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Enhancement of rosehip bioactive compounds by cold plasma pretreatment and application of its extract as a functional ingredient in ketchup

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ARTICLE INFO	A B S T R A C T
Keywords: Cold plasma Rosehip extract Ketchup Bioactive compounds Antioxidant activity	The effect of cold plasma (CP) was investigated on rosehip characterization for 1,2.5, and 5 min. All of the samples that were treated with CP had higher amounts of total phenolic content (TPC), antioxidant activity, vitamin C, and lycopene compared to the control ($P < 0.05$). The extract obtained by rosehip pretreated for 1 min had the highest antioxidant activity as well as bioactive compounds (except anthocyanin) and was selected for application in ketchup. Utilizing the CP-treated rosehip extract (RE) in ketchup successfully enhanced TPC (by 1.44 times), flavonoids (by 1.31 times), antioxidant activity (by 1.21 times), carotenoids (by 1.74 times), lycopene (by 1.11 times), vitamin C (by 1.6 times), and anthocyanins (by 2.46 times) compared to the control ($P < 0.05$). Moreover, the phenolic profile demonstrated that the highest increase belonged to catechin. Therefore,

1. Introduction

Ketchup is one of the highly consumed processed tomato products commonly used with ready to eat foods (Mansouripour, Mizani, Rasouli, Gerami, & Sharifan, 2017). This product is a source of lycopene, a valuable antioxidant with the potency to inhibit cancer cells (Soares et al., 2019). However, it has a low anthocyanin content due to the nutritional profile of tomatoes (Wang, Sun, Zhou, Qiu, & Cui, 2020). Additionally, the loss of some bioactive compounds in ketchup might occur during thermal processing (Jayathunge, Stratakos, Delgado-Pando, & Koidis, 2019). Therefore, there is a need for innovative formulations with enhanced nutritional value due to the demand of healthconscious consumers as well as a high appreciation for ketchup. Fruits and vegetables rich in phytonutrients have the potential to be used in ketchup fortification. In previous research, the use of plant-based ingredients such as strawberry pulp (Ahouagi et al., 2021) and acerola pulp (Prakash, Prabhudev, Vijayalakshmi, Prakash, & Baskaran, 2016) successfully increased the nutritional value of ketchup.

Rosehip is a pseudo-fruit of the *Rosa* species from the Rosaceae family distributed in different regions of the world, such as Asia, the Middle East, Europe, and North America (Zhou et al., 2023). Rosehips are usually used as a fruit, tea, jam, juice, jelly, and food additive. Scientific studies have stated the fruits of *Rosa* species have antioxidant and

anti-inflammatory effects. Various biologically active substances such as polyphenols, flavonoids, carotenoids, ascorbic acid, anthocyanins, tannins, and tocopherols are found in rosehips (Koczka, Stefanovits-Bányai, & Ombódi, 2018; Peña et al., 2023; Zhou et al., 2023). Although a significant amount of rosehips is discarded as horticultural waste, they have potential applications for developing functional foods as a health-promoting ingredient (Zhou et al., 2023). In this regard, rosehips have enhanced the bioactive properties of yogurt (Sahingil & Hayaloglu, 2022).

the RE pretreated by CP has the potential to develop a functional ketchup with high bioactive substances.

Studies have shown that non-thermal processes, such as cold plasma (CP), may cause an increase in bioactive compounds in foods. This increase depends on the conditions of the process such as time, voltage, type of gas, etc. (Sruthi et al., 2022). Cold plasma consists of reactive species of oxygen, nitrogen, and hydrogen, charged particles, positive and negative ions, free radicals, and UV photons (Akaber, Ramezan, & Khani, 2024). This technique is primarily used to inactivate microorganisms and enzymes in food (Sruthi et al., 2022). In previous research, cold plasma has been used in fruits and vegetables to inactivate enzymes and microorganisms, and its effect on quality characteristics such as texture, color, etc. has been investigated (Farooq et al., 2023). To the best of our knowledge, the effect of cold plasma technology on the characteristics of rosehip fruit has not been studied before. Furthermore, there are few studies on adding plasma-treated plant extracts to food

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products. The aim of this study was to find out the effects on the bioactive compounds of rosehip extract when it is treated with cold plasma and how that can be used to improve the bioactive compounds and antioxidant activity of ketchup. The experimental design of the present study was as follows: first, the effect of CP for 1,2.5, and 5 min was investigated on total phenolics, antioxidant activity, vitamin C, lycopene, and anthocyanin contents of rosehip. In the next stage, the selected rosehip extract was used in ketchup formulation and the physicochemical, nutritional, and sensory characteristics were evaluated.

2. Materials and methods

2.1. Materials

Rosehip fruit was provided from Khalkhal city (Ardebil province, Iran). Tomato paste, vinegar, salt, sugar, garlic powder, and cinnamon powder as the ingredients used in the ketchup formulation were purchased from a local store in Tehran. Xanthan gum was purchased by Danisco company (Denmark). All the chemicals were of analytical grade and supplied by Merck Company (Darmstadt, Germany).

2.2. Pretreatment of rosehip by CP

CP pretreatment was carried out using dielectric barrier discharge (DBD) plasma Enhanced Tech 15 A dynamic (Kavoshgaran fan pouya, Iran). This device is equipped with a 100 ml container filled with plasma gas, which allows all sections of the sample surface to be treated uniformly. The plasma generation source consists of two-blade electrodes with dimensions of 1340 mm \times 199 mm. The electrodes are coated with a 10 mm-thick dielectric. The distance between the electrodes was adjusted to 20 mm, and the samples were treated with normal air gas. Dried rosehip fruits were placed on a glass plate and exposed to a voltage of 16 kV for 1, 2.5, and 5 min at atmospheric pressure. The input voltage and frequency of the device were set at 25 kV and 50 Hz, respectively. The untreated sample was regarded as the control (Afshar, Ramezan, & Hosseini, 2022).

2.3. Preparation of rosehip fruit extract

Rosehip extract (RE) was prepared based on the method described by Koczka et al. (2018), with some modifications. The dried fruits without seeds were ground, and 25 g of rosehip powder was extracted for 24 h in 250 ml of 70% ethanol with stirring at 25 °C. The obtained extracts were filtered by Whatman No. 42 filter paper and concentrated using a rotary evaporator (Heidolph Laborota 4003, Germany) under vacuum at 40 °C. The extracts were placed in a dark glass and kept in the refrigerator.

2.4. RE characterization

2.4.1. Total phenol content (TPC) and antioxidant activity

The TPC of RE was determined according to Cho, Kim, Lee, Yeon, and Lee (2020) with slight modifications. A mixture was prepared by combining 300 µl of RE with 300 µl of Folin-Ciocalteu reagent. After 5 min, 600 µl of sodium carbonate 7.5% (w/v) was added and the mixture was rested for 30 min. Then, 5 ml of water was added, and the mixture was incubated in the dark for 30 min. The absorption was measured at 725 nm with an ultraviolet–visible spectrophotometer (VARIAN CARY 100, Australia). The DPPH free radical scavenging method was carried out to evaluate the antioxidant activity (Muniandy, Shori, & Baba, 2016). A mixture containing 1 ml of the RE and 2 ml of 0.1 mM DPPH methanolic solution was prepared followed by the mixture was incubated in the dark at room temperature for 30 min. The absorbance was measured at 515 nm.

2.4.2. Lycopene, ascorbic acid, and anthocyanin

Lycopene was determined according to Mert (2012), with some modifications. First, 1 ml of extract was mixed with 39 ml of hexane, ethanol, and acetone (1:1:1 $\nu/\nu/\nu$). Then the mixture was transferred to the separating funnel. After the separation of hydrophilic and lipophilic phases, the lipophilic phase containing lycopene was separated, and its absorbance was measured at 503 nm.

The ascorbic acid (vitamin C) content of RE was measured according to the method of AOAC 967.21 AOAC (Association of Analytical Chemists) (2005). The method described by Lapornik, Prošek, and Wondra (2005) was used for the determination of anthocyanins. First, 1 ml of extract was added to two tubes. Then, 1 ml of 0.01% HCl solution in ethanol was added to each tube. At the next stage, 10 ml of 2% aqueous HCL was added to the first tube (A1), and 10 ml of a solution with a pH of 3.5 including 0.2 M Na2 HPO4 and 0.1 M citric acid, was added to the other tube (A2). The absorbances of A1 and A2 were measured at 520 nm.

2.4.3. Determination of phenolic compounds

Phenolic compounds were determined using a high-performance liquid chromatography (Agilent 6410, USA) system equipped with a diode-array detector (DAD). Separation was carried out by a C18 column ($100 \times 4.6 \text{ mm}$, 5 µm) at 25 °C using the mobile phase, which consisted of 0.1% formic acid in water (A) and 0.1% formic acid in acetonitrile (B). The injection volume was 5 µL, the flow rate of the mobile phase was 0.3 ml/min, and the gradient was programmed as follows: 0–1 min, 5% B; 1–26 min, 5–13% B; 26–42 min, 13–29% B; 42–46 min, 29–60% B; 46–47 min, 60% B; 47–48 min, 60–5% B; 48–56 min, 5% B. To identify the phenolic acids, the UV spectra were compared with the standards, and the quantification of these compounds was obtained by calibrating curves via the injection of different concentrations of external standards (De Beer, Du Preez, & Joubert, 2021).

2.4.4. Microstructure

The microstructure of rosehip fruits was screened using an SEM (scanning electron microscopy) device (TESCAN VEGA3, Czech Republic). The samples were placed on carbon tape and coated with a thin gold film. The SEM images were obtained at 10 kV and $3000 \times$ magnification.

2.5. Ketchup preparation

In the first stage, the ingredients, including tomato paste (30° Brix, 45%), vinegar (8%), salt (2%), xanthan gum (0.5%), dried garlic powder (0.04%), cinnamon powder (0.07%), and sugar (15%), and water (29.39%) were mixed with an electric blender (Brown, 700 W). The selected RE treated by CP (0.5%) and untreated RE were added in RE-containing ketchups. The prepared treatments were pasteurized in a water bath at 80 °C for 10 min and immediately packed into sterile glass containers. After cooling to 25 °C, the ketchups were refrigerated.

2.6. Analyses of ketchup

2.6.1. Physicochemical tests

The pH was measured using a digital pH meter (86,502 AZ, Taiwan). The total soluble solid (TSS) and ash contents were analyzed by a manual refractometer (Atago, Japan) and the gravimetric method, respectively AOAC (Association of Analytical Chemists) (2005). The amount of vitamin C was determined according to the AOAC method 967.21 AOAC (Association of Analytical Chemists) (2005).

2.6.2. TPC, total flavonoid content (TFC), and antioxidant activity

The ketchup extract was prepared according to the method of Santana et al. (2022), with a slight modification. First, 30 ml of 80% methanol was mixed with 3 g of ketchup on a magnetic stirrer for 1 h. Then, the mixture was centrifuged at 7168g for 20 min. After filtration,

the obtained extract was used to measure the total phenol, flavonoids, antioxidant activity, and determination of phenolic compounds. The TPC and antioxidant activity were evaluated using methods previously explained in 2.4.1. To evaluate the total flavonoid content, the colorimetric assay was used (Chang, Yang, Wen, & Chern, 2002). First, 0.5 ml of extract was mixed with 0.1 ml of 1 M potassium acetate, 0.1 ml of aluminum chloride 10% (w/v), 1.5 ml of ethanol, and 2.8 ml of distilled water. After 30 min, the measurement of absorbance was performed at 415 nm.

2.6.3. Total carotenoid content (TCC), lycopene content, and anthocyanins

The total carotenoids were quantified using the method described by Fernandes, Santos, and Rodrigues (2019) with some modifications. Briefly, 0.8 g of the samples were mixed with 20 ml of hexane-ethanol solvent (9:1). After centrifuging the samples (at 2800 g for 10 min), the absorbance of the samples was measured at 480 nm. The lycopene and total anthocyanin contents were measured according to the methods previously described in 2.4.3.

2.6.4. HPLC analysis of phenolics

Identification and quantification of phenolic compounds in ketchup extracts were performed based on the method explained in 2.4.3.

2.6.5. Color analysis

The color values L* (brightness, 0 = black, 100 = white), a* (+: red, -: green), and b* (+: yellow, -: blue) of ketchup treatments were evaluated by a colorimeter (Konica Minolta), Chroma Meter CR-400, Japan). The a*/b* and total color difference with control or ΔE (Eq. (1)) were calculated (Ghadarloo, Mansouripour, & Saremnezhad, 2023).

$$(1)\Delta E = \sqrt{\left(\Delta L\right)^2 + \left(\Delta a\right)^2 + \left(\Delta b\right)^2}$$

where ΔL , Δa , and Δb are the differences of lightness, redness, and yellowness, respectively.

2.6.6. Sensory evaluation

Sensory characteristics including flavor, color, texture, and overall acceptance were assessed using a 9-point hedonic scale from 1 (strong dislike) to 9 (extremely like) scores. The ketchup samples were removed from the refrigerator and served in transparent plastic cups coded with random three-digit numbers at room temperature. The evaluation was performed by the 10 semi-trained panelists (6 females, and 4 males) from the Food Analysis Laboratory at Tehran Medical Sciences, Islamic Azad University (Tehran, Iran). The panelists rinsed their mouths with water before the evaluation of each sample (Ahouagi et al., 2021).

2.7. Statistical analysis

All the experiments were performed in triplicate, and the results were reported as the mean \pm standard deviation. The statistical analysis was conducted using MINITAB 21 software. The significant differences in results were calculated using analysis of variance (ANOVA), and the means were compared by Tukey's test at the *P* < 0.05 significance level.

3. Results and discussion

3.1. TPC and antioxidant activity

The results of the TPC and antioxidant activity of RE are presented in Table 1. A significant increasing trend was observed in the TPC from 810.67 mg GAE/100 g in control to 827.33 mg GAE/100 g in RE-2.5 (P < 0.05). The difference between the TPC of R-1 and R-2.5 was not significant (P > 0.05). The increase in phenolic compounds can be related to the release of phenolic compounds due to the destruction of the cell membrane of fruits by chemically reactive species, charged particles, and UV photons (Hou et al., 2019). In addition, the activation of the

Table 1

	The effect of cold	plasma on the	bioactive com	pounds of	rosehip fr	uit.
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Treatment	TPC (mg GAE/ 100 gDW)	Antioxidant activity (%)	Vitamin C (mg/100 g DW)	Lycopene (mg/100 g DW)	TAC (mg/ 100 g DW)
R-0	810.67 ± 2.08 ^b	70.15 ± 1.4 c	$6.75~{\pm}$ 0.25 ^c	${0.24} \pm \\ 0.001 \ ^{d}$	$\begin{array}{c} \textbf{7.01} \pm \\ \textbf{0.15}^{\ c} \end{array}$
R-1	$\begin{array}{l} 816 \pm \\ \textbf{7.21}^{ab} \end{array}$	$81.51\pm0.35~^a$	$14.90 \pm 0.50 \ ^{\rm a}$	0.60 ± 0003^{a}	${7.65} \pm \\ 0.08 \ ^{b}$
R-2.5	827.33 \pm 3.06 ^a	$76.49\pm0.79~^{b}$	9.25 ± 0.55 ^b	0.46 ± 0.003 ^b	$7.82~{\pm}$ 0.05 $^{ m b}$
R-5	805.33 ± 3.51 ^b	$78.16\pm0.52\ ^{b}$	9.20 ± 0.60 ^b	0.36 ± 0.004 ^c	$8.15 \pm 0.03 \ ^{a}$

Results are presented as a mean value \pm standard deviation (n = 3), TPC: total phenolic content, TAC: total anthocyanin content. R-0: control (untreated), R-1, R-2.5, and R-5: rosehips treated by cold plasma for 1, 2.5, and 5 min respectively. The different superscript letters are significant in the same column (P < 0.05).

phenylalanine ammonia-lyase enzyme (a key enzyme for the synthesis of phenolic compounds), the increase in the ATP content, and the acceleration of the use of sugars lead to stimulation of the biosynthesis of phenolics (Saremnezhad, Soltani, Faraji, & Hayaloglu, 2021). Similar results regarding the increase of phenolic compounds in cold atmospheric plasma-treated Persian lime fruit and tomato pomace were stated by Akaber et al. (2024). Significant decreasing trends were observed in TPC by elevating the processing time from 2.5 to 5 min (P <0.05). This could be due to the production of high-energy electrons that separate oxygen molecules, leading to ozone generation, which causes the destruction and decomposition of phenolics (Sruthi et al., 2022). The R-1, R-2.5, and R-5 showed significantly higher antioxidant activity (P < 0.05) than the control (70.15%) by 1.16 times (81.51%), 1.09 times (76.49%), and 1.11 times (78.16%), respectively. In addition, a decreasing trend in antioxidant activity was observed after 1 min. This is probably due to the higher reduction of TPC in R-2.5 and R-5 because of the reaction between phenolic compounds with antioxidant properties and reactive oxygen species at longer exposure. The obtained results agreed with a previous study reported by Tappi et al. (2018), where an increase in phenolic compounds and antioxidant capacity of freshly cut apples was observed after 10 min of processing with CP; however, a decreasing trend was reported as the processing time increased to 30 min. Hou et al. (2019) also found that the antioxidant activity of blueberry juice decreased with the prolonged processing time of CP from 2 to 6 min.

3.2. Lycopene, vitamin C, and anthocyanin

Table 1 depicts a significant increase in the lycopene content of RE by CP, followed by a decrease as the time prolonged (P < 0.05). The highest amount of lycopene (0.6 mg/100 g) was observed in R-1, followed by 0.46 mg/100 g (R-2.5) and 0.36 mg/100 g (R-5) compared to 0.24 mg/ 100 g in the control. The higher level of lycopene in CP-treated samples is probably related to the release of membrane-bonded lycopene due to the breakdown of the bond between carotenoids and cell membranes (Fernandes et al., 2019). However, since the oxidative species and radicals could attack the terminals with carbon-carbon double bonds in lycopene (Ranjitha Gracy, Gupta, & Mahendran, 2019), the further reduction occurred as the processing time increased to 5 min.

According to Table 1, applying the CP caused significantly higher levels of vitamin C in R-1, R-2.5, and R-5 by 120.74% (14.90 mg/100 g), 37.03% (9.25 mg/100 g), and 36.29% (9.20 mg/100 g), respectively, than the control (6.75 mg/100 g) (P < 0.05). Further reductions occurred with the increase in processing time. If the rate of ascorbic acid regeneration through the ascorbic-glutathione cycle is higher than the degradation caused by the reaction with species produced by plasma, an increase in ascorbic acid is observed. However, at longer exposure

periods, a decrease occurs due to more production of reactive species in processing (Rodríguez, Gomes, Rodrigues, & Fernandes, 2017). According to the results of Hou et al. (2019), increasing the CP processing time reduced the vitamin C of blueberry juice, which was caused by oxidation with reactive oxygen species.

The results of total anthocyanin content (TAC) are presented in Table 1. The control had a TAC of 7.01 mg/100 g. There was a significant enhancement in TAC of CP-treated extracts by 9.12% (7.65 mg/100 g) in R-1, 11.55% (7.82 mg/100 g) in R-2.5, and 16.26% (8.15 mg/100 g) in RE-5 (P < 0.05). The enhancement of anthocyanin content and increasing trend from 1 to 5 min can be described by the improvement in extractability caused by the destruction of the fruit's cellular structures and the penetration of the solvent into the cellular contents quickly and completely. Indeed, flavonoid compounds need less energy to be released among polyphenols (Sruthi et al., 2022). Therefore, it seems that the rate of increase in anthocyanins as a flavonoid component was higher than their decomposition rate due to more extraction. Consequently, an increasing trend was observed at longer exposure periods in the present study. Similarly, an increase in the anthocyanin content occurred in pomegranate juice and sour cherry marasca juice (Sruthi

et al., 2022).

3.3. Determination of phenolics

Since the RE treated by CP for 1 min had higher antioxidant activity, vitamin C, and lycopene content than the other plasma-treated extracts, it was selected for HPLC and SEM analyses, as well as for utilization in the ketchup formulation. The HPLC analysis of the rosehip extracts revealed nine phenolic compounds, as displayed in Fig. 1. and Table 2. The predominant phenolic substance in the extracts was gallic acid, which is in line with the study of Soltan et al. (2023). The other phenolics identified in rosehip extracts were also previously confirmed (Nadpal et al., 2016; Shameh, Alirezalu, Hosseini, & Maleki, 2019). However, some phenolics reported were not detected in the present study, such as cinnamic acid, ferulic acid, and vanillic acid. The reason is that the phenolic composition of rosehip is affected by different factors such as the genotype, ripening of the fruit, and environmental stresses (Nadpal et al., 2016).

The cold plasma pretreatment of rosehip for 1 min significantly increased the content of gallic acid, catechin, *m*-coumaric acid,



Fig. 1. HPLC chromatogram of phenolic compounds in rosehip extracts and ketchup treatments: untreated RE (a), treated RE by cold plasma for 1 min (b), control ketchup (c), ketchup with un-treated RE (d), and ketchup with RE treated by cold plasma for 1 min (e); Peak: (1) gallic acid, (2) Catechin, (3) Epicatechin, (4) Rutin, (5) Quercitrin, (6) m-Coumaric acid, (7) Quercetin, (8) Caffeic acid, (9) Chlorogenic acid.

Table 2

Retention time and content of phenolic compounds detected in rosehip extracts.

Compound	RT (min)	Content of phenolics (mg/100 g)	
		R-0	R-1
Gallic acid	3.44	$149.64\pm4.08~^{b}$	158.66 \pm 3.54 $^{\rm a}$
Catechin	4.31	$27.64\pm0.78~^{\mathrm{b}}$	40.69 \pm 1.13 $^{\mathrm{a}}$
Epicatechin	5.26	31.30 \pm 2.14 $^{\rm a}$	$30.12\pm1.45~^{\rm a}$
Rutin	7.21	16.97 \pm 1.73 $^{\rm a}$	16.33 \pm 1.53 $^{\rm a}$
Quercitrin	9.67	5.09 ± 0.75 a	6.02 ± 0.48 a
m- coumaric acid	10.18	$12.23 \pm 0.68 \ ^{\rm b}$	$20.33\pm0.46~^{a}$
Quercetin	13.71	$28.15 \pm 1.37 \ ^{\rm b}$	$34.25\pm1.04~^{\rm a}$
Caffeic acid	14.23	$60.57\pm2.48~^{\mathrm{b}}$	$69.55\pm2.06~^{a}$
Chlorogenic acid	15.19	115.81 \pm 3.93 $^{\rm b}$	157.44 \pm 5.17 $^{\rm a}$

Results are presented as a mean value \pm standard deviation (n = 3), R-0: control (untreated), R-1: rosehip treated by cold plasma for 1 min. The different superscript letters are significant in the same row (P < 0.05).

quercetin, caffeic acid, and chlorogenic acid compared to untreated rosehip (P < 0.05). The highest enhancement among phenolic compounds was observed in *m*-coumaric acid (66.23%), followed by catechin (47.21%), chlorogenic acid (35.94%), quercetin (21.66%), caffeic acid (14.82%), and gallic acid (6.02%). This is probably because CP activated PAL and C4H, the crucial enzymes in the phenylpropanoid pathway, leading to the biosynthesis of phenolic compounds such as pcoumaric acid (the precursor of caffeic acid) and gallic acid (Xiaoan Li et al., 2019). This pathway also roles in the biosynthesis of catechin (Xiwang Li et al., 2022) and chlorogenic acid (Tappi et al., 2018).

3.4. Microstructure

The microstructure of control and RE-1 is represented in Fig. 2. The control had a uniform surface and natural morphological structure while a slightly uneven and rougher microstructure was observed for RE-1. Indeed, the cellular structure could be damaged by using CP, which results in the loss of solutes and the rupture of the cell membrane (Gu et al., 2021). Similar findings have been reported regarding the microstructure of plasma-treated banana slices by Gu et al. (2021).

3.5. Analyses of ketchup

3.5.1. Physicochemical tests

The pH, TSS, ash, and vitamin C contents of the ketchup samples are shown in Table 3. The pH of RE-containing ketchups was lower (3.96-3.97) than the control (3.98) (P < 0.05). Rosehip contains various phenolic acids along with organic acids like ascorbic acid, citric acid, and malic acid (Zhou et al., 2023). Therefore, the addition of RE decreased the pH of ketchup. A slightly higher TSS content was observed in ketchup treatments with RE (38.23°Brix) compared to the control (38°Brix) (P < 0.05). The results of pH and TSS are in accordance with the ranges stated by the national standard of Iran for pH (\leq 4) and TSS (≥ 30°Brix) of ketchup INSO (Iran National Organization of Standardization) (2016). The ash content increased significantly from 3% in control to 3.37-3.41% in RE-fortified ketchups. The reason is related to the presence of various minerals such as potassium, calcium, phosphorus, and magnesium in rosehip fruit (Peña et al., 2023). The addition of RE increased vitamin C by 1.6 and 1.4 times in ketchup treatments containing CP-treated RE (K-PRE) and untreated RE (K-RE), respectively

Table 3

The effect of rosehip extract on the physicochemical properties and color values of ketchup treatments.

Parameter	С	K-RE	K-PRE
pH Total soluble solids (°Brix) Ash (%) Vitamin C (mg/100 g) L* a* b* b* AF	$\begin{array}{c} 3.98 \pm 0.006 \ ^{a} \\ 38.03 \pm 0.06 \ ^{b} \\ 3.00 \pm 0.13 \ ^{b} \\ 1.76 \pm 0.21 \ ^{c} \\ 25.08 \pm 0.95 \ ^{a} \\ 36.65 \pm 0.56 \ ^{a} \\ 28.25 \pm 0.86 \ ^{a} \\ 1.30 \pm 0.05 \ ^{a} \end{array}$	$\begin{array}{c} 3.96 \pm 0.006 \ ^{b} \\ 38.23 \pm 0.03 \ ^{a} \\ 3.37 \pm 0.06 \ ^{a} \\ 2.48 \pm 0.42 \ ^{b} \\ 25.41 \pm 1.27 \ ^{a} \\ 36.43 \pm 1.33 \ ^{a} \\ 27.88 \pm 0.59 \ ^{ab} \\ 1.30 \pm 0.03 \ ^{a} \\ 1.87 \pm 0.53 \ ^{a} \end{array}$	$\begin{array}{c} 3.97 \pm 0.006 \ ^{b} \\ 38.23 \pm 0.03 \ ^{a} \\ 3.41 \pm 0.02 \ ^{a} \\ 2.82 \pm 0.28 \ ^{a} \\ 25.29 \pm 0.35 \ ^{a} \\ 35.22 \pm 0.62 \ ^{a} \\ 26.85 \pm 0.50 \ ^{b} \\ 1.31 \pm 0.04 \ ^{a} \\ 2.38 \pm 0.41 \ ^{a} \end{array}$

Results are presented as a mean value \pm standard deviation (n = 3), C: control ketchup, K-RE: ketchup with un-treated RE, K-PRE: ketchup with RE treated by cold plasma for 1 min. The different superscript letters are significant in the same row (P < 0.05).

b



Fig. 2. Microstructure of rosehip fruit: control (a), and treated by cold plasma for 1 min (b).

(P < 0.05). Since vitamin C is rapidly degraded in the presence of heat and oxygen (Jayathunge et al., 2019), the thermal pasteurization of ketchup during its production may lead to a significant decrease in vitamin C. Therefore, the use of RE has partially compensated for its decline.

3.5.2. TPC, TFC, and antioxidant activity

The control ketchup had TPC, TFC, and antioxidant activity of 427.33 mg GAE/100 g, 171.83 mg/100 g, and 77.80%, respectively (Table 4). The use of CP-treated RE elevated the TPC by 1.44 times (617.67 mg GAL/100 g), the TFC by 1.31 times (225.63 mg/100 g), and antioxidant activity by 1.21 times (94.41%) in K-PRE. It should be noted that the K-RE also had higher TPC, TFC, and antioxidant activity than the control by 1.32 times (566 mg GAL/100 g), 1.26 times (217.13 mg/100 g), and 1.18 times (92.11%), respectively (P < 0.05).

Bioactive substances such as polyphenols including flavonoids have the potency to scavenge free radicals, which are responsible for various diseases that are caused by oxidative stress (Sruthi et al., 2022). Sahingil and Hayaloglu (2022) reported that the addition of rosehip pulp (5–20%) to yogurt enhanced TPC and antioxidant activity, and the higher phenolic compounds led to more antioxidant activity, which is consistent with the present study.

3.5.3. TCC, lycopene content, and anthocyanins

According to Table 4, the control had a TCC and lycopene content of 62.23 mg/100 g and 44.77 mg/100 g, respectively. Using CP-treated RE increased the carotenoids by 1.74 times (108.60 mg/100 g) and the lycopene content by 1.11 times (49.82 mg/kg) in K-PRE. In this regard, enhancements by 1.15 times (71.50 mg/100 g) in TCC and by 1.10 times (49.46 mg/kg) in the content of lycopene were observed in K-RE (P < 0.05). It should be noted that the presence of carotenoids such as lycopene, beta-carotene, zeaxanthin, etc. in RE (Koczka et al., 2018) led to higher amounts of these compounds in K-RE compared to the control. Studies have shown that lycopene is a potent antioxidant and inhibits the growth of cancer cells (N. d. C. P. Soares et al., 2019).

As shown in Table 4, the highest enhancement among bioactive compounds was observed for anthocyanins (by 2.46 and 1.94 times in K-PRE, and K-RE respectively) (P < 0.05). Tomatoes contain a slight amount of anthocyanins, which have antioxidant and health-promoting effects due to their ability to absorb oxygen radicals (Wang et al., 2020). Therefore, enhancing the anthocyanin content of ketchup as a high-consumption tomato-based product from 3.83 mg/kg in control to 7.45 mg/kg (K-RE) and 9.43 mg/kg (K-PRE) could be valuable.

3.5.4. Determination of phenolics

The HPLC chromatogram and content of phenolic compounds identified in ketchup treatments are shown in Fig. 1. and Table 5, respectively. Incorporating both rosehip extracts increased the phenolic composition of ketchup (except for *m*-coumaric acid) compared to the control. However, the plasma-treated extract was more effective than

Table 4

TPC, antioxidant activity, TFC, TCC, lycopene, and TAC of control and REfortified ketchup treatments.

Parameter	С	K-RE	K-PRE
TPC (mg GAE/100 g DW) Antioxidant activity (%) TFC (mg/100 g) TCC (mg/100 g) Lycopene (mg/100 g) TAC (mg/kg)	$\begin{array}{c} 427.33 \pm 7.51 \ ^{c} \\ 77.80 \pm 0.77 \ ^{c} \\ 171.83 \pm 0.95 \ ^{c} \\ 62.23 \pm 0.65 \ ^{c} \\ 44.77 \pm 0.12 \ ^{b} \\ 3.83 \pm 0.97 \ ^{c} \end{array}$	$\begin{array}{c} 566 \pm 3.61 \\ ^{b}\\ 92.11 \pm 0.07 \\ ^{b}\\ 217.13 \pm 0.95 \\ ^{b}\\ 71.50 \pm 0.80 \\ ^{b}\\ 49.46 \pm 0.06 \\ ^{a}\\ 7.45 \pm 0.32 \\ ^{b}\end{array}$	$\begin{array}{c} 617.67\pm9.29\ ^{a}\\ 94.41\pm0.36\ ^{a}\\ 225.63\pm5.65\ ^{a}\\ 108.60\pm2.29\ ^{a}\\ 49.82\pm0.06\ ^{a}\\ 9.43\pm0.09\ ^{a} \end{array}$

Results are presented as a mean value \pm standard deviation (n = 3), TPC: total phenolic content, TCC: total carotenoid content, TFC: total flavonoid content, TAC: total anthocyanin content. C: control ketchup, K-RE: ketchup with untreated RE, K-PRE: ketchup with RE treated by cold plasma for 1 min. The different superscript letters are significant in the same row (P < 0.05).

Table 5

Retention time and content of phenolic compounds detected in ketchup treatments.

Compound	RT (min)	Content of phenolics (mg/100 g)		
		С	K-RE	K-PRE
Gallic acid	3.44	$\underset{c}{20.52}\pm1.05$	$\underset{\mathtt{b}}{\textbf{34.28}} \pm \textbf{1.49}$	$\underset{a}{\textbf{38.94}} \pm \textbf{2.08}$
Catechin	4.31	$1.76\pm0.52~^{c}$	$3.92\pm0.58~^{b}$	9.58 ± 0.72 a
Epicatechin	5.26	$5.11\pm0.66\ ^{b}$	$\underset{a}{12.63}\pm0.70$	$\underset{a}{12.52}\pm1.12$
Rutin Quercitrin <i>m</i> - coumaric acid Quercetin Caffeic acid	7.21 9.67 10.18 13.71 14.23	$\begin{array}{c} 2.41 \pm 0.52 \ ^{b} \\ 1.02 \pm 0.10 \ ^{c} \\ 1.85 \pm 0.13 \ ^{a} \\ 2.08 \pm 0.20 \ ^{c} \\ 7.59 \pm 0.73 \ ^{c} \end{array}$	$\begin{array}{c} 6.02 \pm 0.28 \\ ^{a}\\ 1.93 \pm 0.27 \\ ^{b}\\ 1.67 \pm 0.15 \\ ^{ab}\\ 4.76 \pm 0.36 \\ ^{b}\\ 10.52 \pm 1.02 \\ ^{b}\\ \end{array}$	$\begin{array}{c} 6.18 \pm 0.86 \ ^{a} \\ 3.42 \pm 0.39 \ ^{a} \\ 1.45 \pm 0.08 \ ^{b} \\ 5.89 \pm 0.42 \ ^{a} \\ 14.56 \pm 0.84 \\ a \end{array}$
Chlorogenic acid	15.19	$\underset{c}{12.11}\pm1.32$	$\underset{b}{30.51}\pm1.67$	$\underset{a}{\textbf{37.08}} \pm 1.08$

Results are presented as a mean value \pm standard deviation (n = 3), C: control ketchup, K-RE: ketchup with un-treated rosehip extract, and K-PRE: ketchup with rosehip extract treated by cold plasma for 1 min. The different superscript letters are significant in the same row (P < 0.05).

the untreated extract due to the elevation of phenolics caused by the pretreatment. The enhancement of phenolics in K-PRE and K-RE compared to the control was as follows: catechin (444.31% vs. 122.72%), quercitrin (235.29% vs. 89.21%), chlorogenic acid (206.19% vs. 151.94%), quercetin (183.17% vs. 128.84%), caffeic acid (91.83% vs. 38.60%), and gallic acid (89.76 vs. 67.05%).

The antioxidant properties of phenolic substances and their ability to remove excess reactive oxygen species and prevent lifestyle diseases have been validated by earlier scientific studies Matsumura, Kitabatake, Kayano, and Ito (2023). It should be noted that notable losses of phenolic compounds occur during the processing of tomato-based products (Jayathunge et al., 2019). Thus, the improvement of these beneficial substances through the fortification of ketchup by RE, especially CP-treated extract, could be of considerable value.

3.5.5. Color analysis

The results of the color evaluation in Table 3 showed there were no significant differences regarding L* and a* values (P > 0.05). In contrast, according to Sahingil and Hayaloglu (2022), the redness of yogurt with rosehip pulp (5-20%) increased significantly due to the presence of anthocyanin in rosehip, and the brightness decreased. It seems that because of the basic red color of ketchup due to the presence of lycopene, the addition of RE at a level of 0.5% could not lead to a drastic change in the redness. The results indicated no significant difference in the a^*/b^* ratio (P > 0.05). Although the lycopene was increased in RE by the CP process, this enhancement was not enough to cause a significant change in the a*/b* ratio of ketchup. This index is usually used to evaluate the color of tomato-based products, including ketchup (Mert, 2012). The higher a^*/b^* is related to the better color quality of the product. Lehkoživová, Karovičová, and Kohajdová (2009) reported a higher a*/b* ratio for ketchup samples (1.6-2.1) compared to the present study (1.30–1.31). The ΔE was not affected by using untreated or CP-treated extracts in ketchup. Additionally, since the ΔE of K-PRE and K-RE were below 3, the color difference will not be easily seen (Ghadarloo et al., 2023).

3.5.6. Sensory evaluation

The sensory evaluation results in Fig. 3. indicate slight differences in color and texture scores. However, RE decreased the score of flavor and overall acceptance of RE- containing ketchups compared to the control. This is probably due to the bitter taste of RE. Bitterness is a sensory characteristic widely associated with tannins, which are a group of polyphenols (S. Soares et al., 2020). Commonly, foods with sweet, salty, and umami tastes are preferred by consumers (H. Li, Li, Zhang, Wu, &



Fig. 3. Results of the sensory analysis of ketchup treatments; Data are presented as a mean value \pm standard deviation of ten panelists; C: control ketchup, K-RE: ketchup with un-treated RE, K-PRE: ketchup with RE treated by cold plasma for 1 min. The different superscript letters are significant (P < 0.05).

Yu, 2023). Therefore, the control ketchup had higher acceptance than RE-containing ketchups. In contrast, adding 5 to 20% rose hip pulp to yogurt increased sensory scores (Sahingil & Hayaloglu, 2022). It seems that various compositions and contents of phenolics in rosehip fruits caused different results in sensory analyses. To compensate for the flavor and overall acceptance scores, it is suggested to use RE in a microencapsulated form. Moreover, utilizing lower levels of RE and adjusting the ingredients used, such as sweeteners, in the formulation of ketchup could be effective in weakening the bitterness.

4. Conclusion

The CP pretreatment of rosehip for 1 min caused the highest phenolic compounds, antioxidant activity, lycopene, and vitamin C contents. Although the bioactive substances were increased in CP-treated rosehips, a decreasing trend was observed (except for anthocyanins) with elevating the time of processing. Utilization of the extract obtained by the CP-pretreated plant could be a new approach to enhancing the nutritional value of ketchup as a highly consumed product. The fortification of ketchup with the extract of rosehip pretreated with CP showed the highest bioactive contents as well as phenolics compared to the ketchup with untreated rosehip and the control. The enhancement by 2.46 times for anthocyanins in tomato ketchup with low anthocyanin was the most interesting finding. Therefore, CP could be used as a pretreatment to obtain rosehip extract with higher bioactive compounds and to be applied in industrial applications to improve the nutritional value of ketchup as a highly consumed product. It is suggested that future research be focused on Optimizing different variables of cold plasma such as gas, voltage, and time to enhance the bioactive compounds of rosehip.

CRediT authorship contribution statement

Elnaz Yazdi: Resources, Methodology, Investigation, Data curation. **Samar Mansouripour:** Writing – review & editing, Validation, Software, Project administration, Methodology, Conceptualization. **Yousef Ramezan:** Writing – review & editing, Resources, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial

interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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