.4±0.5	80.9±0.6	<0.001	80.9±0.6	80.2±0.6	80.7±0.3	<0.001	0.009
<i>a</i> * (redness)	1.4±0.2	1.5±0.2	0.196	1.4±0.1	1.4±0.1	1.6±0.2	<0.001	0.040
<i>b</i> * (yellowness)	32±1	31.7±0.5	<0.001	32.5±0.9	32.4±0.7	31.0±0.5	<0.001	<0.001
DPPH scavenging (EC ₅₀ , mg/mL)	>400*	134±53	<0.001	237±173	278±125	292±111	<0.001	0.101
Reducing power (EC ₅₀ , mg/mL)	17±2	20±4	<0.001	17±2	19±4	20±4	<0.001	<0.001

In each row, for I and ST, an asterisk (*) or different letters mean significant statistical differences (*p*-value < 0.05) between samples, respectively. The presented standard deviations were calculated from results obtained under different operational conditions. Therefore, these values should not be regarded as a measure of precision, rather as the range of the recorded values.

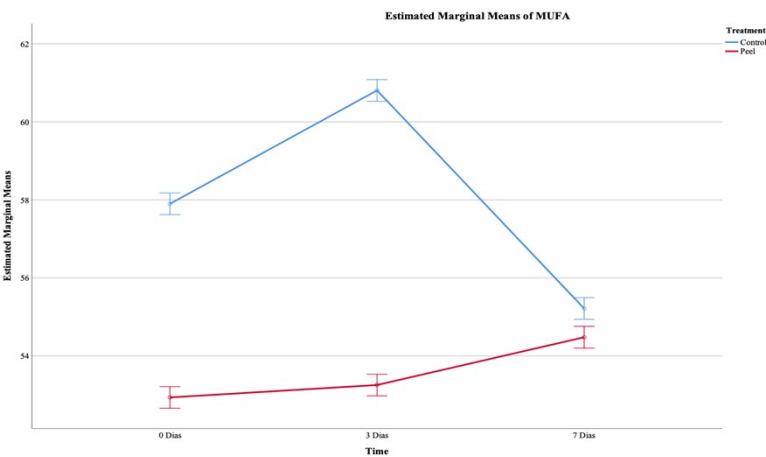
Figure



a)



b)



c)

Figure 1: EMM plots of a) C18:1, b) SFA and c) MUFA.