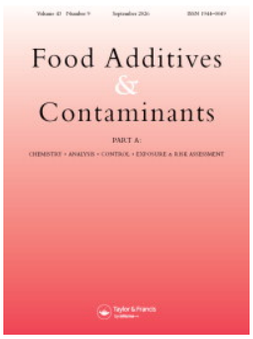

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Cork–polypropylene composites for food contact: a comprehensive study of morphology, migration, chemical safety, and sensory analysis

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ABSTRACT

The development of sustainable food contact materials (FCMs) requires not only reduced environmental impact but also demonstrated safety. Cork–polypropylene composites (CPCs), incorporating unmatured cork and waste generated in cork stopper production, represent a promising alternative; however, their safety remains insufficiently addressed. This study provides a comprehensive safety assessment of CPCs containing 15% cork granulates derived from matured and unmatured cork. A multi-analytical approach combining morphological, thermal, and chemical characterisation (SEM, FTIR-ATR, GC-FID, GC-MS, LC-MS, ICP-OES) was integrated with migration testing under repeat-use conditions and sensory evaluation. Genotoxicity was assessed using the Ames bacterial reverse mutation test on migration extracts. Migration results showed low release of both polypropylene-related additives and cork-derived compounds, with furanic substances identified as process-related migrants but decreasing across repeated use cycles and remaining at low levels. No migration of high molecular weight cork constituents (triterpenoids, sterols) was observed in aqueous simulants. All quantified substances complied with regulatory limits. Ames test results demonstrated no mutagenic activity for any migration extract, confirming the absence of DNA-reactive hazards. Overall, CPCs exhibited stable structure, low sensory impact, and favourable chemical and toxicological profiles. This study provides new evidence supporting the safe use of cork-based composites in food-contact applications and advances the integration of bio-based materials within a circular economy framework.

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Unmatured cork; waste from cork stoppers; metals in bio-based FCM; NIAS; ames test

Introduction

Cork, obtained from the outer bark of *Quercus suber* L., is a renewable natural material widely recognised for its unique cellular structure and multifunctional properties (Silva et al. 2005; Faria et al. 2011; Aroso et al. 2017). Its honeycomb-like morphology, composed mainly of suberin, lignin, and polysaccharides, confers elasticity, low thermal conductivity, and high resistance to gas and liquid permeation (Pereira 1988; Faria et al. 2011; Esteves et al. 2017). These characteristics have traditionally supported applications such as wine stoppers, flooring, insulation, and packaging, while more recent developments have explored cork-derived compounds for functional uses in cosmetics and pharmaceutical products (Ortega-Fernandez et al. 2006; Knapic et al. 2016; Tedjditi et al. 2020; Carriço et al. 2023; Rego et al. 2023).

In cork-producing regions such as Portugal, Spain, and Algeria, cork represents an important economic and ecological resource. However, only a limited fraction of harvested cork is suitable for high-quality natural stoppers, generating substantial quantities of by-products during harvesting and industrial processing (Pereira 2007). Cork oak trees produce three main types of cork during their harvesting cycle: virgin cork from the first harvest, reproduction cork from the second harvest, and matured cork (*Amadia*) from the third harvest onwards (Pereira 2007; Tedjditi et al.

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2020). While *Amadia* cork is widely used in high-performance applications, unmatured cork is generally considered less suitable due to its irregular microstructure and collapsed cellular walls and is therefore typically directed towards lower-value applications (Gil 2015; Knapic et al. 2016; Tedjditi et al. 2020; Svetlana et al. 2024). The effective valorisation of these by-products is therefore essential for improving the economic and environmental sustainability of the cork industry.

The structural and chemical characteristics of cork vary depending on its maturity. Matured cork (MC) generally contains 38–45% suberin, 21–23% lignin, and 15–18% polysaccharides, forming a relatively uniform cellular structure associated with good elasticity and resilience (Pereira 1988; Ferreira et al. 2012; Aroso et al. 2017). In contrast, unmatured cork (UMC) exhibits slightly lower suberin content, higher lignin levels, and greater heterogeneity in cell size and wall thickness, which can influence its mechanical and thermal behaviour (Şen et al. 2014; Mota et al. 2016). Differences in extractive composition are also observed, as early-stage metabolites are more prevalent in UMC and can be detected by techniques such as FTIR-ATR and GC-MS/MS (Svetlana et al. 2024). These compositional variations affect the thermal stability and chemical profile of cork, parameters that become particularly relevant when cork is incorporated into thermoplastic matrices and subjected to high processing temperatures (Gil 2009; 2015; Knapic et al. 2016; Haurie et al. 2023).

The search for renewable and lower-carbon materials has intensified in recent years in response to circular economy policies and European environmental strategies. Packaging and food-contact materials (FCMs) represent a key sector in this transition, as they must simultaneously meet performance requirements, regulatory safety standards, and sustainability goals. European Regulation (EU) 2025/40 promotes the reduction of plastic waste and encourage the development of alternative materials derived from renewable resources (Silva et al. 2020; Gaspar & Braga 2023). Within this context, cork has attracted increasing attention as a bio-based filler capable of partially replacing fossil-derived polymers while contributing to reduced material density and improved environmental performance (Silva et al. 2005; Pereira 2007; Palombini et al. 2023).

Cork–polymer composites (CPCs) have therefore been explored in combination with thermoplastic matrices such as polypropylene (PP), polyethylene (PE), and polylactic acid (PLA) although much less for FCMs (Fernandes et al. 2011; Martins and Gil 2020). The incorporation of cork granulates can enhance thermal insulation, dimensional stability, and acoustic performance while reducing the overall density of the material (Fernandes et al. 2014; Petlitckaia et al. 2024). The final properties of CPCs depend strongly on factors such as cork particle size, dispersion within the polymer matrix, and interfacial compatibility, which can be improved through the use of coupling agents such as maleic-anhydride-grafted polypropylene (PP-g-MA) (Abdallah et al. 2010; Benabdallah et al. 2024). Recent industrial developments demonstrate that cork residues generated during stopper manufacturing can be incorporated into polyolefins using conventional extrusion and injection moulding processes to produce lightweight and recyclable composite materials (Suffo et al. 2024).

Despite these technological advances, the use of cork–polymer composites in food-contact applications requires careful evaluation of chemical safety. According to Regulation (EC) No 1935/2004 and Regulation (EU) No 10/2011, materials intended to come into contact with food must not transfer substances in quantities that could endanger human health or alter the composition or sensory properties of food. Natural materials such as cork contain a wide range of extractable compounds, including phenolic acids, aldehydes, terpenes, and ellagitannins, which may potentially migrate under use conditions (Azevedo et al. 2014; Pinto et al. 2023). In addition, polymer processing at elevated temperatures may generate cork degradation products or oligomeric non-intentionally added substances (NIAS), further increasing the complexity of the chemical profile of CPCs (Vera et al. 2018). Consequently, migration testing combined with advanced analytical techniques is required to

identify and quantify both cork-related and polymer-derived compounds. Additionally, the incorporation of cork particles may alter the migration behaviour of polymer-derived migrants. Therefore, the migration assessment of additives and NIAS of the polymer matrix when used as a CPC, needs a dedicated study.

Complementary analyses are also important for a complete evaluation of food-contact materials. Morphological analysis can reveal potential structural changes in the composite after exposure to food simulants, while sensory evaluation allows the detection of possible odour or flavour transfer, which is critical for consumer acceptance (Cincotta et al. 2018; Gancel et al. 2023). These aspects need to be evaluated under repeat-use migration tests according to EU Regulation 10/2011.

Although numerous studies have examined the mechanical and physical properties of cork–polymer composites (Fernandes et al. 2014; Petlitckaia et al. 2024), information regarding their migration behaviour, chemical safety, and sensory performance in food-contact conditions remains limited. In particular, the influence of cork maturity (unmatured versus matured cork) on the chemical profile and migration behaviour of CPCs has not been investigated.

The objective of this study was to perform an integrated safety assessment of CPC intended for food-contact applications, combining physicochemical characterisation, migration analysis, and toxicological evaluation. Cork granulates from matured and unmatured sources were characterised and incorporated into polypropylene to produce CPCs, which were subsequently evaluated under simulated food-contact conditions. Migration testing, including repeat-use scenarios, was conducted to identify and quantify both intentionally added substances (IAS) and non-intentionally added substances (NIAS). In addition, the genotoxic potential of migration extracts was assessed using the Ames bacterial reverse mutation test. By linking material composition, migration behaviour, and biological response, this study aims to provide a comprehensive and regulatory-relevant evaluation of CPC safety, supporting the use of cork by-products in sustainable food-contact materials.

Materials and methods

Cork granules

Cork samples were supplied by Amorim Cork Solutions, S.A. (Portugal). The cork granulate (BD 0.5/1) used to produce the composite were a mixture of cork obtained from sources with different maturities: (a) Unmatured cork (UMC), derived from the first and second harvests of *Quercus suber,* unsuitable for stopper production; and (b) Matured cork (MC; *Amadia*) originating from residues of natural cork stopper production (third and subsequent extractions) and representative of fully developed cork bark. Cork from both sources was mixed and subsequently ground to produce the granulate, with UMC incorporated at 35-55% and MC at 45-65%. The granulate had a bulk density of 50–60 kg/m^3, particle size of 0.5–1.2 mm and a residual moisture of 4–5%. Samples of granules of UMC and MC separately were also provided for characterisation.

Composite cork-polymer

The cork–polypropylene composite (CPC) was produced *via* extrusion to incorporate 15% (w/w) cork granulate into the polymer matrix PP (PP Sabic® 595 A-00900). This polymer is approved for food contact in accordance with Regulations EC 10/2011 and EC 2023/2006, according to the Declaration of Compliance supplied. No coupling agents, surface compatibilizers, or additional chemical additives were used in the preparation of these composites. The testing specimens were produced by injection moulding. Control samples consisted of 100% PP without cork. Figure 1 shows the PP and CPC testing specimens, moulded as discs with dimensions of 4.5 cm in diameter and 0.5 cm thickness.

These developed CPCs are intended for repeat-use food contact applications, including kitchen utensils (such as cutting boards and handles for ladles and cutlery), as well as reusable

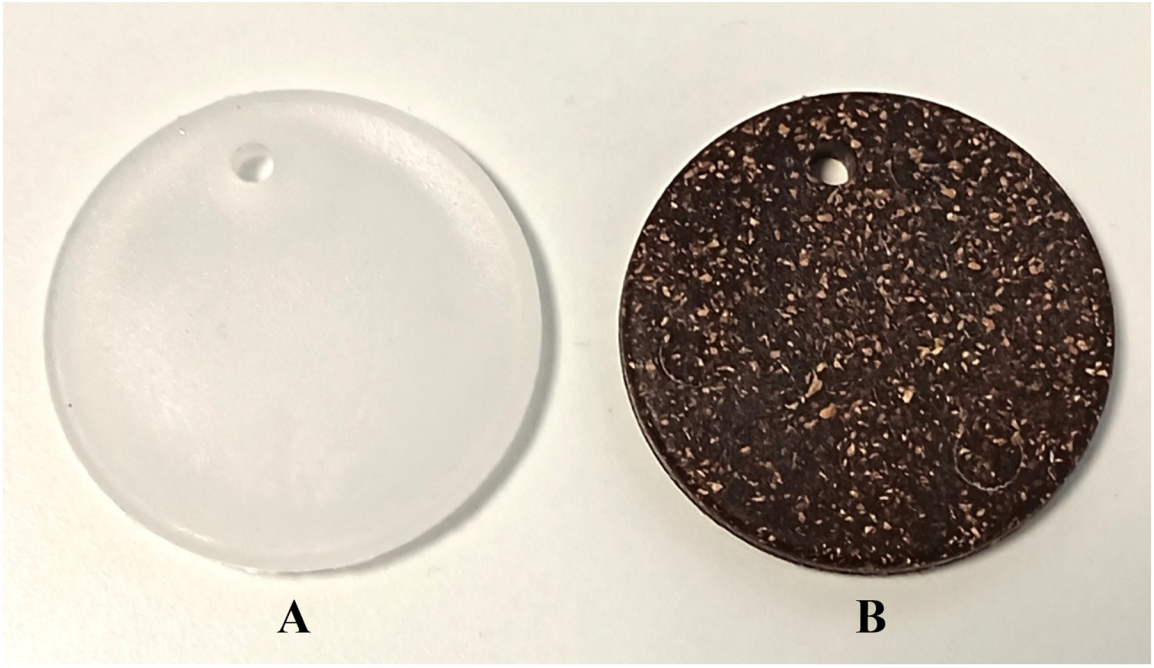

Figure 1. Visual aspect of samples used for testing: (A) PP – polypropylene and (B) CPC – cork–polypropylene composite (Ø 4.5 cm × 0.5 cm).

containers for food transport and storage (such as lunch boxes and screw lids).

Characterisation of the Cork

Morphology (UMC and MC)

Scanning electron microscopy (SEM) was employed to investigate the morphological differences between UMC and MC samples. Both the longitudinal and cross-sections of the cork granules were examined to assess variations in cellular structure and surface characteristics. SEM images were acquired using a Field Emission Gun Scanning Electron Micro-scope (FEG-SEM; JEOL JSM-7001F, Tokyo, Japan) operating at 5 kV, using a secondary electron detector (SED) under vacuum (1.0 Pa). Prior to analysis, cork samples were coated with a 20 nm thick layer of gold/palladium (Au/Pd) using a Quorum Technologies sputter coater (model Q150TES, West Sussex, UK) to enhance surface conductivity. Quantitative image analysis was performed using ImageJ software (version 1.44p, National Institutes of Health, USA). Measurements included the cell base area (μm^2) and radial cell-wall thickness (μm), determined from at least 50 cells per sample, analysed across three independent SEM images, for each cork type.

The apparent density of UMC and MC samples was determined according to ISO 1183-1:2019 using the immersion method (Method A) in deionised water at 22–23 °C. Measurements were performed using an analytical balance (METTLER TOLEDO®) after conditioning the samples at 23 °C and 50% RH. For each cork type, three independent measurements ($n = 3$) were performed, and results are reported as mean ± SD.

Thermal analysis of Cork (UMC and MC)

Thermal analysis of the UMC and MC samples was carried out using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA was performed according to ISO 11358:1997(E) 1997, under a nitrogen atmosphere, from 30 °C to 700 °C at a heating rate of 20 °C·min^{-1}. DSC analysis was conducted using a DSC 204 F1 Phoenix instrument (Netzsch, Germany. Samples were sealed in aluminium crucibles and subjected to a heating–cooling–reheating programme. The temperature was first increased from 20 °C to 200 °C, cooled back to 20 °C, and then reheated to 200 °C, at a rate of 10 °C min^{-1} under nitrogen (40 mL min^{-1} purge). This temperature was selected because according to the manufacturer's technical guidelines, the maximum recommended temperature for injection/extrusion moulding of the PP is 190 °C.

FTIR and multivariate modelling of Cork (UMC and MC)

FTIR spectra of the cork samples were acquired using a PerkinElmer Spectrum BX FTIR System (USA) with a deuterated triglycine sulphate (DTGS) detector. The spectra were collected in diffuse reflectance mode using a PIKE Technologies Gladi attenuated total reflectance (ATR) accessory equipped with a monolithic diamond crystal, covering the wavenumber range of 4000–600 cm^{-1}, with a resolution of 4 cm^{-1} and 32 co-added scans per spectrum. Samples were placed directly on the ATR crystal under constant pressure to ensure consistent contact. The ATR crystal was cleaned, and a background spectrum was acquired before each measurement. To account for cork's natural heterogeneity, multiple regions from the inner layers of both UMC and MC samples were analysed, including areas with higher suberin content. This sampling strategy aimed to provide a representative spectral characterisation of each cork type and to avoid pseudo replication. For each sample, three distinct spots were analysed, and three spectra were acquired per spot (instrumental replicates), resulting in a total of nine spectra per sample.

FTIR spectra were modelled using partial least squares discriminant analysis (PLS-DA) (Alsberg et al. 1998). Prior to modelling, spectra were pre-processed using standard normal variate (SNV) normalisation and the Savitzky–Golay filter (15 smoothing points, 2nd-order polynomial, and 2nd derivative). Data were subsequently mean-centred. Spectral pre-processing and multivariate modelling were performed using MATLAB R2023a (Math-Works, Natick, MA) with PLS Toolbox 9.2.1 (Eigenvector Research, Manson, WA).

Chemical characterisation of Cork (UMC and MC)

Volatiles and semi-volatiles

A semi-quantitative GC-MS screening was conducted using a GC–MS system (Scion 456-GC, Bruker, Massachusetts, USA) following solvent extraction with ethanol and iso-octane. Approximately 0.10 g of UMC and MC samples were extracted in duplicate with 4 mL of each solvent at 60 °C for 24 h. Chromatographic conditions: injection was performed in splitless mode (320 °C, with a splitless time of 0.75 min; 1 µL volume); separation in a Zebron 5 ms Plus column (30 $m \times 0.25$ mm × 0.25 µm); the oven temperature ranged from 50 °C (3 min) to 320 °C at 10 °C/min, held for 15 min. Mass spectrometer conditions: electron ionisation (70 eV) and full scan (m/z 33–700).

Compound identification was supported by NIST 2.4 spectral library matching and retention index (RI) comparison with literature data. Match and reverse match factors were reported to indicate identification confidence, with typical values > 700 considered acceptable for the purpose. For semi-quantification of the compounds detected, deuterated dodecane (26.2 mg/L) was added as an internal standard and analysed under identical conditions. The same relative response factors were assumed, and results were expressed in mg/g of sample. The limit of detection (LOD) was estimated for the internal standard at $S/N = 3$, corresponding to 0.03 mg/L (0.001 mg/g of cork sample).

Metal content of Cork

Metal content of each cork type and of the mixture used to produce the CPC was assessed using the ICP-OES for Al, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Zn. Samples (2 g) were dry-ashed at 450 ± 15 °C, dissolved in concentrated HNO_3, and diluted to 20 mL with ultrapure water and analysed (Avio 220 Max, PerkinElmer, USA) equipped with an S23 autosampler. For hydride-forming elements (As, Sb, Hg), hydride-generation ICP-OES (ICP-OES-HG) was used. Two grams of sample were extracted with 50 mL of 0.556% (v/v) HNO_3 for 24 h, filtered, and pre-reduced with HCl and KI. Hydride-generation standards (0.002–0.010 mg L^{-1}) were prepared by diluting a 1000 mg L^{-1} mercury standard (Merck) and ARISTAR® multi-element stock, followed by the same pre-reduction procedure and 1 h rest in the dark to stabilise hydride-forming species. Calibration for ICP-OES-HG included a reagent blank and five calibration levels, each analysed in duplicate. Instrumental parameters for the hydride flow system and ICP-OES are listed in Table S1.

Screening analyses and semi-quantification of potential migrants from composite

Headspace screening (HS-GC-MS)

Headspace GC–MS analysis was performed using the same GC–MS system described above. Automated sample injection was executed *via* a

static headspace (S-HS) technique using a CombiPAL autosampler equipped with a heated gas-tight syringe (CTC Analytics, Zwingen, Switzerland). Each sample (1 g) in duplicate was cut into small pieces and analysed by headspace GC-MS after 1 h of extraction at 121 °C. Chromatographic conditions: Zebron 5 MS Plus (30 *m* × 0.25 mm ID × 0.25 µm); injector 250 °C; injection volume 1 mL (split 1:10); oven programme 40 °C (5 min), increasing at 10 °C min^{-1} up to 320 °C. Helium was used as carrier gas (1 mL min^{-1}). The mass spectrometer operated in EI mode (70 eV), scanning m/z 33–350. Semi-quantification was based on deuterated dodecane (482 mg L^{-1}) and furfural (208 mg L^{-1}) standards.

Liquid extraction screening (LE-GC-MS)

For liquid extract analysis, 1 g of each sample (in duplicate) was extracted with iso-octane and ethanol (5 mL each) for 24 h at 60 °C, followed by direct GC–MS injection using the system and conditions described in Subsection Volatiles and semi-volatiles.

Overall migration from composite

Migration testing was performed according to the rules established in Regulation (EU) No 10/2011 for OM2 conditions (10 days at 40 °C), which cover long-term storage at room temperature or below, including hot-fill conditions. Furthermore, since the developed CPCs are intended for repeat-use articles (as specified in Section Composite cork-polymer), and in strict compliance with Article 11(3) of Regulation (EU) No 10/2011, a repeated migration testing protocol was carried out. For this purpose, discs of 0.9 dm^2 were immersed in 100 mL of food simulants (10% (v/v) ethanol, 3% (w/v) acetic acid, and vegetable oil) using a surface-to-volume ratio of 6 dm^2 kg^{-1}. The procedure involved three consecutive exposure cycles of 10 days at 40 °C on the same sample, using a fresh portion of food simulant for each cycle, with compliance evaluated based on the migration levels obtained in the third cycle.

For the aqueous simulants (10% ethanol and 3% acetic acid), the overall migration was determined gravimetrically in accordance with the EN 1186 standard; after each exposure cycle, the simulants were evaporated to dryness, and the mass of the non-volatile residue was determined using an analytical balance. For the vegetable oil simulant, overall migration was calculated by the weight loss of the sample, where the absorbed oil residues were quantified by GC-FID using a gas chromatograph (VARIAN CP-3800, Varian, USA). The samples were submitted to pentane extraction, using triheptadecanoin as internal standard, before GC-FID analysis with the following chromatographic conditions: column CP WAX 58 (FFAP) 50 *m* × 0.25 mm; injector/detector temperature 250 °C; oven 100 °C for 1 min increasing at 20 °C min^{-1} up to 160 °C and then at 2 °C min^{-1} up to 220 °C; volume of injection 1 µL. The calibration was performed in the range of 0–80 mg oil, showing linearity (R^2 = 1.0) and a limit of quantification LOQ < 3.0 mg dm^{-2}.

Specific migration from composite

Specific migration of individual substances was determined at 10 days at 60 °C. Discs (0.9 dm^2) in triplicate, were immersed in 100 mL of simulants (10% ethanol, 3% acetic acid and vegetable oil) using the surface-to-volume ratio of 6 dm^2 kg^{-1}. The PP control was tested under identical conditions to differentiate cork-related migrants from those inherent to polypropylene. Repeated use conditions were applied. Determinations after 10, 20 and 30 days of contact (with the same portion of simulant) were also performed. The migration solutions were subsequently analysed by GC-MS and LC-MS for quantitative assessment of identified IAS and NIAS.

Analysis of volatile and semi-volatile migrants

Migration solutions were screened by HS-SPME-GC-MS using a DVB/CAR/PDMS fibre. Samples were incubated at 85 °C (oil) or 40 °C (aqueous simulants) for 45 min, with 15 min desorption prior to analysis. Chromatographic conditions: column Zebron 5 MS Plus (30 *m* × 0.25 mm × 0.25 µm); injector 250 °C; oven temperature 40 °C (10 min), increasing at 5 °C min^{-1} up to 300 °C (3 min); carrier gas helium 1 mL min^{-1}. Detector conditions: EI 70 eV; m/z

33–350. Semi-quantification used deuterated dodecane (0.045 mg L^{-1}) as internal standard and calibration with furfural (0.384 mg L^{-1}). For polar semi-volatiles such as m-diacetylbenzene, aqueous simulants were extracted (1:1) with hexane containing deuterated dodecane (24.6 mg L^{-1}) before GC-MS analysis.

A liquid–liquid extraction GC-MS (LLE-GC-MS) procedure was applied to aqueous simulants for determination of cork triterpenoids and sterols (β-sitosterol, lupenone, lupeol, sitost-4-en-3-one, friedelin). The same chromatographic conditions were applied and the detection was performed by full scan in the range of m/z 33–700. Quantification was made with calibration using deuterated dodecane and analytical standards.

Analysis of non-volatile Cork migrants

For vegetable oil simulant, non-volatile cork-derived compounds were extracted with methanol (1:1), vortexed 2 min, centrifuged (2500 rpm, 2 min), and the supernatant concentrated under nitrogen to 1 mL. LC-MS/MS was operated in ESI positive mode, using 0.1% formic acid in water (A) and 0.1% formic acid in methanol (B) as mobile phases with the gradient: 30% B (0–2 min), 100% B (10–12 min), 30% B (15 min). Chromatographic conditions: injection volume 20 µL; spray voltage 3.5 kV; probe temperature 450 °C. Calibration was performed with reference standards in methanol, and the following LOD depending on analyte (furfural, sitost-4-en-3-one, lupenone, lupeol, friedelin) 0.153–0.954 mg L^{-1}. Table S2 lists the monitored precursor and product ions, with assigned quantifier and qualifier ions, and their corresponding collision energies (CE) for each cork-related compound in CPCs.

Analyses of polypropylene additives migration

Specific migration of 9,9-bis(methoxymethyl)-9H-fluorene (polymerisation aid) and tris (3,5-di-tert-butyl-4-hydroxybenzyl)isocyanurate (antioxidant) was determined by LE-GC-MS after extraction of oil simulant with acetonitrile (1:1). Calibration curves were prepared in blank oil spiked at 0.012–0.120 mg kg^{-1} (fluorene) and 0.05–0.50 mg kg^{-1} (isocyanurate). Chromatographic conditions: Zebron 5 MS Plus (30 $m \times 0.25$ mm × 0.25 µm); injector 280 °C; oven 60 °C (2 min), increasing at 10 °C min^{-1} to 320 °C; helium 1.2 mL min^{-1}. Detection conditions: SIM mode at m/z 165, 178, 194 (fluorene) and 161, 175, 189, 203, 218 (isocyanurate). LOD/LOQ were 0.004/0.015 mg kg^{-1} for the fluorene derivative and 0.015/0.051 mg kg^{-1} for the isocyanurate.

Metals migration from composite

Metal concentrations determined in the cork were conservatively converted into maximum migration values, assuming total transfer of metals from the cork fraction in the CPC to food product and compared to the specific migration limits (SMLs) set in Regulation (EU) No 10/2011.

Impact of migration on composite integrity and sensory properties

Composite integrity

To evaluate the stability of the filler–matrix interface under repeated use, the surface of the CPC samples was examined before and after exposure to migration tests with food simulants. Observations were performed with an Olympus BX51 optical microscope (Olympus, Tokyo, Japan) equipped with an Olympus BX50 digital camera, under direct light at low (40×) and high (100×) magnifications. Surface analysis in the XY plane was conducted to assess macrostructural features, including surface integrity, filler–matrix interactions, and morphological changes resulting from contact with the simulants.

Sensory analysis

The sensory evaluation included both the inherent odour of CPC and PP disc samples and the transfer of odour/flavour from the materials into water as food simulant, following ISO - 13302:2003 and DIN 10955:2004 standards. To evaluate the inherent odour, four specimen discs were placed in a 200 mL glass cup in replicate. Additional glass cups were prepared: one identical cup coded as a blind control and one designated as the reference

(without sample). All cups were stored in the dark at 23 ± 2 °C for 24 h, after which each panellist evaluated the samples directly. For the evaluation of odour/flavour transfer, four specimen discs were immersed in 200 mL of treated water for 10 days at 40 °C. The resulting simulant solutions were compared with a blank sample of the same simulant. Each panellist received two replicates containing simulant in contact with the samples, one reference blank, and one blind control. The sensory evaluation was conducted by a selected panel of nine trained assessors in a test room compliant with ISO 8589:2007. The perceived odour or flavour intensity was scored using a 5-point scale: 0 – no perceptible; 1 – just perceptible; 2 – moderate; 3 – strong; 4 – very strong.

Genotoxicity through AMES test

The mutagenic potential of the samples was evaluated using the Ames bacterial reverse mutation test, according to the OECD Guideline 471 (OECD 2020), using a microplate-based plate incorporation method. This assay detects point mutations in Salmonella typhimurium (TA) strains that carry specific mutations in the histidine operon, rendering them unable to grow on minimal medium unless a reverse mutation restores histidine synthesis. Five TA strains were tested (Trinova Biochem, Giessen Germany): TA98 and TA1537 are sensitive to frame-shift mutagens, TA100 and TA1535 detect base-pair substitution mutagens, and TA102 is particularly sensitive to oxidative and cross-linking agents.

Each strain was exposed to four concentrations of the migration solutions: the undiluted and successive dilutions prepared in sterile migration simulants, corresponding to 1:2, 1:4 and 1:8 dilutions of the migration solution. The test was performed both in the presence and absence of an exogenous metabolic activation system (Mutazyme 30% S9 Mix rat liver PB/BNF (M11-406.3L, Trinova Biochem), which simulates mammalian metabolism and allows the detection of both direct-acting mutagens and compounds requiring metabolic activation (OECD 2020). For each condition, concurrent simulant controls and positive controls appropriate for each strain and metabolic condition were included to verify the validity and sensitivity of the assay. The number of positive wells (His^+ revertants) was automatically scored for each concentration, and results were expressed as the mean number of positive wells per concentration.

A baseline was defined for results evaluation. The fold increase above the zero-dose baseline (mean of zero dose plus one standard deviation) were determined at each dose and testing solution. If the average of the zero-dose was 1 or lower, values lower than or equal to 3 fold were not considered as indicative of equivocal or positive results. If the baseline was higher than 1, the results were considered positive if (i) multiple, dose-dependent increases over 2-fold the baseline or (ii) an increase exceeding the strain-specific threshold was observed at the highest tested dose or at the highest non-toxic dose level. The strain-specific threshold was defined as ≥ 3-fold the baseline for strains TA1535 and TA1537, and ≥ 2-fold the baseline for strains TA98, TA100 and TA102 (Spiliotopoulos and Koelbert 2020).

Statistical analysis

Semi-quantitative profiling of compounds in ethanol extracts of unmatured (UMC) and matured (MC) cork by LE-GC-MS/MS was performed using Welch's two-sample t-test for compounds common to both cork types, assuming unequal variances. Fold change (UMC/MC) was calculated, and values below detection limits or with insufficient replicates were excluded.

Metal contents in UMC and MC cork were reported as mean ± standard deviation ($n = 5$) and compared using Welch's t-test. Statistical significance was defined as $p < 0.05$ (*), $p < 0.01$ (**), or NS (not significant). Elements with insufficient data were not analysed (Co and Ni: all values below detection limits; Pb: single value in MC). All analyses were performed using OriginPro 2024 (OriginLab, USA).

Sensory data were evaluated using mean and median values, and differences between sample types (PP vs. CPC) or between replicates and blind controls were assessed with a non-parametric Wilcoxon signed-rank test, suitable for the ordinal scoring scale and limited replicates. Differences were considered statistically significant at $p < 0.05$.

Results and discussion

Characterisation of Cork granulates (UMC and MC)

Key analytical parameters of the granulate include SEM-based morphology, extractive profiles, FTIR-ATR spectra, TGA/DSC thermograms and ICP-OES metallic elements content. These results are summarised in Table 1.

Morphology

SEM analysis revealed distinct microstructural differences between the UMC and MC cork (Figure 2). UMC exhibits larger, irregular cells (514 ± 311 μm^2), relatively uniform thinner walls (1.67 ± 0.43 μm), and slightly higher apparent density (0.230 ± 0.047 g/cm^3), whereas MC shows smaller, more uniform cells (306 ± 147 μm^2) with a broader distribution of wall thickness (1.94 ± 0.80 μm) and lower apparent density (0.201 ± 0.009 g/cm^3). Histograms of cell base area and radial wall thickness confirm these differences, reflecting the incomplete tissue maturation in UMC versus the organised, honeycomb-like architecture of MC (Silva et al. 2005; Gil 2009; Fernandes et al. 2011). These morphological traits may directly impact CPC performance. The large standard deviation observed for the cell base area, especially in UMC, highlight a high intrinsic structural heterogeneity. For the cork material properties, this wide variability implies that UMC will exhibit less predictable, anisotropic

Table 1. Summary of key parameters of unmatured (UMC) and matured (MC) cork as source of cork granulate incorporated into the CPC, determined by GC–MS, FTIR-ATR-PLSDA, TGA, DSC, and ICP-OES.

Parameter	UMC	MC	Observation / Relevance for CPC
Cork morphological features[1]:			
Base cell area (μm^2)	514 ± 311	306 ± 147	UMC: variable dispersion; MC: uniform dispersion
Radial cell-wall thickness (μm)	1.67 ± 0.43	1.94 ± 0.80	MC: stronger reinforcement; UMC: easier polymer infiltration
Apparent density (g/cm^3)	0.230 ± 0.047	0.201 ± 0.009	UMC: higher extractive retention; MC: stable filler–matrix
GC–MS (Extractives)[2]:			
Volatile terpenes & terpenols	0.007–0.009 mg/g	n.d.	UMC enriched; thermally reactive → potential oxidation NIAS
Ferulic acid derivatives	0.001 mg/g	n.d.	UMC contains known furan precursor structures
Alkene-type volatile	0.024 mg/g	n.d.	UMC traces of alkenes → reactive NIAS precursors
Quinones (catechol, hydroquinone, benzoquinone)	n.d.	0.003–0.017 mg/g	MC stable, oxidised signature; low migration
Galactopyranoside	n.d.	0.128 mg/g	Polar and non-volatile
Triterpenoid ester	trace	2.406 mg/g	Non-volatile; stabilises MC
FTIR-ATR:			
1737 cm^{-1} (C = O suberin ester)	Moderate	Stronger	Higher esterified suberin in MC
1603–1510 cm^{-1} (lignin aromatics)	Slightly stronger	Lower	Higher lignin/polysaccharides in UMC
1150–1000 cm^{-1} (C–O–C polysaccharides)	Strong	Moderate	More carbohydrates in UMC
Overall spectral trend	More lignin/polysaccharides	More oxidised/ esterified suberin	Reflects maturity differences
TGA[1]:			
Initial mass loss (%)	3.0 ± 0.8	7.1 ± 0.9	Volatile/moisture fraction; early emissions
T_{onset} (°C)	241.2 ± 4.4	265.6 ± 3.1	UMC → earlier degradation, more NIAS precursors
$T_{10\%}$ (°C)	285 ± 5	265 ± 6	Mass-loss profile differences
T_{max} (°C)	313 ± 3.5	356 ± 18	MC more thermally stable
Ash content (%)	15 ± 1	23 ± 2	Higher inorganic residue in MC
DSC[1]:			
1st cycle, 20–200 °C			
$T_{Low\text{-}range\ transition}$ (°C)	80.7 ± 1.8	66.3 ± 5.9	Bound-water/suberin differences; extra peak 97.4 ± 5.3 for MC due to bound-water evaporation
$T_{High\text{-}range\ transition}$ (°C)	153.2 ± 12.5	n.d.	Hemicellulose relaxation, minor lignin–carbohydrate changes
2nd cycle re-heating, 20–200 °C			
$T_{Low\text{-}range\ transition}$ (°C)	69.2 ± 1.01	61.6 ± 0.6	Reproducible low-range transition
ICP-OES (Metals)[3]:			
Al (mg/kg)	94.1 ± 57.4	21.2 ± 24.4	Higher in UMC; within historical cork ranges
Ba (mg/kg)	16.1 ± 3.6	4.9 ± 0.8	Higher in UMC; within reported variability
Fe (mg/kg)	55.2 ± 35.0	10.5 ± 5.7	Higher in UMC; typical cork constituent
Mn (mg/kg)	23.4 ± 6.7	7.3 ± 1.1	Higher in UMC; consistent with cork composition
Cr (mg/kg)	0.2 ± 0.08	0.11 ± 0.04	Slightly higher in UMC; within reported cork levels
Cd (mg/kg)	0.22 ± 0.05	0.04 ± 0.002	Higher in UMC; within reported cork variability
Li (mg/kg)	5.1 ± 3.7	9.9 ± 5.7	Higher in MC; likely related to post-harvest factors

Values are presented as mean ± standard deviation. Sample size: [1]n = 3,[2]n= 2, [3]n = 5. Full analytical data supporting this characterisation, including GC–MS extractive profiles, FTIR-ATR spectra, PLS-DA outputs, TGA/DSC thermograms, and ICP-OES metal results, are provided in the Supplementary Material.

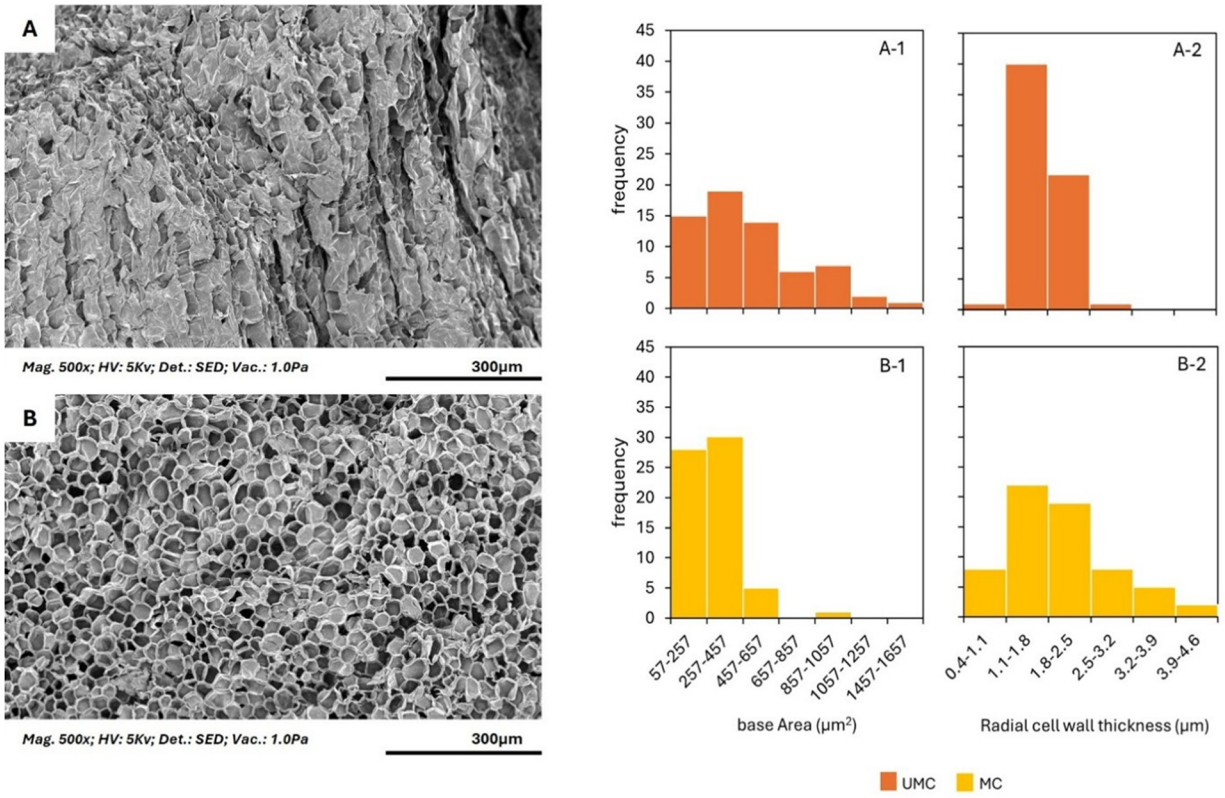

Figure 2. Structural characterisation of cork source samples from cork granulates incorporated into the CPC. SEM images of cork cellular structure at 500× magnification: (A) unmatured cork (UMC) and (B) matured cork (MC, Amadia); (A-1, B-1) Histograms of cell base area; (A-2, B-2) Histograms of radial cell-wall thickness. Data represent ≥ 50 cells per sample.

mechanical behaviour, where exceptionally large cells can act as localised stress concentrators or initiation sites for microstructural deformation under load. On the other hand, the narrower distribution in MC ensures a more isotropic response and uniform stress distribution across the material. The dense, heterogeneous UMC structure provides abundant sorption sites and enables polymer infiltration, enhancing mechanical interlocking and stress transfer, as well as localised flexibility and toughness. In contrast, MC uniform, ordered cell structure promotes consistent dispersion within the polymer matrix and limits migration of extractable compounds, supporting composite stability (Gil 2009; Fernandes et al. 2011; Serra et al. 2022).

Chemical characteristics

GC–MS analysis of ethanol extracts revealed higher number of detectable compounds than iso-octane extracts and compositional distinction between UMC and MC (Figure S1, Table 2). UMC is enriched in volatile terpenes (0.007–0.009 mg/g), ferulic acid derivatives (0.001 mg/g), and low-molecular alkenes (0.024 mg/g), indicative of thermally labile and reactive constituents. These classes of compounds are known to undergo thermal degradation or secondary reactions at high temperatures and therefore may act as precursors of NIAS during extrusion and injection moulding when cork is incorporated in the CPC. Terpenes and terpenoids, in particular, are well-documented VOCs emitted during extrusion of plant-based/lignocellulosic materials due to their thermal lability and tendency to oxidise or rearrange (Hejna 2022; Mironeasa et al. 2023). Ferulic acid and its derivatives can also undergo partial decomposition or structural transformation under high-temperature processing (Llevot et al. 2016; Kasmi et al. 2019; Khan et al. 2023). Therefore, these extractives are relevant to the CPC behaviour, as the presence of ferulic-acid-type structures in UMC provides known precursors for the furanic compounds, later detected in CPC screening and migration testing (Section Headspace screening and section Migration of cork related substances). Similarly, low-molecular alkenes

Table 2. Semi-quantitative profiling of key compounds detected in ethanol extracts of unmatured (UMC) and matured (MC) cork by LE-GC-MS/MS.

RT (min)	CAS	Compound	MW (g/mol)	Match / RM	RI (library / calc.)	ΔRI	UMC (mg/g)[a]	MC (mg/g)[a]	Observation
Compounds differing between cork types:									
6.283	106-51-4	*p*-Benzoquinone	108.09	806 / 864	927 / 928	−1	n.d.	0.017 ± 0.004	Phenolic oxidation marker
10.943	120-80-9	Catechol	110.11	782 / 847	1208 / 1197	11	n.d.	0.014 ± 0.0002	Phenolic oxidation product
12.162	123-31-9	Hydroquinone	110.112	783 / 847	1283 / 1281	2	n.d.	0.003 ± 0.0003	Phenolic; Suggest advanced oxidation
14.082	–	Unknown	–	– / –	– / 1423	–	0.024 ± 0.003	n.d.	Unidentified compound
15.363	–	Galactopyranoside	–	– / –	– / 1525	–	n.d.	0.128 ± 0.009	Specific glycoside; possible hemicellulose derivative
15.551	645-08-9	Isovanillic acid	168.15	724 / 842	– / 1541	–	n.d.	0.005 ± 0.0003	Phenolic; Lignin oxidation product
16.683	–	Fatty acid ester	–	– / –	– / 1637	–	n.d.	2.406 ± 0.532	Lipophilic extractive
18.954	537-98-4	*trans*-Ferulic acid	194.18	603 / 716	1871 / 1846	25	0.001 ± 0.0001	n.d.	Aromatic acid extractive
29.449	–	Terpene/Terpenol	–	– / –	– / 3175	–	0.009 ± 0.005	n.d.	Specific volatile compound
30.020	–	Terpene/Terpenol	–	– / –	– / 3269	–	0.007 ± 0.002	n.d.	Specific volatile compound
Compounds common to both cork types:									
30.468	83-46-5	β-Sitosterol	414.71	824 / 841	3187 / 3340	153	0.295 ± 0.003	0.370 ± 0.072	Plant sterol marker; higher in MC; NS (t-test, p = 0.38; n = 2); FC = 0.80
32.317	559-74-0	Friedelin	426.72	917 / 919	3533 / 3582	49	3.749 ± 1.114	4.472 ± 0.350	Triterpenoid marker; slightly higher in MC; NS (t-test, p = 0.52; n = 2); FC = 0.84
33.829	–	Friedelin-related	–	– / –	– / 3731	–	2.066 ± 0.496	2.710 ± 0.555	Likely Friedelin oxidation product; higher in MC; NS (t-test, p = 0.35; n = 2); FC = 0.76

[a]Values reported as mean ± standard deviation, $n = 2$; Semi-quantification based on deuterated dodecane (26.2 mg/L) as internal standard. Estimated LOD = 0.001 mg/g ($S/N = 3$); Compound identification supported by spectral match (NIST 2.4) and retention index (RI) comparison; Compounds common to both cork types were compared using Welch's two-sample t-test; Differences were not statistically significant ($p > 0.05$); Legend: RM – Reverse match, n.d.– not detected, UMC – unmatured cork, MC – matured cork, NS – not statistically significant, Fold change (FC) calculated as UMC/MC.

readily participate in thermal and oxidative reactions, forming oligomers or secondary byproducts commonly reported as NIAS in thermoplastic systems (Llevot et al. 2016; Miralles et al. 2025). MC extracts contained oxidised phenolics, including catechol, hydroquinone, and p-benzoquinone (0.003–0.017 mg/g), galactopyranoside (0.128 mg/g), and lipophilic esters (2.406 mg/g), reflecting a more chemically stable and oxidised profile. Their present is consistent with oxidative transformations of lignin and suberin derivatives during maturation (Irmak et al. 2020; Sun et al. 2021). Galactopyranoside presence in MC may arise from hemicellulose degradation during thermal processing of cork (e.g. boiling) or from biological maturation (Rocha et al. 1996; Alañón et al. 2011). Given their polar nature and reactive hydroxyl groups, galactose-based glycosides could potentially enhance interfacial adhesion with polar polymer matrices like PLA, as reported for other natural fibre composites (Petinakis et al. 2009; Larguech et al. 2021). Plant sterols and triterpenoids (β-sitosterol, friedelin, friedelin-related compounds) were present in both cork types, slightly higher in MC, and subtle variations may reflect either natural compositional differences or extraction efficiency (Pereira 2007; Castola et al. 2002). Taken together, these results suggest that MC possesses a more oxidised and

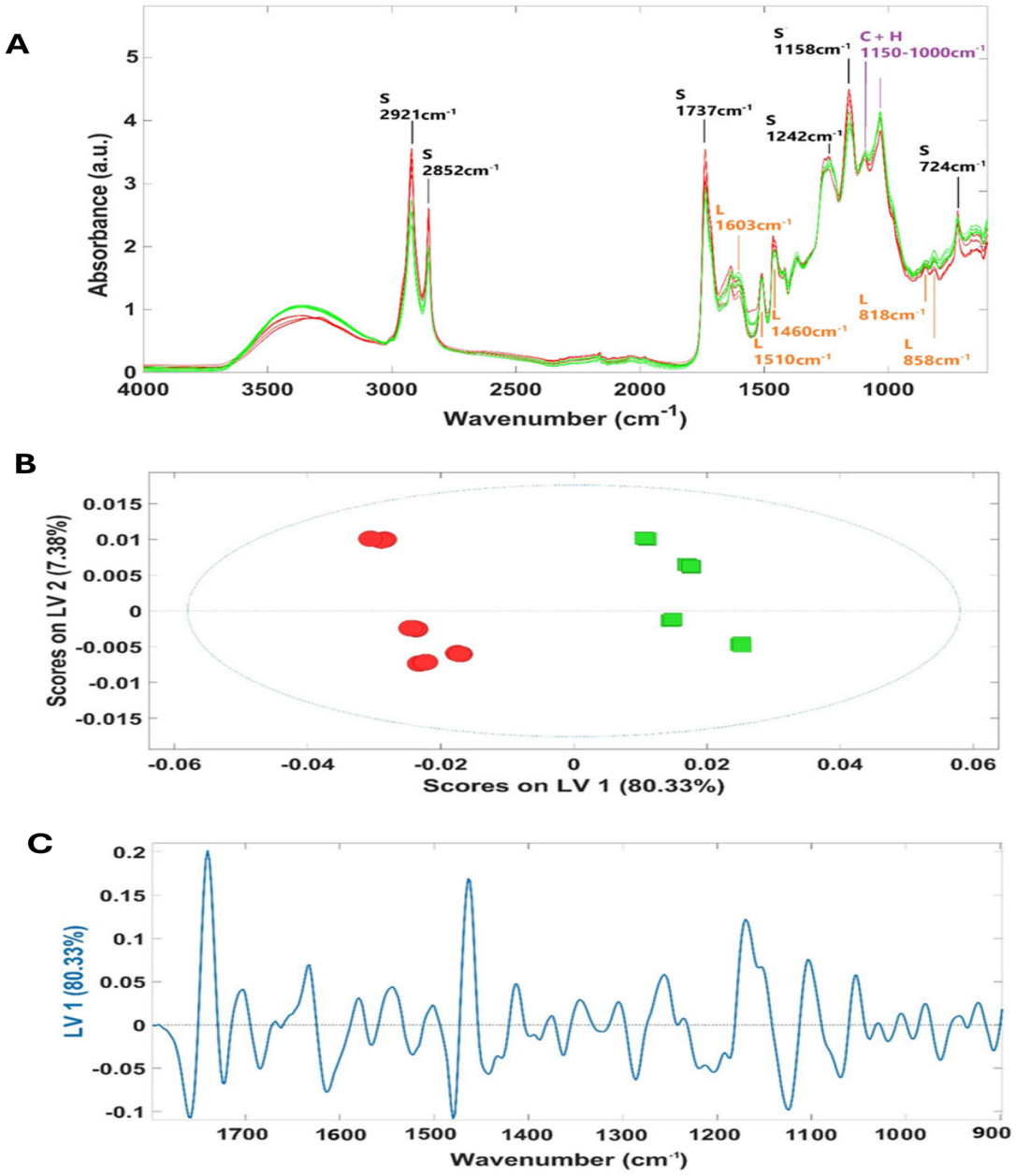

Figure 3. A- FTIR-ATR spectra of cork samples. Legend: red lines- UMC samples; green lines- MC samples. B - Scores map of the PLS-DA regression model obtained with all FTIR-ATR spectra. Legend: red circles- UMC sample; green squares- MC sample. C- PLS-DA model loadings. Legend: blue line- LV1 loadings.

thermally stable extractive profile, remaining more resistant to degradation during polymer processing. In contrast, UMC appears to retain a higher proportion of reactive phenolic precursors, particularly ferulic-acid-type constituents.

FTIR-ATR spectroscopy, supported by PLS-DA regression, confirmed these differences (Figure 3; Table S3). UMC exhibits stronger lignin- and polysaccharide-related bands (1603–1510 cm^{-1} and 1150–1000 cm^{-1}), whereas MC shows more intense esterified suberin signals (1737 cm^{-1}). The multivariate PLS-DA model highlights these chemical fingerprints as discriminators between the cork types, consistent with GC–MS data. The PLS-DA model was validated using venetian blinds cross-validation (10 splits, blind thickness = 1). Both sample classes (MC and UMC) achieved sensitivity and specificity of 1.000 in the calibration and cross-validation sets. RMSEC and RMSECV were 0.01268 and 0.02044, respectively, with R^2 values of 0.99935 and 0.99831, confirming the model's high accuracy and robustness. The first two latent

variables (LVs) of the PLS-DA regression model explained nearly 88% of the spectral variability, achieving clear separation between cork types. This performance is comparable to previous NIR-based PLS-DA studies on cork powder extracts, with reported 80–100% accuracy depending on the compound considered (Páscoa et al. 2024).

The main compositional differences between UMC and MC relate to the relative abundance of suberin, lignin, and polysaccharides. Variations in lipid-derived ester groups and aromatic lignin structures are particularly influential, and these differences help explain the distinct functional performance of corks in composites. Importantly, the stronger lignin and polysaccharide signals observed in UMC also support the higher Fe and Mn contents reported later, since these metals are linked to lignin biosynthesis and oxidative processes. In addition, the less oxidised suberin profile of UMC helps explain the earlier onset of thermal degradation described later, while the more oxidised, phenolic-rich profile of MC underlies its delayed thermal transitions. These cross-relations confirm that FTIR signatures are consistent with the chemical and thermal behaviours observed across the study.

Metal analysis

The total content of twelve selected metals in UMC and MC samples was quantified (Table 3). These elements were chosen based on historical bibliographical data for cork showed in (Supplementary information Table S4) and the list of metals regulated for plastic food contact materials under Regulation (EU) No 10/2011 (European Commission, 2011).

The historical data reveal considerable variation in metal concentrations across decades (1965–2023), likely reflecting differences in cork sources, environmental exposure, and analytical methods over time. Elements such as aluminium (Al), iron (Fe), and zinc (Zn) have consistently been detected in cork materials, often at relatively high concentrations, whereas data for other elements, including barium (Ba), beryllium (Be), and vanadium (V), are only available in more recent studies (2014–2023). This historical comparison highlights the inherent variability of cork's metal composition, reinforcing the need to contextualise current findings within this broader spectrum.

In the present study, statistically significant differences ($p < 0.05$) were observed between UMC and MC samples for seven metals: Al, Ba, Cd, Cr, Fe, lithium (Li), and manganese (Mn). UMC samples exhibited higher concentrations of Al, Ba, Cd, Cr, Fe, and Mn. This can be attributed to their denser and more lignified microstructure, which provides additional binding sites and promotes retention of metals, particularly Mn and Fe, which are closely associated with lignin biosynthesis and oxidative processes (Onnerud et al. 2002; Champ et al. 2021). In contrast, the elevated Li levels observed in MC are unlikely to result from structural differences, as Li is highly mobile and interacts weakly with lignin or suberin (Ponte-e-Sousa and Neto-Vaz 2011). Instead, they may reflect post-harvest treatments, such as boiling or prolonged storage, which can alter elemental distribution or concentrate Li relative to other components.

Although Al and Fe were significantly higher in UMC, their levels remain within the historical

Table 3. Metal content in cork samples (UMC and MC), mg/kg.

Element	UMC[a]	MC[a]	*t-statistic*	p-value	Significant
Al	94.1 ± 57.4	21.2 ± 24.4	−2.615	0.031	*
Ba	16.1 ± 3.6	4.9 ± 0.8	−6.872	0.002	**
Cd	0.22 ± 0.05	0.04 ± 0.002	−5.825	0.001	**
Co	< LOD	< LOD	N/A	N/A	
Cr	0.2 ± 0.08	0.11 ± 0.04	−2.921	0.019	*
Cu	9.9 ± 1.4	11.2 ± 2.1	−0.968	0.363	NS
Fe	55.2 ± 35.0	10.5 ± 5.7	−2.816	0.023	*
Li	5.1 ± 3.7	9.9 ± 5.7	2.559	0.031	*
Mn	23.4 ± 6.7	7.3 ± 1.1	−5.264	0.001	**
Ni	< LOD	< LOD	N/A	N/A	
Pb	0.14 ± 0.05	0.07	N/A	N/A	
Zn	2.9 ± 0.5	2.3 ± 0.4	−1.992	0.103	NS

[a]Mean ± standard deviation, $n = 5$. Legend: UMC – unmatured cork; MC- matured cork (*Amadia*); N/A indicates too few non-censored data points (Co and Ni: all values below the limits of detection, Co: 0.010 mg/kg; Ni: 0.009 mg/kg; Pb: only one measured value of total replicates in MC sample). Significance levels: $p < 0.05$ = *, significant; $p < 0.01$ = **, highly significant; NS – not significant, were determined using Welch's two-sample t-test.

ranges reported (Table 3). Cd and Cr concentrations in UMC, while below historical maxima, approach the upper bounds of recent datasets (Cd ~0.1 mg/kg; Cr ~0.7 mg/kg), supporting the need for migration testing. No significant differences were observed for Cu and Zn, while Co and Ni were generally below quantification limits, precluding statistical analysis. Pb was below the quantification limit for MC samples; however, occasional detectable levels in UMC suggest possible localised accumulation linked to matrix heterogeneity and warrant further investigation through migration testing. These findings align with historical values and suggest minimal contamination typically associated with environmental or processing factors (Silva et al. 2005; Pereira 2013). Arsenic (As) and mercury (Hg) have historically low concentrations in cork (average values of 0.3 mg/kg for As and 0.1 mg/kg for Hg in 2014–2023, with no data prior to 2003).

Thermal properties

TGA and DSC results are summarised in Table 1. The thermograms are presented in Figures S2 and S3. UMC shows lower Tonset (241.2 ± 4.4 °C) and Tmax (313 ± 3.5 °C) compared with MC (Tonset 265.6 ± 3.1 °C, Tmax 356 ± 18 °C), reflecting earlier decomposition of labile constituents. DSC low-range endothermic transitions indicate differences in bound-water and suberin mobility between cork types (UMC: 80.7 ± 1.8 °C; MC: 66.3 ± 5.9 °C). UMC also exhibits a distinct high-range transition at 153.2 ± 12.5 °C, attributed to hemicellulose relaxation and minor lignin–carbohydrate rearrangements. During the second heating cycle (reheating to 200 °C), the low-range transitions shifted to 69.2 ± 1.0 °C for UMC and 61.6 ± 0.6 °C for MC, confirming that these endothermic events reflect inherent material characteristics rather than artefacts of thermal history. No additional high-range transitions emerged upon reheating, and cooling cycles showed no exothermic or crystallisation events, consistent with the amorphous nature of cork polysaccharides and suberin domains (Şen et al. 2014; Paiva and Magalhães 2018). These thermal characteristics relate with the chemical behaviour of cork during CPC processing: the lower Tonset and earlier decomposition of UMC promote the formation of thermal degradation products, such as furfural and 5-methylfurfural, whereas the higher thermal stability of MC reduces the generation of processing-related NIAS, supporting safer and more consistent composite properties (Silva et al. 2005; Fernandes et al. 2011; Şen et al. 2014). The MC's more stable profile reduces risks of off-gassing, discolouration, or interaction with the PP matrix during extrusion, whereas UMC may require tighter processing control to maintain composite integrity.

Screening of potential migrants in Cork–polypropylene composite

Screening analyses were performed to identify substances potentially migrating from the cork and polypropylene phases of the CPCs vs PP (control) samples. The results of the screening are presented in Table 4.

Headspace screening

The HS-GC-MS chromatograms (Figure S4) show the volatile compounds detected from the CPC and PP control samples. Furfural and 5-methylfurfural were identified exclusively in the CPC samples, indicating their formation during extrusion or injection moulding, as they were not detected in raw cork. This is consistent with the presence of thermally labile constituents including ferulic-acid derivatives previously identified, confirming their origin from cork-derived precursors rather than from PP. The remaining detected compounds, predominantly substituted benzenes and phenols, were present at similar or higher levels in PP alone compared with CPC, indicating their polymer-derived origin. The similarity of these profiles between CPC and PP confirms that these compounds result from PP additives or intrinsic degradation mechanisms, rather than from cork matrix.

Liquid extraction screening

The chemical profiles of CPC and PP were investigated using two solvent extractions, iso-octane and ethanol, followed by LE-GC-MS analysis (Table 4). The chromatograms are shown in Figure S5. Both extractions revealed the presence

Table 4. Volatile and extractable compounds detected in PP and CPC samples by HS-GC-MS and LE-GC-MS (ng/g).

				HS-GC-MS		LE-GC-MS			
						Iso-octane		Ethanol	
Peak ID	Compound	CAS No.	MW (g/mol)	PP	CPC	PP	CPC	PP	CPC
1A	Acetic acid	64-19-7	60.05	–	4310	–	–	–	–
2A	Furfural	98-01-1	96.08	–	40094	–	–	–	–
3A	5-Methylfurfural	620-02-0	110.11	–	3260	–	–	–	–
–	Ethanone, 1-(2,3-dihydro-1H-inden-5-yl)- OR 1-[4-(1-methylethenyl)phenyl]-	4228-10-8 / 5359-04-6	160.09 / 160.21	438	855	801	2030	290	1292
1B	m-Diacetylbenzene (or isomer)	6781-42-6	162.19	31419	2650	43704	16811	18525	17155
–	Ethanone, 1-[4-(1-hydroxy-1-methylethyl)phenyl]-	54549-72-3	178.23	–	–	18796	3877	5178	4320
–	m-Diisopropenylbenzene (or isomer)	3748-13-8	158.24	83	81	–	–	–	–
–	2,6-Di-tert-butylbenzoquinone	719-22-2	220.31	319	42	–	–	–	–
–	Benzyl alcohol, α,α-dimethyl-p-isopropyl-	3445-42-9	178.27	6069	309	–	–	–	–
–	2,4-Di-tert-butylphenol	96-76-4	206.32	2544	956	2493	1614	584	870
–	9,9bis(methoxy-methyl)fluorene	182121-12-6	254.33	–	–	1322	782	453	600
–	n-Hexadecanoic acid	57-10-3	257.42	–	–	–	241	104	606
–	Squalene	111-02-4	410.72	–	–	–	665	–	833
2B	Phenol, 2,4-bis(1,1-dimethylethyl)-, phosphite (3:1)	31570-04-4	646.92	–	–	150200	97674	22820	24204
–	Lupenone	1617-70-5	424.7	–	–	–	2910	–	1447
–	γ-Sitostenone	84924-96-9	412.0	–	–	–	9322	–	8169
3B	Tris(2,4-di-tert-butylphenyl) phosphate	95906-11-9	662.92	–	–	76270	49921	11708	11078
4B	Friedelan-3-one	559-74-0	426.72	–	–	–	255908	–	237430
–	Stigmastane-3,6-dione (5α)	22149-69-5	428.69	–	–	–	1320	–	1788
5B	3,12-Oleandione	n.a.	–	–	–	–	20088	–	25189
–	Furan, 2-(diethoxymethyl)-	13529-27-6	170.21	–	–	–	–	–	218
–	Vanillin (or isomer)	121-33-5	152.15	–	–	–	–	–	1180
–	3,5-Di-tert-butyl-4-hydroxybenzoic acid, methyl ester	2511-22-0	264.37	–	–	–	–	3573	–
–	Octadecanoic acid	57-11-4	284.0	–	–	–	–	120	638
–	Unknown	n.a.		–	–	–	–	–	10178

of polymer-related additives and natural cork compounds, highlighting the contributions of each component in the CPC matrix. The iso-octane extract showed both polymer-related and cork-related compounds. Detected cork markers included lupenone, friedelan-3-one, and 3,12-oleandione, characteristic triterpenoids of cork, and γ-sitostenone and stigmastane-3,6-dione, derived from oxidised sterols. These triterpenoids markers correspond directly to the non-volatile extractives quantified in the raw cork materials (Table 2). Polypropylene-related additives and degradation products were also observed, such as tris(2,4-di-tert-butylphenyl) phosphate, phenol, 2,4-bis(1,1-dimethylethyl)-, phosphite (3:1), and m-diacetylbenzene or isomer, consistent with antioxidant stabilisers and catalyst residues (Table 4). These findings indicate that cork contributes natural triterpenes and sterols, while polypropylene contributes additive-related substances.

The ethanol extract showed a similar profile, with additional detection of furan, 2-(diethoxymethyl)- and vanillin (or isomer), found only in CPC. This indicates partial reaction of furanic compounds with ethanol. Some PP-related additives exhibited lower intensity in CPC extracts compared to neat PP, likely due to matrix effects during extraction (Table 4). Cork triterpenoids and sterols were consistently detected also in ethanol extract.

Overall, both extraction solvents identified the main cork markers and PP additives. Iso-octane preferentially extracts nonpolar compounds, while ethanol can solubilise slightly more polar components, including reaction products like vanillin and furan derivatives. The compounds identified in this screening analysis were used to define the set of target migrants subsequently determined in the migration experiments.

Overall migration

Migration values for all food simulants (10% ethanol, 3% acetic acid, and vegetable oil, after 10 days at 40 °C) were below the respective limits of quantification (< 2.0 mg/dm^2 for aqueous simulants and < 3.0 mg/dm^2 for oil). These values are far below the overall migration limit of 10 mg/dm^2 established

in Regulation (EU) No 10/2011. No increase in migration was observed between the contact cycles, confirming the stability of the CPC during repeated use (Brandsch and Schuster 2020). This stability is consistent with the morphological and extractive characteristics of the cork granules (section Characterization of cork granulates (UMC and MC)), where both UMC and MC components become embedded within the polymer matrix, limiting the release of extractable species. The absence of measurable migration across all simulants demonstrates that cork incorporation at 15% (w/w) does not compromise the polymer matrix or enhance global substance release, indicating excellent material integrity and compliance with regulatory requirements for food-contact applications.

Specific migration

The migration solutions after 10 days at 60 °C of contact under repeated use conditions were analysed by HS-SPME-GC-MS for volatile and semi-volatile compounds, and by LE-GC-MS for triterpenoids, sterols, and PP additives. Compounds detected in CPC but absent in PP were attributed to cork-related, whereas substances detected in both materials were considered PP-related. The objective was also to evaluate if the incorporation of cork granules affects the inherent migration profile of the PP matrix.

Migration of Cork related substances

A set of volatiles and semi-volatiles originating from thermal degradation of cork were detected in CPC but absent in the PP control. These included furfural, 5-methylfurfural, 2-nonanone, ethyl esters of C7–C9 fatty acids, and octanoic acid (Table 5). Among these, furfural and 5-methylfurfural dominated the migration profile and were the only substances consistently above trace levels. Migration of both compounds decreased markedly across repeated cycles, confirming stability of the CPC under repeated-use conditions (Brandsch and Schuster 2020) (Figure 4). In 10% ethanol, furfural decreased from 5.32 mg/kg (1st cycle) to 1.58 mg/kg (3rd cycle), and 5-methylfurfural from 0.68 mg/kg to 0.17 mg/kg (Table 4). Migration into 3% acetic acid was lower, ranging from 2.77 to 1.14 mg/kg for furfural and 0.40 to 0.13 mg/kg for 5-methylfurfural. The lowest migration occurred in vegetable oil, with furfural decreasing from 0.0.48 to 0.15 mg/kg and 5-methylfurfural from 0.05 to 0.01 mg/kg. The migration of these furanic species is mechanistically consistent with the thermal

Table 5. Migration of cork-related volatile/semi-volatile compounds and non-volatile (terpenoids/sterols) into food simulants, detected in CPC samples but absent in PP control samples after 10 days at 60 °C under repeated-use conditions, determined by HS-SPME-GC-MS and LE-GC-MS (mg/kg food or µg/kg food*).

Migrant	CAS	MW (g/mol)	1st migration	2nd migration	3rd migration
				Ethanol 10%	
Furfural	98-01-1	96.08	5.32 ± 1.629	2.77 ± 0.485	1.58 ± 0.170
5-Methylfurfural	620-02-0	110.11	0.68 ± 0.159	0.32 ± 0.063	0.17 ± 0.022
2-Nonanone	821-55-6	142.24	0.03 ± 0.02*	0.01 ± 0.02*	< LOQ
Heptanoic acid, ethyl ester	106-30-9	158.24	0.02 ± 0.01*	0.01 ± 0.02*	< LOQ
Octanoic acid	124-07-2	145.20	0.17 ± 0.11*	0.10 ± 0.14*	0.03 ± 0.04*
Octanoic acid, ethyl ester	106-32-1	172.26	0.06 ± 0.04*	0.05 ± 0.07*	0.01 ± 0.02*
Nonanoic acid, ethyl ester	123-29-5	186.29	0.04 ± 0.03*	0.05 ± 0.06*	0.01 ± 0.01*
				Acetic acid%	
Furfural	98-01-1	96.08	2.77 ± 0.187	1.07 ± 0.265	1.14 ± 0.322
5-Methylfurfural	620-02-0	110.11	0.40 ± 0.029	0.15 ± 0.040	0.13 ± 0.034
				Vegetable oil	
Furfural	98-01-1	96.08	0.48 ± 0.100	0.18 ± 0.040	0.15 ± 0.031
5-Methylfurfural	620-02-0	110.11	0.05 ± 0.010	0.01 ± 0.004	0.01 ± 0.003
				Ethanol 10% and acetic acid 3%	
β-Sitosterol	83-56-5	414.7	< LOD[1]	< LOD[1]	< LOD[1]
			< LOD[2]	< LOD[2]	< LOD[2]
Lupenone	1617-70-5	424.7	< LOD[3]	< LOD[3]	< LOD[3]
Lupeol	545-47-1	426.72	< LOD[3]	< LOD[3]	< LOD[3]
Sitost-4-en-3-one	1058-61-3	412.69	< LOD[3]	< LOD[3]	< LOD[3]
Friedelin	559-74-0	426.72	< LOD[3]	< LOD[3]	< LOD[3]

Migration was evaluated over three consecutive contact cycles (1st, 2nd, and 3rd migration) using ethanol 10% (hydroalcoholic), acetic acid 3% (acidic), and vegetable oil (fatty) as food simulants. Values represent the mean ± standard deviation of three replicate analyses. LOD – limit of detection: 1- 0.010 mg/kg food (for ethanol 10%); 2–0.020 mg/kg food (for acetic acid 3%); 3 - 0.005 mg/kg food (for both food simulants). LOQ – limit of quantification: 0.01 µg/kg food. For the molecular structures and CAS numbers of the identified migrants, see Figure S6.

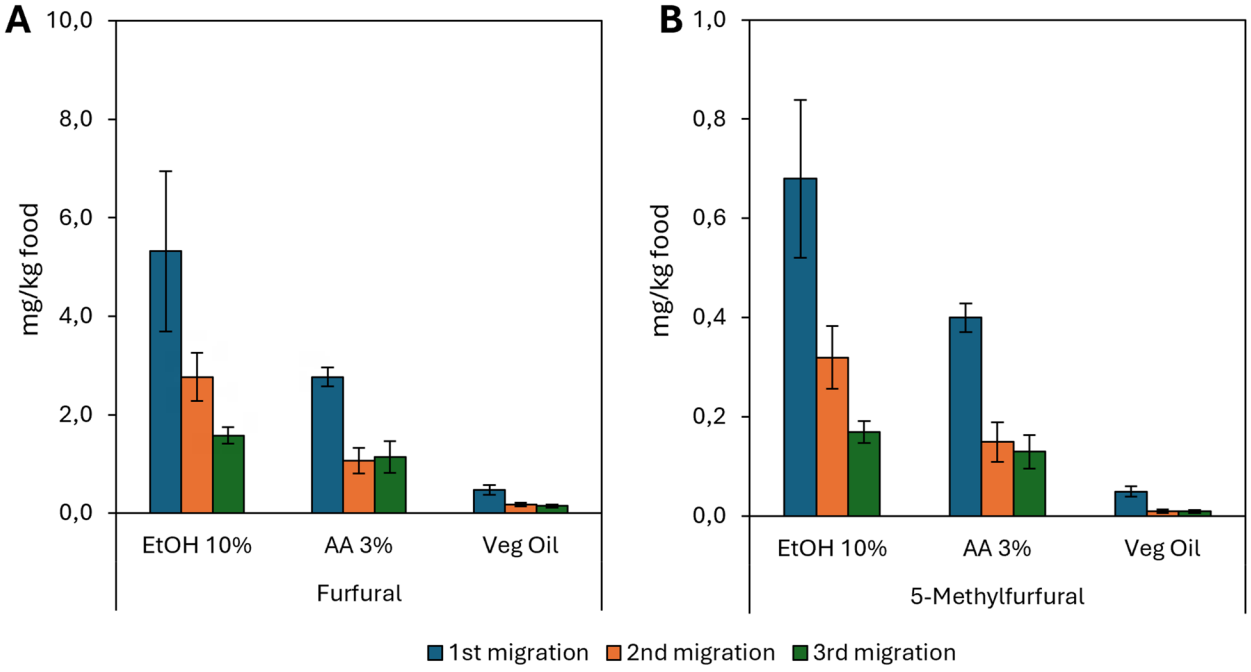

Figure 4. Migration of key cork-related volatile compounds into different food simulants (EtOH 10% = ethanol 10%; AA 3% = acetic acid 3%; Veg. Oil = vegetable oil) after 10 days at 60 °C under repeated-use cycles, detected in CPC samples but absent in PP control samples, determined by HS-SPME-GC-MS (Average ± SD, $n = 3$). Panel **A**: Migration of furfural. Panel **B**: Migration of 5-methylfurfural. Results show the trend from the 1st to the 3rd repeated-use migration cycle. Full quantitative data are provided in Table 5.

decomposition of UMC extractives (Section Characterization of cork granulates (UMC and MC)), and their decreasing concentration across cycles confirms their finite pool and the stabilisation of the composite matrix (Brandsch et al. 2021).

These substances are well-known process NIAS formed through acid hydrolysis or thermal degradation of polysaccharides containing pentose or hexose units (Maarse et al. 1994). Their formation is associated with Maillard and caramelisation pathways occurring during thermal processing of sugar-rich materials, particularly at temperatures above 120 °C and under low-moisture conditions (Esteban et al. 2020; Lee et al. 2022; Gao et al. 2024;). Both compounds naturally occur in a wide variety of heat-processed foods at concentrations far exceeding those detected here. Reported levels include 55–255 mg/kg in coffee and cocoa (Kowalski et al. 2013; Esteban et al. 2020), up to 26 mg/kg in whole-grain bread and bakery products (Ortu and Caboni 2017; Kobelev et al. 2023), and 1–33 mg/L in alcoholic beverages, with additional occurrences in fruit juices, honey, and thermally concentrated products (Bharani et al. 2024; Gao et al. 2024). Toxicological studies indicate that furfural and 5-methylfurfural may display genotoxic or cytotoxic properties at high experimental doses; however, typical dietary exposures remain orders of magnitude lower and are not considered a health concern under normal consumption patterns (Zhang et al. 2012; Farag et al. 2020; Meng et al. 2024). Migration studies from food-contact bamboo materials similarly show that consumer exposure from FCMs is low and within safe margins (Guan et al. 2023). Given their ubiquitous occurrence in foods, well-established formation mechanisms during heating, and substantially higher concentrations present in the regular diet compared with the trace amounts detected here, it is not expected that the incorporation of cork in the present FCM application will contribute meaningfully to consumer exposure to furfural or 5-methylfurfural.The highest migration value in CPC (1.58 mg/kg furfural into ethanol) remains orders of magnitude below typical dietary exposure. EFSA established an ADI of 0.5 mg/kg bw/day for furfural (EFSA AFC Panel 2004), and thus the contribution from CPC use is negligible.

Cork triterpenoids and phytosterols (β-sitosterol, lupenone, lupeol, sitost-4-en-3-one, and friedelin)

showed no detectable migration into aqueous food simulants in any migration cycle (Table 5), consistent with their high molecular weight, low polarity, strong affinity for the cork suberin matrix, and low volatility, properties of UMC and MC constituents detected in raw materials, which support their complete retention within the CPC. Detection limits for for β-Sitosterol were 0.010 mg/kg food in ethanol 10% and 0.020 mg/kg food in acetic acid 3% and 0.005 mg/kg food for the remaining compounds. Similar negligible migration of cork-derived triterpenoids into aqueous simulants has been reported in the literature (Busta et al. 2020). In vegetable oil, these same compounds were detected in the blank oil controls, a result fully aligned with their natural occurrence in edible plant oils. β-Sitosterol alone typically ranges between 974–4,350 mg/kg in canola, corn, walnut and other vegetable oils, while lupeol and friedelin are also commonly present at trace-to-µg/g levels in many nuts and seeds (Abdallah et al. 2015; Yoon et al. 2025). Because these substances are intrinsic constituents of plant oils, their presence in the oil simulant cannot be attributed to migration from CPC, rendering oil-based migration testing analytically invalid for these analytes (Busta et al. 2020; Störmer et al. 2024; Yoon et al. 2025). Given the lack of detectable migration into aqueous simulants, combined with the well-documented natural abundance of these sterols and triterpenoids in common dietary oils, incorporation of cork into CPCs is not expected to contribute meaningfully to consumer exposure. These findings support the conclusion that migration assessment for these compounds should focus on aqueous simulants and that oil-based measurements are not suitable for exposure estimation.

Migration of PP-related substances

A limited number of PP-related volatile and semi-volatile compounds were detected in both PP and CPC samples, including acetophenone-related substances, m-diacetylbenzene, and antioxidants/degradation products (Table 6). Migration into aqueous simulants was very low and decreased across cycles. For example, migration of 1-[4-(1-methylethenyl)phenyl]ethanone (cas no. 5359-04-6) decreased to ≤0.00012 mg/kg in ethanol and ≤0.00005 mg/kg in acetic acid by the 3rd migration cycle (Figure 5(A)). CPC migration values were generally equal to or lower than those of PP, indicating that incorporation of cork does not

Table 6. Migration of PP-related volatile and semi-volatile compounds into food simulants (determined by HS-SPME-GC-MS) and target PP additives into vegetable oil (determined by LE-GC-MS) from CPC and PP control samples after 10 days at 60 °C under repeated-use conditions (mg/kg food or µg/kg food*).

Migrant	CAS	MW (g/mol)	Sample	1st migration	2nd migration	3rd migration
					Ethanol 10%	
Ethanone, 1-[4-(1-methylethyl)phenyl]-	645-13-6	162.23	PP	< LOQ	< LOQ	< LOQ
			CPC	< LOQ	< LOQ	< LOQ
Ethanone, 1-[4-(1-methylethenyl) phenyl]-	5359-04-6	160.21	PP	0.27 ± 0.08*	0.15 ± 0.023*	0.09 ± 0.004*
			CPC	0.41 ± 0.12*	0.24 ± 0.03*	0.12 ± 0.01*
m-Diacetylbenzene	6781-42-6	162.19	PP	0.10 ± 0.03*	0.05 ± 0.00*	0.02 ± 0.00*
			CPC	0.10 ± 0.01*	0.04 ± 0.01*	0.02 ± 0.00*
2,6-Di-tert-butyl-4-hydroxy-4-methylcyclohexa-2,5-dien-1-one	10396-80-2	236.35	PP	0.01 ± 0.00*	< LOQ	< LOQ
			CPC	0.01 ± 0.01*	< LOQ	< LOQ
					Acetic Acid 3%	
Ethanone, 1-[4-(1-methylethenyl) phenyl]-	5359-04-6	160.21	PP	0.09 ± 0.01*	0.06 ± 0.01*	0.05 ± 0.01*
			CPC	0.17 ± 0.03*	0.08 ± 0.02*	0.08 ± 0.03*
m-Diacetylbenzene	6781-42-6	162.19	PP	0.48 ± 0.07*	0.32 ± 0.01*	0.19 ± 0.06*
			CPC	0.35 ± 0.05*	0.12 ± 0.05*	0.10 ± 0.04*
					Vegetable Oil	
Ethanone, 1-[4-(1-methylethenyl) phenyl]-	5359-04-6	160.21	PP	0.0124 ± 0.0012	0.0067 ± 0.0008	0.0041 ± 0.0000
			CPC	0.0131 ± 0.0008	0.0054 ± 0.0009	0.0059 ± 0.0005
m-Diacetylbenzene	6781-42-6	162.19	PP	0.534 ± 0.0332	0.293 ± 0.0191	0.182 ± 0.0328
			CPC	0.048 ± 0.0138	0.0028 ± 0.0008	0.028 ± 0.0149
9,9-Bis(methoxymethyl)-9H-fluorene	182121-12-6	254.33	PP	0.025 ± 0.001	0.023/0.017/<LOQ	0.011/< LOQ/<LOQ
			CPC	0.020 ± 0.002	< LOQ (all values)	< LOQ (all values)
Tris(3,5-di-tert-butyl-4-hydroxybenzyl) Isocyanurate (**)	27676-62-6	784.10	PP	0.217 ± 0.017	0.092 ± 0.060	0.101 ± 0.006
			CPC	0.274 ± 0.012	0.118 ± 0.011	0.096 ± 0.017

All values are expressed as mean ± standard deviation of three replicate analyses where measurable. When 1 or more replicate is not measurable individual values are presented. (**) – Volatile fraction of the substance. LOQ - Limit of quantification: 0.01 µg/ kg food. For the molecular structures and CAS numbers of the identified migrants, see Figure S6.

cause an increase in the release of PP-related volatiles (Brandsch et al. 2021).

Migration into vegetable oil was higher, as expected, for hydrophobic PP-related substances. The highest values were observed for m-diacetylbenzene (Figure 5(B)), decreasing from 0.534 mg/kg (PP, 1st cycle) to 0.182 mg/kg (3rd cycle). CPC values were consistently lower, reaching 0.028 mg/kg in the 3rd cycle. Ethanone, 1-[4-(1-methylethenyl)phenyl]- was also detected in vegetable oil, decreasing across repeated uses from 0.0124 to 0.0041 mg/kg in PP and from 0.0131 to 0.0059 mg/kg in CPC (Figure 5(C)). Additional LE-GC-MS quantification confirmed m-diacetylbenzene at 0.02 µg/kg (PP and CPC) in ethanol and 0.19 vs 0.10 µg/kg in acetic acid, respectively (Table 6). An unknown trace compound (< 0.01 µg/kg, below the LOQ) was detected in both PP and CPC.

Targeted LE-GC-MS analysis quantified the migration of two PP additives: 9,9-bis(methoxymethyl)-9H-fluorene and tris(3,5-di-tert-butyl-4-hydroxybenzyl) isocyanurate—into vegetable oil (Table 6). The fluorene derivative, a polymerisation aid authorised under Regulation (EU) No 10/2011 with an SML of 0.05 mg kg^{-1}, and the isocyanurate antioxidant (SML 5 mg kg^{-1}) both showed decreasing migration across the three repeated-use cycles. For CPC, migration levels remained below the respective SMLs and equal to or lower than the the PP control in the third cycle. In addition, 9,9-bis(methoxymethyl)-9H-fluorene was below the LOQ in CPC during the second and third cycles, indicating minimal release upon repeated contact.

Overall, the migration behaviour of CPC demonstrates stable performance under repeated-use conditions. Cork-derived process contaminants (furfural and 5-methylfurfural) migrated at low µg–mg/kg levels but decreased consistently across cycles and remained well below toxicological thresholds. Natural cork constituents (triterpenoids and sterols) did not migrate into aqueous simulants, and migration into oil could not be reliably assessed due to natural background levels in the blank oil.

PP-related substances migrated at very low levels into aqueous simulants and at expected levels into vegetable oil, with CPC systematically showing equal or reduced migration compared to PP. All quantified PP additives complied with their respective SMLs. Taken together, the data indicate that incorporation of cork granules into PP does not increase migration of PP-related substances, does not introduce safety-relevant levels of cork-derived migrants, and leads to an overall stable migration profile compatible with food-contact use under repeated-use conditions.

Metal migration

To assess compliance with the specific migration limits (SMLs) defined in Regulation (EU) No 10/2011, potential migration was conservatively estimated assuming total release of metals from the cork fraction of a CPC article containing 15% cork (Table 7).

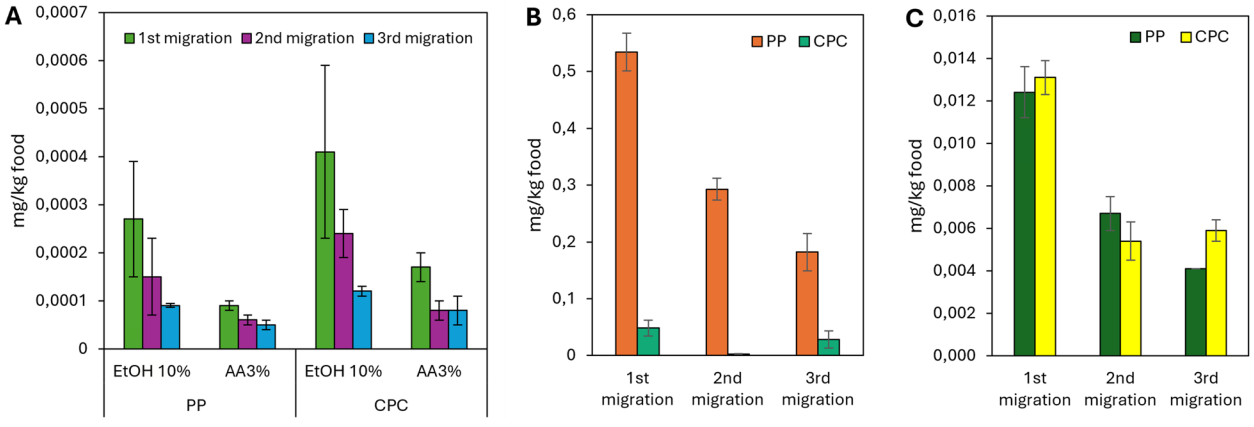

Figure 5. Migration of representative PP-related substances, from CPC and PP control samples across repeated-use cycles, determined by HS-SPME-GC-MS (Average ± SD, $n = 3$). Panel **A**: Migration of 1-[4-(1-methylethenyl)phenyl]ethanone into aqueous simulants (EtOH 10%, AA 3%). Panel **B:** Migration of m-diacetylbenzene into vegetable oil. Panel **C**: Migration of 1-[4-(1-methylethenyl)phenyl]ethanone into vegetable oil. Full quantitative data are provided in Table 6.

Table 7. Metals migration estimation from the CPC sample, with 15% of incorporated cork, (mg/kg food).

Metal	Al	Ba	Cd	Co	Cr	Cu	Fe	Li	Mn	Ni	Pb	Zn	As	Hg	Sb
Mean	0.9590	0.2116	0.0010	<LOD[1]	0.0024	0.2177	0.4410	0.1134	0.3641	<LOD[2]	0.0029	0.0877	< LOD[3]	< LOD[3]	< LOD[3]
St dev	0.1248	0.0192	0.0001	–	0.0002	0.0342	0.0345	0.0667	0.0471	–	0.0003	0.0225	–	–	–
SML (Reg. 10/2011)	1	1	ND (LOD[4])	0.05	ND (LOD[5])	5	48	0.6	0.6	0.02	ND (LOD[5])	5	ND (LOD[5])	ND (LOD[5])	0.04

LOD - Limit of Detection: 1 - 0.0002; 2 - 0.0002; 3 - 0.001; 4 - 0.002; 5 - 0.010; ND – not detectable.

Among all elements, only Al (0.96 mg kg^{-1} food) approached its SML of 1 mg kg^{-1}, while all others were well below their corresponding limits. Elements such as Cd, Cr, Ni, and Pb, subject to stricter SMLs or non-detectable (ND) thresholds, remained below the respective limits of detection (0.0002–0.010 mg kg^{-1} food). No elements exceeded their SMLs, confirming a favourable safety margin even under the conservative total-migration assumption.

Integrity and sensory properties of composite

Optical microscopy evaluation

Optical microscopy analysis of the CPC samples after repeated use (3 sequential contacts with fresh simulants) and after contact during 10, 20 and 30 days revealed that the surface structure remained largely unaltered compared to control samples with no simulant exposure (Figure 6). No fractures or relevant morphological changes were observed, indicating that the cork remained well embedded in the polypropylene matrix and that the composite maintained a stable structure under repeated-use conditions. The main differences detected were limited to surface colour and contrast variations under direct optical microscopy illumination. Samples in contact with 10% ethanol exhibited blue tones, those exposed to 3% acetic acid showed brown hues, and vegetable oil contact led to a more vivid brown colouration. Contrast changes were more noticeable for aqueous simulants (10% ethanol and 3% acetic acid) relative to the control. For samples exposed to vegetable oil, residual fat contributed to a slight reduction in apparent surface roughness, which may give the impression of interaction between the oil and the composite material due to the polyolefin nature of the matrix.

Sensory analysis

The inherent odour of sample discs and the odour and flavour transmitted to water simulant were evaluated for PP and CPCs.

Inherent odour (24 h, 23 °C)

PP discs exhibited minimal inherent odour, with average scores ranging from 0.0 (control) to 0.7

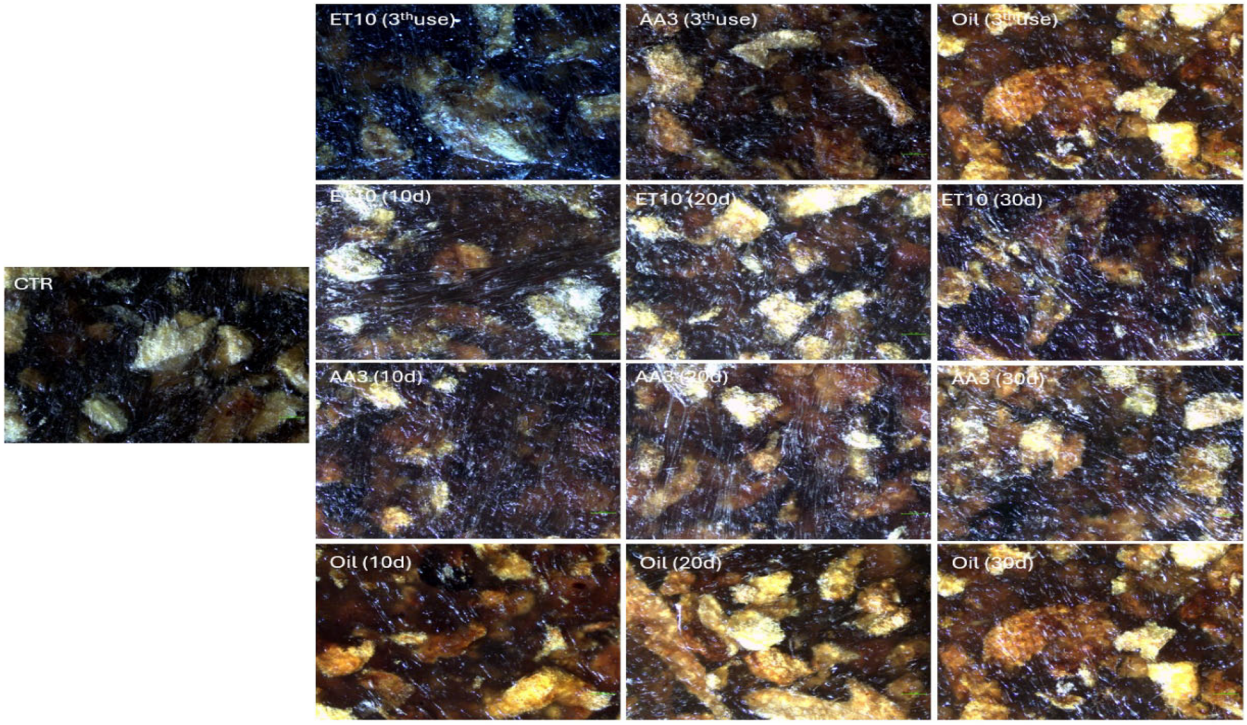

Figure 6. Optical microscopy images (40X magnification) of CPC composite surfaces after repeated use (3rd contact) and kinetic migration tests with food simulants: Ethanol 10% (Et10), Acetic Acid 3% (AA3), and Vegetable Oil (Oil), at intervals of 10, 20, and 30 days under 60 °C compared to a control sample (CTR) with no simulant contact.

(replicates) and descriptors indicating only a slight plastic note. Statistical analysis indicated that the hypothesis of no perceptible difference compared to the control was rejected for inherent odour at 95% probability, although the overall intensity remained low ('absence of odour/just perceptible odour'). In contrast, CPCs samples showed higher inherent odour scores (0.0–1.6), with descriptors such as 'wood/toasted.' The hypothesis of no perceptible difference was rejected, confirming that cork contributes significantly to the composite's odour (Table S5).

Odour and flavour transferred to simulant (10 days, 40 °C)

PP discs did not impart detectable odour or flavour, with average scores ≤0.2. The hypothesis of no perceptible difference was not rejected, indicating negligible sensory transfer (Table S5). Conversely, CPCs released low but perceptible odour (0.7–0.8) and flavour (1.7–1.8) into the simulant, with descriptors including chemical/plastic and plastic/wood. For CPCs, the hypothesis of no perceptible difference was rejected, confirming minor sensory transfer of volatile components from cork. Incorporation of cork into PP increased both inherent odour and the potential for sensory transfer to water simulant. While PP alone showed minimal sensory impact, CPCs exhibited moderate inherent odour and low-level odour and flavour release. These results are consistent with the presence of cork extractives, which may contribute volatile compounds detectable by sensory analysis. No defined EU acceptance criteria exist for sensory thresholds in this context; based on common practice, level 2 is considered the limit (Störmer et al. 2024), which aligns with the observed results for CPCs samples. Overall, the sensory impact of CPCs remains mild, supporting its suitability for food contact applications where slight cork character is acceptable.

Ames test

Table 8 presents the results for the Ames test performed over the migration solutions (ethanol - 10%, acetic acid - 3% and oil). Across all Salmonella strains evaluated (TA98, TA100, TA102, TA1535 and TA1537) none of the tested

Table 8. Ames test results. TA - *Salmonella typhimurium*. S9 - Mutazyme 30% S9 Mix rat liver PB/BNF. (-) negative. (-/+) weak mutagenic response.

	TA98	TA100	TA102	TA1535	TA1537
Ethanol 10% S9-	(i)	(i)	(iii)	(i)	(i)
Ethanol 10% S9+	(i)	(i)	(iii)	(i)	(i)
Ac acid S9-	(i)	(i)	(iii)	(i)	(i)
Ac acid S9+	(i)	(i)	(iii)	(i)	(i)
Oil S9-	(i)	(ii)	(iii)	-/+(iv)	-(i)
Oil S9+	(i)	(i)	(iii)	(i)	(i)

(i) The values remain close to simulant control and error bars overlap extensively with the controls. No dose-related increase was observed.
(ii) The oil sample shows consistently high revertant numbers for S9- at all tested concentrations, except at the lowest dose. The pattern is uniform, suggesting a matrix-related effect rather than a genotoxic effect (e.g. increased background noise, or stress effects on the bacteria (Vasetska et al. 2025).
(iii) All migration solutions and respective dilutions showed intermediate revertant counts without S9 and values with S9 at levels close to the controls; the values did not show a dose-dependent response.
(iv) The oil sample tested without S9 showed a slight dose-related increase (for at least three doses) in revertant numbers meeting the positivity criterion defined for the assay. Although the absolute counts remained low compared with the positive control, the highest response reached three times the solvent control value. This pattern was not observed in the presence of S9.

migration solutions induced a mutagenic response under the experimental condition, either with or without metabolic activation (S9 mix), with the exception of a weak increase observed for the oil extract in TA1535 in the absence of S9. Positive controls consistently produced robust increases in revertant numbers, confirming the sensitivity and proper performance of the assay. Simulant controls remained within expected limits, aside from a minor elevation in the ethanol baseline for TA1535 (S9–), which did not compromise assay validity (maron and Ames 1983). This indicates that the absence of DNA-reactive mutagenic substances resulting from the contact between the CPC and the food simulant.

Conclusions

This study provides a comprehensive and integrated safety evaluation of CPCs for food-contact applications, combining physicochemical characterisation, migration analysis, sensory evaluation, and genotoxicity assessment.

The results demonstrate that the incorporation of cork granulates, including both matured and unmatured fractions, does not compromise the structural integrity or migration performance of polypropylene. Migration levels of both cork-derived and polymer-related substances remained low, decreased under repeated-use conditions, and complied with applicable regulatory limits. Cork-derived furanic compounds were identified as process-related NIAS, but their occurrence at low levels and rapid depletion across cycles indicate limited relevance for consumer exposure. Furthermore, the outcomes of the Ames test demonstrate the absence of DNA-reactive mutagenic hazards even considering that other substances including NIAS, undetected by the chromatographic techniques used, are potentially present. Overall, the combined chemical and toxicological evidence support the safe use of this bio-based composites for food-contact applications, supporting the circular valorisation of cork by-products.

To successfully transition these sustainable cork-polypropylene composites from a laboratory scale to commercial food contact applications, a multi-step roadmap focusing on technical optimisation, targeted food-pairing, and industrial scaling would be needed. In particular, it is recommended to validate the assessments over samples obtained at industry scale with analyses of batch-to-batch and corresponding sensitivity analyses, as well as long-term durability and stress testing to assess the potential impact in safety.

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Author contributions

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Methodology, Supervision, Validation, Writing – original draft; **Lisete Moutinho**: Project administration, Resources, Validation, Visualization, Writing – review & editing; **Clara Sousa**: Investigation, Methodology, Writing – original draft; **Fátima Poças**: Conceptualization, Funding acquisition, Resources, Supervision, Validation, Writing – review & editing.

Disclosure statement

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References

[EC] European Commission. 2004. Regulation (EC) No 1935/2004 of the European Parliament and of the Council of 27 October 2004 on materials and articles intended to come into contact with food. Off J Eur Union L. 338:4–17.

[EC] European Commission. 2006. Regulation (EC) No 2023/2006 of 22 December 2006 on good manufacturing practice for materials and articles intended to come into contact with food. Off J Eur Union L. 384:75–78.

[EC] European Commission. 2025. Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food (Consolidated version of 16 March 2025). Off J Eur Union. 12:1–89 (2011 Jan 15). https://eur-lex.europa.eu/eli/reg/2011/10/2025-03-16/eng

[OECD] Organisation for Economic Co-operation and Development. 2020. Guidelines for the testing of chemicals. Section 4: test No. 471: bacterial reverse mutation test.

Abdallah F, Cheikh B, Baklouti M, Denchev Z, Cunha A. 2010. Effect of surface treatment in cork reinforced composites. J Polym Res. 17(4):519–528. https://doi.org/10.1007/s10965-009-9339-y

Abdallah IB et al. 2015. Content of carotenoids, tocopherols, sterols, triterpenic and aliphatic alcohols, and volatile compounds in six walnuts (*Juglans regia* L.) varieties. Food Chem. 173:972–978. https://doi.org/10.1016/j.foodchem.2014.10.095

Alañón M et al. 2011. Antioxidant capacity and phenolic composition of different woods used in cooperage. Food Chem. 129(4):1584–1590. https://doi.org/10.1016/j.foodchem.2011.06.013

Alsberg BK, Kell DB, Goodacre R. 1998. Variable selection in discriminant partial least-squares analysis. Anal Chem. 70(19):4126–4133. https://doi.org/10.1021/ac980506o

Aroso I, Araújo A, Pires R, Reis R. 2017. Cork: current technological developments and future perspectives for this natural, renewable, and sustainable material. ACS Sustainable Chem Eng. 5(12):11130–11146. https://doi.org/10.1021/acssuschemeng.7b00751

Azevedo J et al. 2014. Migration of phenolic compounds from different cork stoppers to wine model solutions: antioxidant and biological relevance. Eur Food Res Technol. 239(6):951–960. https://doi.org/10.1007/s00217-014-2292-y

Benabdallah F, Safra I, Ghachem K, Alwadai N, Kolsi L. 2024. Micromechanical modeling and experimental characterization of thermoplastic cork filler composites. Polym Polym Compos. 32:1–16. https://doi.org/10.1177/09673911241246625

Bharani K, Khatoon R, Lalchandani D, Chenkual L, Porwal P. 2024. Determination of α-oxo-aldehydes and furfurals using HPLC-PDA-ELSD method in sugar-containing food products. Food Chem Adv. 4:100614. https://doi.org/10.1016/j.focha.2024.100614

Brandsch R, Pemberton M, Schuster D, Welle F. 2021. Impact of partitioning in short-term food contact applications focused on polymers in support of migration modelling and exposure risk assessment. Molecules. 27(1):121. https://doi.org/10.3390/molecules27010121

Brandsch R, Schuster D. 2020. Repeated use food contact materials: a categorisation approach in support of risk assessment. Food Addit Contam Part A Chem Anal Control Expo Risk Assess. 37(12):2184–2203. https://doi.org/10.1080/19440049.2020.1798512

Busta L et al. 2020. Oxidosqualene cyclases involved in the biosynthesis of triterpenoids in *Quercus suber* cork. Sci Rep. 10(1):8011. https://doi.org/10.1038/s41598-020-64913-5

Carriço CM et al. 2023. Added-value compounds in cork by-products: methods for extraction, identification, and quantification of compounds with pharmaceutical and cosmetic interest. Molecules. 28(8):3465. https://doi.org/10.3390/molecules28083465

Castola V et al. 2002. Composition and chemical variability of the triterpene fraction of dichloromethane extracts of cork (Quercus suber L.). Ind Crops Prod. 15(1):15–22. https://doi.org/10.1016/s0926-6690(01)00091-7

Champ T et al. 2021. Lignin-derived non-heme iron and manganese complexes: catalysts for the on-demand production of chlorine dioxide in water under mild conditions. Inorg Chem. 60(5):2905–2913. https://doi.org/10.1021/acs.inorgchem.0c02742

Cincotta F, Verzera A, Tripodi G, Condurso C. 2018. Non-intentionally added substances in PET bottled mineral water during the shelf-life. Eur Food Res Technol. 244(3):433–439. https://doi.org/10.1007/s00217-017-2971-6

DIN 10955. 2004. Food analysis – sensory analysis – evaluation of odour and flavour transfer from food contact materials. Germany: Deutsches Institut für Normung eV.

EFSA AFC Panel. 2004. Opinion of the scientific panel on food additives, flavourings, processing aids and materials in contact with food (AFC) on a request from the Commission related to furfural and furfural diethyl acetal. EFSA J. 67:1–27. https://doi.org/10.2903/j.efsa.2004.67

Esteban J, Vorholt A, Leitner W. 2020. An overview of the biphasic dehydration of sugars to 5-hydroxymethylfurfural and furfural: a rational selection of solvents using COSMO-RS and selection guides. Green Chem. 22(7):2097–2128. https://doi.org/10.1039/C9GC04208C

Esteves B et al. 2017. Cork liquefaction for polyurethane foam production. BioResources. 12(2):2339–2353. https://doi.org/10.15376/biores.12.2.2339-2353

European Commission. 2011. Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food (Text with EEA relevance). Off J Eur Union L. 12:1–89.

European Parliament and Council. 2025. Regulation (EU) 2025/40 of the European Parliament and of the Council of 19 December 2024 on packaging and packaging waste, amending Regulation (EU) 2019/1020 and Directive (EU) 2019/904 and repealing Directive 94/62/EC. Off J Eur Union L. 40 (2025 Jan 22). https://eur-lex.europa.eu/eli/reg/2025/40/oj

Farag M et al. 2020. The toxicological aspects of the heat-borne toxicant 5-hydroxymethylfurfural in animals: a review. Molecules. 25(8):1941. https://doi.org/10.3390/molecules25081941

Faria DP, Fonseca AL, Pereira H, Teodoro OMND. 2011. Permeability of cork to gases. J Agric Food Chem. 59(8):3590–3597. https://doi.org/10.1021/jf200491t

Fernandes E, Correlo V, Chagas J, Mano J, Reis R. 2011. Properties of new cork–polymer composites: advantages and drawbacks as compared with commercially available fibreboard materials. Compos Struct. 93:3120–3129. https://doi.org/10.1016/j.compstruct.2011.06.020

Fernandes E, Correlo V, Mano J, Reis R. 2014. Polypropylene-based cork–polymer composites: processing parameters and properties. Compos Part B-Eng. 66:210–223. https://doi.org/10.1016/j.compositesb.2014.05.019

Ferreira R et al. 2012. Suberin isolation from cork using ionic liquids: characterisation of ensuing products. New J Chem. 36(10):2014–2024. https://doi.org/10.1039/c2nj40433h

Gancel A, Jourdes M, Pons A, Teissèdre P. 2023. Migration of polyphenols from natural and microagglomerated cork stoppers to hydroalcoholic solutions and their sensory impact. OENO One. 57(3):13–16. https://doi.org/10.20870/oeno-one.2023.57.3.7393

Gao Q et al. 2024. Investigation of the formation of furfural compounds in apple products treated with pasteurization and high pressure processing. Food Res Int. 190:114546. https://doi.org/10.1016/j.foodres.2024.114546

Gaspar M, Braga M. 2023. Edible films and coatings based on agrifood residues: a new trend in the food packaging research. Curr Opin Food Sci. 50:101006. https://doi.org/10.1016/j.cofs.2023.101006

Gil L. 2009. Cork composites: a review. Materials. 2(3):776–789. https://doi.org/10.3390/ma2030776

Gil L. 2015. New cork-based materials and applications. Materials (Basel). 8(2):625–637. https://doi.org/10.3390/ma8020625

Guan M-Y et al. 2023. Formation and migration of 5-hydroxymethylfurfural and furfural from food contact bamboo sticks during heating and their safety evaluation. J Food Compos Anal. 117:105146. ISSN 0889-1575 https://doi.org/10.1016/j.jfca.2023.105146

Haurie L, Malet R, Realinho V. 2023. The components' roles in thermal stability and flammability of cork powder. Materials. 16:3829. https://doi.org/10.3390/ma16103829

Hejna A. 2022. More than just a beer—the potential applications of by-products from beer manufacturing in polymer technology. Emergent Mater. 5(3):765–783. https://doi.org/10.1007/s42247-021-00304-4

Irmak S, Kang J, Wilkins M. 2020. Depolymerization of lignin by wet air oxidation. Bioresour Technol Rep. 9:100377. https://doi.org/10.1016/j.biteb.2019.100377

ISO 11358:1997(E). 1997. Plastics—thermogravimetry (TG) of polymers—general principles. Switzerland: ISO.

ISO 1183-1. 2019. Plastics—methods for determining the density of non-cellular plastics—part 1: immersion method, liquid pycnometer method and calculation. Switzerland: ISO.

ISO 13302. 2003. Sensory analysis—methodology—selection, training and monitoring of assessors. International Organization for Standardization.

ISO 8589. 2007. Sensory analysis—general guidance for the design of test rooms. Switzerland: International Organization for Standardization.

Kasmi S, Gallos A, Beaugrand J, Paës G, Allais F. 2019. Ferulic acid derivatives used as biobased powders for a convenient plasticization of polylactic acid in continuous hot-melt process. Eur Polym J. 110:293–300. https://doi.org/10.1016/j.eurpolymj.2018.11.036

Khan M et al. 2023. Effect of ferulic acid derivative concentration on the release kinetics, antioxidant capacity, and thermal behaviour of different polymeric films. Food Chem. 410:135395. https://doi.org/10.1016/j.foodchem.2023.135395

Knapic S, Oliveira V, Machado J, Pereira H. 2016. Cork as a building material: a review. Eur J Wood Prod. 74(6):775–791. https://doi.org/10.1007/s00107-016-1076-4

Kobelev KV et al. 2023. Study of Brewer's Spent grain environmentally friendly processing ways. Molecules. 28(11):4553. https://doi.org/10.3390/molecules28114553

Kowalski S, Lukasiewicz M, Duda-Chodak A, Zięć G. 2013. 5-hydroxymethyl-2-furfural (HMF) – heat-induced formation, occurrence in food and biotransformation – a review. Pol J Food Nutr Sci. 63(4):207–225. https://doi.org/10.2478/v10222-012-0082-4

Larguech S, Triki A, Ramachandran M, Kallel A. 2021. Dielectric properties of jute fibers reinforced poly(lactic acid)/poly(butylene succinate) blend matrix. J Polym Environ. 29(4):1240–1256. https://doi.org/10.1007/s10924-020-01927-0

Lee J et al. 2022. Unravelling caramelization and Maillard reactions in glucose and glucose + leucine model cakes: formation and degradation kinetics of precursors, α-dicarbonyl intermediates and furanic compounds during baking. Food Chem. 376:131917. https://doi.org/10.1016/j.foodchem.2021.131917

Llevot A, Grau E, Carlotti S, Grelier S, Cramail H. 2016. From lignin-derived aromatic compounds to novel biobased polymers. Macromol Rapid Commun. 37(1):9–28. https://doi.org/10.1002/marc.201500548

Maarse H, Visscher CA et al. 1994. Volatile compounds in foods and beverages. Marcel Dekker.

Maron DM, Ames BN. 1983. Revised methods for the Salmonella mutagenicity test. Mutation. Mutat Res. 113(3-4):173–215. https://doi.org/10.1016/0165-1161(83)90010-9

Martins C, Gil V. 2020. Processing–structure–properties of cork polymer composites. Front Mater. 7:297. https://doi.org/10.3389/fmats.2020.00297

Meng D, Zhang F, Jia W, Jiao J, Zhang Y. 2024. Understanding the health implications of furan and its derivatives in thermally processed foods: occurrence, toxicology, and mixture analysis. Curr Opin Food Sci. 57:101151. ISSN 2214-7993. https://doi.org/10.1016/j.cofs.2024.101151

Miralles P, Fuentes-Ferragud E, Socas-Hernández C, Coscollà C. 2025. Recent trends and challenges on the non-targeted analysis and risk assessment of migrant non-intentionally added substances from plastic food contact materials. Toxics. 13(7):543. https://doi.org/10.3390/toxics13070543

Mironeasa S, Coțovanu I, Mironeasa C, Ungureanu-Iuga M. 2023. A review of the changes produced by extrusion cooking on the bioactive compounds from vegetal sources. Antioxidants (Basel). 12(7):1453. https://doi.org/10.3390/antiox12071453

Mota G et al. 2016. Cellular structure and chemical composition of cork from *Plathymenia reticulata* occurring in the Brazilian Cerrado. Ind Crops Prod. 90:65–75. https://doi.org/10.1016/j.indcrop.2016.06.014

Onnerud H, Zhang L, Gellerstedt G, Henriksson G. 2002. Polymerization of monolignols by redox shuttle-mediated enzymatic oxidation: a new model in lignin biosynthesis. Plant Cell. 14(8):1953–1962. https://doi.org/10.1105/TPC.001487

Ortega-Fernandez C, Gonzalez-Adrados JR, García-Vallejo MC, Calvo-Haro R, Caceres-Esteban MJ. 2006. Characterization of surface treatments of cork stoppers by FTIR-ATR. J Agric Food Chem. 54(14):4932–4936. https://doi.org/10.1021/jf0529823

Ortu E, Caboni P. 2017. Levels of 5-hydroxymethylfurfural, furfural, 2-furoic acid in sapa syrup, Marsala wine and bakery products. Int J Food Prop. 20(sup3):S2543–S2551. https://doi.org/10.1080/10942912.2017.1373668

Paiva D, Magalhães FD. 2018. Dynamic mechanical analysis and creep-recovery behavior of agglomerated cork. Eur J Wood Prod. 76(1):133–141. https://doi.org/10.1007/s00107-017-1158-y

Palombini F et al. 2023. 3D cellular characterization and finite element analysis of cork compressive behavior based on high-resolution X-ray microtomography. Wood Sci Technol. 57(4):903–928. https://doi.org/10.1007/s00226-023-01483-5

Páscoa RNMJ et al. 2024. Application of NIR spectroscopy for the valorisation of cork by-products: a feasibility study over the screening and discrimination of chemical compounds of interest. Pharmaceuticals (Basel). 17(2):180. https://doi.org/10.3390/ph17020180

Pereira H. 1988. Chemical composition and variability of cork from *Quercus suber* L. Wood Sci Technol. 22(3):211–218. https://doi.org/10.1007/BF00386015

Pereira H. 2007. Cork biology (Part 1). In: Pereira H, editor. Cork: biology, production and uses. Elsevier. p.7–99. https://doi.org/10.1016/B978-0-444-52967-1.X5000-6

Pereira H. 2013. Variability of the chemical composition of cork. BioResources. 8(2):2246–2256. https://doi.org/10.15376/biores.8.2.2246-2256

Petinakis E et al. 2009. Effect of matrix–particle interfacial adhesion on the mechanical properties of poly(lactic acid)/wood-flour micro-composites. J Polym Environ. 17(2):83–94. https://doi.org/10.1007/s10924-009-0124-0

Petlitckaia S et al. 2024. Valorization of cork and high-density polyethylene and polypropylene wastes in cork–plastic composites: their morphology, mechanical performance, and fire properties. J Compos Sci. 8(6):195. https://doi.org/10.3390/jcs8060195.

Pinto C et al. 2023. Cork by-products as a sustainable source of potential antioxidants. Sustainable Chem Pharm. 36:101252. https://doi.org/10.1016/j.scp.2023.101252

Ponte-e-Sousa J, Neto-Vaz AM. 2011. Cork and metals: a review. Wood Sci Technol. 45:183–202. https://doi.org/10.1007/s00226-009-0288-8

Rego L et al. 2023. *Quercus suber* bark as a sustainable source of value-added compounds: experimental studies with cork by-products. Forests. 14(3):543. https://doi.org/10.3390/f14030543

Rocha S, Delgadillo I, Correia A. 1996. GC-MS study of volatiles of normal and microbiologically attacked cork from Quercus suber L. J Agric Food Chem. 44(3):865–871. https://doi.org/10.1021/jf9500400

Şen A, Bulcke JV, Defoirdt N, Acker JV, Pereira H. 2014. Thermal behaviour of cork and cork components. Thermochim. Acta. 582:94–100. https://doi.org/10.1016/j.tca.2014.03.007

Serra G, Fernandes F, De Sousa R, Noronha E, Ptak M. 2022. New hybrid cork-STF (Shear thickening fluid) polymeric composites to enhance head safety in micro-mobility accidents. Compos Struct. 301:116138. https://doi.org/10.1016/j.compstruct.2022.116138

Silva F, Dourado F, Gama M, Poças F. 2020. Nanocellulose bio-based composites for food packaging. Nanomaterials (Basel). 10(10):2041. https://doi.org/10.3390/nano10102041

Silva SP et al. 2005. Cork: properties, capabilities and applications. Int Mater Rev. 50(6):345–365. https://doi.org/10.1179/174328005X41168

Spiliotopoulos D, Koelbert C. 2020. Assessment of the miniaturized liquid Ames microplate format (MPF™) for a selection of the test items from the recommended list of genotoxic and non-genotoxic chemicals. Mutat Res Genet Toxicol Environ Mutagen. 856-857:503218. https://doi.org/10.1016/j.mrgentox.2020.503218

Störmer A, Hetzel L, Franz R. 2024. A critical review of test methods and alternative scientific approaches to compliance and safety evaluation of paper and board for food contact. Front Chem. 12:1397913. https://doi.org/10.3389/fchem.2024.1397913

Suffo M, Pérez-Muñoz C, Alba G, Villar M. 2024. An innovative polypropylene/waste cork composite material for spirit and wine stopper caps. Appl Sci. 14(7):3014. https://doi.org/10.3390/app14073014

Sun L et al. 2021. Facile synthesis and performance of pH/temperature dual-response hydrogel containing lignin-based carbon dots. Int J Biol Macromol. 175:516–525. https://doi.org/10.1016/j.ijbiomac.2021.02.049

Svetlana P, Toussaint B, Paul-Antoine S, Yann Q. 2024. Valorisation of virgin cork by the design of cork–plaster composites. Inorg Chem Commun. 161:111782. https://doi.org/10.1016/j.inoche.2023.111782

Tedjditi A, Ghomari F, Taleb O, Belarbi R, Bouhraoua R. 2020. Potential of using virgin cork as aggregates in development of new lightweight concrete. Constr Build Mater. 265:120734. https://doi.org/10.1016/j.conbuildmat.2020.120734

Vasetska O, Bubalo V, Usenko T, Zhminko P, Prodanchuk M. 2025. Antimutagenic effects of 2,6-dimethylpyridine-N-oxide using fluctuation ames test. Toxicol Rep. 14:101989. https://doi.org/10.1016/j.toxrep.2025.101989

Vera P, Canellas E, Nerín C. 2018. Identification of non-volatile migrant compounds and NIAS in polypropylene films used as food packaging characterized by UPLC-MS/QTOF. Talanta. 188:750–762. https://doi.org/10.1016/j.talanta.2018.06.022

Yoon B-H, Truong V-L, Jeong W-S. 2025. Phytosterols: extraction methods, analytical techniques, and biological activity. Molecules. 30(12):2488. https://doi.org/10.3390/molecules30122488

Zhang Y, , Song Y, Li Q. 2012. A Review on formation mechanism, determination and safety assessment of furfural and 5-hydroxymethylfurfural (HMF) in foods. Food Sci. 33(5):275–280. https://doi.org/10.7506/spkx1002-6630-201205059