



UV-LED irradiation of black pepper: A non-thermal approach to preserve sensory quality

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ARTICLE INFO

Keywords:

Non-thermal
Sensory analysis
Food quality
UV LED irradiation
Spices

ABSTRACT

The impact of ultraviolet light-emitting diode (UV-LED) irradiation on the chemical and sensory properties of black peppercorns is investigated in this study. Samples were treated with 280-, 300-, and 365-nm light, as well as at 280 + 300 nm. Total phenolic content, antioxidant activity, phenolic compounds, volatiles, piperine, fatty acids, and tocopherols were analysed, followed by a sensory analysis. Chemical and instrumental analyses revealed moderate changes (up to 40%) in phenolic compounds, volatiles, fatty acids, and tocopherols, with the combined wavelength treatment often resulting in a greater deterioration, and agitation being the main driver for quality degradation. However, sensory evaluation showed no significant differences in colour, smell, or flavour, indicating that organoleptic properties remained unaffected. The findings suggest that UV-LEDs offer a valuable and feasible alternative to conventional heat and water vapour treatments, avoiding humidity-related issues whilst preserving the product's organoleptic integrity. UV-LEDs demonstrated potential for non-thermal spice treatment with minimal sensory impact.

1. Introduction

Black pepper (*Piper nigrum*) is one of the most widely consumed spices globally, valued not only for its pungent flavour and aroma but also for its bioactive compounds, including piperine, phenolics, and essential oils (Ravindran & Kallapurackal, 2012). However, like many dried spices, black pepper is susceptible to microbial contamination during post-harvest handling, storage, and distribution. Traditional decontamination methods, such as heat and steam treatments, have been used to ensure microbial safety, although, they often compromise the product's sensory and nutritional quality (Choi et al., 2020; Polovka & Suhaj, 2010). However, they present significant drawbacks. Beyond their high energy consumption, which makes them a non-environmentally friendly option, these conventional processes compromise product quality. Thermally sensitive nutrients, such as vitamins and carbohydrates, can be partly lost or transformed into other compounds, and flavour and smell are affected by the acceleration in the volatilisation of important components of essential oils. For this reason, alternative non-thermal treatments have led to a growing interest in

recent years.

Ultraviolet (UV) radiation has emerged as a promising non-thermal technology for microbial inactivation in food systems. Among the UV treatments available, the use of conventional mercury-based lamps emitting at UV-C (254 nm) has been extensively studied for its germicidal properties (Arcos-Limiñana, Brunet, et al., 2025). However, the recent development of UV light-emitting diodes (UV-LEDs) offers several advantages over conventional mercury-based UV lamps, including tuneable wavelengths, lower energy consumption, longer lifespan, and environmental safety (Soro et al., 2023). UV-LEDs operating at specific wavelengths have shown potential for microbial reduction in various food matrices (Koutchma et al., 2019; Nyhan et al., 2021), resulting in a significant decrease of a number of foodborne pathogens in black pepper (Arcos-Limiñana et al., 2026), but their impact on the chemical and sensory attributes of spices remains underexplored.

The application of UV-LEDs to spices such as black pepper presents a unique challenge. Spices are complex matrices rich in volatile and other compounds that contribute to their characteristic flavour, aroma, and health-promoting properties (Ravindran & Kallapurackal, 2012). Any

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<https://doi.org/10.1016/j.ifset.2026.104758>

Received 18 March 2026; Received in revised form 14 July 2026; Accepted 24 August 2026

Available online 25 August 2026

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treatment that alters these compounds may affect consumer acceptance and market value. Although the combination of UV radiation with mild thermal treatments has been explored to enhance microbial reduction on food matrices, its application to spices remains scarce and limited. Recent attempts to combine UV-C with mild heat on whole peppercorns or red pepper powder have only evaluated superficial parameters, such as instrumental colour or specific capsaicinoids (Cheon et al., 2015; Park et al., 2019). Since black pepper is rich in volatile compounds, any thermal treatment can accelerate their volatilisation and chemical oxidation. Furthermore, the physical configuration of the treatment system, such as the use of reactors –i.e., containers that move the sample by means of stirring, vibration, air circulation, or any other means can influence the extent of chemical changes during UV exposure (Arcos-Limiñana, Brunet, et al., 2025).

In a previous work, it was observed that the microbial disinfection efficacy of using a multi-wavelength UV-LED system against key food-borne pathogens on black peppercorns, establishing its potential for non-thermal decontamination (Arcos-Limiñana et al., 2026). On the basis of these findings, the main objective of this study was to evaluate the comprehensive phytochemical, volatile, and sensory impact of multi-wavelength UV-LED radiation (280, 300, 365 nm and their combination) on black peppercorns using a mechanical reactor. This work aims to characterise the chemical stability and consumer acceptability of peppercorns subjected to UV treatments under different wavelengths, ensuring its overall viability and progressing into the industrial development of this technology for food processing.

2. Methodology

2.1. Reactants

Gallic acid (> 97.2%), caffeic acid (>98%), quercetin (>95%) and trans-ferulic acid (>99%) were obtained from Sigma Aldrich, (Shanghai, China). *cis*-3-Hexenyl acetate (>98%), trans-cinnamic acid (>99%), *p*-Hydroxybenzoic acid (>99%), vanillic acid (>97%), *p*-coumaric acid (>98%) and syringic acid (98%) were purchased from Thermo Fisher Scientific, (Idstein, Germany). Folin-Ciocalteu reagent (2 M), 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox, 97%) were obtained from Sigma Aldrich (Buchs, Switzerland). Vanillin (99%) was obtained from Sigma Aldrich (Lyon, France). Succinic acid (>99.5%) was purchased from Sigma Aldrich (Vienna, Austria). Sodium methoxide (25% w/v), *n*-Hexane (>99%), NaCO₃, D-(–)-quinic Acid (98%) and protocatechuic acid (>99%) were obtained from Sigma Aldrich (Idstein, Germany). FAME 37 multistandard, alpha-tocopherol (neat) and Delta-tocopherol (neat) were obtained from Supelco (Bella-monte, PA, USA). Ethanol (96%) was obtained from VWR international (Radnor PA, USA). (–)-Epicatechin (>99%) and (+)-catechin (>99%) were purchased from Extrasynthèse (Genay, France). Sodium methoxide (25% w/v) and *n*-Hexane (>99%) were obtained from Sigma Aldrich, USA. Sulfuric acid (96%) was obtained from Panreac (Barcelona, Spain).

2.2. Samples and UV-LED treatment

A single batch of black peppercorns originating from Vietnam (4 to 4.5 mm of diameter), purchased from a local market in Brussels, Belgium, was subjected to a UV treatment at different wavelengths (280, 300, and 365 nm), utilising a UV-LED device (PearlLab Beam, Aquisense Technologies, NC, USA). The experimental configuration consisted of a 90-mm diameter petri dish containing the sample, placed on an MIT000420 vibrating shaker (Technoflux, Barcelona, Spain) to facilitate the movement and rotation of the peppercorns during irradiation, which is necessary for an even dose distribution and efficient microbial disinfection, as shown in our previous works (Arcos-Limiñana, Brunet, et al., 2025; Arcos-Limiñana, Maestre-Pérez, & Prats-Moya, 2025). The reactor vibrated at a frequency of 10 Hz, an amplitude of 4.5 mm and a RMS

acceleration of approximately 12.11 m/s². The UV-LED source was placed 5 cm above the sample surface, as shown elsewhere (Arcos-Limiñana et al., 2026). UV irradiance was quantified using an optical meter, ILT2400 Optometer (International Light Technologies, USA), and the irradiance levels and doses are presented in Table 1. Fifteen-gram samples were exposed to UV treatment durations of 10, 20, and 30 min using each of the three individual wavelengths, as well as simultaneous irradiation with 280 and 300 nm, in accordance with the findings of a preceding experiment that found those two wavelengths to be the most effective against bacteria (Arcos-Limiñana et al., 2026). To ensure safety and maintain dark conditions, the experimental setup was enclosed in a box during irradiation. A control setup was prepared to evaluate the impact of vibration and differentiate it from the radiation effect. The experimental setup was identical to the rest of the experiments, with the shaker on but the LEDs turned off. Three different samples were subjected to each treatment and then pooled. The analyses were carried out in triplicate and were compared to the untreated peppercorn.

2.3. Total phenolic content and antioxidant activity

Before the analysis of total phenolic content (TPC) and antioxidant activity, an ethanolic extraction was performed on the samples. A hundred milligrams of ground pepper were vortexed with 5 mL of 70% ethanol in water (v/v) for 30 s. Afterwards, the mixture was placed in an ultrasonic bath (JP Selecta, Spain) for 20 min. After that, it was centrifuged at 5000 rpm for 10 min, and the supernatant was collected. The pellet was resuspended in 5 mL of 70% ethanol, and the extraction process was repeated, combining both supernatant extracts. All extractions were performed in triplicate. The extracts were filtered through a 0.45 µm pore size nylon membrane (Filtros Anolía, Barcelona, Spain) and stored at –15 °C.

The FRAP method was carried out according to Benzie and Strain (1996), utilising 100 µL of extract and Trolox as the standard. Absorbance was measured at 593 nm after 30 min of dark incubation with the FRAP reagent. A Trolox solution was employed as a reference standard, ranging between 1 and 7 ppm, and 0.3 and 1.2 absorbance units, respectively. The TPC was determined using the Folin-Ciocalteu method (Singleton & Rossi, 1965). For this procedure, 200 µL of extract was mixed with 100 µL of Folin reagent and 4.5 mL of distilled water. After one minute, 500 µL of 7% Na₂CO₃ was added. The solution was allowed to rest for 90 min at room temperature, and absorbance was measured at 760 nm, with concentrations of gallic acid as the standard ranging between 3 and 13 ppm, and 0.2 and 1.2 absorbance units, respectively. Results were expressed as mg of gallic acid equivalents.

2.4. Phenolic composition

A phenolic profile analysis was conducted using an Agilent 1290 Infinity Online UHPLC equipped with a triple quadrupole spectrometer featuring JetStream and iFunnel technology (UHPLC-1290/QQQ-6490; Agilent, CA, USA). Five microliters of the ethanolic extract were injected into the UHPLC and eluted at a flow rate of 0.5 mL/min using a Poroshell 120 EC-18 2.7 µm 3 × 100 mm column (Agilent, CA, USA). The mobile phase consisted of a mixture of two solvents. Phase A consisted of

Table 1

UV irradiance and dosage, depending on the wavelength and the time of exposure at a 5 cm distance from the sample.

Wavelength (nm)	Irradiance (W/cm ²)	Dose 10 min (J/cm ²)	Dose 20 min (J/cm ²)	Dose 30 min (J/cm ²)
280	0.068	40.8	81.6	122.4
300	0.049	29.4	58.8	88.2
365	0.043	25.8	51.6	77.4
280 + 300	0.112	67.2	134.4	201.6

ultrapure water with 0.1% formic acid while phase B was acetonitrile with 0.1% formic acid. The gradient of the mobile phase is detailed in Table 2. Both negative and positive ion modes were employed with the following parameters: gas temperature of 275 °C, flow rate of 11 mL/min, and nebuliser pressure of 40 psi. Standard curves were generated using various dilutions of stock solutions ranging from 0.01 to 8 ppm for each analysed compound (epicatechin (Ep), catechin (Cc), vanillin (Vn), succinic acid (Su), trans-ferulic acid (Fe), p-hydroxybenzoic acid (Hb), caffeic acid (Ca), quercetin (Qt), protocatechuic acid (Pr), gallic acid (Ga), trans-cinnamic acid (Ci), p-coumaric acid (Cu), syringic acid (Sy), vanillic acid (Vc), and quinic acid (Qa)). Additionally, neochlorogenic acid (Nc), chlorogenic acid (Ch), rutin (Ru), phlorizin (pH), kaempferol (Ka), and hesperidin (He) were qualitatively analysed. The results were expressed as the retention percentage of organic acids in black peppercorns after the UV treatment. The results were expressed as the retention percentage of phenolic compounds in black peppercorns after the UV treatment.

2.5. Volatile composition

A HS-SPME GC–MS method was used for volatile determination following a modified version of the methodology described by Baky et al. (2024). A hundred milligrams of powdered sample was placed in 15 mL SPME screw-cap vials and spiked with 5 µL of cis-3-hexenyl acetate as an internal standard (IS). Analyses were conducted using an adapted version of the methodology by Juan-Polo et al. (2021) with an Agilent 7890B gas chromatograph (Palo Alto, CA, USA) coupled to an Agilent 5977B mass spectrometer (Palo Alto, CA, USA). Compounds were separated on a fused silica capillary J&W DB-624 GC Column (30 m, 0.25 mm i.d., 1.40 µm film thickness) from Agilent (Palo Alto, CA, USA); injector port and interface temperature were set at 250 °C. Helium was used as the carrier gas at a flow rate of 1 mL/min, and samples were analysed in triplicate. Samples were equilibrated at 75 °C for 25 min. Subsequently, the SPME fiber (DVB/CAR/PDMS SPME, 50/30 µm, StableFlex, 1 cm long) fixed on the mechanical arm was exposed to the headspace of the sample vial for compound extraction. After 60 min of exposure, the fiber was withdrawn and inserted into the GC injector for desorption (3 min) and analysis. The column temperature was programmed to start at 70 °C (held for 2 min) and increase to 250 °C (held for 12 min) at a rate of 5 °C/min. Samples were injected into the column with a 1:50 split ratio. Mass spectrometric acquisition parameters included: electron energy of 70 eV, source temperature of 230 °C, quadrupole temperature of 150 °C, mass range of m/z 15–350, scan rate of 3.62 s/scan, and EM voltage of 1190 V. Mass spectra were compared with the National Institute of Standards and Technology (NIST) database. Signal standardisation was performed by dividing the raw area by the IS and then by the mass of the sample in grams in the vial. All volatile data analyses were conducted using the standardised signal per gram and were expressed as retention percentage after the UV treatment in the figures.

The selection criteria for the volatile compounds selection was based on signal intensity and smell relevance (Baky et al., 2024; Hinds et al., 2021; Izcarra et al., 2022; Lee et al., 2020; Murthy et al., 1996). Nineteen peaks were identified and included for data analysis. The standardised area per gram of every compound is shown in Table S1.

2.6. Fatty acid and tocopherol composition

The fatty acid and tocopherol composition were determined using an adapted version of the direct analysis methodology described by Juan-Polo et al. (2022). In brief, 3 mL of n-hexane containing 150 ppm BHT were added to 1 g of powdered pepper, and 50 mg of ascorbic acid were added to prevent tocopherol oxidation. This mixture was stirred with a magnetic stirrer at 900 rpm and 50 °C for 10 min. The procedure was repeated after adding 1.5 mL of 0.2 M sodium methoxide, and again for one minute after adding 1.5 mL of a 5% v/v sulfuric acid solution in methanol. Finally, the supernatant was collected and filtered through a 0.22 µm nylon-membrane syringe filter, diluted 1:10 with hexane for fatty acid methyl esters (FAMES) analysis, but no dilution was employed for the tocopherol analysis.

FAMES were analysed using an Agilent 7890 N gas chromatograph (GC) coupled to a 5977B mass spectrometer (MS) (Agilent Technologies, Palo Alto, CA). The separation was performed on a BPX70 column (60 m × 0.25 mm × 0.25 µm). The ion source and GC–MS transfer line temperatures were set at 250 °C and 280 °C, respectively. Helium was used as the carrier gas at a flow rate of 1 mL/min, and samples were injected with a 1:100 split ratio. FAMES standards consisted of a series of dilutions ranging from 2 to 150 ppm of each FAME. FAMES were identified by matching against the NIST MS library and by considering the retention times of all compounds present in the standards. FAME data were expressed as mg of fatty acid per kg of sample.

Alpha, gamma and delta tocopherol standard curves were prepared for their quantification, and the sample was diluted 1:1 (w/w) with hexane. Tocopherol analysis was conducted using an Agilent 1260 Infinity Binary System ultra-high-performance liquid chromatography (UHPLC) equipped with a fluorescence detector (Agilent Technologies, Palo Alto, CA). The separation of target analytes was achieved using an Inertsil NH₂ normal-phase HPLC column (5 µm, 250 × 4.6 mm I.D.) maintained at 40 °C. The mobile phase consisted of a mixture of n-hexane and isopropanol (98:2 v/v). The separation was performed in isocratic mode at a flow rate of 1.2 mL/min, with a sample injection volume of 20 µL. The excitation and emission wavelengths were set at 298 nm and 325 nm, respectively. Tocopherols in the samples were identified by comparing their retention times with those of the standards, which were prepared as a series of dilutions ranging from 0.1 to 15 ppm for each tocopherol.

2.7. Piperine

Quantification of piperine in the samples was performed using a 1260 Infinity Liquid Chromatograph (Agilent, Santa Clara, CA, USA) equipped with a diode array detector (DAD) and a Kinetex 5 µm EVO C18 100 Å column (Phenomenex, Torrance, CA, USA). The 70% ethanol extracts were filtered through a 0.22 µm nylon membrane prior to analysis, and 10 µL of each extract was injected into the HPLC system. The mobile phase consisted of 30% acetonitrile and 0.5% formic acid in ultrapure water (solvent A) and acetonitrile (solvent B), as detailed in Table 3. The flow rate was maintained at 0.8 mL/min, and detection was carried out at 324 nm. Piperine quantification was based on a calibration curve constructed using standard solutions ranging from 10 to 200 ppm.

Table 2

Gradient and solvent composition of the UHPLC experiment for the phenolic profile determination.

t (min)	A (%)	B (%)
6	65	35
10	55	45
12	90	10

Table 3

Mobile phase gradient percentage for piperine determination.

t (min)	A (%)	B (%)
0	100	0
18	20	80
22	100	0
24	100	0

2.8. Sensory analysis

The sensory analysis was conducted in accordance with the ISO 4120:2021 standard for triangle tests in sensory evaluation. Triangle tests were employed as discrimination tests to determine whether perceptible differences existed between treated and untreated pepper samples. Independent triangle tests were conducted for each sensory modality (colour, smell, and flavour), with panellists instructed to focus on the specified attribute in each session when identifying the odd sample. A triangle test was conducted for each treatment (280, 300, 365, and 280 + 300 nm) and for all sensory attributes. Each test comprised a set of three subsamples, each labelled with a randomly generated three-digit code. Among the three subsamples, one differed from the other two—one treated and two untreated samples—or vice versa, and participants were asked to identify the distinct pepper.

For the colour assessment, three 13.5 mL vials were used, each containing 1 g of peppercorns. A separate set of three vials, each containing 1 g of peppercorns, was prepared for the smell evaluation. For the flavour assessment, three servings of long white rice were used, as described by Dawodu et al. (2023), each seasoned with either treated or untreated finely ground pepper. The rice servings were heated to 45 °C in an oven and served immediately before the flavour evaluation. Each rice serving contained 8 to 10 g of rice, with a seasoning ratio of 0.1 g of pepper and 1.5 g of salt per 100 g of rice. Participants were provided with a cup of water and instructed to rinse their mouths after tasting each rice sample to avoid cross-sample interference. The order in which the three samples were presented to each participant was randomized to minimize order bias. In addition to the triangle test, participants were asked to provide a basic qualitative evaluation by comparing the smell and flavour of treated and untreated samples. These qualitative observations were collected solely for contextual interpretation and were not subjected to statistical analysis nor used to quantify sensory attributes. All tests were conducted in the morning, between 10:00 and 13:00. To avoid bias in data analysis, participant identities were pseudonymized.

A convenience sample of 58 participants was recruited from the University of Alicante, Spain, either in person or via email. The sample included 37 women and 21 men, aged 18 to 65 years, with a mean age of 28.4 years, all of whom were untrained panellists. Prior to participation, each individual received a printed informed consent form and was free to withdraw from the study at any time. Participants with any medical condition—temporary or permanent—that could interfere with the accurate evaluation of sensory attributes were excluded before testing began.

All participants were required to complete all tests on the same day. To be included in the final analysis, each participant had to respond to all three sensory attributes (colour, smell, and flavour) for at least two different treatments. A total of 40 valid responses per sample were obtained, in accordance with the ISO 4120:2021 standard parameters ($\alpha = 0.05$; $\beta = 0.2$; $pd. = 30\%$). The effect of each treatment was evaluated using ten sets of subsamples: half contained two untreated and one treated sample, and the other half contained one untreated and two treated samples.

The sensory analysis protocol was reviewed and approved by the University of Alicante's Ethical Committee (Ref. 2023-12-21).

2.9. Statistical analysis

Results were expressed as mean and standard deviation. To determine the effects of UV radiation and agitation on the pepper samples, data were subjected to a factorial analysis of variance (ANOVA) followed by Tukey's multiple-comparison post hoc test ($p < 0.05$). Statistical analysis was performed using SPSS, version 25 (IBM, NY, USA). Significant differences in the sensory analysis were determined in accordance with ISO 4120:2021.

3. Results and discussion

3.1. Total phenolic content and antioxidant activity

TPC and antioxidant activity are often used as indicators of chemical and nutritional changes in food matrices (Eichholz et al., 2007; García-Mosqueda et al., 2023; Hinds et al., 2021). In the present work, only the radiation treatment at 280 nm resulted in a significant difference in TPC. For this wavelength, an increase of 11 and 13% was observed after the 10- and 20-min treatments, as shown in Fig. 1. This trend has been observed in many vegetables when irradiated with 254 or 280 nm radiation (Arcos-Limiñana, Brunet, et al., 2025; Hassan et al., 2020; Kamel et al., 2022). UV irradiation has been observed to increase the synthesis of phenolic compounds (Emus-Medina et al., 2023) not only under UV-C radiation, but across the whole UV spectrum (Ghasemzadeh et al., 2016; Hinds et al., 2021). The 30-min agitated control sample, which contained 580 ± 50 mg GAE, did not differ significantly from the control, suggesting that shaking did not influence the TPC.

Regarding antioxidant activity, after 10 min of treatment with 280 nm radiation, an increase was observed, followed by a decrease to previous levels. No other significant difference was observed due to treatment time or wavelength, as depicted in Fig. 2. Although no significant differences in the results were found between the control and the reactor control, the latter was significantly lower than the 280, 300 and 365 nm 30-min treatments, indicating that the agitation might have mitigated the increase in antioxidant activity resulting from UV irradiation. In one of our previous works, when black peppercorns were irradiated with UV-C using a less aggressive reactor, antioxidant activity increased by 44% (Arcos-Limiñana, Brunet, et al., 2025). Although the FRAP method was not employed in the Hinds et al. (2021) study of black pepper irradiation, the use of UV-B and UV-A radiation increased antioxidant activity by around 25%, especially at 280 nm. This result might be due to the defensive synthesis of phenols and other antioxidant compounds by the peppercorn cells, enhanced at shorter wavelengths, or because of cell wall breakage and phenolic depolymerization, which may have increased their extractability (Arcos-Limiñana, Maestre-Pérez, & Prats-Moya, 2025; Chowdhury et al., 2017; Ortiz Araque et al., 2019). However, after a certain dose, the photodegradation of the phenolic and antioxidant compounds may overcome this effect and start decreasing, as consistently observed in previous experiments with peppercorns and other low water activity foodstuff (Arcos-Limiñana, Brunet, et al., 2025; Arcos-Limiñana, Prats-Moya, & Maestre-Pérez, 2025; Hassan et al., 2020; Hinds et al., 2021; Kamel et al., 2022).

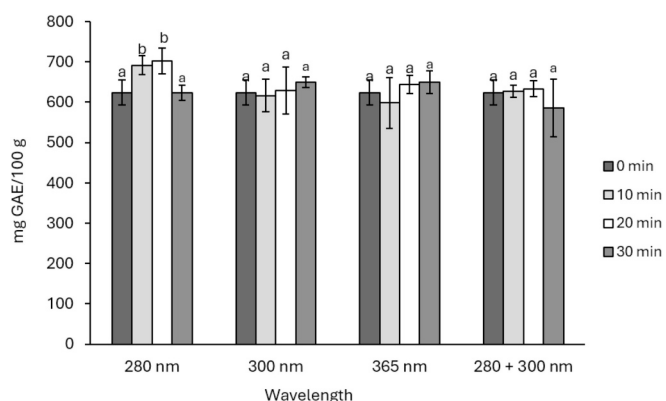


Fig. 1. Total phenolic content (TPC) in gallic acid equivalents (mean $\pm$ SD) of black peppercorns treated for up to 30 min with different wavelengths. Notes: Different letters within the same wavelength indicate significant differences between mean values ($p < 0.05$). The 0 min sample represents the unshaken control.

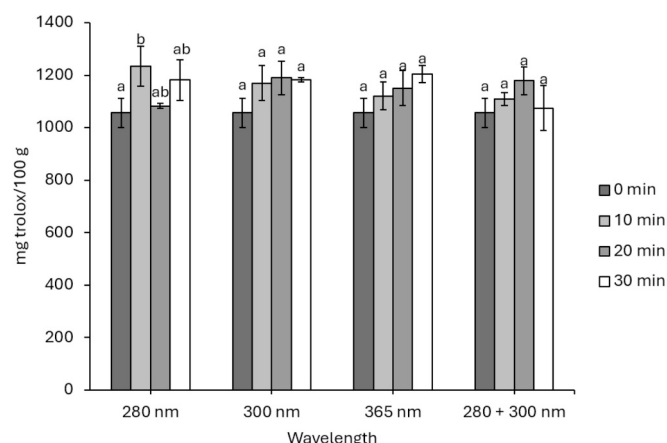


Fig. 2. Antioxidant activity (FRAP) in trolox equivalents (mean $\pm$ SD) of black peppercorns treated for up to 30 min with different wavelengths. Notes: Different letters within the same wavelength indicate significant differences between mean values ($p < 0.05$). The 0 min sample represents the unshaken control.

3.2. Phenolic composition

The phenolic composition plays an essential role in food flavour, providing bitterness and pungency (Issaoui et al., 2020). The 30-min UV treatment significantly affected many of them, as shown in Fig. 3. The concentrations of Su, Pr, Hb, and Fe decreased by 16–40% in all samples. Qa also significantly decreased by 23–35% in all samples except in the

280 nm treated sample, as well as in the case of Ci, which was the most affected, decreasing 61 to 75% of its original content under all treatments except in the 365 nm treatment. Some significant increases were also observed. For instance, neochlorogenic acid increased by 80% under 280 nm UV radiation, and Cc increased by 51% after the 280 + 300 nm combined treatment.

In contrast to the observations made in the TPC and antioxidant activity tests, the phenolic composition of the sample subjected to shaking without irradiation exhibited a nearly identical effect to that of the 30-min UV treatments. This finding suggests that the primary factor driving the observed changes was not the irradiation itself but rather the shaking process. However, certain exceptions were observed, including Vc, which exhibited significant decreases of 60% and 76% following 280- and 300-nm treatments, respectively, but no statistical difference was detected in the reactor control. Furthermore, the study revealed that Ep levels decreased by 30% following 280 nm irradiation, regardless of the shaker's influence. However, the Nc and Cc increases were produced without significant changes in the reactor control. The quantification of the phenolic compounds is shown in Table S2, and the qualitative analysis of the additional 6 phenolic compounds is shown in Table S3.

One of our previous studies showed a lower impact of 254 nm UV-C treatments on the phenolic composition of black pepper when using a gentler movement (Arcos-Limiñana, Brunet, et al., 2025). Furthermore, when using UV-LEDs without agitation, the composition of phenolic compounds in black pepper tended to increase instead of decrease (Hinds et al., 2021), indicating the potential implications of the election of the reactor on the chemical quality. Other UV-treated matrices have shown similar results. Hernandez-Aguilar et al. (2022) investigated the effects of irradiation on turmeric bread and reported increases in Ch, Fe,

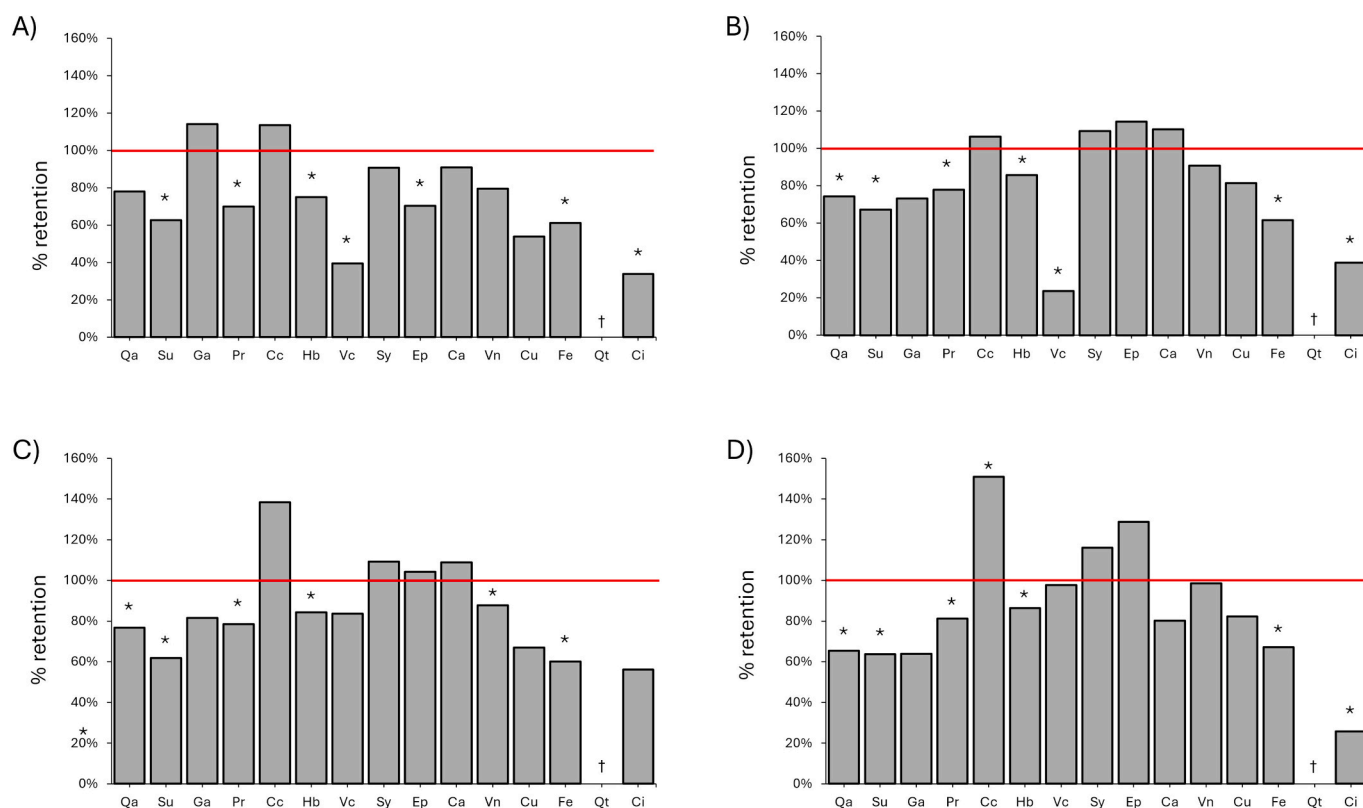


Fig. 3. Retention of phenolic compounds in black peppercorns after 30 min of treatment with 280 nm (A), 300 nm (B), 365 nm (C) and 280 + 300 nm (D) radiation relative to the control.

Notes: Asterisks (*) stand for significant differences between treated and untreated samples, and crosses (†) for below the limit of detection (LOD). Qa: quinic acid. Su: succinic acid. Ga: gallic acid. Pr: protocatechuic acid. Cc: catechin. Hb: p-hydroxybenzoic acid. Vc: Vanillic acid. Sy: syringic acid. Ep: epicatechin. Ca: caffeic acid. Vn: vanillin. Cu: coumaric acid. Fe: ferulic acid. Qt: quercetin. Ci: cinnamic acid. Nc: neochlorogenic acid. Ch: chlorogenic acid. Ru: rutin. Ph: phlorizin. Ka: kaempferol. He: hesperidin.

Pr, Hb, and Ga acids. Similarly, Vergne et al. (2018) studied the irradiation of green tea, finding no change in Ep levels but a 65% increase in Cc. Conversely, Islam et al. (2016) observed that UV irradiation of apple juice resulted in a slight reduction in Ch acid and Ep, while Cc levels increased modestly. This variability in the results has been reported previously, and several recommendations were given to increase reproducibility, which included providing information such as sample thickness and reactor dimensions, but also the use of reactors to increase the area of exposition or accurately measuring the irradiance or dose, as recommended in our previous review (Arcos-Limiñana, Maestre-Pérez, & Prats-Moya, 2025).

3.3. Volatile composition

Volatile compounds are a key asset for the commercialisation of black pepper, as smell is essential for spices, and many identified volatile compounds were significantly affected by the treatment.

Under all wavelengths, 3-Carene, o-Cymene, and Linalool were observed to increase significantly, while 9 others decreased by up to 60% after the 30-min treatment, as shown in Fig. 4. Although every wavelength produced a similar outcome, the 300 nm treatment resulted in higher volatile retention than the other treatments. This wavelength-dependent preservation contrasts with some literature trends. For instance, while some authors reported no change or even increases in volatile fraction using conventional UV-C lamps (Arcos-Limiñana, Prats-Moya, & Maestre-Pérez, 2025; Lee et al., 2020), others observed significant losses in 3-carene, limonene, β - and α -pinene, and α -phellandrene when employing broader UV-B and UV-A wavelengths (Hinds et al., 2021).

The significant changes in β -Pinene, 3-Carene, D-Limonene,

β -Phellandrene, o-Cymene, β -Myrcene, α -Phellandrene, α -Pinene, and α -Terpinolene compounds were simultaneously influenced by both agitation and irradiation. Sabinene, on the other hand, decreased only when irradiated. Other molecules, such as α -Copaene, β -Bisabolene, Linalool, Germacrene-D, and δ -Cadiene, were affected only by reactor agitation, not by irradiation. Caryophyllene, Caryophyllene oxide, β -Elemene, and Humulene remained unaltered after agitation and irradiation. While the mechanisms of molecule degradation from UV light are the generation of reactive oxygen species (ROS), photoisomerization and other matrix-dependant interactions (Arcos-Limiñana, Maestre-Pérez, & Prats-Moya, 2025; Koutchma & Popović, 2019; Soro et al., 2023), the movement might have facilitated the oxidation of these molecules. On the other hand, the oxidation resulting from UV radiation can induce complex photochemical reactions, including isomerisation and radical-mediated rearrangements of terpenes (Goti et al., 2024; Pasik et al., 2025), contributing to a redistribution of the monoterpene fraction rather than simple depletion (Friedman & Farmer, 2018).

Hydrocarbon monoterpenes containing structural strain, such as Sabinene, are particularly susceptible to photochemical and oxidative transformations (BenchChem, 2026). The absorption of UV photons and the exposition to the UV-generated ozone promotes electronically excited states that can facilitate the cleavage of the strained cyclopropane moiety present in Sabinene, leading to radical-mediated ring-opening reactions and skeletal rearrangements. These intermediates can subsequently recombine or undergo rearrangement to form more thermodynamically stable monocyclic or bicyclic products (e.g., p-Cymene-type aromatics or rearranged C10 skeletons). This behavior may explain the selective decrease of Sabinene under irradiation, supporting its role as a primary precursor of secondary transformation products formed during treatment. Monoterpenes such as D-Limonene and α -Pinene are

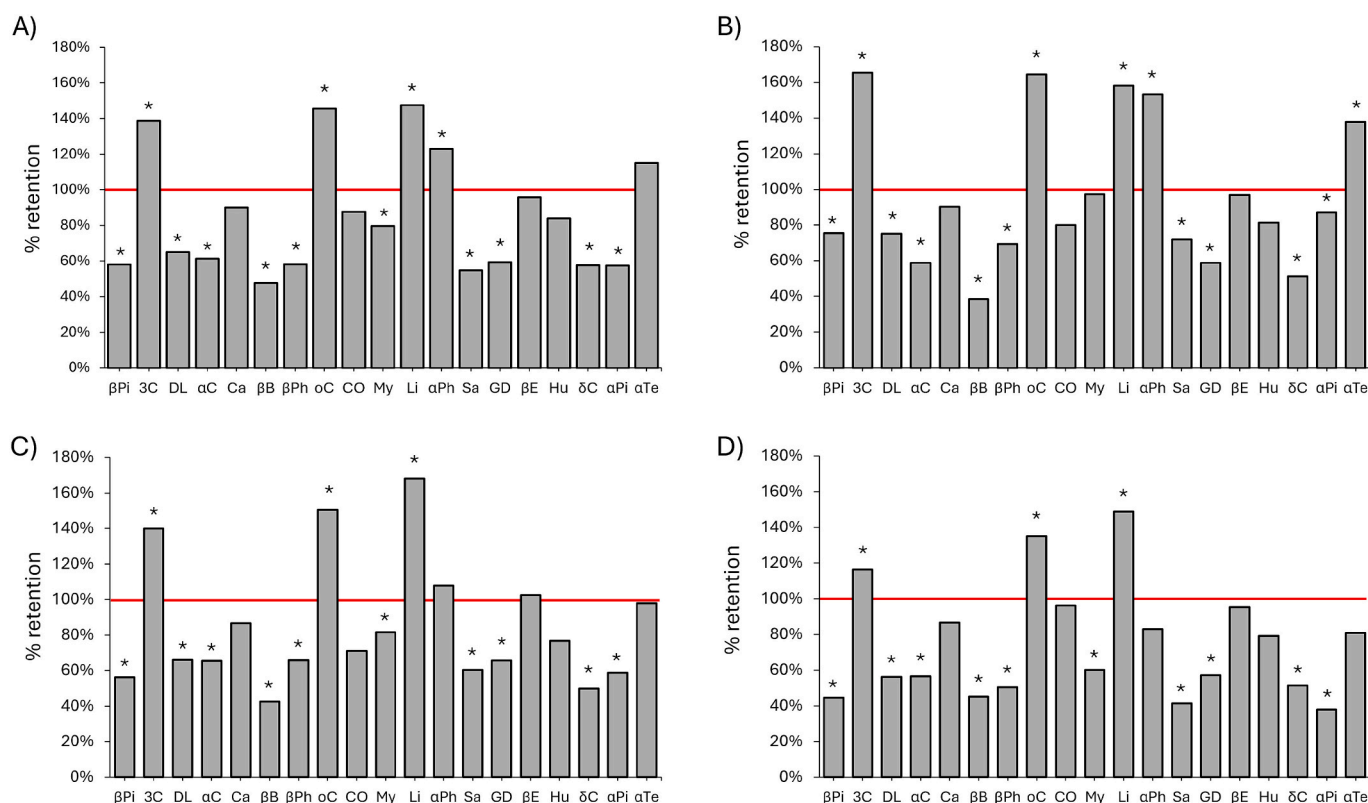


Fig. 4. Retention of volatile compounds in black peppercorns after 30 min of treatment with 280 nm (A), 300 nm (B), 365 nm (C) and 280 + 300 nm (D) radiation relative to the control.

Notes: Asterisks (*) stand for significant differences between treated and untreated samples. β Pi: β -Pinene. 3C: 3-Carene. DL: D-Limonene. α C: α -Copaene. Ca: Caryophyllene. β B: β -Bisabolene. β Ph: β -Phellandrene. oC: o-Cymene. CO: Caryophyllene oxide. My: Myrcene. Li: Linalool. α Ph: α -Phellandrene. Sa: Sabinene. GD: Germacrene-D. β E: β -Elemene. Hu: Humulene. δ C: δ -Cadiene. α Pi: α -Pinene. α Te: α -Terpinolene.

well known to react readily with reactive oxygen species (ROS), including singlet oxygen, ozone, and OH radicals, due to their electron-rich double bonds (Wang et al., 2023; Zeinali et al., 2019). These reactions typically initiate autooxidation mechanisms, involving successive O₂ addition and peroxy radical formation, which lead to highly oxygenated products (Benoit et al., 2023). As a result, the formation of oxygenated monoterpenes—such as alcohols, ketones, and epoxides—can occur, with compounds like Linalool or related tertiary alcohols being commonly detected among oxidation products depending on reaction pathways and conditions.

Interestingly, the influence of reactor agitation observed in our data is consistent with the role of oxygen mass transfer and mixing in promoting radical oxidation chains, thereby accelerating autooxidation reactions and increasing the yield of oxygenated derivatives. Conversely, the relative stability of the sesquiterpene fraction (e.g., β -Caryophyllene, Caryophyllene oxide, β -Rlemene, or Humulene) can be attributed to their C15 skeleton and lower volatility, as well as their generally reduced susceptibility to rapid oxidation compared to more reactive monoterpenes (Zeng et al., 2025). In addition, sesquiterpenes typically require higher energy input for photochemical degradation and tend to exhibit greater thermal and oxidative stability, which explains their persistence under UV treatment conditions.

A similar mechanistic explanation was given by Hinds et al. (2021) to the increase in Caryophyllene after 280 and 365 nm irradiation, but this change was not observed in the present work. However, different sample treatment methods were used prior analysis, potentially leading to different results.

3.4. Fatty acid and tocopherol composition

Following the UV-LED treatments, the fatty acid profile changed. The total fatty acids accounted for 2300 ± 100 mg/kg in the raw black pepper, which significantly decreased to 1800 ± 200 mg/kg in the reactor control, closely followed by the 300 and 365 nm treated samples, and decreasing further to 1550 ± 50 mg/kg in the case of the 280 + 300 nm treatment. However, in the 280 nm irradiated pepper, the concentration remained unchanged (2300 ± 200 mg/kg). The major fatty acid, palmitic acid, was reduced by 16% in the reactor control, indicating that its degradation was solely due to the shaker. This trend was also observed in stearic and oleic acid, while the combined 280 + 300 nm was the only one to demonstrate a significant decrease in palmitic and stearic acids beyond that observed in the reactor control, as illustrated in Fig. 5. Notably, the 300 nm treatment was the least aggressive to fatty acids, which only resulted in one significantly changed: stearic acid. A

general decline was observed in all major fatty acids, although in some cases, it was not statistically significant. However, irradiation at 280 nm mitigated the effect of movement in most cases and increased linoleic acid by 22%. While the minor fatty acids were generally less impacted by the treatment, the 280 nm wavelength also increased the concentration of dodecanoic, eicosanoic, and tricosanoic acids in the black pepper, as shown in Fig. 6. At the same time, the combined wavelength resulted in a significant decrease of dodecanoic acid, which was further than the results of the reactor control.

However, in most cases, the treatment had no observable effect, or the effect was attributable to the agitation. This result led to the conclusion that, under the experimental conditions employed, the impact of irradiation was negligible. A significant effect was only observed after 280 + 300 nm irradiation, suggesting the need for twice the dose for this to happen. This study represents a novel approach in the field of research, as it is the first time that black pepper or a similar matrix has been exposed to UV-LEDs to observe alterations in the fatty acid composition. When black pepper was irradiated with a conventional 254 nm lamp using a reactor, a decrease in linoleic, docosanoic, and tetracosanoic acids was observed (Arcos-Limiñana, Prats-Moya, & Maestre-Pérez, 2025), which was similar to the decline observed in the reactor control. These findings provide evidence that movement, rather than irradiation, is the primary source of fatty acid degradation by facilitating their oxidation (Geng et al., 2023), although not the only one. The 280 nm radiation might have degraded more efficiently cellular structures, either by direct photooxidation or formation of singlet oxygen, disrupting the membrane, releasing intracellular lipids and, thus, facilitating the extraction of the fatty acids (Li et al., 2019; Ortiz Araque et al., 2019), resulting in a higher output. Although the erosion of the pepper skin leads to the decrease in fatty acids concentration, very small amounts of powder were observed after the treatments, rendering this explanation unlikely. The available scientific literature also reports no significant change in the fatty acid composition of peanuts, fish or meat when no reactor was used (Juan-Polo et al., 2023; Monteiro et al., 2017; Soro et al., 2022).

Vitamin E, also known as tocopherol, is a fat-soluble antioxidant molecule that exhibits distinctive characteristics and is indispensable for human health (Azzi, 2007). Tocopherols are naturally present as a range of isomers, mainly alpha-, delta-, and gamma-tocopherol, although alpha-tocopherol has been documented to have optimal antioxidant activity (Khan et al., 2022). The tocopherol composition of the samples was found to be significantly affected by the treatments, as illustrated in Fig. 7. On the one hand, alpha-tocopherol was reduced 24–32% using individual wavelengths and 39% using the combined 280 + 300 nm

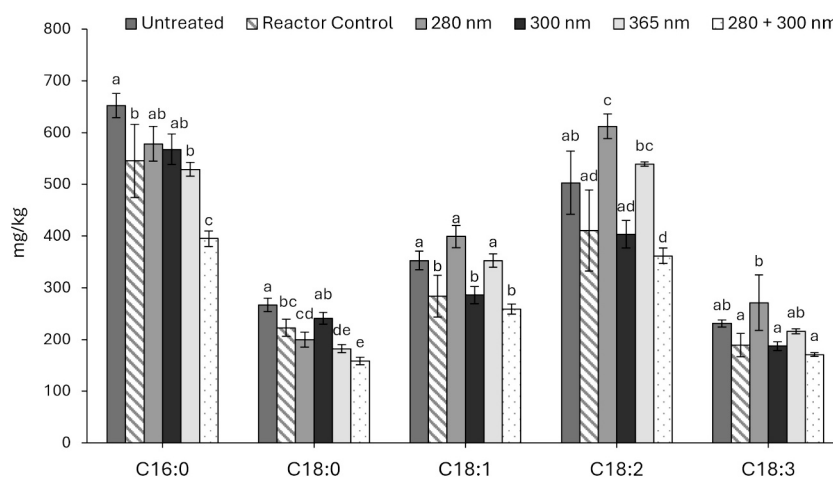


Fig. 5. Major fatty acid composition of black pepper after 30 min of treatment with every wavelength (mean $\pm$ SD).

Notes: Different letters within each fatty acid indicate significant differences between mean values ($p < 0.05$). C16:0 – palmitic acid. C18:0 – stearic acid. C18:1 – oleic acid. C18:2 – linoleic acid. C18:3 – alpha linolenic acid.

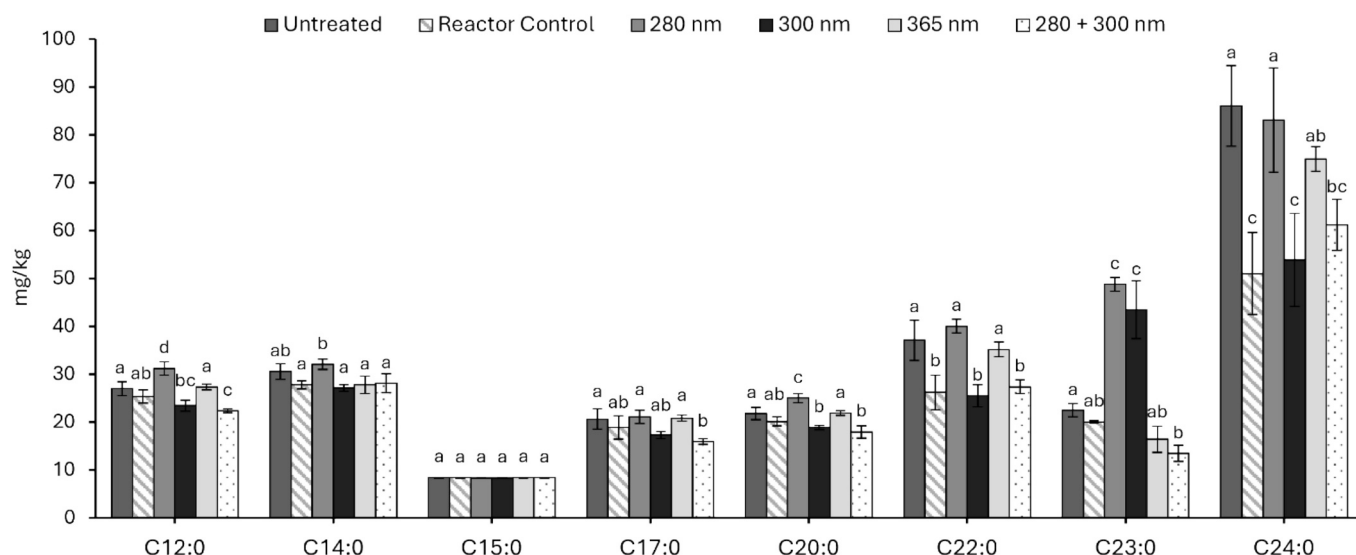


Fig. 6. Minor fatty acid composition of black pepper after 30 min of treatment with every wavelength (mean $\pm$ SD).

Notes: Different letters within each fatty acid indicate significant differences between mean values ($p < 0.05$). C12:0 – dodecanoic acid. C14:0 – tetradecanoic acid. C15:0 – pentadecanoic acid. C17:0 – heptadecanoic acid. C20:0 – eicosanoic acid. C22:0 – docosanoic acid. C23:0 – tricosanoic acid. C24:0 – tetracosanoic acid.

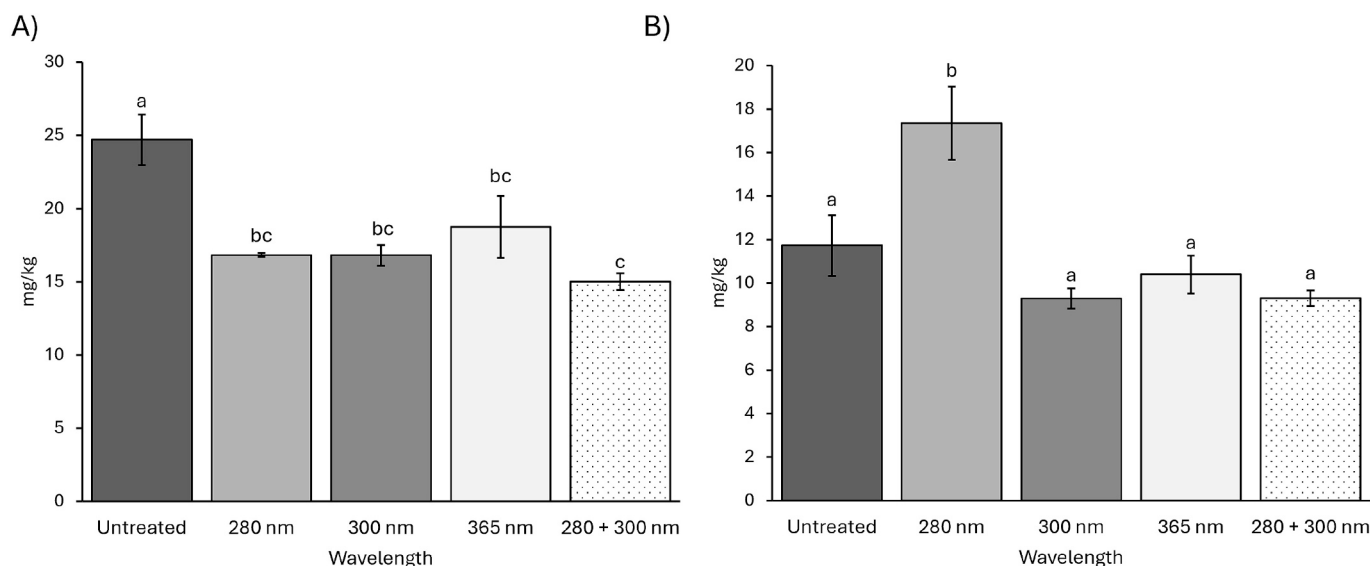


Fig. 7. Alpha (A) and gamma (B) tocopherol concentration in black pepper samples after irradiation with different wavelengths for 30 min. Different letters within the same wavelength indicate significant differences between mean values ($p < 0.05$).

treatment. However, the reactor control experiment yielded a 32% reduction, indicating that the alteration of the tocopherol was attributable to the agitation of the sample during the treatment. Conversely, gamma-tocopherol exhibited a significant difference only after the 280-nm treatment, resulting in an irradiation-induced increase of 48%. The presence of delta-tocopherol was not detected in any of the samples.

3.5. Piperine content

Piperine is the molecule responsible for the characteristic pungent flavour of black pepper (Ashokkumar et al., 2021). Piperine was not significantly affected by the treatment overall, as shown in Fig. 8. However, the reactor control showed a slight decrease in piperine content, although this change remained not statistically significant. Nevertheless, it was considerably lower than all the UV-treated samples, suggesting that UV irradiation may have contributed to maintaining or increasing piperine levels, while the effect of agitation alone might have

mitigated that increase.

Nevertheless, piperine was increased considerably by 23% when irradiated with the 280 nm LEDs for 20 min. These results are consistent with previous experiments, in which piperine in black peppercorns remained at similar levels after UV-B, although UV-A increased it by up to 25% (Hinds et al., 2021). Even with long irradiation times UV-C irradiation resulted in minimal changes (Arcos-Limiñana, Brunet, et al., 2025; Collings et al., 2018). In contrast, other non-thermal methods have been observed to result in a decrease between 40 and 50% (Bang et al., 2021; Fine & Gervais, 2004), likely altering their organoleptic characteristics.

3.6. Sensory analysis

Although chemical analysis helps establish a theoretical basis for potential changes in consumer sensory perception and assess possible changes in the nutritional quality of black pepper, sensory analysis must

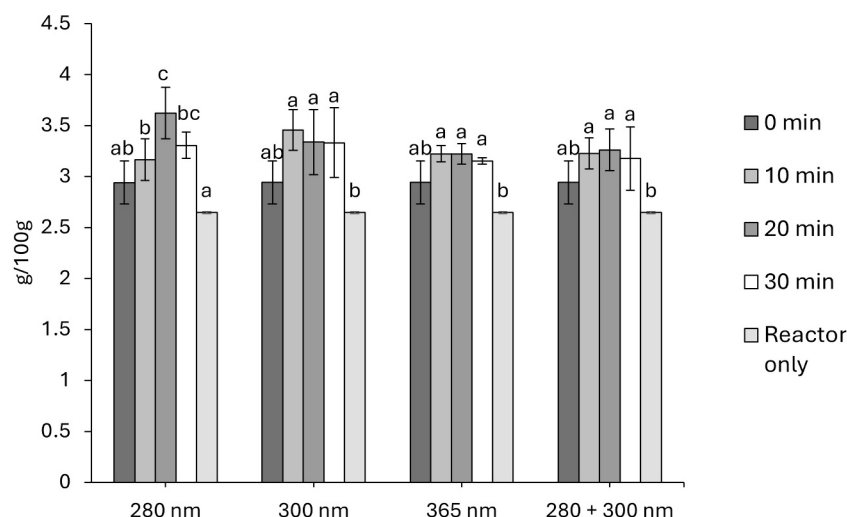


Fig. 8. Piperine content (mean $\pm$ SD) of black peppercorns treated for up to 30 min with different wavelengths. Different letters within the same wavelength indicate significant differences between mean values ($p < 0.05$).

eventually be performed to evaluate the perceptible differences in humans that the treatment may have caused. A comprehensive study must ascertain whether, even in the absence of apparent chemical changes, irradiation has produced off-flavours or discolouration.

As indicated in Table 4, and following the guidelines of ISO 4120:2021, no significant differences could be observed in any of the UV-LED 30-min treatments for colour, smell and flavour, compared to the untreated black pepper, which accounted for 19 correct answers out of 40. Although significant differences in the phenolic, volatile and fatty acid profiles were found, and slight differences in the piperine content, antioxidant activity and TPC were measured, it appears not to be sufficient to result in a perceptible alteration of the qualities of black pepper. Although basic qualitative information was also collected in the sensory analysis, the evaluation of the smell and flavour differences between treated and untreated samples were observed to differ among participants, often leading to contradictory outcomes, preventing a general conclusion to be drawn.

The phenolic compounds were the most significantly affected by the UV-LED treatments, with decreases of up to 60%. The inability of participants to perceive such differences can be indicative of two possibilities: (1) the most affected phenolic compounds, such as cinnamic acid or vanillic acid, were not relevant for the perception of black pepper's flavour, or (2) the differences of the organoleptically relevant phenolic compounds were not enough to be perceived, at least by an untrained panel. Fatty acid oxidation may have influenced black pepper's taste (Brühl, 2014). However, the low fat content in black pepper and the slight differences observed may have prevented its perception. Nevertheless, the nature of the pepper flavour test makes it more difficult for participants to distinguish between them, thereby reducing the test's sensitivity. Furthermore, the flavour is also dependent on volatile compounds (Prescott, 2015), which were significantly affected by the treatments.

Table 4

Number and percentage of correct answers in the triangular test of colour, smell and flavour of black pepper after the 30-min UV-LED treatments ($N = 58$).

Sensory parameter	280 nm ($n = 40$)	300 nm ($n = 40$)	365 nm ($n = 40$)	280 + 300 nm ($n = 40$)
Colour	14 (35%)	16 (40%)	8 (20%)	13 (33%)
Smell	15 (38%)	17 (43%)	10 (25%)	13 (33%)
Flavour	12 (30%)	14 (35%)	16 (40%)	15 (38%)

Notes: Significant differences ($p < 0.05$) were established based on the number of correct answers (19 or more), according to ISO 4120:2021.

The characteristic aroma of black pepper is predominantly influenced by α -pinene, β -myrcene, and linalool (Jagella & Grosch, 1999a; Jagella & Grosch, 1999b). However, the levels of α -pinene (fresh, piney) decreased by around 50% in most samples, while β -myrcene (earthy, herbal) decreased by up to 40%. In contrast, linalool (floral, sweet) increased by another 50 to 70%. However, 2,3-diethyl-5-methylpyrazine and 2-isopropyl-3-methoxypyrazine, which produce off-flavours (Jagella & Grosch, 1999b), were not detected. Olfactory perception is a complex sensory process that involves detecting information from a wide range of compounds. The chemical composition of these compounds is often a simplified version of the complex sensory data perceived by the individual. A substantial alteration in composition does not invariably result in a discernible sensory distinction. It is acknowledged that specific volatiles may undergo changes in quantity; however, as long as these remain below the odour threshold, they are unlikely to impact perception (Ma et al., 2021). Moreover, the presence of other dominant aromas has the potential to mask odours, thereby compensating for any observed changes. Furthermore, the presence of synergistic or suppressive effects between volatiles suggests that even if individual compounds undergo change, the overall aroma perception may remain relatively stable. Additionally, the release dynamics of key aroma compounds may not be accurately reflected in the chromatogram when HS-SPME is used.

3.7. Limitations, industrial implications, and future research

While this study offers novel insights into the impact of UV-LED treatments on the quality of spices such as black pepper, it is constrained by limitations that limit the generalizability of the findings. Notwithstanding the need for a reactor to achieve a safer level of disinfection, it is essential to note that several reactor types can adapt to different circumstances. The reactor utilised in this study mimics the functionality of a vibratory conveyor, thereby enabling uninterrupted treatment lines. However, this reactor appears to result in greater deterioration of volatile and phenolic compounds, as well as fatty acids, than the laboratory-scale rotary drum reactor used in one of our previous studies (Arcos-Limiñana, Brunet, et al., 2025), particularly given that the treatment time was threefold longer in the drum reactor. The irradiation time is, in fact, another limitation, since the LED lamp could not be left on for more than 30 min due to the potential overheating of sensitive LED compounds, although this could be easily solved with a customised setup. However, it is always essential to exercise caution when interpreting results from laboratory-scale reactors. The investigation of practical, cost-effective, and viable reactors that are

compatible with UV-LEDs is strongly encouraged, and this is a first step for that purpose. Additionally, scaling up these reactors is recommended to validate the extrapolation of results.

Nevertheless, should a more effective or more suitable reactor be developed, UV-LEDs have the potential to address numerous industrial challenges. The superiority of UV radiation in minimising chemical and organoleptic alterations compared with heat and vapour treatments has been widely reported (Arcos-Limiñana, Maestre-Pérez, & Prats-Moya, 2025; Soro et al., 2023). The use of heat treatments result in a significant deterioration of the quality of solid food products, including spices, which discards them as a fit-for-all process for disinfection in the food industry (Estrada-Beltrán et al., 2024). Furthermore, the use of water vapour results in product humidification, which in turn increases the risk of mould and bacterial growth during storage. The use of gamma irradiation to disinfect spices has also been a subject of considerable interest (Choi et al., 2020). However, implementing this technique requires sophisticated facilities and strict regulation of radioactive materials, which can be expensive and even impractical. Moreover, it is subject to public animosity, which hinders their implementation. Nevertheless, the scientific literature lacks studies on UV-LED treatments examining the effects of the treatments on microbial load, chemical compounds, and organoleptic properties after storage. Despite observing a similar disinfection yield in 280 and 280 + 300 nm treatments in a preceding work (Arcos-Limiñana et al., 2026), the latter has resulted in a more substantial overall impact, frequently leading to further deterioration of chemical quality, particularly with regard to phenolic, volatile, and fatty acid compounds. It remains unclear whether this is due to the higher dose or a synergistic effect, and more research is necessary. Nonetheless, the resultant outcome frequently approximates or is slightly inferior to that of the 300 nm treatment, which suggests that the 300 nm irradiation was the main driver of the degradation, worsened by the action of a higher overall UV dose.

In relation to sensory testing, an untrained panel was utilised. While this panel does resemble the average consumer, it is recommended that a trained panel be employed in the future to detect subtle changes in flavour or smell. Also, the participation of more panellists could improve the reliability of the results. Additionally, the inherent intricacy of flavour evaluation for black pepper may have hindered untrained panellists' ability to discern differences. As scientific knowledge on this topic progresses, the employment of a trained panel to detect subtle variations and correlate them with quantitative chemical analyses is recommended.

4. Conclusion

This study confirms that UV-LED treatments can induce moderate changes in the chemical composition of black pepper without significantly affecting its sensory qualities, suggesting that the employed UV-LED irradiation system did not compromise black pepper's sensory quality. Although some alterations were observed in the total phenolic content, the antioxidant activity, the phenolic composition, and the volatile and fatty acids profile, these were generally minor and often comparable to those caused by mechanical agitation alone, which can be improved. Although the simultaneous 280 and 300 nm irradiation proved to be only slightly more effective than 280 nm against bacteria in previous studies, often led to further deterioration of chemical quality, especially of phenolics, volatile compounds, and fatty acids. Despite the observed changes, the organoleptic properties remained unaltered, highlighting UV-LEDs as a viable and feasible alternative to conventional water vapour and heat treatments. Unlike thermal methods, UV-LEDs avoid increasing product humidity, offering a promising non-thermal treatment strategy for the spice industry. After analysing the results, 280 nm appears to be the most suitable wavelength for black pepper treatment, balancing microbial safety with the preservation of nutritional and sensory quality.

CRedit authorship contribution statement

Víctor Arcos-Limiñana: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Arturo B. Soro:** Writing – review & editing, Supervision, Methodology. **Brijesh K. Tiwari:** Writing – review & editing, Supervision. **Koenraad Van Hoorde:** Writing – review & editing, Supervision, Conceptualization. **Salvador Maestre-Pérez:** Writing – review & editing, Validation, Supervision, Methodology, Conceptualization. **Soledad Prats-Moya:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Compliance with ethical standards

This study was conducted in accordance with the Declaration of Helsinki and approved by the Ethics Committee of the University of Alicante (Exp. UA-2023-12-21, approved on 30 May 2024). Informed consent was obtained from all participants involved in the study.

Funding

This research was supported financially by the University of Alicante through a predoctoral contract from the Vicerrectorado de Investigación y Transferencia de Conocimiento (UAFPU21-05).

The Agilent 7890B gas chromatograph (Palo Alto, CA, USA) coupled with an Agilent 5977B mass spectrometer (Palo Alto, CA, USA) and also the Agilent 1290 Infinity Online UHPLC with a triple quadrupole spectrometer featuring JetStream and iFunnel technology (UHPLC-1290/ QQQ-6490; Agilent, CA, USA) used in this work were financed 80% by Fondos del Programa Operativo FEDER 2007/2013 de la Comunitat Valenciana and 20% by Generalitat Valenciana.

The authors also thank the University of Alicante for the financial support (UAUSTI24-11) for the use of the equipment.

Declaration of competing interest

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

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ifset.2026.104758>.

Data availability

Data will be made available on request.

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