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Integration of Annurca apple peels into wheat crackers through the co-formulation with lemon fibres: focus on physical quality and phenolic recovery

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ABSTRACT

Apple peels are an underutilized by-product and a rich source of dietary fibres and phenolic compounds, thus enhancing as a dried ingredient the nutritional profile of baked goods. However, phenolic compounds, on baking, can degrade and lose their potential health benefits. Remarkably, the interaction between dietary fibres and phenolic compounds is well documented. In this sense, the binary combination within bakery formulation of apple peels and different sources of dietary fibres could represent an effective strategy to mitigate polyphenol thermal degradation.

The present research investigates how co-incorporating Annurca peel powder (APP) and different lemon polysaccharides (as a source of soluble and insoluble fibres) may affect the nutritional and physical properties of low-moisture bakery products, such as crackers. It examines how lemon polysaccharides influence the recovery of apple peel polyphenols during baking, potentially improving their retention in crackers. APP inclusion did not affect the texture of the crackers, whereas insoluble lemon fibres increased the breaking force as opposed to pectin. A non-targeted analysis of the phenolic profile of APP via UHPLC-ESI-QTOF-MS/MS revealed 59 compounds, with flavonols being the most abundant. Baking led to anthocyanin loss but increased the recovery of hydroxybenzoic acids. Notably, co-formulating with lemon polysaccharides (both soluble and insoluble) helped preserve flavonols. Dihydrochalcone, hydroxycinnamic acids, and flavan-3-ols recovery improved with pectin addition, while proanthocyanidin content decreased with either lemon insoluble polysaccharides or pectin addition. This study demonstrates that it is possible to achieve greater stability of apple phenolic compounds in low-moisture baked goods through co-formulation with lemon polysaccharides.

1. Introduction

Apples are among the most consumed fresh fruits, ranking 4th in the world after oranges, bananas, and grapes (Lyu et al., 2020). In the manufacture of some products, such as applesauce and canned apples, apples are peeled, and the apple peels become a by-product (Wolfe & Liu, 2003). The most common disposal methods for apple by-products, including peels, are to discard them directly into the soil in a landfill, incinerating or composting them, and formulating low-quality animal feedings. However, the dumping of these by-products can have several adverse impacts: greenhouse gas emissions, unpleasant odors due to uncontrolled microbial fermentation, and land spreading due to contaminated groundwater (Dhillon et al., 2013). In addition, land spreading can create breeding grounds for many human disease vectors, which can cause epidemics, and industries incur losses due to waste treatment and transportation costs for dumping into landfills, which is not cost-effective (Dhillon et al., 2013).

Most of the documented beneficial effects of apples are attributed to their dietary fibre and phenolic fractions (Patocka et al., 2020; Wojdyło et al., 2008; Łata et al., 2009). As confirmed by pre-clinical studies,

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apple polyphenols exert antioxidant, anti-inflammatory, and lipid-lowering effects. These documented health-promoting properties are ascribed to five main classes, namely, flavanols, flavonols, phenolic acids, dihydrochalcones and anthocyanins (Patocka et al., 2020). Isolated apple polyphenols have been proven to be effective in preventing/treating hypercholesterolemia (Aprikian et al., 2002; Sekhon-Loodu et al., 2014), Atherosclerosis in ApoE-deficient mice (Soleti et al., 2020), colorectal cancer (Marzo et al., 2021), non-alcoholic hepatitis (Skinner et al., 2019), ulcerative colitis (Yeganeh et al., 2018) and indomethacin-induced gastric damage (Lee et al., 2017). More recently, Cano-Lou et al. (2025) reported how polyphenols extracted from the peels of 6 different apple cultivars showed antidiabetic and antioxidant properties (Cano-Lou et al., 2025). Moreover, dietary fibre consumption improves the bowel transit of feces and the feeling of satiety, reduces blood cholesterol levels, and prevents obesity (Deng et al., 2011; Llobera & Cañellas, 2007; Tseng & Zhao, 2013; Zhang et al., 2018).

Given the potential of apple by-products as a source of dietary fibre and different polyphenol subclasses, their incorporation into baked goods has been studied during the past decades (Kruczek et al., 2023; Kırbaş et al., 2019; Lyu et al., 2020; Mir et al., 2017; Wolfe & Liu, 2003). However, the successful incorporation of apple by-products, such as fruit pomaces, into food products presents a major technological challenge in maintaining consumer acceptability. Like other insoluble fibres, fruit pomaces have a generally high-water absorption capacity, leading to competition with gluten and starch for available moisture (Bianchi et al., 2021; Franco et al., 2026; Rainero et al., 2021; Rodríguez et al., 2024; Tolve et al., 2021). Notably, the negative water dynamics imparted by apple pomace in some products, such as bread, can be counteracted by the co-inclusion of exogenous soluble polysaccharides like psyllium or pectin (Franco et al., 2026). However, there are no studies exploring the integration of apple peel by-product into baked goods, nor the effect of apple peel inclusion into low-moisture baked systems such as crackers. At the same time, polyphenols when subjected to high temperatures, including baking, can degrade and thus lose their potential bioactivity (Kruczek et al., 2023; Rupasinghe et al., 2008). Notably, Franco et al. (2026) observed that the co-formulation of pomace-containing breads with soluble polysaccharides prevented the loss of dihydrochalcones and flavanols during the bread-making process. However, a comprehensive omics-based approach providing insights into apple phenolics' stability and how the co-integration of lemon polysaccharides affects polyphenol recovery during the manufacturing of low-moisture bakery products is missing.

The present study aimed to investigate the chemical stability of the extractable fraction of Annurca peel polyphenols after the baking of crackers and to investigate the dual potential of exogenous polysaccharides to prevent the loss of apple peel polyphenols during baking. Annurca apple variety was selected because it is a local variety, its peel is rich in anthocyanins, and several studies have shown its nutraceutical properties (D'Angelo et al., 2017; Tenore et al., 2013, 2016). Crackers were formulated with 10% apple peel powder (APP), with and without the co-addition of 10% lemon pectin or lemon insoluble polysaccharide, which are two of the most widely used and commercially available ingredients for food formulation or fortification. Using UHPLC-ESI-QTOF -MS/MS, the chemical stability during baking of phenolics at subclass and compound levels was studied. Crackers were also analysed for physical and colorimetric properties, texture, and dietary fibre content.

2. Materials and methods

2.1. Materials and chemicals

Annurca apples (*M. pumila* Miller cv Annurca**)** were manually peeled off, and peels were immediately dried in a professional heat drier at 50 °C for 16 h (Biosec Pro, Tauro Essiccator S. r.l., Vicenza, Italy). Then, dried peels were milled with a knife mill (Grindomix gm 200, Retsch, Germany), thus obtaining the Annurca peel powder (APP). The APP was stored in plastic bags at −80 °C until used.

Common wheat flour was kindly supplied by Macinazione Lendinara SPA ("Anima di Pane" flour, starch 71%, fibre 1.4%, fat 1.7%, protein 11%, ash < 0.55%), whereas unsalted butter (Lactis, Parmalat SPA) and dried yeasts (Lesaffre Italia) were purchased from local markets. Silvateam Spa kindly supplied lemon polysaccharides (namely, Aglufiber™, Aglupectin LC-S12P™, respectively). Aglufibers™ is obtained from citrus fruit after juice, oil, and pectin are extracted. Aglupectin LC-S12P™ is extracted from citrus peel; it is a low methoxyl pectin (29-33%) and is mainly composed of polygalacturonic acids (min 65%). From here on, we refer to Aglufibers as lemon fibres and Aglupectin as lemon pectin.

Acetonitrile, methanol, water, and formic acid utilized were all LC-MS analytical grade and purchased from Sigma-Aldrich (St. Luis, USA). Polyphenol standard protocatechuic acid, 4-hydroxybenzoic acid, caffeic acid, ferulic acid, p-coumaric acid, chlorogenic acid, epicatechin, (+)-catechin, procyanidin B2, hyperoside, quercetin, phloretin, phloridzin, and cyanidin galactoside chloride were purchased from Extrasynthese (Extrasynthese, Lyon, France).

2.2. Cracker-making and formulation

Dry ingredients were mixed with melted butter in a commercial planetary mixer (Kenwood, Chef XL KVL4110S, Treviso, Italy) (speed set at 1). Subsequently, tap water was slowly added to the bowl at room temperature. After that, the speed was set at 2 for 2 min to develop a homogeneous dough, which rested in the bowl for 30 min to ferment at room temperature. The dough was then split into several pieces and pressed with a manual sheeter to obtain a homogeneous layer of 2 mm height. Then, a cracker mold (9 × 5 cm) was used to obtain a rectangular shape, and a fork was used to pierce the surface of the cracker's dough. Raw samples were baked in a preheated oven (Icombi Pro, Rational, Italy) at 220 °C for 17 min. APP, lemon pectin, and lemon insoluble fibres were added as alone or in different combination by substituting 10% or 20% of wheat flour from the formulation of the control sample (CC), thus obtaining CM (10% APP), LC (10% lemon pectin), LCM (10% APP + 10% lemon pectin), FA (10% lemon fibre), FAM (10% APP + 10% lemon fibre) as reported in Table 1. A total of 3 independent batches per sample were prepared.

2.3. Proximate composition of APP and experimental crackers

The proximate composition of APP was evaluated in triplicate following standard procedures. The moisture content was determined using method 44–15A (AACC, 2000), protein content using method 976.05, the total, soluble and insoluble dietary fibre contents (TDF, SDF, and IDF, respectively) were determined according to method 991.43,

Table 1
Formulation of control (CC) and experimental crackers (CM, LC, LCM, FA, FAM). Ingredients' weights are expressed in grams.

Sample	APP	Lemon fibre[a]	Lemon pectin[b]	Wheat flour	Butter	Yeast	Water
CC	0	0	0	540	54	9	360
CM	54	0	0	486	54	9	360
LC	0	0	54	486	54	9	360
LCM	54	0	54	432	54	9	360
FA	0	54	0	486	54	9	360
FAM	54	54	0	432	54	9	360

CC: control cracker; CM: cracker with 10% APP; LC: cracker with 10% of Lemon Pectin; LCM, cracker with 10% of APP and 10% of Lemon Pectin; FA: cracker with 10% Lemon Fibres; FAM: cracker with 10% of APP and 10% of Lemon Fibre.
APP: Annurca peel powder.
[a] Aglufiber™.
[b] Aglupectin LC-S12P™.

total fat using method 948.22, ash using method 942.05 (AOAC, 2007), and free sugars using the Megazyme assay kit K-SUFRG 06/14 (Megazyme, Wicklow, Ireland). Cracker samples and commercial lemon polysaccharides were characterized for their moisture and dietary fibre content, in triplicate, following the above-described procedures. All the measured chemical components were expressed as g/100 g of dry matter (DM).

2.4. Physicochemical analyses of crackers

The crackers' thickness was measured with a caliper. The volume was determined using the AACC rapeseed displacement method 10-05.01 (AACC, 2000).

The color analysis was carried out with a reflectance colorimeter (Illuminant D65) (Minolta Chroma meter CR-300, Osaka, Japan) according to the CIE-L^* a^* b^* system. In the CIE-L^* a^* b^* system L^* value represents the lightness ranging from 0 (black) to 100 (white), while a^* and b^* are chromaticity coordinates. The a^* represents red and green values, while b^* represents yellow and blue components. Cracker samples were placed on a clear worktable, and measurements were taken at 9 points on the upper surface of the sample.

The texture profile analysis was performed using a TA-XT plus Texture Analyzer (TX-700, Lamy Rheology, Champagne au Mont d'Or, France) equipped with a 5 kg load cell and "three-point bending" geometry (Lamy Rheology, ref. 130091R) to determine the breaking force. Crackers were subjected to a single compression cycle (or breaking cycle) using a T probe (Lamy Rheology, ref. 130162) at 1 mm/s. The breaking force was calculated as the maximum force (N) to break cracker samples in half.

2.5. Extraction of phenolic compounds from APP and cracker samples

For polyphenols extraction, 50 mg of APP or 500 mg of crackers (previously lyophilized and milled) were mixed in a 15 mL falcon with 7.5 mL of a methanolic solution (80% methanol, 19% distilled water, and 1% formic acid), covered to avoid light exposure and shaken on a rotating wheel for 16 h set at 60 rpm, at room temperature (25 °C). The supernatants were recovered by a plastic pipette, centrifuged, and stored at −80 °C before analyses (Bianchi et al., 2024; Tolve et al., 2021). Five hundred mg of crackers of crackers was used to maintain the ratio between APP and the estimated APP content in crackers (i.e., 6.0 g/L).

2.6. Untargeted screening, semi-quantification of phenolic compounds in APP, and experimental crackers

A high-performance liquid chromatography-mass spectrometry method for profiling the polyphenol from APP and experimental crackers was used, adapting the methods from Bianchi et al. (2022) and Rocchetti et al. (2021). Before injection, samples were filtered using a 0.22 μm syringe filter (Phenomenex, Germany). Polyphenols were chromatographically separated by employing an Agilent 1260 HPLC system coupled to a 6540 electrospray-ionization quadrupole time-of-flight mass spectrometer HPLC-ESI-QTOF-MS/MS (Agilent, Germany) and an Eclipse XDB-C18 column (150 × 2.1 mm, 5 μm particle size, Agilent Technologies, Germany). Columns were maintained at 30 ∘C, and a linear elution gradient was applied, using a mobile phase consisting of 0.1% of formic acid in water (A) and 0.1% formic acid in acetonitrile (B) as follows: 0-5% B, 0-5 min; 5-30% B, 27 min; 30-95% B, 27-29 min. The column was re-equilibrated between injections for 3 min, applying the initial mobile phase conditions. Flow rate was 500 μL/min, and the injection volume was 5 μL. The HPLC system autosampler was thermostated at 5 ∘C to prevent degradation reactions during analysis. For comprehensive polyphenol identification, total ion spectra were collected in negative mode over the mass range of m/z 100–1500 at an acquisition rate of 2.0 spectra/s. A capillary voltage of 5 kV was applied. Subsequently, MS/MS analyses were executed in the all-ion mode with varying collision energies (10, 20, and 30 eV) for fragmentation. The ionization source was operating at 325 ∘C, using high-purity nitrogen (99.998%) at a pressure of 30 psi and a flow rate of 8 L/min. Octapole RF and fragment voltage were set at 750 V. Data collection and analysis were conducted using Agilent Mass Hunter Qualitative Software V. 10.0 (Agilent Technologies, USA).

The annotation was done via spectral matching against the databases FoodDB and Phenol-Explorer. The identification step was based on mass accuracy (setting a 5-ppm tolerance for m/z values), isotopic pattern, and spectral matching. Then, putative compounds were semi-quantified with corresponding external standards based on the peak area from the extracted ion chromatograms (EIC) in negative mode using standard calibration curves from 0.16 to 40 mg/L. The semi-quantification was based on the structural similarity of the selected phenolic compounds with the selected external standards. Thus, protocatechuic acid, 4-hydroxybenzoic acid were employed for the semi-quantification of hydroxybenzoic acids (HBAs); caffeic acid, ferulic acid, p-coumaric acid, and chlorogenic acid were employed for hydroxycinnamic acids (HCAs); epicatechin and catechin for flavan-3-ols; procyanidin B2 for Proanthocyanidins, Hyperoside, and quercetin for flavonols; phloretin and phloridzin for dihydrochalcones, and cyanidin galactoside chloride for anthocyanins (Extrasynthese, Lyon, France). Quantities were expressed as mg of standard equivalents per 100 g dry weight (DW, crackers, or APP).

2.7. Recovery of phenolic compounds after the baking of experimental crackers

The recovery of phenolic compounds is calculated as the ratio between the experimentally determined phenolic content in crackers and the theoretical phenolic content of crackers due to APP incorporation. The amount of APP (expressed as dry weight, g/100 g DW) in experimental crackers has been estimated by considering the moisture contributions of each individual ingredients and the final moisture of the samples. The recovery of phenolic compounds after baking was calculated as follows:

$$Recovery\ (\%) = \frac{C_c}{C_a} \times 100$$

where C_c is the apple phenolic compounds concentration (mg/100g of crackers DW) in experimental cracker CM, FAM, LCM, and C_a is the theoretical phenolic compounds concentration in crackers, calculated as follows:

$$C_a = C_i \times \frac{APP_d}{S_d} \times (100 - M)$$

Where C_i is the phenolic concentration in APP (mg/100g DW), APP_d is the dry weight of APP in the formulation, S_d is the sum of the dry weights of all the ingredients in the formulation (i.e., wheat flour, APP, lemon polysaccharides, butter, yeast), and M is the moisture percentage of the crackers. Moisture contents of ingredients are for wheat flour 15%, APP 9%, lemon polysaccharides 10%, butter 16%, dry yeast 5%, and experimental crackers 5%.

2.8. Statistical analyses

Data are presented as mean and standard deviation from three independent batches. Prior to statistical analyses, data were tested for normality using the Shapiro–Wilk test and for homoscedasticity using Levene's test (OriginLab, version 2025b). A total of 30 crackers per batch were tested for volume, texture, baking loss, color, and thickness. The variance analysis (ANOVA) was carried out between the mean values of 30 crackers of each batch, and the significant differences were determined by applying Tukey's multiple comparison test (Tukey, HSD). Then, crackers were pooled per batch, ground, and the resulting powder

was used extract polyphenols. Extractions were made in triplicate per batch and injected once in the HPLC. The ANOVA was conducted on the mean values of each batch, and significant differences were determined using Tukey's multiple comparison test. Pearson's correlation analyses were carried out between sample means. Agglomerative hierarchical clustering (AHC) and heat maps were done with OriginPro (OriginLab, version 2025b). Prior to the analyses, recovery data were scaled using Z-score normalization. AHC was performed using Euclidean distances to compute dissimilarities and Ward's method to cluster phenolic compounds. The heat map generated by AHC of phenolic compounds displayed the magnitude of each value in color, from blue (low) to red (high), based on their normalized recovery in the samples. The horizontal axis of the dendrogram has been transformed (log2) to improve the clarity of the image.

3. Results and discussion

3.1. Proximate composition of ingredients and crackers

Commercial lemon fibre and pectin were mainly composed of insoluble and soluble fibres, respectively, with a low protein content. In this study, lemon pectin is composed of polygalacturonic acid with low levels of methoxyl substituents. Lemon insoluble fibres, as reported by the manufacturer, are depleted of pectin and thus are putatively mainly composed of cellulose, hemicelluloses, and low amounts of lignin (R. Xiao et al., 2024). APP is composed by 42% of free sugars, a low amount of protein, fat, and ashes (<5%), and for 10.8% soluble fibres, and for 24.5% insoluble fibres. APP soluble fibres are putatively composed of pectin, while the insoluble fraction is composed of cellulose, hemicellulose, and lignin (Lyu et al., 2020).

As expected, the co-integration of different sources of dietary fibre influenced the amount of soluble, insoluble, and total dietary fibres in the resulting crackers (Table 2), whereas moisture content did not change among different crackers with a mean value of 5%. Adding APP increased CM's total, soluble, and insoluble fibre content by 3.5-, 3.3-, and 3.5-fold, respectively, compared to CC. LCM had the highest content of total and soluble fibres (9.8-fold and 24-fold compared to control crackers CC, respectively), whereas FAM had the highest content of insoluble fibres (11-fold compared to CC). Considering the European regulation (EC) N.◦ 1924/2006 on nutrition and health claims made on foods, CM can benefit from the claim “source of fibres” while FAM, FA, LCM, and LC can benefit from the claim “high fibres”, since they have more than 3g and 6g/100g of dietary fibre each 100g of product, respectively. Mir et al. (2027) reported an increased level of soluble, insoluble, and total dietary fibres in rice crackers with increasing amount of apple pomace (Mir et al., 2017).

Table 2
Soluble (SF), insoluble (IF), and total dietary fibre (TDF) content and percentage in the ingredients and experimental crackers.

Sample	TDF	IF	SF	IF(%)	SF (%)
APP	35.3 ± 0.2	24.5 ± 0.3	10.8 ± 0.2	70	30
Lemon Fibre*	81 ± 1.2	75 ± 2.3	6.0 ± 1.1	93	7
Lemon Pectin**	88.2 ± 1.4	0.9 ± 0.0	87.3 ± 2.1	1	99
CC	1.3 ± 0.3^{e}	0.9 ± 0.2^{f}	0.4 ± 0.2^{f}	70	30
CM	4.6 ± 0.3^{d}	3.4 ± 0.1^{c}	1.4 ± 0.0^{d}	70	30
LCM	12.8 ± 0.2^{a}	3.0 ± 0.1^{d}	9.8 ± 0.4^{a}	23	77
FAM	11.7 ± 0.2^{b}	10.0 ± 0.2^{a}	1.7 ± 0.0^{c}	84	16
LC	9.8 ± 0.4^{c}	1.1 ± 0.0^{e}	8.7 ± 0.2^{b}	11	89
FA	9.2 ± 0.3^{c}	8.3 ± 0.3^{b}	0.9 ± 0.1^{e}	90	10

CC: control cracker; CM: cracker with 10% APP; FA: cracker with 10% Lemon Fibre; FAM: cracker with 10% of APP and 10% of Lemon Fibre; LC: cracker with 10% of Lemon Pectin; LCM: cracker with 10% of APP and 10% of Lemon Pectin. APP: Annurca peel powder. *Aglufiber™. **Aglupectin LC-S12P™.
Different superscripts within the same column indicate statistically different means.

3.2. Physical, textural, and colorimetric characterization of experimental crackers

CM crackers were slightly thicker (similarly, in volume) than the control sample CC, even though they accounted for 4.6g/100g of total fibres (Table 2). Indeed, APP had a total sugar content of 43g/100g (Table S1), representing a fermentable substrate that could help the yeast produce gases, which can lead to a puffer product once cooked. The addition of lemon soluble or insoluble fibres influenced the thickness and the volume of experimental crackers (Table 3, Fig. 1), and remarkably, the total dietary fibre content was inversely correlated with volume and thickness ($r = -0.89$, $r = -0.92$, respectively; $p < 0.05$, Table S1). Lemon fibre or pectin-enriched crackers were less thick than the control sample, especially the ones richer in lemon insoluble fibre, namely FAM and FA. Indeed, the lemon insoluble fibres had the greatest worsening effect, reducing thickness by 2-fold compared to CC. Yilmaz et al. (2017) obtained thicker crackers supplemented with a low amount of orange seed fibres (around 2.9% and almost insoluble fibres) (Yilmaz & Karaman, 2017), whereas Rainero et al. reported how higher grape pomace powder (rich in insoluble fibre) addition dramatically reduces breadsticks' volume (Rainero et al., 2021). The addition of insoluble fibres could generate a stiffer structure of the dough and thus compromise the ability to hold the air bubble during leavening (Fu et al., 2015). Even though pectin might counteract the gluten matrix development as well, for instance, by hydration competition (Franco et al., 2026), it can make the dough more viscous, thus promoting the retention of gas bubbles during fermentation and kneading steps. On the contrary, in the work of Schmidt et al. (2018), adding 10% of black currant pomace did not affect the volume increase of savory crackers (Schmidt et al., 2018). The same authors argued that the added amount is ineffective in altering the protein structure of crackers, thereby preventing protein aggregation.

Crackers tested in this study were brittle, and the fracture occurred in a clean cut without bending. Addition of APP slightly increased the maximum breaking force required to snap the crackers, but the difference between CC and CM is statistically not significant. Lemon pectin addition lowered the force necessary for breaking a cracker by almost 60% compared to the control sample, while it increased with the lemon insoluble fibres but not in the presence of APP, such as in the case of FA crackers (26% more compared to CC). Indeed, the breaking force and soluble fibre content are negatively correlated (Table S2), suggesting that the added pectin can soften the cracker structure, preventing the development of a solid gluten structure. Indeed, the gluten matrix can be altered in several ways: by interacting with food components and competing for optimal hydration. Mir et al. (2017) and O'Shea et al. (2012) reported that apple pomace (rich in soluble fibre) decreases the hardness of rice crackers by acting as a lubricant, thus reducing the hardness of puffed snacks (Mir et al., 2017; O'Shea et al., 2012). Meanwhile, FA crackers require more force to break. Yilmaz et al. (2017) reported how citrus seed fibres and wheat fibres, rich in insoluble fractions, increased the breaking force of supplemented crackers (Yilmaz & Karaman, 2017). On the contrary, adding APP and insoluble fibres (FAM) simultaneously did not change the maximum force required to break the FAM sample in half. It is assumed that sugar crystals do not initiate fractures, but fat and entrapped air bubbles can (van der Sman & Renzetti, 2019). We suppose that, by helping yeasts during fermentation, sugars within APP can increase the number of air bubbles in the dough, thereby generating more points of fracture. Therefore, a compensation effect between the stiff effect of insoluble fibres, the “soften” effect of sugars, and the lubricant effect of natural apple pectin might occur. Similarly, sugar substitution in cookies increased their breaking strength (Ashwath Kumar & Sudha, 2021). Therefore, the force required to snap into two pieces the crackers in the present study could depend on the three-ways of interaction between the factors soluble, insoluble fibres, and APP sugars.

The L^* parameter was higher in crackers without APP (namely, CC,

Table 3
Physical, textural, and colorimetric characterization of control and experimental crackers.

Sample	Thickness(mm)	Volume(cm^3)	Breaking Force (N)	L^*	a^*	b^*
CC	4.8 ± 0.1^{b}	17.7 ± 0.6^{a}	21.3 ± 2.9^{b}	73.1 ± 2.1^{a}	4.2 ± 1.2^{d}	31.6 ± 0.5^{a}
CM	5.0 ± 0.1^{a}	18.2 ± 1.3^{a}	23.5 ± 1.7^{b}	63.1 ± 2.1^{c}	8.6 ± 0.9^{a}	30.7 ± 1.0^{a}
FA	2.7 ± 0.2^{d}	10.3 ± 0.7^{b}	27.0 ± 1.8^{a}	67.2 ± 3.1^{b}	5.2 ± 1.7^{c}	23.3 ± 0.7^{b}
FAM	2.3 ± 0.1^{f}	8.6 ± 0.2^{c}	21.3 ± 1.0^{b}	57.8 ± 2.6^{d}	8.2 ± 0.7^{a}	19.5 ± 0.3^{c}
LC	3.0 ± 0.1^{c}	10.3 ± 0.2^{b}	12.1 ± 1.5^{c}	74.2 ± 1.9^{a}	1.1 ± 0.1^{e}	19.4 ± 0.4^{c}
LCM	3.0 ± 0.2^{c}	10.4 ± 0.5^{b}	12.7 ± 1.5^{c}	64.4 ± 1.8^{bc}	7.0 ± 0.2^{b}	17.1 ± 0.9^{d}

CC: control cracker; CM: cracker with 10% of APP; FA: cracker with 10% of Lemon Fibre; FAM: cracker with 10% of APP and 10% of Lemon Fibre; LC: cracker with 10% of Lemon Pectin; LCM, cracker with 10% of APP and 10% of Lemon Pectin; APP: Annurca peel powder.
Different superscripts within the same column indicate statistically different means.

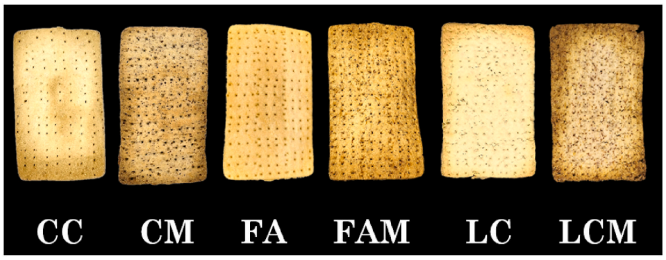

Fig. 1. Picture of the experimental crackers. From Left to right: CC: control cracker; CM: cracker with 10% of APP; FA: cracker with 10% of lemon fibre; FAM: cracker with 10% of APP and 10% of lemon fibre; LC: cracker with 10% of lemon pectin; LCM, cracker with 10% of APP and 10% of lemon pectin. APP: Annurca peel powder.

FA, and LC), and lower in crackers with only lemon insoluble fibres, whereas pectin addition showed no statistically relevant effect. Oppositely, Sivam et al. (2011) reported that bread enriched with 3% high-methoxy or low-methoxy had lower lightness than control bread. Moreover, adding apple phenolic extract or a mix of pectin and apple phenolic extracts to bread increased the lightness parameter. The APP addition clearly decreased the L* parameter. Indeed, the APP sugars and polyphenols can promote Maillard reaction with amino acids and darken the surface of the crackers (Mir et al., 2017; Rainero et al., 2021; Tolve et al., 2021). The a^* parameter increased when APP was added to the formulation. Similarly, Bianchi et al. (2022) reported how muffins enriched with grape pomace had increased a* value (Bianchi et al., 2022). The presence of pigments, like polyphenolic compounds (e.g., anthocyanins), in the Annurca apple peels can explain such effect (Bianchi et al., 2022; Rainero et al., 2021). The b^*parameter negatively correlates with total fibres content. Indeed, samples FA, FAM, LC, and LCM showed the lowest yellowness compared to CC and CM samples. Sensory analyses should be conducted to assess the sensorial aspects and acceptability of developed experimental crackers.

3.3. Semi-quantification of APP polyphenols

In the present study, any phenolic compounds were detected in wheat flour and lemon polysaccharides, while 59 phenolic compounds were determined in APP, for a total of 751 mg/100g, (Table S3), distributed in 7 phenolic subclasses: Hydroxycinnamic acids, Hydroxybenzoic acids, Flavan-3-ols, Proanthocyanidins, Anthocyanins, Flavonols, Dihydrochalcones (Table 4).

Anthocyanin accounted for a total of 4.20 mg/100g DW in APP, equivalent to 0.6% of identified compounds (Table 4), and the most abundant was the cyanidin galactoside. Zahid et al. (2023) reported 5.9 mg/100g DW of anthocyanins in apple peels (Zahid et al., 2023). Generally, in apple cultivars with red skins (as in the case of Annurca apples), anthocyanins are the phenolic subclass that imparts the typical pigmentation. However, UV-B irradiation and low-temperature treatment can affect the intensity and pattern of anthocyanin accumulation in apple peels (De Paepe et al., 2015).

Dihydrochalcones accounted for 12.2% of all phenolic compounds found in APP, corresponding to 91.8 mg/100g. Similarly, Lopez-Rodulfo et al. (2024) reported 10.2% of dihydrochalcones in 'Jonagold' apple pomace after juice extraction. Oppositely, a lower fraction was found by Fernandez-Jalao et al. (2020), who reported an overall 4% in high hydrostatic pressure (HPP) -treated and not-treated 'Golden Delicious' apple cultivars (Fernández-Jalao et al., 2020). Phloretin xyloglucoside was the most abundant dihydrochalcones in APP, accounting for 49%, while Phlorizin was around 31%, and these two compounds have been reported to be found in apple samples (Horvacki et al., 2022). Fernandez-Jalao et al. (2020) and Alongi et al. (2025) recover a higher percentage of Phloridzin than Phloretin xyloglucoside in whole apples, apple puree, or homogenized apples (Alongi et al., 2025; Fernández-Jalao et al., 2020). Moreover, the dihydrochalcones in Jonagold pomace and juice fractions showed different contents, suggesting that both apple cultivars, type of extractions, and specific apple tissue (e.g., pomace, whole apple, or juice) might influence their final content in apple processed products (Fernández-Jalao et al., 2020; Kaeswurm et al., 2022; Lopez-Rodulfo et al., 2024).

Flavan-3-ols accounted for a total of 4.3% of APP's phenolic compounds. Epicatechin is the major flava-3-ols in APP, followed by catechin (85% and 10% respectively, catechin/epicatechin ratio 0.12). Alongi et al. (2025), Kaeswurm et al. (2022), and Lopez-Rodulfo et al. (2024) reported similarly higher percentages of epicatechin vs catechin content in apples, independently of the applied treatment or technological processes, but with different ratios (Alongi et al., 2025; Kaeswurm et al., 2022; Lopez-Rodulfo et al., 2024).

In the present study, flavonols were the major class of APP's phenolic compounds, with a total of 63.3%. Higher flavonol content was also reported by Lopez-Rodulfo et al. (2024) in "Jonagold" pomace vs juice. Isoquercetin and Hyperoside (i.e., quercetin galactoside and glucoside) are the major compounds in flavonols, with a total content of 198 mg/100g, followed by quercetin arabinofurinoside (Avicularin), and isorhamnetin arabinoside. However, Kaeswurm et al. (2022) and Lopez-Rodulfo et al. (2024) reported a higher content of quercetin rhamnoside compared to other flavonols (Kaeswurm et al., 2022; Lopez-Rodulfo et al., 2024). Oppositely, Zhang et al. (2025) reported more Isoquercetin in freeze-dried Red Fuji apples, while other authors reported a range in apple peels between 0.1 and 8.7mg/g DW (Kalinowska et al., 2014).

Hydroxybenzoic acids accounted for 3.1% of phenolic compounds in APP. 4-hydroxybenzoic acid glucoside was the primary compound of the subclass, followed by protocatechuic acid glucoside (49% and 18% respectively).

Hydroxycinnamic acids accounted for 5.2% of all found phenolic compounds in APP, and the chlorogenic acid was the most represented. Similarly, Bouayed et al. (2012) and Fernandez-Jalao et al. (2020) reported high concentrations of chlorogenic acid in different apple varieties (Jonaspritz, Jonagold, Mutzu, and Golden Delicious) (Bouayed et al., 2012; Fernández-Jalao et al., 2020). In the present study, the amount of hydroxycinnamic acids is lower than that reported by Lopez-Rodulfo et al. (2024), who utilized whole apples.

Table 4
Polyphenol content detected in APP expressed as mg/100g of dry weight.

Phenolic compounds in APP	mg/100g DW
Cyanidin galactoside	2.49 ± 0.18
Cyanidin glucoside	0.88 ± 0.10
Cyanidin arabinoside	0.80 ± 0.02
Σ Anthocyanins	4.20 ± 0.30
3-hydroxy-Phloretin pentose-hexose	9.06 ± 0.60
Hydroxyphloridzin	4.68 ± 0.25
Phloretin	0.26 ± 0.01
Phloretin xyloglucoside	45.6 ± 1.2
Phloridzin	32.3 ± 1.8
Σ Dihydrochalcones	91.8 ± 2.8
Catechin	3.33 ± 0.26
Catechin glucoside	0.35 ± 0.04
Epicatechin	27.3 ± 3.2
Epicatechin glucoside	1.22 ± 0.11
Σ Flavan-3-ols	32.2 ± 2.0
Hyperoside + Isoquercetin	198 ± 5
Isorhamnetin arabinofuranoside	73.0 ± 0.9
Isorhamnetin galactoside	9.67 ± 0.12
Isorhamnetin glucoside	20.1 ± 1.3
Kaempferol arabinoside	1.11 ± 0.15
Kaempferol glucoside	8.08 ± 0.60
Kaempferol rhamnoside	0.88 ± 0.09
Quercetin	5.76 ± 0.38
Quercetin rhamnoside	12.7 ± 1.8
Quercetin arabinopiranoside	26.7 ± 2.1
Quercetin arabinoglucoside	0.44 ± 0.02
Quercetin arabinofurinoside (Avicularin)	90.9 ± 0.9
Rutin	28.6 ± 3.8
Taxifolin glucoside	1.77 ± 0.22
Σ Flavonols	478 ± 12
4-Hydroxybenzoic acid glucoside	10.9 ± 1.5
4-Hydroxybenzoic acid	1.89 ± 0.65
Hydroxytirosol glucoside	3.51 ± 0.62
Protocatechuic acid	1.79 ± 0.18
Protocatechuic acid glucoside	4.19 ± 0.38
Vanillic acid	0.46 ± 0.06
Vanillic acid glucoside	0.48 ± 0.21
Σ Hydroxybenzoic acids	23.21 ± 1.1
Coumaroilquinic acid	2.18 ± 0.35
Ferulic acid glucoside	3.94 ± 0.61
p-coumaric acid hexoside	1.47 ± 0.01
Caffeic acid	0.40 ± 0.03
Chlorogenic acid	28.9 ± 3.3
Cryptoclorogenic acid	2.69 ± 0.26
Σ Hydroxycinnamic acids	39.58 ± 0.52
Procyanidin dimer B2	37.48 ± 2.93
Procyanidin dimer #1	6.13 ± 0.81
Procyanidin dimer #2	0.90 ± 0.00
Procyanidin trimer #1	3.31 ± 0.27
Procyanidin trimer #2	4.91 ± 0.40
Procyanidin trimer #3	18.9 ± 1.9
Procyanidin trimer #4	2.22 ± 0.10
Procyanidin tetramer #1	1.25 ± 0.19
Procyanidin tetramer #2	1.02 ± 0.08
Procyanidin tetramer #3	1.39 ± 0.14
Procyanidin tetramer #4	3.77 ± 0.06
Procyanidin pentamer #1	0.66 ± 0.04
Procyanidin pentamer #2	0.75 ± 0.08
Σ Proanthocyanidins	82.7 ± 6.5
Σ APP	751 ± 25

Hydroxycinnamic content could depend on apple variety (Jonagold vs Annurca), the apple tissue under consideration (peels, pulp, or whole apple), or the technological process applied. Indeed, Kaeswurm et al. (2022) showed how the content of hydroxycinnamic acids in ancient apple varieties is concentrated in the flesh, while the content is lower in the peels.

Proanthocyanidins were 11.0% of all APP's phenolic compounds. Procyanidin B2 is the most prevalent compound in the proanthocyanidin class, which was also reported by other authors in different apple varieties (Fernández-Jalao et al., 2020; Kaeswurm et al., 2022; Lopez-Rodulfo et al., 2024).

3.4. Apple phenolic compounds' recovery after baking

The heat stability of polyphenols is complex and could depend on several parameters like the chemical structure of the phenolic compounds (rate of hydroxylation, glycosylation, acylation, and pigmentation (J. Xiao, 2022)), the temperature, the type of cooking (boiling, roasting, baking, frying), but also on the condition of the surrounding environment (e.g., extracellular condition, pH, presence of degrading enzymes, etc …). Phenol oxidases play a major role in phenolic stability (Langston et al., 2023; Palermo et al., 2014); however, in the present study, we can consider phenol oxidases almost degraded after the thermal treatment of Annurca by-products and the baking of the cracker samples. Palermo et al. (2013) reported that boiling water is the main cause of phenol leaching and, consequently, phenol losses, especially in noodles or pasta added with plant by-products rich in polyphenols (Liberatore et al., 2025). Roasting or baking decreased the phenolic compound content, but less extensively (Palermo et al., 2014). In addition, the phenolic compounds participate in the interplay of the Maillard reaction, which increases the level of Maillard reaction products and decreases the phenolic level (Langston et al., 2023; Palermo et al., 2014).

Anthocyanins were not detected, suggesting that heating caused an extensive degradation. For instance, purple maize extracts resulted in a high loss of anthocyanins at temperatures ranging from 120 °C to 180 °C (Slavu et al., 2020). Hirth et al. (2014) and Barti et al. (2015) reported lower anthocyanin content, thus lower thermal stability, in starch-based matrices with bilberry anthocyanins and purple and blue wheat flours, respectively, after the baking process (Bartl et al., 2015; Hirth et al., 2014). In contrast, Karakaya et al. (2016) reported how baking buns or biscuits did not affect anthocyanin content at all (Karakaya et al., 2016), while the addition of raspberry, strawberry, black currant, and sour cherry pomaces to muffins showed different anthocyanin content in the conventional oven baking (Górnaś et al., 2016). Even though there is consensus on the degradation of anthocyanins, the plant matrix, the temperature, and the type of baking product formulations might affect anthocyanins' stability. Thus, consideration should be done from case to case.

The hydroxybenzoic acid aglycons (namely, protocatechuic acid and 4-hydroxybenzoic acid) increased after baking (Table 5, CM sample). The protocatechuic acid content was almost 2 times in every experimental sample compared to the expected amount in APP. Hydroxybenzoic acids could represent the end products of the degradation of anthocyanins (like protocatechuic acid, 4-hydroxybenzoic acid) (Mäkilä et al., 2016) and flavonols like quercetin (4-hydroxybenzoic acid) (Chaaban, Ioannou, Paris, et al., 2017). For this reason, the above-cited aglycons have been omitted from the recovery calculation, depicted in Fig. 2. Regarding glycosides, the protocatechuic acid glucoside was more stable than 4-hydroxybenzoic acid glucoside, having fewer hydroxyl groups. Indeed, an increased number of hydroxyls substitutes, absence of sugar moieties, and presence of galloyl groups can reduce the thermal stability of polyphenols (Chaaban, Ioannou, Chebil, et al., 2017; Davidov-Pardo et al., 2011; S. Lin et al., 2023). For the above-cited reason, hydroxytirosol glucoside showed similar thermal behaviour to the protocatechuic acid glucoside after baking.

Caffeic acid was not recovered in the baked crackers (Table 5). Mukundur et al. (2017) reported a recovery of caffeic acid in bread supplemented with green coffee extracts of around 0.5% (Mukkundur Vasudevaiah et al., 2017). Chlorogenic acid recovery was lower than expected (60%) in CM crackers compared to APP. Similarly, Budryn et al. (2013) showed that chlorogenic acid degraded due to thermal conditions of about 60% in donuts with green coffee extracts (Budryn et al., 2013), while Rupasinghe et al. (2008) reported a recovery of 55% in muffins fortified with apple skins (Rupasinghe et al., 2008). Oppositely, Mukkundur et al. (2017) reported a higher recovery of Chlorogenic acid (75%) in bread (Mukkundur Vasudevaiah et al., 2017). Cryptochlorogenic (4-caffeoylquinic acid) acid was less stable than

Table 5
Phenolic compounds content and recovery in CM, FAM, and LCM experimental crackers. The recovery is calculated as reported in section 2.7.

Class	Compounds	CM	FAM	LCM
Anthocyanins	Cyanidin galactoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Cyanidin glucoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Cyanidin arabinoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
Hydroxybenzoic acid	4-Hydroxybenzoic acid glucoside	0.26 ± 0.01 26.8	0.37 ± 0.05 37.8	1.12 ± 0.07 114
	4-hydroxybenzoic acid	0.30 ± 0.04 174	1.05 ± 0.06 610	1.11 ± 0.00 645
	Hydroxytirosol glucoside	0.35 ± 0.04 110	0.35 ± 0.03 110	0.38 ± 0.00 121
	Protocatechuic acid	0.48 ± 0.02 294	0.55 ± 0.01 337	0.48 ± 0.03 294
	Protocatechuic acid glucoside	0.40 ± 0.02 104	0.55 ± 0.02 144	0.46 ± 0.01 120
	Vanillic acid	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Vanillic acid glucoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
Hydroxycinnamic acids	Caffeic acid	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Chlorogenic acid	1.61 ± 0.20 60.2	1.09 ± 0.13 41.6	0.67 ± 0.03 25.6
	Coumaroilquinic acid	0.18 ± 0.01 90	0.22 ± 0.01 110	0.18 ± 0.00 90
	Cryptoclorogenic acid	0.09 ± 0.01 36.7	0.10 ± 0.00 40.8	0.10 ± 0.01 40.8
	Ferulic acid glucoside	0.13 ± 0.01 36.3	0.16 ± 0.01 44.6	0.34 ± 0.03 94.5
	p-coumaric acid hexoside	0.15 ± 0.00 111	0.15 ± 0.02 111	0.10 ± 0.01 76.4
Dihydrochalcones	3-hydroxy-Phloretin pentose hexose	0.43 ± 0.05 52.2	0.48 ± 0.03 58.2	0.45 ± 0.01 54.6
	Hydroxyphloridzin	0.31 ± 0.03 72.9	0.39 ± 0.02 91.7	0.34 ± 0.01 80
	Phloretin xyloglucoside	2.69 ± 0.14 64.9	3.41 ± 0.04 82.3	3.23 ± 0.18 78
	Phloridzin	1.38 ± 0.12 47	2.35 ± 0.28 80	3.61 ± 0.07 123
	Phloretin	0.09 ± 0.01 375	0.28 ± 0.01 1166	1.27 ± 0.04 5291
Flavan-3-ols	Catechin	0.23 ± 0.04 75.9	0.58 ± 0.05 191	0.59 ± 0.02 195
	Catechin glucoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Epicatechin	1.55 ± 0.23 62.4	1.48 ± 0.25 59.6	2.98 ± 0.09 120
	Epicatechin glucoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
Flavonols	Isorhamnetin arabinoside	4.77 ± 0.30 71.8	9.89 ± 0.88 150	5.83 ± 0.43 87.8
	Isorhamnetin galactoside	0.77 ± 0.09 87.6	1.02 ± 0.03 116	1.09 ± 0.08 124
	Isorhamnetin glucoside	0.86 ± 0.09 47	1.20 ± 0.04 64.7	1.34 ± 0.02 73.3
	Kaempferol arabinoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Kaempferol glucoside	0.43 ± 0.04 58.5	0.61 ± 0.00 83.1	0.58 ± 0.01 79
	Kaempferol rhamnoside	0.11 ± 0.00 137	0.14 ± 0.00 175	0.14 ± 0.00 175
	Quercetin arabinopiranoside	1.15 ± 0.15 47.4	1.61 ± 0.17 66.3	0.97 ± 0.03 40
	Quercetin arabinofurinoside (Avicularin)	5.76 ± 0.62 69.7	8.59 ± 0.90 103	4.84 ± 0.14 58.6
	Quercetin arabinoglucoside	0.00 ± 0.00 0	0.00 ± 0.00 0	0.00 ± 0.00 0
	Isoquercetin + Hyperoside	17.25 ± 1.32 95.8	20.10 ± 0.84 111	18.99 ± 0.35 105
	Quercetin rutinoside (Rutin)	1.33 ± 0.14 51.1	1.55 ± 0.15 59.6	0.99 ± 0.07 38
	Quercetin rhamnoside	0.62 ± 0.07 53.7	0.93 ± 0.02 80.6	0.78 ± 0.00 67.5

(*continued on next page*)

Table 5 (*continued*)

Class	Compounds	CM	FAM	LCM
	Taxifolin glucoside	0.12 ± 0.01	0.14 ± 0.00	0.16 ± 0.00
		74.5	86.9	99.3
	Quercetin	2.97 ± 0.20	8.75 ± 0.44	10.01 ± 0.15
		566	1700	1910
Proanthocyanidins	Procyanidin B2	0.92 ± 0.00	0.85 ± 0.00	0.79 ± 0.00
		27	25	23.2
	Procyanidin dimer #1	0.42 ± 0.00	0.29 ± 0.00	0.27 ± 0.00
		75.4	52	48.5
	Procyanidin dimer #2	0.09 ± 0.01	0.14 ± 0.00	0.11 ± 0.00
		109	220	134
	Procyanidin trimer #1	0.15 ± 0.00	0.14 ± 0.00	0.13 ± 0.00
		49.8	46.5	43.2
	Procyanidin trimer #2	0.20 ± 0.00	0.19 ± 0.00	0.18 ± 0.00
		44.8	42.6	40.3
	Procyanidin trimer #3	0.40 ± 0.04	0.36 ± 0.05	0.29 ± 0.01
		23.4	21	17
	Procyanidin trimer #4	0.12 ± 0.01	0.12 ± 0.01	0.12 ± 0.00
		59.4	59.4	59.4
	Procyanidin tetramer #1	0.09 ± 0.00	0.11 ± 0.01	0.10 ± 0.00
		78.9	96.5	87.7
	Procyanidin tetramer #2	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
		0	0	0
	Procyanidin tetramer #3	0.07 ± 0.00	0.08 ± 0.01	0.09 ± 0.02
		55.5	63.4	71.4
	Procyanidin tetramer #4	0.07 ± 0.11	0.08 ± 0.10	0.09 ± 0.05
		20.4	23.3	26.2
	Procyanidin pentamer #1	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
		0	0	0
	Procyanidin pentamer #2	0.00 ± 0.00	0.00 ± 0.00	0.00 ± 0.00
		0	0	0

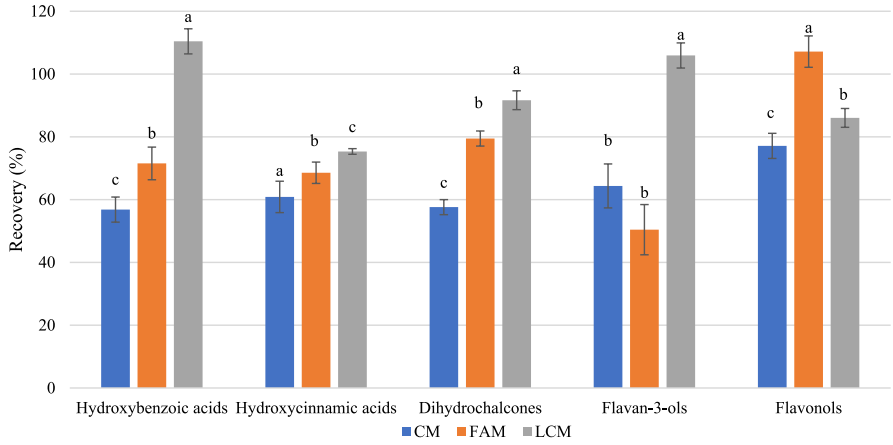

Fig. 2. Recovery of phenolic compounds in crackers compared to Annurca by-product powder. CM: cracker with 10% of APP; FAM: cracker with 10% of APP and 10% of Lemon Fibre; LCM: cracker with 10% of APP and 10% of Lemon Pectin. APP: Annurca peel powder. Bars with different superscript letters are statistically different for $p < 0.05$. Aglycon hydroxybenzoic acids and chlorogenic acids have not been included.

chlorogenic (3-caffeoilquinic acid), which is also reported by Y. J. Li et al. (2015) in aqueous solution. Overall, coumaric acid derivatives were more stable to heat, probably due to a less hydroxylated phenolic ring.

Dihydrochalcones in CM were 57% compared to APP content, like the recovery of dihydrochalcones attained by Franco et al. (2026) in apple pomace-containing bread. Dihydrochalcones, unlike most flavonoids, have A and B-ring not conjugated together, but linked with a flexible chain at C3, which is supposed to affect molecular changes or breakdown during heating (Human et al., 2021; W. Li et al., 2020). Phloretin xyloglucoside and hydroxyphloridzin stability were higher (64.9% and 72.9%) compared to other dihydrochalcones, due to the disaccharide linked at the C2 position, which can provide steric hindrance and thus a protective effect from chemical reaction. The lower recovery of 3-hydroxy-phloretin pentose hexose compound suggests that sugar moieties are supposedly linked to a different carbon of the A ring. Phloretin content was relatively low in APP (Table 1), and its content increased severalfold in crackers, which is also reported by Rupasinghe et al. (2008), and by Franco et al. (2026). We suppose that the aglycone is likely to increase during the baking due to the deglycosylation of the respective glycoside forms phloridzin or phloretin xyloglucoside.

Only the aglycon forms of flavan-3-ols was detected in the baked sample, namely catechin and epicatechin (Table 5). This suggests that glycosidic bonds between the sugar moiety and catechin or epicatechin can be easily degraded during baking.

Table 5 shows that the proanthocyanidin content decreased, independent of the polymerization degree. In addition, pentamers were not detected in any experimental samples. Similarly to Le Bourvellec et al. (2012), we hypothesize that heat treatment during the baking of the experimental crackers may cause APP procyanidins to depolymerize and

form carbocations, which then form covalent bonds with the nucleophilic groups of fibres (Le Bourvellec et al., 2012), which in the case of CM is the natural pectin and cellulose present in APP.

Flavonols in the CM sample were lower than the expected amount added with APP. Bedrníček et al. (2020) showed how the recovery levels of flavonols (mainly quercetin glucoside and quercetin diglucoside) were lower in dried onion peels supplemented bread (around −24% *vs* bread dough) (Bedrníček et al., 2020). Furthermore, they showed that adding untreated onion peels to bread increased flavonol content by 156%, suggesting that applying a sequential thermal treatment to onion peels might reduce their stability (the first for thermal stabilization and the second for bread baking). Moreover, Zhao et al. (2017) reported a retention of around 85% and 74% for two quercetin hexosides and one pentoside, respectively (after baking blueberries for 15 min at 176 °C) (Zhao et al., 2017). The quercetin glycosides in the present study followed a similar trend in CM sample, considering crackers were baked at higher temperatures and slightly longer. In another study with applesauce (12 varieties), Le Bourvellec et al. (2011) observed that flavonol content increased in three varieties and decreased in 9 varieties after thermal treatment (Le Bourvellec et al., 2011). The same authors argued that differences in the skin of the apple varieties, structures, mechanical resistance, and cuticle thickness might influence the recovery of quercetin derivatives in the applesauce.

Flavonols have a fully conjugated C-ring compared to flavanols, flavan-3-ols, and dihydrochalcones. This structural motif can promote electron delocalization of the ring and enhance its stability, which can partially explain the higher retention (77%, Fig. 2) compared to other flavonoid subclasses (i.e., flavanols, flavan-3-ols, dihydrochalcones, and proanthocyanidins). In addition, the sugar moiety at C3 of the C ring is supposed to discourage carbocation formation and thus enhance the compound stability. Taxifolin glucoside lacks the double bond between C2 and C3, and thus, a lower retention compared to hyperoside + isoquercetin was expected. Among quercetin derivatives, quercetin arabinoglucoside and quercetin rutinoside showed the least recovery. These compounds have two O-glycosidic bonds at positions C3 and C7 of the C and B rings, respectively, which influence their solubility and potentially their sensitivity. In this sense, our findings agree with Bedrníček et al. (2020) who reported extensive heat-induced degradation of di or tri-glycosylated quercetins, and less extensive degradation of the aglycon form, resulting in an increase in quercetin content.

Substitution patterns like glycosylation, acylation, methylation, and hydroxylation have been proven to affect polyphenols' stability (Cao et al., 2021). Nevertheless, it is rather complex to elucidate how multiple and discrete structural differences among polyphenols can affect their stability during baking.

3.5. *Apple phenolic compounds recovery after the addition of lemon soluble or insoluble fibres*

Fig. 2 shows the recovery for each APP polyphenols' subclasses in CM, FAM, and LCM crackers (not proanthocyanidins and anthocyanins, discussed later). Hydroxybenzoic acids, hydroxycinnamic acids, dihydrochalcones, and flavan-3-ols' recoveries were higher in crackers with added lemon pectin, whereas flavonols recovery was higher with the addition of insoluble fibres. The correlation analyses (Table S4) support these hypotheses. This is the first time, to the author's knowledge, that the protective effect of different types of fibres on phenolic compounds in crackers has been reported, even after processing and baking steps.

The recoveries of the hydroxybenzoic glycosides were close to the expectations. The increased content of aglycons in FAM and LCM samples (Table 5) can be ascribed both to the degradation of other phenolic compounds and to an improved release from the APP matrix of covalent or non-covalent bound polyphenols due to kneading and heating.

In FAM and LCM crackers, the recovery of chlorogenic acid was slightly lower than that of CM. In contrast, Ferracane et al. (2008) reported an increased amount of chlorogenic acid in heated artichokes. They debated that isomerization and hydrolysis, due to the massive trans-esterification phenomenon, occurred during the processing (Ferracane et al., 2008). We supposed that fibre addition (especially lemon pectin) can facilitate the epimerization shifting or possible dimerization with chlorogenic acid and its derivatives, forming new compounds like hydroxylated 5-o-caffeoylquinic acid derivatives or 4, 5-dicaffeoylquinic acid, which have not been identified with UHPLC-MS/MS in the present study (Dawidowicz & Typek, 2010). For this reason, in Fig. 2, the chlorogenic acid was not considered. In contrast, cryptoclorogenic acid, which structurally differs from chlorogenic by the carboxyl bond in position 4 of the quinic acid instead of position 3, has similar recovery in every experimental cracker sample, independent of the added fibres. Cumaroylquinic acid had higher recovery in FAM compared to CM and LCM, suggesting a protective effect from thermal degradation due to the insoluble fraction of lemon fibres but also reinforcing the fact that the coumaric is mostly more heat-stable than caffeic acid. Similarly, Mukkundur et al. (2017) reported the 75% recovery of cumaroylquinic acid in bread (Mukkundur Vasudevaiah et al., 2017). The p-coumaric acid hexoside showed insignificant degradation when insoluble lemon fibre was added (FAM) but lower recovery in crackers with added pectin (LCM), oppositely ferulic acid glucoside recovery was higher.

Adding insoluble and soluble lemon polysaccharides increased dihydrochalcone content to 79% and 91%, respectively (Fig. 2). 3-hydroxy-phloretin pentose-hexose was slightly lower in CM than in FAM and LCM, but there was no significant difference between the crackers with added insoluble lemon fibres/pectin (Table 5). As expected, phloretin xyloglucoside had similar behavior, whereas hydroxyphlorizin content was higher in FAM. Phloridzin was more thermally stable (123% recovery) in crackers with added lemon pectin than in those with added insoluble lemon fibres (FAM). However, its higher recovery, exceeding 100%, can be explained by different and interdependent factors such as the chemical breakdown of other polyphenols, increased resealing from APP matrix, and the protective effect of fibres. Crackers with added lemon pectin and fibres showed a higher phloretin content than CM. This effect could represent further evidence of the protective effect of fibres. Indeed, Padayachee et al. (2012) reported that a bacterial cellulose-pectin composite could form hydrophobic pockets that enhance binding affinity with polyphenols through hydrophobic interactions. Therefore, we postulate that glycosylated phloretins lose their sugar moieties during baking. Still, the resulting aglycon (phloretin), being more hydrophobic, can better fit the hydrophobic pockets formed by pectin or insoluble fibres (such as cellulose), thereby limiting their subsequent thermal degradation.

Catechin/epicatechin contents found in LCM crackers were higher compared to CM. Moreover, the catechin content was higher than expected, exceeding 190%, which can be explained by the case of phloridzin: by increased release from the APP matrix, the protective effect of fibres, and extensive decomposition of proanthocyanidins, as observed in Fig. 3. Similarly, Franco et al. (2026) reported increased recovery of catechin and epicatechin in bread with apple pomace and psyllium or apple pomace and pectin. In addition, it has been reported that epicatechins and catechins can undergo epimerization when heated (Ananingsih et al., 2013; De Taeye et al., 2014; Huang et al., 2025; Lončarić et al., 2018).

Overall, for the hydroxycinnamic, hydroxybenzoic acids, dihydrochalcones, and flavan-3-ols, both insoluble and soluble fractions of lemon exerted a protective effect during baking, with pectin demonstrating the greatest effect. Indeed, lemon pectin can form a gel in water, which increases its molecular surface area, therefore exposing more functional groups (such as carboxyl and hydroxyl groups) that can form hydrogen bonds with polyphenols. In addition, pectin units are linked by a flexible α(1 → 4) glycosidic bond, which allows a better structural adaptation for phenolic compounds (Assifaoui et al., 2024; Hu et al., 2025). These two features of pectin (structural flexibility and gelling behaviour) can elucidate why it outperforms lemon insoluble fibres,

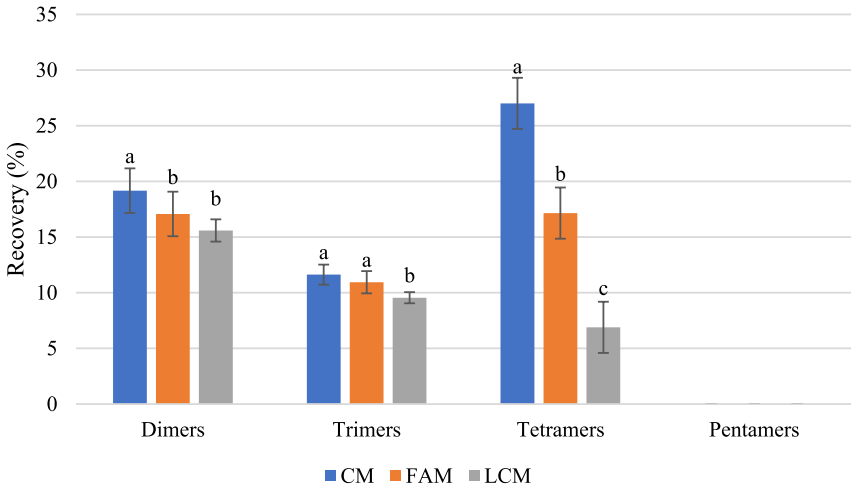

Fig. 3. Proanthocyanidin content in crackers compared to Annurca by-product powder. CM: cracker with 10% of APP; FAM: cracker with 10% of APP and 10% of Lemon Fibre; LCM: cracker with 10% of APP and 10% of Lemon Pectin. APP: Annurca peel powder. Bars with different superscript letters are statistically different for $p < 0.05$.

which are mostly made of a less flexible and non-gelling polymer like cellulose.

Fig. 3 shows that proanthocyanidin content decreased, independent of the polymerization degree and more pronouncedly with lemon fibre addition. Considering the above-cited effects of heat depolymerization and carbocation formation (Le Bourvellec et al., 2012), we can postulate that adding fibres amplifies the number of possible binding sites. Especially in the case of low methoxyl pectin, the carboxyl groups can provide binding sites for carbocations, thereby reducing the soluble and quantifiable fraction of polyphenols. Inversely, Franco et al. (2026) reported slightly higher recovery of proanthocyanidins with a degree of polymerization between 2 and 4 (dimers to tetramers) in bread with high methoxyl pectin. In this sense, we hypothesize that methoxylation can constitute a barrier to the nucleophilic binding, thus resulting in higher proanthocyanidin recovery.

Isorhamnetin arabinoside content was higher in FAM crackers, while galactoside and glucoside were higher in LCM crackers. Kaempferol rhamnoside content increased after the baking treatment and in the presence of fibres (Table 5). Kaempferol glucoside was higher in FAM and LCM, while Kaempferol arabinoside was not recovered. On the contrary, the content of Kaempferol glucoside in cherry pomace-enriched muffins was almost 100% (Górnaś et al., 2016). Except for quercetin arabinoglucoside (not recovered after the baking), all quercetins bonded with sugar showed higher content when insoluble fibres were added. Quercetin content increased by 4-, 12-, and 14-fold compared to APP in CM, FAM, and LCM, respectively, and similarly as reported in Franco et al. (2026). Surprisingly, given the trend in the other studied subclasses of apple polyphenols, the greater protective effect is exerted by insoluble lemon fibres, which are putatively composed mainly of cellulose. However, due to the stiffness of cellulose glycosidic bond (β(1 → 4)) and its inability to gel, we can exclude that it can "encapsulate" polyphenols like pectin can do (D. Lin et al., 2021). We hypothesize that hydrogen bonds and hydrophobic interactions are the driving forces for flavonols higher recovery, especially for quercetin and its derivatives having more than five -OH groups. Nonetheless, flavanols have different architectural features compared to other flavonoids: they have a planar aromatic ring with can potentially favor CH or OH – π interactions with cellulose (Krysa et al., 2025), or even π – π interactions with other polyphenols (like flavanones) already bound to lemon insoluble fibres.

3.6. Considerations about the combined effect of baking and fibres on individual phenolic compound recovery

Fig. 2 and the correlation Table S4 highlighted the fibres' roles in protecting and modulating the phenolic subclasses of crackers after baking; instead, the hierarchical cluster heatmap (Fig. 4) underlined how individual compounds grouped based on their recovery and trends when lemon insoluble and soluble fibres were added. It is evident that the addition of lemon fibres can modulate the recovery of phenolic compounds and thus the profile of CM, FAM, and LCM samples. Examining the clusters (Fig. 4), we can observe that apple polyphenols have been grouped into three main response patterns:

- Phenolic compounds' recovery is higher with the addition of soluble lemon fibres (Cluster 3),
- Phenolic compounds' recovery is lower without fibre addition and higher with lemon soluble or insoluble fibres addition (Cluster 2),
- Phenolic compounds' recovery is lower with lemon soluble fibre addition (Cluster 1).

In cluster 1 (Fig. 4), the lower recovery of proanthocyanidins in LCM crackers can be explained by their tendency to react with nucleophilic sites of sugars at high temperature and subsequent formation of a glycosidic bond; or their interaction with those sugar residues through non-covalent bonds, both of which led to lower extractability. However, structurally similar compounds such as procyanidin dimer #2 belonged to cluster 2 and demonstrated a different behaviour, more closely related to hydroxyploridzin. If we consider that proanthocyanidins can undergo epimerization, depolymerization, or polymerization reactions (De Taeye et al., 2014), the reduced content of procyanidin dimer #2 in CM (compared to other proanthocyanidins from the green cluster) can be partially explained. At the same time, polyphenols with different structures and chemical makeups are part of cluster 1 (e.g., avicularin and protocatecuic acid). This trend can be observed across all clusters, with an uneven distribution of phenolic subclasses. In this sense, chemical features of polyphenols and the proposed reactions only partially explained their recovery. It should be considered all the complex and interdependent factors that could influence the APP polyphenols recovery, like chemical breakdown and transformation, the positive/antagonistic effect of fibres that can impact polyphenols extractability or protect them from thermal degradation, and at the same time, the potential release of bound compounds from the APP matrix

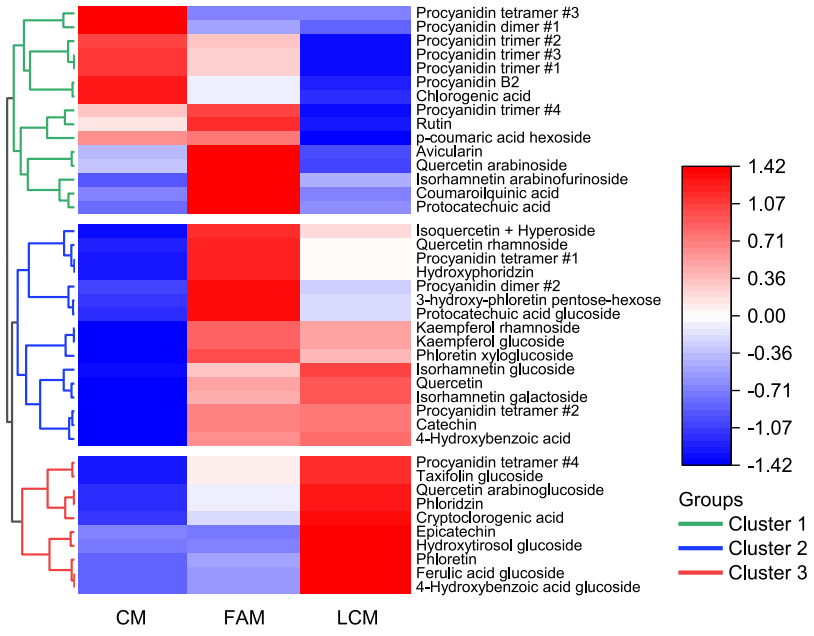

Fig. 4. Heat maps resulting from the Agglomerative Hierarchical cluster analysis of the normalized recovery of phenolic compounds in CM (10% of APP), FAM (10% of APP and 10% of lemon fibre) and LCM (10% of APP and 10% of lemon pectin) cracker samples. The horizontal axis of the dendrogram has been log-transformed (log2). Colored cells on the heat map correspond to a normalized value of the recovery, with samples in columns and phenolic compounds in rows. APP: Annurca peel powder.

during crackers' processing.

In the present study, we increased the recovery of APP polyphenols by adding lemon polysaccharides and showed that adding soluble or insoluble lemon fibres can differentially affect the APP phenolic profile of the final product. However, it is difficult to discern the real contribution of the proposed factors and their interdependencies. In addition, the polyphenols-polysaccharide interaction mechanisms reported are inferred from the literature and from differences in the extractability of the APP phenolic compounds. Future studies should explore the factors that contribute to the polyphenols recovery and investigate the intermolecular forces between lemon polysaccharides and APP polyphenols, using techniques such as FTIR, DSC, and molecular docking.

4. Conclusions

This study developed five fibre-enriched cracker formulations by incorporating soluble and insoluble lemon polysaccharides along with Annurca apple peel powder (APP). From a technological viewpoint, insoluble lemon polysaccharides reduced cracker volume, while APP increased thickness, probably because its sugar content supported yeast activity. Texture analysis showed that pectin softened the crackers, whereas insoluble fibres increased the breaking force. However, a combination of both fibres types resulted in a balanced texture.

The phenolic profile of APP was characterized, identifying 59 compounds across seven subclasses, predominantly flavonols. Baking significantly influenced phenolic recovery: anthocyanins were not retained, whereas certain compounds, such as protocatechuic acid, 4-hydroxybenzoic acid, quercetin, and phloretin, increased in abundance, likely due to degradative reactions (e.g., deglycosylation) of more complex polyphenols. Overall, flavonols demonstrated the greatest thermal stability, attributed to their conjugated structure.

The addition of lemon pectin improved the recovery of several phenolic subclasses, particularly the aglycons of hydroxybenzoic acids and flavan-3-ols, likely due to its structural properties (e.g., chain flexibility and reactive functional groups). In contrast, proanthocyanidins decreased, likely because they tended to bind to lemon polysaccharides covalently. Insoluble lemon fibres enhanced the recovery of most phenolics, especially flavonols, possibly involving hydrogen bonding and π-interactions with lemon cellulose. However, future studies should verify the present hypotheses by investigating the intermolecular forces between polysaccharides and apple polyphenols.

Cluster analysis suggested that phenolic stability depended less on subclass structural similarities and more on factors such as single-molecule features, interactions with fibres, chemical reactions during baking, and the possible release of bound polyphenols due to crackers processing steps.

In conclusion, combining APP with lemon-derived fibres in low-moisture bakery products is a promising strategy to enhance dietary fibre content, increase the recovery of phenolic compounds, and formulate innovative antioxidant-rich baked goods targeting health-conscious consumers. This approach not only improves the nutritional value of the final product but also supports sustainability by valorizing apple peel waste, reducing environmental, social, and economic impacts.

CRediT authorship contribution statement

Federico Bianchi: Conceptualization, Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing. **Gianluca Giuberti:** Supervision, Visualization, Writing – review & editing. **Maria Franco:** Investigation, Methodology. **Mario M. Martinez:** Methodology, Supervision, Validation, Writing – review & editing. **Barbara Simonato:** Supervision, Visualization, Writing – review & editing.

Declaration of competing interests

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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fbio.2026.109675.

Data availability

Data will be made available on request.

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