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Wine lees as functional ingredient to produce biscuits fortified with polyphenols and dietary fibre

Giusy Rita Caponio [a], Roberta Miolla [b], Mirco Vacca [b], Graziana Difonzo [b,*], Maria De Angelis [b]

[a] Department of Bioscience, Biotechnology and Environment, University of Bari Aldo Moro, Via Orabona 4, 70125 Bari, Italy
[b] Department of the Soil, Plant and Food Science DiSSPA, University of Bari Aldo Moro Via Amendola, 165/a, I-70126 Bari, Italy

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ABSTRACT

Wine lees, a by-product of winemaking process, still contains bioactive molecules (especially fibres and polyphenols) with potential functional properties. Considering the high frequency of consumption and high versatility of biscuits, the purpose of this work was to replace wheat flour with 10% (F10) and 20% (F20) of wine less flour (WLF) to produce biscuits with an improved nutritional profile. This study assessed the impact of substituting 10 and 20% of WLF on the physico-chemical, nutritional, textural, sensory, and volatile aroma compound characteristics. These results were compared with those of a control biscuit exclusively made with wheat flour (F0). The statistical comparison allowed for demonstrating how the fortified biscuits (F10 and F20) had significantly higher values of fibres, proteins, phenolic compounds, antioxidant activity, and higher oxidative stability than the control (F0). Different flavonoids and anthocyanins were detected in F10 and F20, such as ellagic acid and malvidin-3-acetil glucoside with a concentration greater than 200 mg/kg. The increased fibres and polyphenols in the F20 led to a significant lowering in glycaemic index (pGI) and starch hydrolysis (HI). Based on simulated colonic fermentation, F20 also exhibited a slight prebiotic effect supported by the significant increase in lactic acid bacteria cell density compared to F0.

1. Introduction

The bakery sector, in particular that of biscuits, is remarkably diversified in terms of product variety (Misra & Tiwari, 2014). Due to their ease of consumption (ready-to-eat) and affordable price, biscuits stand out the most popular baked goods, enjoyed by people from all levels of society (Arepally, Reddy, Goswami, & Datta, 2020; Kumar et al., 2019). Although different types of biscuits have become widespread in recent years (Bravo-Núñez & Gómez, 2023; Caponio, Difonzo, et al., 2022; Zielińska & Pankiewicz, 2020), these are usually made by mixing flour, eggs, butter, and sugar. In addition to differences related to the formulation of the product itself, variations in production technologies impact the texture and rheological features of biscuits, which are crucial in consumers' perception and evaluation (Sai Manohar & Haridas Rao, 2002). Furthermore, the increasing consumer interest in health has spurred the development of novel foods designed to determine specific beneficial effects (Caponio, Difonzo, et al., 2022; Lippolis, Cofano, Caponio, De Nunzio, & Notarnicola, 2023). In this field, playing a key role in human health due to the presence of bioactive compounds (Granato et al., 2020), innovative additives are required to functionalize foods. One of the most critical issues concerning biscuits is fat oxidation, which contributes to the deterioration and alteration of product quality compromising the shelf-life (Patrignani, Conforti, & Lupano, 2014). This process can lead to rancidity, resulting in the formation unpleasant flavours due to aliphatic aldehydes or other volatile compounds (Calasso et al., 2023; Marzano et al., 2022). In this context, antioxidants and polyphenols could act as regulators of oxidation stability (Rather, Masoodi, Akhter, Rather, & Shiekh, 2016) interfering with radicals and, thus, interrupting the chain reaction (Santos-Sánchez, Salas-Coronado, Villanueva-Cañongo, & Hernández-Carlos, 2019). Besides the antioxidant activity, polyphenols can also improve the functional/nutritional value of biscuits ensuring rheology, sensory, and shelf-life improvements (Difonzo et al., 2021). Furthermore, traditional biscuits – which involve the use of wheat flour, sugar, and fat – are characterized by a high glycaemic index and low dietary fibre content (Devi & Khatkar, 2016) leading to an increased risk of adverse cardiovascular events as well as the onset of certain diseases (Jenkins et al., 2021). In addition, dietary fibre plays a beneficial role in the human body by reducing the

* Corresponding author. Università degli Studi di Bari Aldo Moro, Via Amendola 165/a, I-70126 Bari, Italy.
E-mail addresses: giusy.caponio@uniba.it (G.R. Caponio), roberta.miolla@uniba.it (R. Miolla), mirco.vacca@uniba.it (M. Vacca), graziana.difonzo@uniba.it (G. Difonzo), maria.deangelis@uniba.it (M. De Angelis).

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glycaemic response and, thus, improving blood glucose control in diabetic subjects (Zhu, Du, Zheng, & Li, 2015). As the results of these considerations, it appears clearly how the developing of new formulations of biscuits with high biological value and nutritionally enhanced with concentrated content of antioxidants, fibres, and vitamins should be a promising tool to reach the goal of supporting the human wellbeing. In this context, the winemaking process generates a considerable volume of different residues, characterized by the presence of biodegradable compounds and suspended solids that require specific disposal. Among oenological by-products, wine lees represent "residues formed at the bottom of wine vessels, after fermentation, during storage or after treatments, as well as residues obtained after wine filtration or centrifugation" (Pérez-Serradilla & De Castro, 2008). The solid fraction of wine lees consists mainly of yeast cells (*Saccharomyces cerevisiae*), organic acids (mainly tartaric acid), insoluble carbohydrates, inorganic salts, phenolic compounds, and pulp and other parts of the grape (Braga, Silva, & Alves, 2002; Gómez, Igartuburu, Pando, Luis, & Mourente, 2004; Pérez-Serradilla & De Castro, 2008) allowing us to consider it an excellent source of antibacterial substances, dietary fibre, and phenolic compounds with antioxidant activity that can be used to produce nutritionally improved foods with positive health deliverables (Rivas et al., 2021).

A considerable number of previous studies have been conducted using wine lees to evaluate their antioxidant activity and nutritional value in different food matrices, such as ice cream, yogurt, and cereal bars (Borges et al., 2021; Hwang, Shyu, & Hsu, 2009; Sharma & Aglawe, 2022; Sharma, Kumar, Azad, & Adsule, 2015). However, to our best knowledge, no studies have been carried out concerning the incorporation of wine lees into biscuits and evaluating, at the same time, technological, sensory, antioxidant, and nutritional profiling. Since by-products of winemaking process, especially grape pomace and grape seeds, are widely used in the agri-food sector, the choice of use wine lees allows to valorize also another by-product of the winemaking process not yet explored in biscuits production. The exploitation of food by-products would encourage an environmentally sustainable approach, promoting the development of closed-loop industrial processes in a circular economy vision. It would also allow the development of new industrial segments aimed at innovative food production (Campos, Gómez-García, Vilas-Boas, Madureira, & Pintado, 2020). In the current study, according with results provided from the characterization of wine less flour (WLF), this functional ingredient was used in combination with traditional flour to formulate two innovative experimental biscuits (with 10 % and 20 % of WLF) and compared to a control biscuit.

The aim of this study was to exploit wine lees to improve the nutritional profile of biscuits by adding evidence about their impact on glycaemic index, simulated faecal microbiota, phenolic profile, as well as physico-chemical and sensory features. The actual applicability of wine lees will be investigated by evaluating possible changes in the aromatic profile thanks to sensory analysis coupled with the determination of volatile compounds. It will also be possible to confirm the improvement of the nutritional profile and the slowing down of oxidative processes.

2. Materials and methods

2.1. Wine lees flour preparation

Wine lees from different Apulian red grape cultivars of *Vitis vinifera* L. (Primitivo, Negramaro, Susumaniello) were supplied by the winery Tenuta Viglione Wine located in Santeramo in Colle (Bari). Lees from the first ranking, at the end of the alcoholic fermentation, were filtered and then freeze-dried to reach a final moisture less than 3% (Buchi Lyovapor ™L-200, Switzerland). The freeze-dried lees were crushed and sieved (Giuliani, Turin, Italy) separating particles with equal or smaller than 300 μm, according to previously study conducted on muffins and pizza base enriched with grape pomace (Difonzo, Troilo, Allegretta, Pasqualone, & Caponio, 2023; Troilo, Difonzo, Paradiso, Pasqualone, & Caponio, 2022).

2.2. Biscuit preparation

Three biscuit formulations were developed by substituting the wheat flour 00 (F0) with WLF at 10 (F10) and 20 % (F20). The amount of WLF equal to 10% and 20% were chosen to achieve the nutritional claims "source of fibre" and "high fibre" respectively according to EC Regulation 1924/2006. The ingredients used were (i) wheat flour 00 – containing 9.5 g/100 g of proteins, 1.5 g/100 g of lipid, 0.3 g/100 g of saturated fatty acids, 1.9 g/100 g of fibre, and 73 g/100 g of carbohydrates, of which 1.5 g/100 g of sugars – used in quantities of 250, 225, and 200 g in F0, F10 and F20, respectively; (ii) WLF used in quantities of 0, 25, and 50 g in F0, F10 and F20, respectively; (iii) partially skimmed milk (80 g); (iv) olive oil (70 g); (v) sugar (70 g); (vi) ammonium bicarbonate (3 g). The process consisted of (i) mixing wheat flour 00, WLF, sugar, and oil with the mechanical beater (Bomann, Knetmaschine KM 370 C B, Kempen, Deutschland) for 5 min; (ii) adding partially skimmed milk and ammonium bicarbonate and mechanical kneading for 12 min; (iii) shaping the dough manually; iv) cooking in ventilated electric oven (Smeg SI 850 RA-5 oven, Smeg S. p.A., Guastalla, Italy) for 16 min at 160 °C.

2.3. Proximate composition

Moisture content was determined by a thermobalance (Ladwag MAC 110/NP, Radwag, Wagi Elektroniczne, Poland). Protein content (total nitrogen × 6.25), ash, lipid, and total dietary fibre were determined using the AOAC, 2000, methods 979.0, 923.03, 945.38 and, 985.29 (Horwitz, 2000) respectively. Carbohydrates was calculated as a difference subtracting the total dietary fibre, protein, ash, moisture, and lipid contents from 100. All the analyses mentioned were done in triplicate.

2.4. Total phenol content and antioxidant activity

The extraction of polyphenols from WLF was carried out as described by Difonzo et al. (2023) and Troilo, Difonzo, Paradiso, Pasqualone, and Caponio (2022) with some modifications. A mixture of methanol/water (80:20, v/v) in a ratio of 1:10 (v/w) was added to 3 g of sample. After a stirring for 10 min, samples were sonicated (Ultrasonic cleaner CP104, EIA, Arezzo) at 50 Hz for 15 min at room temperature, then stirred for 30 min. Later, samples were centrifugated at 12,000 *g* for 15 min at 4 °C (MOD SL 16 R, Centrifuge, Thermo Scientific Massachusetts, USA) and the supernatants were filtered with a nylon filter (pore diameter 0.45 μm, Sigma, Ireland).

The total phenol content (TPC) was determined according to the Folin-Ciocalteu method according to Difonzo et al. (2017): 980 μL of deionized water, 20 μL of sample and 100 μL of reactive of Folin-Ciocalteu were injected into a cuvette; after 3 min, 800 μL of Na_2CO_3 to 7,5% were added to each cuvette and stored at room temperature in the dark for 60 min. After incubation, the reading of the absorbance was carried out at 720 nm and the results was expressed as mg of gallic acid equivalents (GAE)/g of WLF.

Extracts were utilized for determination of antioxidant activity, by ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid)) and DPPH (2,2-diphenil-1-picrylhydrazyl) tests, according to (Noviello et al., 2022). The ABTS assay was carried out in reaction cuvettes suitable for spectrophotometry; a quantity of sample equal to 50 μL and 950 μL of final solution of ABTS was introduced into each cuvette. After 8 min in the dark, the absorbance reading took place in the Cary 60 spectrophotometer (Agilent, Cernusco, Milan) which allows to determine the decrease in absorbance at 734 nm.

For the DPPH assay, 50 μL of each extract were added to 950 μL of DPPH solution. The absorbance was read at 517 nm using a Cary 60

spectrophotometer (Agilent, Cernusco, Milan) after 30 min of incubation in the dark. The ABTS and DPPH test results were expressed in μmol Trolox equivalents (TE)/g of WLF. Each sample was analysed in triplicate.

2.5. Phenolic compounds and anthocyanins determination

The identification and quantification of single phenolic compounds was carried out according to Troilo et al. (2022) using a UHPLC Dionex Ultimate 3000 system (HPG 3200 R S binary pump, WPS-3000 TRS autosampler, TCC-3000 R S column thermostat compartment, DAD detector, Thermo Fischer Scientific, Germering, Germany). An Acclaim ™ 120 Å C18 column (3 μm particle size, 3 mm × 150 mm length, Thermo Fischer Scientific) was used for the separation of phenolic compounds; to maintain column temperature at 30 °C was used a phase consisting of (A) water/formic acid (90:10, v/v) and (B) acetonitrile/formic acid/-water (59.9:0.1:40, v/v/v), at the constant flow rate of 0.709 mL/min. The gradient program solvent B used as follows: 0–1 min 2%; 1–5 min 12%; 5–13 min 30%; 13–21 min 100%; 21–28 min 100%; 28–32 min 2%. UV absorbance was measured at 280, 320 and 370 nm and the calibration were conducted using external standards. The standards used were the following: gallic and syringic acids, quercetin, quercetin-3-*O*-glucoside, myricetin, rutin, kaempferol, catechin, resveratrol and viniferin. Peak identification was performed by comparing the retention times and spectral characteristics of the diode array with external standards. Quantification of the phenolic compounds was expressed as mg/kg of WLF, and the analysis was performed in triplicate.

The identification of anthocyanins was carried out by HPLC-DAD according to Troilo et al. (2022). The instrument used is a Thermo Scientific (Dionez, Germering, Germania) equipped with a WPS-3000 R S autosampler, an HPG-3200 R S pump, a TCC-3000 column compartment and an L-2450 array detector diode. The mobile phase consisted of (A) water/formic acid (90:10, v/v) and (B) acetonitrile, at the constant flow rate of 0.709 mL/min. The gradient elution program for solvent B consisted of: 0–1 min 5%; 1–10 min 13%; 10–20 min 15%; 20–30 min 25%; 30–35 min 5%. The total duration was 35 min with a 20 μL injection volume; The detection was conducted at 520 nm and quantitative analysis was carried out using the external standard method based on a calibration curve obtained by injecting different concentrations of malvidin-3-glucoside. The results are expressed as mg/kg of malvidin-3-glucoside equivalent. The analysis was carried out in triplicate.

2.6. Colour and texture analyses

The CM-600 d colorimeter (Konica Minolta, Tokyo, Japan) associated with Spectramagic NX software (Konica Minolta, Tokyo, Japan) was used to determine colour of biscuits. Lightness (L*), redness (a*) and yellowness (b*) were measured. The scale used was CIE (International Commission on Illumination) L*a*b*, under a D65 illuminate. The total colour variation (ΔE), to compare the colorimetric differences between the biscuits, was calculated using the following equation:

$$\Delta E = \left[(L* - L*_0)^2 + (b* - b*_0)^2 + (b* - b*_0)^2 \right]^{1/2}$$

where $L*_0$, $a*_0$ and $b*_0$ represented the coordinates for the reference cookies while L*, a* and b* were the colour coordinates of the other samples. In the calculation, the mean values were taken as a reference; therefore, the following ΔE scale was followed to evaluate the results obtained: 0–2.0 = unrecognizable difference; 2.0–3.5 = difference recognizable by an experienced observer; >3.5 = clear difference (Brochard, Correia, Barroca, & Guiné, 2021).

Texture parameters (hardness and brittleness) of biscuits were determining by a 3-point bending test according to Pasqualone et al. (2019), providing some modifications. A texture analyser (Z1.0 TN, Zwick Gmbh & Co., Ulm, Germany) equipped with a 1 KN load cell was used. The distance between the distance bars was 4 cm with a probe speed of 5 mm/min. Hardness is the force (N) required to break the biscuits and brittleness (mm) is the distance crossed by the blade before biscuits broke (mm). Six replicated were made for each sample. The data was processed from TestXPertII software version 3.41 (Zwick Roell, Ulm, Germany).

2.7. In vitro starch hydrolysis and predicted glycaemic index

In vitro starch hydrolysis of biscuits was determined according to Difonzo et al. (2022), simulating the *in vivo* digestion of starch. Briefly, aliquots of biscuits, containing 1 g of starch, were subjected to an enzymatic process (pancreatic amylase and pepsin-HCl), and the released glucose content was measured with D-fructose/D-glucose Assay Kit (Megazyme, Wicklow, Ireland). Simulated digests were dialyzed (cut-off of the membrane: 12,400 Da) for 180 min. Aliquots of dialysate, containing free glucose, and partially hydrolysed starch were sampled every 30 min and further treated with amyloglucosidase. Then, free glucose was determined using the above-mentioned enzyme-based kit and finally converted into hydrolysed (digested) starch in biscuits. Control white wheat bread was used as the control to estimate the hydrolysis index (HI = 100). The predicted glycaemic index (pGI) was calculated using the equation pGI = 0.549 × HI + 39.71 as described by Caponio, Difonzo, et al. (2022). Each sample was analysed in triplicate.

2.8. Simulated colonic fermentation

Before to proceed with the colonic fermentation *in vitro*, retentates from dialysis were used to constitute the faecal media. By following previous standardized procedures (Vacca et al., 2021), K_2HPO_4 2 g/L, $C_2H_3NaO_2$ 5 g/L, $C_6H_{17}N_3O_7$ 2 g/L, $MgSO_4$ 0.2 g/L, $MnSO^4$ 0.05 g/L, glucose 2 g/L, inulin 4 g/L, fructo-oligosaccharides 4 g/L, Tween 80 polysorbate 1 mL/L were homogenized in distilled-water before to be sterilized (121 °C for 15 min), while retentates were cold-sterilized with filters (0.22 μm). Into the final faecal medium formulations, biscuits were present at the 2.5% (w/v). The faecal slurry was obtained as previously detailed (Vacca et al., 2023). Fresh faeces were provided by a healthy volunteer who had no antibiotics or probiotics treatments in the last 3 months before sample delivery. After collection in sterile tubes filled until 4/5 of the total volume, faeces were processed within 1 h from sampling in bags with filter (250 μm) added with distilled water (in a final ratio of 32% w/v), homogenized in a lab stomacher for 3 min, centrifuged (8000×*g*, 20 min, 4 °C). Pellets were recovered while supernatants were replaced by an equal volume of faecal medium to constitute the faecal batch. Faecal batches were incubated anaerobically at 37 °C, for 42 h, under gentle stirring conditions (150 rpm).

2.9. Viable faecal microbiota profiling

At the end of the faecal batch incubation time, viable faecal microbiota was profiled by plate-counts. After serial 10-fold dilutions in sterile saline (NaCl 0.9%) solution, to inspect the viable microbiota, Plate Count Agar (PCA); Wilkins-Chalgren anaerobe agar (WCAn); de Man, Rogosa and Sharpe (MRS) agar; M17 agar; Violet Red Bile Glucose Agar (VRBGA); and modified Bifidobacterium agar (mBifA) were used as culture media for total aerobes microbial (TAMC), total anaerobes microbial (TANMC), lactic acid bacteria (LAB – bacilli); LAB (cocci); Enterobacteriaceae; and faecal Bifidobacterium counts, respectively. Exception made for mBifA, which was purchased by Becton Dickinson GmbH (Heidelberg; Germany), all other media were purchased by Oxoid Ltd. (Basingstoke, Hampshire, England). WCAn and mBifA were anaerobically incubated, while other media were incubated aerobically. Time and temperature of incubation followed those defined by the related manufacturers.

2.10. Determination of volatile compounds

Volatile compounds of biscuits were analysed using an Agilent 6850 gas chromatograph (GC), equipped with an Agilent 5975 mass spectrometer (MS) (Agilent Technologies Inc, Santa Clara, CA, USA) and the solid phase microextraction was performed in the hand space (HS-SPME) as described by Pasqualone et al. (2019). The sample grounded was weighed into a 12 mL vial and added to 100 μL of 1-propanol solution as internal standard and a 4 mL of saturated aqueous solution of NaCl. Subsequently the vials were sealed with butyl rubber septa and aluminium caps. Before volatile compounds extraction, samples were homogenized for 2 min using a vortex shaker. Volatile compounds were extracted by exposing at 75 μm SPME fibre on carboxin/polydimetilsiloxane (CAR/PDMS) in the hand space of the sample at 40 °C for 50 min. The fibre was desorbed for 6 min in the injection port of the gas chromatograph, operating in split-less mode at 230 °C for 3.5 min. The separation of volatile compounds was done on an HP-Innowax (Agilent Technologies Inc., Santa Clara, CA, USA) polar capillary column (60 m long × 0.25 mm ID × 0,25 μm film thickness in the following conditions: inlet temperature 250 °C, flow rate of 1.5 mL/min, vector pressure (helium) equal to 30 kPa. The oven temperature was set at 35 °C for 5 min, after being increased by 5 °C per min to 50 °C and kept under isothermal conditions for 5 min; then increased to 5.5 °C and finally held constant at 210 °C for 5 min. The mass detector was set at the following conditions: interface temperature 230 °C, source temperature 230 °C, ionization energy 70eV and, scan range 33–260 amu. The internal standard used for the standardization of peaks was 1 propanol. The analysis was carried out in triplicate.

2.11. Sensory analysis

Sensory analysis was carried out by a semi-trained group of ten panellists. The analysis was carried out according to the ethical guidelines of the Laboratory of Food Science and Technology of the Department of Plant, Soil and Food Science of University of Bari, Italy. The intensity of appearance, smell, taste, and structural attributes was measured using a 9-point scale using eleven sensory descriptors. Visual and tactile analyses was evaluated through biscuits colour (0 = yellow; 9 = light brown/violet) and friability respectively (0 = very hard; 9 = very crumbly). The gustatory attributes subjected to evaluation were sweetness, salty, acidity, bitterness, astringency, and off taste intensity according to the following score (0 = unperceived; 9 = very intense). Finally, the material attributes perceived upon tasting was assessed by evaluating the following parameters: hardness (0 = soft; 9 = hard) dryness (0 = humid; 9 = very dry) and granularity (0 = no perceived particle; 9 = many particles of various sizes).

2.12. Test of oxidation stability (RapidOxy)

The oxidation test was conducted using RapidOxy (Anton Paar, Blankenfelde-Mahlow, Germany) equipped with a microprocessor that allows you to quickly measure the oxidative stability of lipid matrices when subjected to forced oxidation by increasing the temperature and pressure of oxygen. Induction time was calculated by determining the duration required to attain a 10% decrease in oxygen pressure under conditions of T = 140 °C, P = 700 kPa (De Angelis, Pasqualone, Squeo, & Summo, 2023). One gram of biscuits was shredded and crushed and subsequently analysed in triplicate.

2.13. Statistical analysis

The results were expressed as the mean ± standard deviation (SD) of two lots for each type of biscuits. Significant differences ($P < 0.05$) were determined by analysis of variance unidirectional (ANOVA), followed by Tukey test for multiple comparisons. The statistical analysis was using the statistical software Minitab (Minitab Inc., State College, PA, USA).

3. Results and discussion

3.1. Chemical and bioactive characterization of wine lees flour

Wine lees are a sludge material made of intact or partially degraded yeast cells and other insoluble particles that accumulates at the bottom of wine tanks after alcoholic fermentation. Wine lees contain significant amounts of polysaccharides, proteins, lipids and other organic molecules (De Iseppi et al., 2021). Table 1 shows proximate composition, antioxidant activity and total phenol content of WLF. The moisture content after the freeze-drying treatment was 3.18 g/100 g and the pH 3.70 attributable to the presence of tartaric acid salts precipitating together with yeasts following the alcoholic fermentation (Pérez-Serradilla & De Castro, 2008). The considerable protein content (17.26 ± 0.02 g/100 g) probably resulted from residues of yeast cell autolysis, whereas the detected ash content (15.23 ± 0.51 g/100 g) was in line with previously provided results on WLF (Rivas et al., 2021). As also confirmed by other authors, WLF showed a good amount of fibre (48.01 g/100 g) (Rivas et al., 2021; Rubio et al., 2021). Wine lees represents a source of polyphenols that exert antioxidant activity (De Iseppi, Lomolino, Marangon, & Curioni, 2020, 2021; Devesa-Rey et al., 2011; Kontogiannopoulos, Patsios, Mitrouli, & Karabelas, 2017); the main phenolic compounds and anthocyanins have been detected and quantified in WLF (Table 2). Two phenolic acids and three flavonoids were detected by HPLC-DAD; the ellagic acid was found to be the most abundant (1899.43 mg/kg) followed by quercetin (480.08 mg/kg) that is one of the main flavonols of grapes (Giacobbo et al., 2019; Iacopini, Baldi, Storchi, & Sebastiani, 2008). Still, ten anthocyanins were also identified, and the most representative concentration was provided by malvidin (955.79 mg/kg) and malvidin-3-acetyl glucoside (1080.78 mg/kg) in agreement with Romero-Díez et al. (2018) that previously discussed the impact of aging wine lees on anthocyanins concentration. Polyphenols are typical components of vegetables, fruits and cereals; in particular they are synthesized by plants in response to internal or environmental stress conditions (Pandey & Rizvi, 2009a). Polyphenols, including anthocyanins and flavonoids, are known to counteract free radicals, such as reactive oxygen and reactive nitrogen species (Abdel-Aal et al., 2008; Nimse & Pal, 2015). The beneficial health effects of anthocyanins have been demonstrated *in vitro*, in animals and in humans in the spectrum of antagonistic roles against oxidative stress, antimicrobial activity, and prevention or combating of neurodegenerative, cardiac, and metabolic disease (Khoo, Ng, Yap, Goh, & Yim, 2019; Tarone, Cazarin, & Marostica Junior, 2020; Tosti, Bertozzi, & Fontana, 2018). In addition, anthocyanins have a protective effect on sight along with carotenes and vitamin A (Khoo et al., 2019; Matsumoto, Nakamura, Iida, Ito, & Ohguro, 2006) and modulate the gut microbiota by

Table 1
Chemical composition, antioxidant activity, total phenol content of WLF.

Parameters	WLF
Moisture (g/100 g)	3.18 ± 0.10
pH	3.70 ± 0.01
Protein (g/100 g)	17.26 ± 0.02
Lipid (g/100 g)	1.76 ± 0.09
Fibre (g/100 g)	48.01 ± 0.03
Carbohydrates (g/100 g)	14.56 ± 0.32
Ash (g/100 g)	15.23 ± 0.51
TPC (mg GAE/g)	9.60 ± 0.38
ABTS (μmol TE/g)	46.60 ± 5.67
DPPH (μmol TE/g)	44.27 ± 1.07

Data are represented as means ± SD of three lots of WLF. Abbreviations: ABTS, 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid); DPPH, 2,2-diphenil-1-picrylhydrazyl; TPC, total phenol content; WLF, wine lees flour.

Table 2
Phenolic compounds and anthocyanins detected in WLF by HPLC-DAD.

Phenolic acids	WLF
Syringic acid	53.88 ± 1.13
Ellagic acid	1899.43 ± 57.63
Flavonoids	
Quercetin	480.08 ± 29.90
Myricetin	87.20 ± 0.87
Rutin	322.63 ± 21.04
Anthocyanins	
Delphinidin-3-glucoside	126.02 ± 1.66
Cyanidin-3-glucoside	55.54 ± 0.70
Petunidin-3-glucoside	187.72 ± 0.61
Peonidin-3-glucoside	97.36 ± 2.02
Malvidin-3-glucoside	955.79 ± 54.56
Delphinidin-3-acetil-glucoside	107.94 ± 1.84
Cyanidin-3-acetil-glucoside	59.84 ± 2.46
Petunidin-3-acetil-glucoside	43.58 ± 0.10
Peonidin-3-acetil-glucoside	138.00 ± 1.26
Malvidin-3-acetil-glucoside	1080.78 ± 4.45

The results are expressed in mg/kg. Data are represented as means ± SD of three lots of WLF. Abbreviation: WLF, wine lees flour.

promoting the increase of probiotics, such as bifidobacteria and lactobacilli, with beneficial effects on human health (Mattioli, Francioso, Mosca, & Silva, 2020; Park, Cho, Jin, Yang, & Yi, 2019; Zhu et al., 2018).

3.2. Characterization of experimental biscuits

3.2.1. Proximate composition, antioxidant activity, and total polyphenol content of biscuits

Table 3 reports the results of proximate composition, antioxidant activity, and TPC of biscuits. The WLF enriched biscuits showed moisture values significantly higher than the control (9.05 and 9.23 *vs* 4.8 g/100 g). This increase was probably due to the presence of fibre in the WLF, which are known for the ability to have water holding and binding capacities (Mudgil & Barak, 2019), enabling the absorption of more water than the basic ingredients, thus suggesting a potential effect on the water absorption capacity of the resulting biscuits. Similar results were also observed by Maner, Sharma, and Banerjee (2017) and Rainero et al. (2022) and after the addition of grape marc to breadsticks and biscuits, respectively. The protein content increased in biscuits added with reaching a value of 9.57 g/100 g in F20. This result is in line with the increase observed by Sharma and Aglawe (2022) when wine lees were added to yogurt. The protein increase observed in experimental biscuits is due to the protein content of wine lees, as reported in Table 1 (Delgado De La Torre, Priego-Capote, & Luque de Castro, 2015). An increase in dietary fibre proportional to the addition of WLF was observed in F10 and F20, allowing them to be labeled as "source of fibre" and "high fibre" according to the current Regulation 1924/2006 (European Commission, 2006). As reported in Table 3, experimental biscuits reached an increase of ash compared to the control biscuit, due to the ash in WLF (Table 1). This could indicate a higher mineral content, resulting in a possible biscuit enrichment in essential micronutrients (Maner et al., 2017; Sancho-Galan, Amores-Arrocha, Jiménez-Cantizano., & Palacios, 2020). The replacement of wheat flour with WLF significantly affected the TPC and antioxidant activity of biscuits as shown in Table 3. A dose-dependent increase in TPC and antioxidant activity with a higher percentage of WLF replacement was found in the F10 and F20 biscuits formulation. The highest TPC and antioxidant activity values were observed in F20. Similar results were also highlighted in the previous literature on different foods incorporated with wine lees (Alarcón et al., 2020; Hwang et al., 2009; Sharma et al., 2015).

Table 3
Proximate composition, antioxidant activity, and total polyphenol content of biscuits.

Parameters	F0	F10	F20
Moisture (g/100 g)	4.80 ± 0.03 ^{b}	9.05 ± 0.06 ^{a}	9.23 ± 0.20 ^{a}
Protein (g/100 g)	8.46 ± 0.01 ^{b}	9.94 ± 0.15 ^{a}	9.57 ± 0.32 ^{a}
Lipid (g/100 g)	16.50 ± 0.15 ^{b}	17.82 ± 0.29 ^{a}	17.25 ± 0.04 ^{ab}
Ashes (g/100 g)	0.45 ± 0.01 ^{c}	1.53 ± 0.04 ^{b}	2.68 ± 0.01 ^{a}
Carbohydrates (g/100 g)	67.12 ± 0.02 ^{a}	56.96 ± 0.02 ^{b}	53.23 ± 0.91 ^{c}
Total dietary fibre (g/100 g)	2.67 ± 0.09 ^{c}	4.70 ± 0.12 ^{b}	8.04 ± 0.33 ^{a}
ABTS (μmol TE/g)	1.16 ± 0.09 ^{c}	2.43 ± 0.08 ^{b}	4.27 ± 0.08 ^{a}
DPPH (μmol TE/g)	0.47 ± 0.00 ^{c}	2.63 ± 0.05 ^{b}	4.77 ± 0.13^{a}
TPC (mg GAE/g)	0.25 ± 0.03 ^{c}	0.70 ± 0.05 ^{b}	1.45 ± 0.01 ^{a}

Data are represented as means ± SD of three lots. Different letters (a, b, and c) in the same row indicate significant differences at $P < 0.05$ according to one-way ANOVA followed by the Tukey's HSD test. Abbreviations: ABTS, 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid); DPPH, 2,2-diphenil-1-picrylhydrazyl; F0, control biscuit without wine lees flour; F10, F20 biscuits with 10% and 20% wine lees flour replacing wheat flour; TPC, total phenol content.

3.2.2. Quantitative UHPLC-DAD analysis of phenolic compounds of biscuits

The results of quantified phenolic compounds by UHPLC-DAD are shown in Table 4. F20 contained a prevalence of ellagic acid (316.73 mg/kg), malvidin 3-glucoside (187 mg/kg) and malvidin 3-acetil-glucoside (472 mg/kg). Troilo et al. (2022) also observed the abundant presence of malvidin-3-glucoside in muffins enriched with grape pomace. Therefore, the addition of WLF allowed to obtain cookies rich in phenols and anthocyanins with potential health-related properties (Piccolella, Crescente, Candela, & Pacifico, 2019; Pojer, Mattivi, Johnson, & Stockley, 2013).

Several studies have confirmed the antioxidant, anti-inflammatory, and protective action against metabolic disorders and chronic diseases of polyphenols (Ganesan & Xu, 2017; Grosso et al., 2017; He & Sun, 2016; Liu et al., 2014; Liu et al., 2017). In addition, polyphenols act on the immune system by counteracting cellular inflammation and tumor angiogenesis (Compaore, Bakasso, Meda, & Nacoulma, 2018; Ding, Xu, Fang, & Jiang, 2020). Among the polyphenols, ellagic acid is known for its health benefits, such as high antioxidant power (Devipriya, Srinivasan, Sudheer, & Menon, 2007; Tosovic & Bren, 2020) even at low concentrations (Galano, Francisco Marquez, & Perez-Gonzalez, 2014), anti-inflammatory power (Gil, Hong, & An, 2021; Rogerio et al.,

Table 4
Phenols, flavonoids and anthocyanins detected and quantified by HPLC-DAD in biscuits.

Phenolic acids	F0	F10	F20
Syringic acid	81.30 ± 0.86 ^{a}	16.03 ± 1.12 ^{b}	16.58 ± 0.41 ^{b}
Flavonoids			
Quercetin	n.d	32.05 ± 0.93 ^{b}	75.55 ± 0.49 ^{a}
Ellagic acid	n.d.	282.94 ± 0.29 ^{b}	316.73 ± 1.56 ^{a}
Anthocyanins			
Delphinidin-3-glucoside	n.d.	57.95 ± 0.34 ^{b}	69.40 ± 2.03 ^{a}
Petunidin-3-glucoside	n.d.	52.07 ± 0.10 ^{b}	70.27 ± 1.25 ^{a}
Peonidin-3-glucoside	n.d.	49.10 ± 0.08 ^{a}	49.60 ± 0.51 ^{a}
Malvidin-3-glucoside	n.d.	139.56 ± 0.32 ^{b}	187.70 ± 0.21 ^{a}
Delphinidin-3-acetil-glucoside	n.d.	52.04 ± 0.90 ^{b}	66.77 ± 0.78 ^{a}
Peonidin-3-acetil-glucoside	n.d.	52.91 ± 0.51 ^{b}	58.88 ± 0.71 ^{a}
Malvidin-3-acetil-glucoside	n.d.	267.92 ± 1.61 ^{b}	472.83 ± 5.60 ^{a}

Data are represented as means ± SD of three lots. Different letters (a, b, and c) in the same row indicate significant differences at $P < 0.05$ according to one-way ANOVA followed by the Tukey's HSD test. Abbreviations: F0, control biscuit without wine lees flour; F10, F20 biscuits with 10% and 20% wine lees flour replacing wheat flour; n.d., not detected.

2008) antimutagenic activity (Ramadan, Ali Mohamed, Yahya Shaymaa, & ElSayed, 2019; Smart et al., 1986) and antiproliferative (Duan et al., 2020). Several studies have focused on the regulatory role of ellagic acid on cellular spectra to prevent, attenuate, or slow the growth of chronic diseases such as cardiovascular and neurodegenerative diseases (Aslan et al., 2020; Javaid et al., 2021; Kiasalari et al., 2017; Salinger-Martinovic et al., 2020), cancer (Harikrishnan, Jantan, Alagan, & Haque, 2020; Wang et al., 2019), and diabetes (Amor, Gomez-Guerrero, Ortega, Sala-Vila, & Lazaro, 2020).

Anthocyanins (such as malvidin, definidin, and peonidin) are also known for their several biological activities, particularly antioxidant activity through free radical scavenging (Khoo, Azlan, Tang, & Lim, 2017; Tena, Martín, & Asuero, 2020) and anti-inflammatory properties related to their ability to counteract cardiological, diabetic, and neuronal diseases (Alam et al., 2021; Seymour et al., 2009). Indeed, anthocyanins act on the mechanism of regulation of inflammatory cytokine signalling associated with maintaining homeostatic balance in the body (Diaconeasa et al., 2020; Khoo et al., 2017; Mattioli et al., 2020). Therefore, the addition of wine lees in the biscuit could be confer an excellent nutritional composition and potential health benefits related to the consumption of biscuits themselves (Sharma et al., 2015). However, with a view to industrial production, it will be necessary to evaluate the optimal storage strategy for proper handling of wine lees after their delivery in order to preserve bioactive and functional molecules until their use in biscuits.

3.3. *In vitro starch hydrolysis*

Considering the promising chemical composition of wine-making by-products (Caponio, Minervini, Tamma, Gambacorta, & De Angelis, 2023), such as WLF, and considering the remarkable dietary fibre content, it resulted pivotal the inspection of its impact on the pGI. Nowadays, there is an increasingly widespread trend to reduce the consumption of glycaemic carbohydrates while increasing the levels of indigestible carbohydrates and slowly digestible starch to target of broad range of beneficial effects (Agama-Acevedo, Islas-Hernández, Pacheco-Vargas, Osorio-Díaz, & Bello-Pérez, 2012; Lal, Singh, Sharma, Singh, & Kumar, 2021).

Starch hydrolysis percentage indicates the percentage of starch that is converted to glucose as a result of enzymatic digestion processes. The higher the percentage of starch hydrolysis, the more glucose is released. The glycemic index is related to the rate of increase in glucose values and thus to the speed and extent of starch digestion; in fact, reducing sugars are released during starch digestion with an increase in blood glucose (Lal et al., 2021).

Fig. 1 shows the HI and pGI of F0 and WLF biscuits (F10 and F20). According to other studies (Di Cairano et al., 2022; Ogunka-Nnoka, Ben-piakor, & Mepba, 2020), control biscuits (F0) achieved pGI and HI values of about 87.62 ± 2.14 and 87.27 ± 3.89. Interestingly, the addition of 10% of WLF (F10) determined a reduction of pGI and HI values (83.62 ± 2.16 and 79.99 ± 3.92, respectively), although not statistically significant. Instead, pGI and HI values of samples with 20% of WLF (F20) (81.26 ± 3.09 and 75.73 ± 5.62, respectively) were statistically lower than F0. Although the lipid contents of F10 and F20 were higher than F0 (data shown above in Table 3), the F20 sample achieved a lower pGI value than F0 (Heredia-Sandoval et al., 2020; Wee & Henry, 2020). Therefore, biscuits with added WLF with a lower hydrolysis rate than the control biscuits cause a moderate increase in blood sugar levels. In fact, dietary fiber in the form of fruits, vegetables, or plant-based ingredients – which are increasingly added to foods – has been shown to influence the glycemic response (Wee & Henry, 2020). This result was in line with previous studies which estimated the glycemic index of biscuits with added bioactive compounds derived from by-product of the brewing industry (Heredia-Sandoval et al., 2020).

In addition to the higher fibre content, the lower pGI in samples with WLF (F10 and F20) could also be related to the higher levels of total

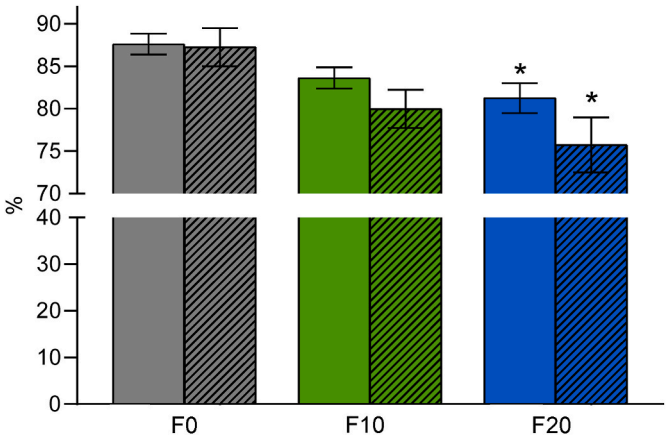

Fig. 1. Results of predicted glycemic index (pGI) (smooth bar) and hydrolysis index (HI) (textured bar) of control biscuits prepared with 100% wheat flour 00 (F0) and wine less flour 10% and 20% (F10 and F20, respectively). The values represent means of triplicates ± standard deviation; * indicates significant differences ($P < 0.05$) vs. F0, according to one-way ANOVA followed by Tukey's HSD test.

polyphenols and flavonoids compared to the control sample (F0). This hypothesis is supported by studies attributing a modulatory effect of polyphenols on digestion and carbohydrate metabolism (Hanhineva et al., 2010), as polyphenols, anthocyanins, and catechins allow to delay the starch digestion through the inhibition of digestive enzymes (i.e., α-amylase α-glucosidase), resulting in a reduction of the glycaemic index of foods (Adedayo, Adebayo, Nwanna, & Oboh, 2018; Caponio, Noviello, et al., 2022; Uğur et al., 2022). Moreover, evidence resulting from experimental intestinal cell models underlined how dietary polyphenols modulate glucose transport (Caponio, Lippolis, et al., 2022; Sun & Miao, 2020), while the reduction of pGI again confirmed the beneficial role of WLF fibre and polyphenols in controlling glycaemic response (Korompokis, Verbeke, & Delcour, 2021; Riccardi, Rivellese, & Giacco, 2008; Tosh & Bordenave, 2020). Overall, epidemiological evidence demonstrates that consumption of foods with a lower glycemic index is positively related to better health status, reduced risk of developing chronic lifestyle disorders such as type II diabetes mellitus and cardiovascular diseases (Korompokis et al., 2021).

3.4. *Viable faecal microbiota profiling after simulated colonic fermentation*

After 42 h of simulated colonic fermentation, viable cells from the faecal microbiota were profiled to investigate how a different polyphenol concentration led to differences in bacterial densities because of previous studies demonstrated how polyphenols can affect gut microbiota exerting a broad spectrum of roles and activities (Rodríguez-Daza et al., 2021). Polyphenols can serve as prebiotics promoting the growth and activity of beneficial gut bacteria like *Bifidobacterium* and lactobacilli (Plamada & Vodnar, 2021), leading to the production of short-chain fatty acids (SCFAs), as well as inhibiting the growth of harmful ones (Rana, Samtiya, Dhewa, Mishra, & Aluko, 2022). In turn, gut microbiota is pivotally involved in polyphenols metabolism because polyphenols themselves are not absorbed in the upper gastrointestinal tract while reach the colon largely intact where they can be further metabolized by gut bacteria, leading to the production of metabolites with potential health benefits (Cortés-Martín, Selma, Tomás-Barberán, González-Sarrías, & Espín, 2020). Except for Enterobacteriaceae in F20 samples, all plated bacterial groups exhibited increased densities compared to the initial inoculum (Fig. 2A). This was in line with previous studies, which assessed a decreasing trend in Enterobacteriaceae – and other bacterial groups, such as *Bacteroides fragilis* and clostridia – densities based on the

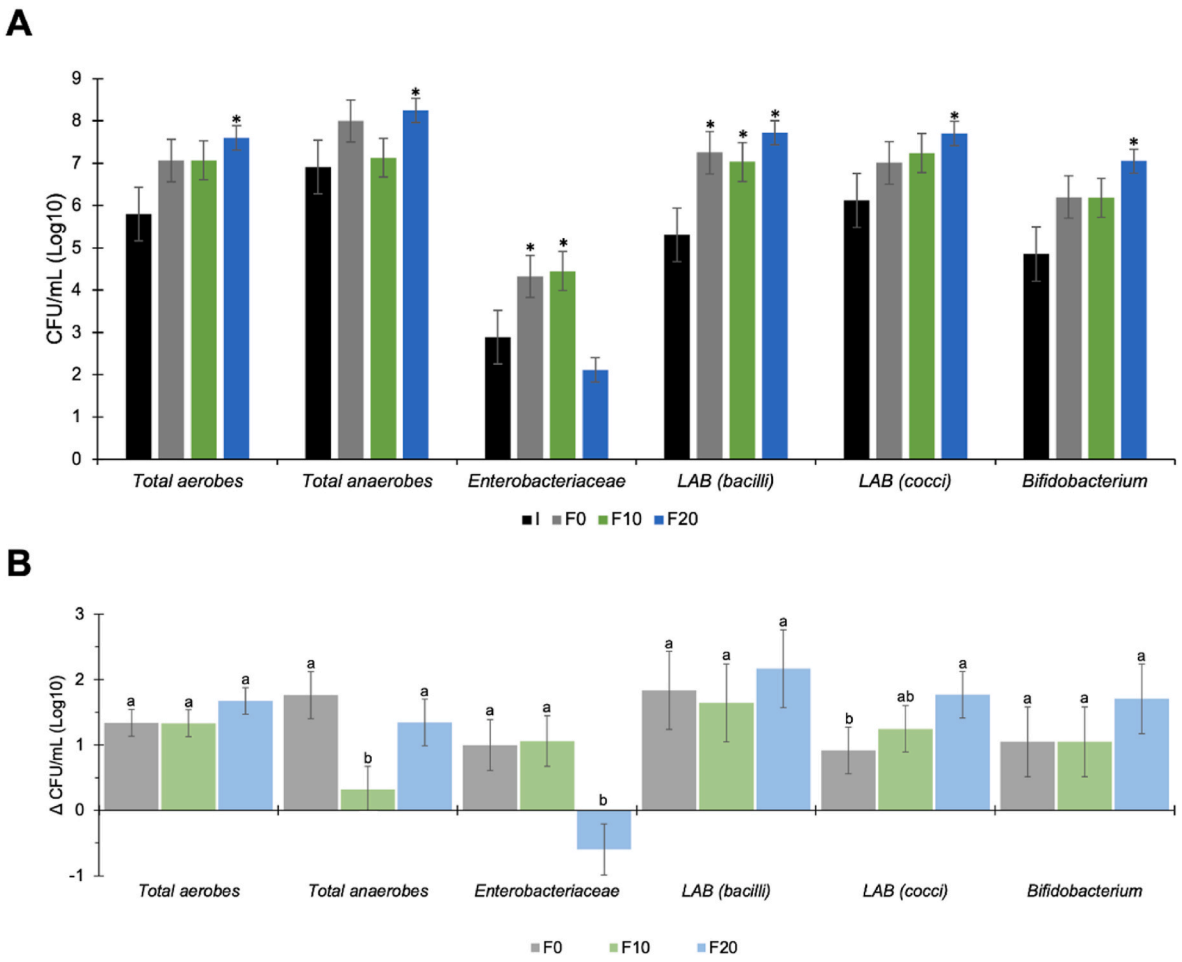

Fig. 2. Viable bacterial counts (Log10 UFC/mL) observed in fermented biscuits (F0, F10, F20) under simulated colonic conditions. The I-sample represents the cell density used as the initial inoculum. In panel A, an asterisk (*) represents a significant difference ($P < 0.05$; two-tailed Student's t-test) compared to the inoculum cell density. ($^{a\text{-}b}$) In panel B, distinct lowercase letters indicate significant differences ($P < 0.05$; two-way ANOVA).

presence of ellagic acid in samples in dose-dependent manner (Li et al., 2015). The same study noticed how both LAB and *Bifidobacterium* were, instead, able to utilize this substrate and glycosyl ellagic acid supporting the proof of concept about their prebiotic role within the gut. Focusing on quercetin-based exposure in mice, the study carried out by Ju et al. (Ju et al., 2018) stated a similar anti-microbial effect onto the abundance of *E. coli* and proteobacteria. Apart from assessing viable cell density, when compared to the initial inoculum (I), both F0 and F10 led to a significant ($p < 0.05$) increase in Enterobacteriaceae and bacillus-shaped LAB. In contrast, F20 showed increases in total aerobes and anaerobes, LAB (both bacilli and cocci), and bifidobacterial cells. In fact, as previously discussed describing health implications associated to LAB and *Bifidobacterium* activity, additional gut microbes can be positive modulated by flavonoids, likely Ruminococcaceae and *Clostridium ramosum,* leading to an increased metabolism of SCFAs (Qin et al., 2022), the same metabolites for which both LAB and *Bifidobacterium* can be flagged as health promoting bacteria. For a more detailed comparison between biscuit samples, we assessed differences using delta values (Δ, i.e., T42-T0) of cell density (Fig. 2B). Because of we used retentates from *in vitro* starch hydrolysis, the low bioavailability in sugars probably led to the absence of significant differences for total aerobes, LAB (bacilli), and *Bifidobacterium* cell density between biscuit samples. However, total anaerobes were lower ($P < 0.05$) in F10 compared to F0. Moreover, the use of retentates highlighted the role of high polyphenol concentrations found in F20 samples. This observation is consistent with prior studies (Wang et al., 2015) and resulted in a significant decrease in potentially harmful bacteria, specifically those belonging to the Enterobacteriaceae family, in comparison to both F0 and F10. As additional focus on health implications associated to the potential of these innovative biscuits, it should be considered that, although Enterobacteriaceae family belongs to the core gut microbiota, a bloom in its density were usually identified as markers of disease (Baldelli, Scaldaferri, Putignani, & Del Chierico, 2021; Boopathi et al., 2023). By contrast, it is worth noting that F20 exhibited a slight prebiotic effect, as evidenced by the significant increase in LAB (cocci) and *Bifidobacterium* cell density when compared to F0. Therefore, considering that polyphenols can either inhibit or promote the growth of lactic acid bacteria and bifidobacterial cells, depending on their concentration and composition (Plamada & Vodnar, 2021; Tabasco et al., 2011), the findings provided by our experiments *in vitro* holds promise for potential health benefits associated to the consumption of F20-biscuits.

3.5. Colour and texture analysis

The colour and texture of baked goods represent key parameters affecting the consumer acceptance based on the common expectation that light colours and friable structures should characterize these products (Conte et al., 2021).

Table 5 shows the colour and texture parameters of biscuits. The colour of experimental biscuits was strongly influenced by the addition of WLF. In particular, the colour analysis showed a progressive reduction in lightness (L*), redness (a*) and yellowness (b*) with increasing WLF amount in biscuits. The luminosity (L*) reduction of experimental biscuits is strictly associated to the colour of WLF added in the

Table 5
Colourimetric indices and textural parameters of biscuits.

Parameter	F0	F10	F20
Colour			
a*	12.19 ± 0.67 [a]	5.73 ± 0.16 [b]	3.89 ± 0.16 [c]
b*	37.88 ± 0.74 [a]	4.5 ± 0.22 [b]	1.85 ± 0.07 [c]
L*	65.29 ± 2.71 [a]	30.17 ± 0.57 [b]	23.45 ± 0.56 [c]
ΔE	–	48.92	55.94
Texture			
Hardness (N)	53.28 ± 2.03 [a]	25.70 ± 0.26 [b]	22.93 ± 0.30 [c]
Brittleness (mm)	1.72 ± 0.12 [a]	1.36 ± 0.17 [b]	1.26 ± 0.12 [b]

Data are represented as means ± SD of three lots. Different letters (a, b, and c) in the same row indicate significant differences at $P < 0.05$ according to one-way ANOVA and the Tukey's HSD test for multiple comparison. Abbreviations: F0, control biscuit without wine lees flour; F10, F20 biscuits with 10% and 20% wine lees flour replacing wheat flour.

formulations; the purple/brown colour of WLF is conferred by anthocyanins, pigments present in grape skins and grape seeds (Aksoylu, Çağindi, & Köse, 2015; Camire, Chaovanalikit, Dougherty, & Briggs, 2002). Similar results were obtained by Kohajdová, Karovičová, & Lauková. (2018) in biscuits enriched with preparations based on grape skins and seeds, which caused a reduction in L*, a* and b* parameters.

Lastly, the colour difference (ΔE) between the control biscuits (F0) and experimental biscuits with WLF (F10, F20) shows a growing difference with an increasing amount of WLF added. Similar ΔE values were observed by Kohajdová, Karovičová, and Lauková (2018) in biscuits enriched with grape peel and seed preparations.

Freshness and wholesomeness of baked goods are evaluated by analysing the hardness and brittleness of products (Liu, Cao, & Liu, 2019). Both parameters of the biscuits were determined by breaking the samples through the three-point bending test. The addition of WLF significantly affected textural parameters of biscuits; lower hardness and brittleness values were found in experimental biscuits (F10, F20) than in the control sample (F0); these reductions may be due to the higher moisture and fibre content in enriched biscuits probably relate (De Gennaro, Difonzo, Summo, Pasqualone, & Caponio, 2022; Mancebo, Rodríguez, Martínez, & Gómez, 2018; Min, Bae, Lee, Yoo, & Lee, 2010). In addition, the partial replacement of wheat flour with oenological flour may have reduced the gluten content present in the biscuit contributing to the reduction of the hardness of the biscuits as observed by Chauhan, Saxena, and Singh (2016) in biscuits enriched with amaranth flour.

3.6. Determination of volatile compounds

The identification of volatile compounds contained in the biscuits is reported in Table 6. 33 volatile compounds were identified: 3 alcohols, 10 aldehydes, 3 chitons, 5 acids, 4 esters, 3 furans and 5 pyrazines. In general, alcohols are metabolites synthesized by yeasts during fermentation and are then released into wine (Trani, Verrastro, Punzi, Faccia, & Gambacorta, 2016), which is why we find them in the lees; in fact, 1-hexanol and 1-octen-3-ol were only present in experimental biscuits. Therefore, the presence of 1 hexanol may have contributed to the appearance of hints of resin, flower, green that are typical of this compound (Ferreira, López, & Cacho, 2000). On the contrary, 2-ethyl-1-hexanol, responsible for rose and green scents (Ferreira et al., 2000), derived by lipid oxidation (Pasqualone et al., 2014) decreased in experimental biscuits probably due to more oxidative resistance conferred by polyphenols (Gutiérrez-del-Río et al., 2021). The data reported a general reduction of most aldehydes in biscuits added with WLF (F10, F20) compared to F0 (control sample without WLF). This reduction may be due to the presence of polyphenols with antioxidant activity capable of inactivating free radicals by stabilizing them through the

Table 6
Volatile compounds of biscuits.

Volatile compounds	F0	F10	F20
Alcohols			
1-hexanol	n.d.	1.81 ± 0.13 [a]	2.12 ± 0.27 [a]
2-ethyl-1-hexanol	4.80 ± 0.54 [a]	2.27 ± 0.09 [b]	2.32 ± 0.10 [b]
1-octen-3-ol	n.d.	1.07 ± 0.09 [b]	1.70 ± 0.20 [a]
Aldehydes			
Butanal, 2-methyl	16.10 ± 1.08 [a]	4.96 ± 0.31 [b]	0.27 ± 0.02 [c]
Butanal, 3-methyl	31.28 ± 2.81 [a]	2.08 ± 0.16 [b]	0.89 ± 0.11 [b]
Pentanal	2.29 ± 0.19 [b]	2.85 ± 0.15 [b]	7.59 ± 0.88 [a]
Hexanal	21.51 ± 2.79 [b]	15.63 ± 0.56 [b]	38.97 ± 5.50 [a]
Octanal	1.95 ± 0.18 [a]	1.35 ± 0.06 [b]	1.63 ± 0.17 [ab]
(E)-2-octenal	0.82 ± 0.01 [b]	1.23 ± 0.05 [a]	1.24 ± 0.10 [a]
Nonanal	12.82 ± 1.71 [a]	8.77 ± 0.52 [b]	4.73 ± 0.35 [c]
(E)-2-nonenal	n.d.	0.93 ± 0.08 [a]	0.51 ± 0.02 [b]
Decanal	2.11 ± 0.10 [a]	1.25 ± 0.05 [b]	-
Benzaldehyde	6.03 ± 0.57 [a]	4.75 ± 0.43 [b]	4.12 ± 0.48 [b]
Ketones			
Methyl-ethyl ketone	6.30 ± 0.54 [a]	2.10 ± 0.23 [b]	2.46 ± 0.08 [b]
6 metil-5-epten-2-one	n.d.	2.29 ± 0.23 [a]	1.30 ± 0.16 [b]
Acetophenone	0.82 ± 0.10 [a]	0.43 ± 0.01 [b]	0.26 ± 0.02 [c]
Acids			
Butanoic acid	n.d.	0.35 ± 0.01 [b]	0.51 ± 0.04 [a]
Acetic acid	n.d.	3.17 ± 0.11 [b]	6.17 ± 0.59 [a]
Pentanoic acid	n.d.	0.70 ± 0.06 [a]	0.77 ± 0.03 [a]
Hexanoic acid	n.d.	9.09 ± 1.22 [a]	11.19 ± 1.19 [a]
Octanoic acid	n.d.	3.05 ± 0.34 [b]	4.09 ± 0.59 [a]
Esters			
Butanedioic acid, diethyl ester	n.d.	1.72 ± 0.07 [a]	1.73 ± 0.19 [a]
Hexanoic acid, ethyl ester	n.d.	n.d.	1.15 ± 0.10 [a]
Octanoic acid, ethyl ester	1.75 ± 0.20 [c]	4.70 ± 0.35 [b]	6.44 ± 0.67 [a]
Decanoic acid, ethyl ester	2.52 ± 0.22 [c]	3.03 ± 0.30 [b]	3.45 ± 0.33 [a]
Furans			
Furfural	5.20 ± 0.58 [a]	1.03 ± 0.05 [c]	2.69 ± 0.19 [b]
Furan, 2-penthyl	2.16 ± 0.18 [a]	1.86 ± 0.20 [a]	1.08 ± 0.09 [b]
2-Furanmethanol	8.19 ± 0.77 [a]	0.47 ± 0.05 [b]	0.22 ± 0.02 [b]
Pyrazines			
Pyrazine	10.50 ± 1.76 [a]	0.50 ± 0.02 [b]	0.13 ± 0.01 [b]
Pyrazine, methyl	29.58 ± 3.36 [a]	2.04 ± 0.24 [b]	n.d.
Pyrazine, ethenyl-	2.99 ± 0.28 [a]	0.53 ± 0.02 [b]	n.d.
Pyrazine, 2,6-dimethyl	3.22 ± 0.03 [a]	2.13 ± 0.17 [b]	0.78 ± 0.06 [c]
Pyrazine, 2-ethenyl-6-methyl	1.33 ± 0.09 [a]	0.48 ± 0.05 [b]	0.16 ± 0.02 [c]

The results are expressed in μg/g. Data are represented as means ± SD of three lots. Different letters (a, b, and c) in the same row indicate significant differences at $P < 0.05$ according to one-way ANOVA followed by the Tukey's HSD test. Abbreviations: F0, control biscuit without wine lees flour; F10, F20 biscuits with 10% and 20% wine lees flour replacing wheat flour; n.d., not detected.

transfer of a hydrogen atom or a single electron (Gutiérrez-Del-río et al., 2021). Specifically, octanal, (E)-2-octenal, nonanal, (E)-2-nonenal, decanal, and benzaldehyde decreased in experimental biscuits. Octanal and nonanal are saturated aliphatic aldehydes defined as lipid oxidation markers (Marco, Navarro, & Flores, 2007), so their reduction indicates a slowdown in the oxidative process confirming the results obtained from the analysis of oxidative stability. Benzaldehyde derives from the metabolism of phenylalanine, which is an essential amino acid present in wheat (Pripis-Nicolau, De Revel, Bertrand, & Maujean, 2000), consequently the replacement of a part of wheat flour with WLF could cause its decrease. As a result, were observed a reduction in the typical almond taste of benzaldehyde (Servili et al., 2009), citrus-like, pungent, and sweet typical of octanal, nonanal and decanal, respectively (Morales, Luna, & Aparicio, 2005; Reiners & Grosch, 1998). Butanal, 2-methyl and butanal, 3-methyl fall into the Strecker aldehydes class,

and their formation occurs during the Maillard reaction involving α di-carbonyl compounds formed as a result of carbohydrate dehydration and amino acids (Delgado, Hidalgo, & Zamora, 2016; Pico, Bernal, & Gómez, 2015). Data show a decrease of 2-methyl-butanal and 3-mmethyl-butanal, responsible for the malt aroma (Reiners & Grosch, 1998), with increasing amount of added WLF and as explained by Low et al. (2006), the reduction may be associated with low pH values that counteract the formation of Strecker aldehydes.

A significant increase of hexanal, with green and grassy notes (Kesen, Kelebek, Sen, Ulas, & Selli, 2013), was observed in F20 compared to F0 and F10 probably due to the higher content of wine lees. As reported in Table 6, acids – attributable to the fermentation process of wine (Pittari, Moio, & Piombino, 2021) – were detected only in experimental biscuits, and in many cases are considered responsible of pungent notes (Morales et al., 2005).

Esters are secondary or tertiary aroma compounds formed in wine through the process of esterification, ester hydrolysis (Waterhouse, Sacks, & Jeffery, 2016, pp. 60–62) or primary or secondary fermentation (Antalick, Perello, & de Revel, 2012; Sumby, Grbin, & Jiranek, 2010). This is in line with our results in which we observed a significant increase in esters in fortified biscuits compared to the control biscuits certainly associated with the addition of wine lees as winemaking by-products.

In addition, WLF can retain esters in the matrix (Gallardo-Chacón, Vichi, López-Tamames, & Buxaderas, 2009), as was also observed by Martín-Garcia, Riu-Aumatell, and López-Tamames (2022) in bread supplemented with wine lees. In addition to WLF, the addition of other oenological by-products such as grape mark in extract in biscuits (Pasqualone et al., 2014) leads to an increase in esters. The esters significantly contribute to the aromatic profile of the wine giving floral and fruity notes (Lasik-Kurdy's, Majcher, & Nowak, 2018; Waterhouse et al., 2016, pp. 60–62). Consumer appreciation of a food product is the key factor for product success. Among volatile compounds, those formed during the Maillard reaction have a greater influence on the aroma of baked goods (Caponio et al., 2022; Starowicz, 2021), in particular their formation depends on temperature and time, pH, the amount of water, and the sugars and amino acids involved in the reaction (Jousse, Jongen, Agterof, Russell, & Braat, 2002). Among the compounds that play an important role in the conferring of aromas of baked goods there are pyrazines (hints of cooked, toasted, toasted, baked cereals) and furans (hints of sweet, burnt, pungent, caramel-like) (Van Boekel, 2006). Therefore, aldehydes, furans, pyrazines, and ketones reduction in experimental biscuits compared to control could also be associated with the presence of phenolic compounds capable of scavenging radicals produced following the Maillard reaction (Mildner-Szkudlarz, Siger, Przygoński, Radziejewska-Kubzdela, & Zawirska-Wojtasiak, 2022). Mildner-Szkudlarz et al. (2017) also found the same trend, in fact they observed the inhibitory action of polyphenols towards the production of pyrazines.

3.7. Test of oxidation stability

Lipid oxidation is the main deterioration reaction of the fat fraction of foods (Calligaris, Pieve, Kravina, Manzocco, & Nicoli, 2008) that negatively affects shelf-life, promotes the development of off-flavours, and increases the possibility of occurrence of potentially harmful compounds (Van Dyck, Verleyen, Dooghe, Teunckens, & Adams, 2005).

The potential antioxidant activity of wine lees to counteract lipid oxidation in biscuits was tested by the oxidative stability test. Fig. 3 shows the results of the oxidative test expressed as the induction time – in minutes – that positively correlated to oxidative resistance (Caruso et al., 2017).

The induction time was equal to 34.01, 45.13, and 52.10 in F0, F10 and F20, respectively, and, therefore, it was observed a significant increase in induction time when WLF was added to the biscuits. A direct proportionality between the amount of added WLF and the induction time was observed.

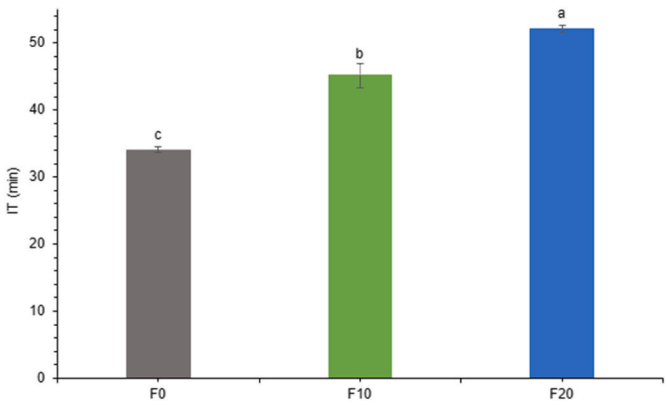

Fig. 3. Oxidative stability (RapidOxy) of biscuits. Data are represented as means ± SD of three lots and different letters (a, b, and c) mean a significant difference at $P < 0.05$ according to one-way ANOVA followed by Tukey's HSD test. Abbreviations: F0, control biscuit without wine lees flour; F10, F20 biscuits with 10% and 20% wine lees flour replacing wheat flour; IT, induction time.

Data obtained from the RapidOxy confirmed the results of the analysis of antioxidant activity because the increased induction time observed in F10 and F20 suggested the protective effect of WLF on the oxidation of experimental biscuits. Similar results were observed by Difonzo et al. (2018) and Gruppi, Giuberti, Duserm Garrido, & Spigno, (2023) in baked snacks and biscuits enriched with olive leaf extract and different fibres respectively. Noteworthy, the addition of WLF in biscuits leads to a greater oxidative stability. Increased oxidative resistance results in a potential increase in shelf-life and better preservation of certain health properties (Caruso et al., 2017; Van Dyck et al., 2005).

3.8. Sensory analysis

Fig. 4 shows the sensory profile of the biscuits. In general, WLF addition significantly influenced sensory properties of experimental biscuits. F10 and F20 showed an increase in colour proportional to the addition of WLF. The loss of colour typical of conventional biscuits observed in experimental samples (F10, F20), in accordance with instrumental analysis, is due to the addition of WLF as also observed by Sharma et al. (2015) in ice cream enriched with wine lees. The friability showed differences among experimental biscuits (F10, F20) compared to the control sample (F0), specifically, the WLF addition led to a friability increase directly proportional to the increase in the percentage of substitution.

Regarding gustatory descriptors, no significant differences were found in the sweetness and saltiness taste between enriched biscuits (F10, F20) and control sample (F0) as observed by Rainero et al. (2022) in breadsticks fortified with red grape pomace. The acidity, bitterness, astringency, and off-taste were more perceived by increasing the levels of WLF adde 2023d. The increase in off-taste intensity and acid taste in experimental biscuits, as reported by Olt et al. (2023) and Tolve, Pasini, Vignale, Favati, & Simonato (2020) in biscuits and pasta enriched with grape pomace, detected during the sensory analysis confirm the results of the volatile profile influenced by acids. The bitterness and astringency sensation can be attributed to the presence of polyphenols in WLF due to the interaction that occurs between polyphenols and saliva (Davidov-Pardo et al., 2012). Similar results were found in previous studies which respectively used a marc extract, a preparation based on grape skins and grape seeds in biscuits and red grape pomace in breadsticks (Kohajdová et al., 2018; Mohamed Ahmed et al., 2020; Rainero et al., 2022).

Finally, texture attributes such as hardness, dryness, and granularity were influenced by WLF addition. Specifically, the lower hardness of the experimental biscuits (F10, F20) compared to the control sample (F0)

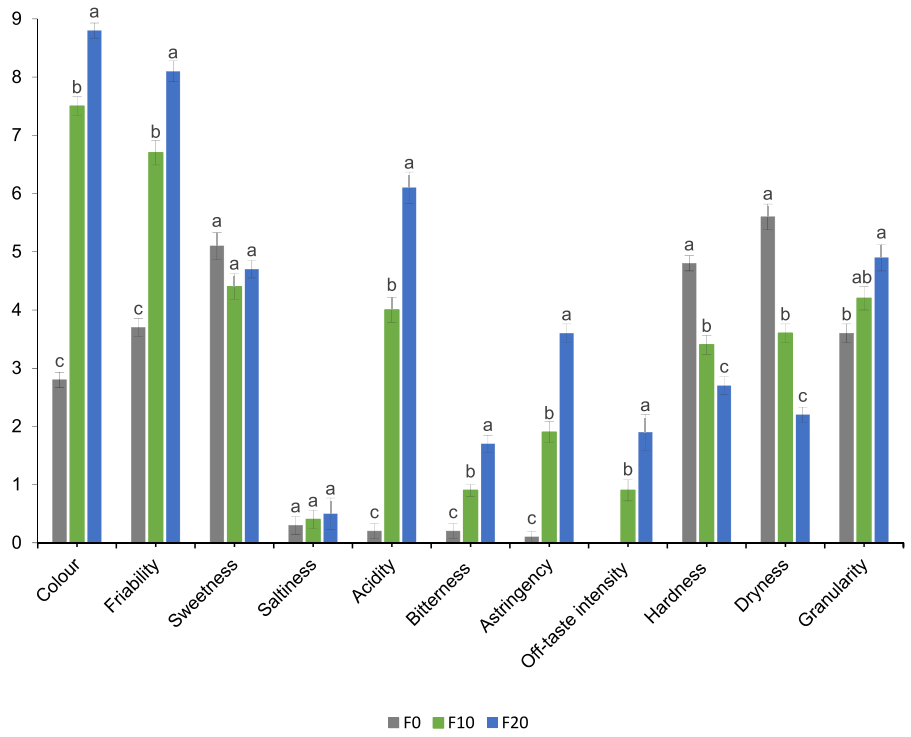

Fig. 4. Results of the sensory analysis of the biscuits; data are represented as means ± SD of ten panellists. Different letters (a, b, and c) mean a significant difference at $P < 0.05$ according to one-way ANOVA followed by Tukey's HSD test.

agrees with the instrumental results, probably due to increased moisture (Table 5) and to the dilution of gluten caused by WLF which does not contain gluten (Petchoo, Jittinandana, Tuntipopipat, Ngampeerapong, & Tangsuphoom, 2021). Similarly, the dryness of F10 and F20 was less than the control; this could be associated with the increased moisture of the experimental biscuits. Therefore, as reported by Miolla et al. (2023) it is necessary to identify consumer segments that are attentive to health aspects and would be more inclined to accept foods with different characteristics, provided they are improved nutritionally.

4. Conclusions

In the current study wine lees were exploited to develop fortified biscuits with polyphenols and dietary fibre. The substitution of wheat flour with 10% and 20% WLF improved the nutritional composition of biscuits, allowing the nutritional claims "source of fibre" and "high fibre", as well as increasing the amount of bioactive compounds such as phenolic acids, flavonoids and anthocyanins. The addition of WLF positively influenced pGI of experimental biscuits, reaching lower value compared to the control biscuits. Viable faecal microbiota profiling after simulated colonic fermentation resulted in a reduced cell density of Enterobacteriaceae and increased growth coccus-shaped LAB, and Bifidobacterium. Moreover, the fortified biscuits seem to be more stable to the oxidation, as highlighted by RapidOxy. Based on these promising results, the formulation will be further optimized to improve the sensory features of biscuits and carry out a consumer acceptability evaluation. Finally, it will be necessary to select the storage conditions of the wine lees that allow to use a sustainable environmental and economic approach while preserving the bioactive and functional molecules.

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CRediT authorship contribution statement

Giusy Rita Caponio: Writing – review & editing, Formal analysis, Data curation. **Roberta Miolla:** Writing – review & editing, Formal analysis, Data curation. **Mirco Vacca:** Writing – review & editing, Formal analysis, Data curation. **Graziana Difonzo:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Maria De Angelis:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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