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Low-Pressure and Atmospheric-Pressure Cold Plasma Treatment as a Pretreatment for Extracting Volatile Oils from Black Pepper (*Piper nigrum*) Seeds

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ABSTRACT

Volatile Oils (VOs) are essential contributors to the aroma and flavor of black pepper seeds, making their analysis crucial after sterilization processes. This study aimed to qualitatively and quantitatively analyze VOs in black pepper seeds that have been subjected to the following two modes of Cold Plasma (CP) treatments to evaluate its efficacy as a pretreatment for hydro distillation. CP generating setups, Low-Pressure Cold Plasma (LPCP) and Gliding Arc Plasma Discharge (GAPD) were used for the study. A commercially available black pepper (Piper nigrum)variety (MB12) underwent CP treatments with varying time durations (5, 10, and 15 minutes). The VOs were extracted using hydro-distillation and identified and quantified by GC-MS and GC-FID respectively. The study examined 12 major components of VOs, including α-pinene, β-caryophyllene, and D-limonene. Results revealed that LPCP at 5 minutes and GAPD at 10 minutes yielded the highest VO content, while prolonged treatment times led to decrease oil yields in both treatments. No significant differences were observed in individual VO components between the two CP treatments. The refractive index of VOs after LPCP and GAPD treatments did not significantly differ, affirming the GC-FID results. Importantly, the study concluded that both 5-minute treatment time of LPCP and10-minute treatment time of GAPD did not adversely affect the quality of VOs in black pepper seeds. The results of the study highlighted the necessity of insights into optimal processing conditions and highlighting the utility of LPCP and GAPDas pretreatments for VO extraction.

Keywords: black pepper seeds, volatile oils extraction, cold plasma, gliding arc plasma, low pressure plasma

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INTRODUCTION

The distinctive flavor and aroma of black pepper are attributed to its essential oil, constituting approximately 3–6% of the black pepper (*Piper nigrum*)(Pepper et al., 2019). The composition of volatile oil content depends upon the maturity and are reported as 8.6 ml/100g at 22.5 weeks mature in Sri Lankan black pepper and the volatile oils are contained in the inner layers of the mesocarp (Jansz et al., 1984). Due to the significant economic and medicinal importance of volatile oils of black pepper seeds, numerous studies have been conducted to explore the methods of volatile oil extraction and characterization. The oil, ranging from colorless to greenish, emanates a spicy (peppery) fragrance. Extraction methods such as distillation, simultaneous distillation-extraction (SDE), solid phase microextraction (SPME), or supercritical fluid extraction are commonly employed to obtain black pepper oil from *P. nigrum* fruits(Can Başer & Buchbauer, 2015; Pepper et al., 2019). Nevertheless, these approaches come with certain drawbacks, such as an extended extraction duration, substantial energy consumption, potential contamination by solvents, the necessity for supplementary purification steps, and an adverse impact on the caliber of extracts and overall amount of extracted material (Alashti et al., 2024). The process of hydro-distillation stands as the oldest and frequently employed technique for obtaining aromatic essences such as volatile oils from botanical substances. This method holds distinct advantages over alternative extraction techniques, as the equipment is cost-effective and straightforward to construct, simple to use and feasible for mass production. Consequently, it finds extensive utilization within the essential oil industry (Do, 2022). Therefore, a pretreatment to increase the oil extraction yield coupled to hydro-distillation is a timely requirement.

The potential of cold plasma technology for diversified applications in the food industry such as microbial decontamination, increasing seed germination yield and producing plasma activated water are being experimented worldwide (Adamovich et al., 2022; Afshari & Hosseini, 2014; Cherif et al., 2023; Chizoba Ekezie et al., 2017) and is now being assessed as a pretreatment for drying (Loureiro et al., 2021; Shishir et al., 2020; Tabibian et al., 2020) and extraction(Heydari et al., 2023; Kodama et al., n.d.). Cold plasma is generated through the electrical breakdown of gases at varying pressure gradients (ranging from low pressure to atmospheric pressure), resulting in a gas that is partially or fully ionized (Shishpanov et al., 2020; Ansari et al., 2022; Chizoba Ekezie et al., 2017). The makeup of cold plasma is distinctively determined by the setup and operational parameters, including the power source, voltage, frequency, pressure circumstances, gas composition, and environmental factors (Valencia et al., 2022). In contemporary research, a variety of methods for generating cold plasma are employed, including low-pressure cold plasma glow discharge, dielectric barrier discharge, atmospheric plasma jet, corona discharges, gliding arc discharge, and plasma torch (Chiappim et al., 2023; Misra et al., 2011; Thirumdas et al., 2017b; Yang et al., 2024). Cold plasmas, commonly known as non-equilibrium plasmas exhibit distinctive temperature variations among plasma species (Akishev et al., 2010; Kogelschatz, 2003; Kuzminova et al., 2017; Misra et al., 2016). Within cold plasma, the electron temperature remains unbalanced with the resultant ion temperature, even though the supplied energy is sufficient to sustain electron flow and induce partial ionization of the gas. The generation of cold plasma is achievable under both low-pressure and atmospheric pressure conditions (Sarangapani et al., 2015).Non-equilibrium plasma persists across a spectrum of pressures, typically spanning from approximately 0.1 to 100 Pa for low-pressure plasma, or at atmospheric pressure (around 10^5 Pa).

A pivotal contrast lies in the behaviour of reactive gaseous species between low-pressure and atmospheric-pressure plasmas, notably in their lifespans. Reactive species often endure for mere microseconds at atmospheric pressure but can extend beyond a second under low pressure. At atmospheric pressure, predominant loss mechanisms involve homogeneous reactions in the gas phase, while at low pressure, varied reactions in the surface dominate(Primc, 2022).

Nevertheless, cold plasma, as a surface treatment method, exhibits restricted penetration depth, influenced by factors such as substrate structure and composition, power input, distance between the plasma source and the surface, as well as the half-lives of reactive species (Hertwig et al., 2018). Recent studies have shown penetration depths in various biofilms ranging from 1 nm to 50 µm, reaffirming the efficacy of cold plasma technology in surface treatment (Bußler, n.d.;Ziuzina, 2015).

Plasma has wide applications in removing of materials in nano - micro levels which is referred to as “etching”. During cold plasma treatment surface etching is one of the key phenomena occur by physical sputtering or chemical reactions in material processing industry. Plasma etching, reactive ion etching and sputtering etching are the key types of etching processes which are referred related to the mode of action (Thirumdas et al., 2017b). Different commercial surface modifications such as increasing hydrophilicity, hydrophobicity and functionalization are carried out with specific plasma designs in polymer, plastic and metal industries (Chizoba Ekezie et al., 2017; Fitzpatrick et al., 2016; Sarangapani et al., 2016; Submitted et al., 2011; Thirumdas et al., 2017a). However, the phenomenon is widely applicable in the food industry as a surface treatment method especially as a pretreatment method(Annor et al., 2014; H. S. Lee et al., 2020; Yu et al., 2014).

The extraction yield of plant components is subject to various factors, encompassing the extent of cell wall disruption, plant/botanical species, surfaces, and the chemical makeup of active biological compounds inherent to the plant. Cold plasma processes have been shown to induce alterations in the physical properties of different plant matrices especially in the surfaces. This results in the development of fissures and indentations on the surface cracks, facilitating an enhanced exudation of substances and thereby elevating the extraction yield (Heydari et al., 2023).For the utilization of atmospheric pressure plasma in material processing, in etching and deposition applications, the necessity extends beyond achieving a uniformly distributed plasma over a large area and need to have a high plasma density (Y. H. Lee & Yeom, 2005).

With that background, the study was intentional to investigate the potential of low-pressure and atmospheric pressure cold plasma technology to use as pretreatment methods to extract volatile oils from black pepper seeds using hydro-distillation.

METHODOLOGY

Sample Selection

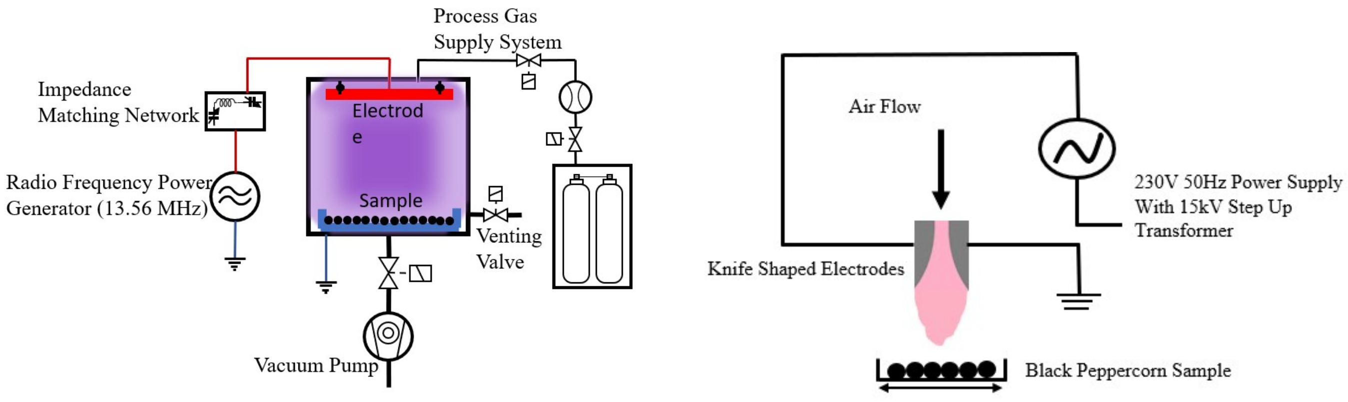
Samples of black pepper seeds, weighing 100g each and meeting export quality standards, were acquired from a processing facility specializing in black pepper in Padukka, Western Province, Sri Lanka. The facility processes the MB12 variety of black pepper cultivated in Matale, Central Province, Sri Lanka. The diameters of 100 black pepper seeds were measured individually using a calibrated Vernier caliper (GENERAL, USA). The chosen black pepper seeds for the study have a mean diameter of 4.72 ± 0.39 mm and an average mass of 0.053 ± 0.013 g.

Cold plasma treatment for black pepper seeds

Two cold plasma generating setups, Low-Pressure Cold Plasma treatment (LPCP) and Gliding Arc Plasma Discharge (GAPD) were used for the study (Figure 02 (a) and (b)). The descriptive methods carried out for each treatment are described below.

Low Pressure Cold Plasma Treatment

Utilizing a 300 W, 30 L low pressure plasma chamber operating at radio frequency in a plasma setup with parallel plate capacitance coupling, the Low-Pressure Cold Plasma Treatment (depicted in Figure 02 (b)) was executed. Oxygen, with a purity of 99.99%, served as the treatment gas. The black pepper seeds sample was evenly distributed in a metal tray with a rectangular shape with a packing seeds density of 0.22 g/cm^2 (250 mm x 150 mm), strategically placed at the central point within the low-pressure chamber. To regulate the process, the oxygen gas supply rate was maintained at 120 cm^3/min, ensuring a pressure range between 0.35 mbar and 0.45 mbar. The treatment parameters included a wattage setting of 250 W, and treatment durations of 5, 10, and 15 minutes.



(a) **(b)**

Figure 1: (a) Low Pressure Cold Plasma Treatment (b) Gliding Arc Plasma Discharge Treatment

Gliding Arc Plasma Discharge Treatment

The study employed a Gliding Arc Plasma Discharge system procured from Eltech Pvt Ltd in Mumbai, India. Cold plasma, generated at a fixed supply voltage of 15 kV and Alternating Current at 50 Hz, utilized atmospheric air with a flow rate of 0.009 m^3/s. In the experimental setup, 20g of pepper seeds sample was evenly dispersed as a single layer of black pepper seeds (0.22 g/cm^2) in a petri dish and it was placed in a holder made up of poly acrylic acid. The holder with the petri dish having black pepper seeds was placed 4 cm below the plasma discharge. Manual horizontal shaking was done wearing electrically insulated gloves twice a minute. The study operated under atmospheric conditions with air at 26°C and 65% humidity. Treatment durations were established at 5 minutes, 10 minutes, and 15 minutes, each repeated three times for analysis.

Volatile Oil Extraction

To obtain pepper seed oils in accordance with AOAC 17th edition 2000, 962.17, hydro-distillation was conducted with a sample size of three (n=3). The process involved crushing 50 g of pepper seeds in a dry grinder for five seconds, followed by heating the crushed seeds with distilled water amounting 500 ml. The vapor generated during this process was underwent condensation and refluxing for about 4 hours until the oil collected in the trap had no alteration in the volume in one-hourtime interval. Subsequently, the amountof volatile oil accumulated in the trap was quantified using a calibrated measuring cylinder. The amount of oil extracted was determined using the following equation (Equation 1).

$$Voltile\ Oil\ Content = \frac{Volume\ of\ oils\ extracted\ in\ the\ hydrodistillation\ unit\ (ml)}{Dried\ weight\ of\ crushed\ black\ pepper\ used\ (g)} \ldots (1)$$

Analysis of Volatile Oils by Gas Chromatography – Mass Spectroscopy

Volatile oil in black pepper seeds was both identified and quantified through Gas Chromatography-Mass Spectrometry (GC-MS) and Gas Chromatography with Flame Ionization Detection (GC-FID), respectively. Qualitative analysis of the volatile oil was conducted using a Gas Chromatograph (Thermo Scientific TRACE 1300) coupled with a Mass Spectrometer (ISQ-QD, Single Quadrupole). This setup included an auto injector AI 1310 (Thermo Scientific) and a Thermo Scientific fused silica capillary column (DB-WAX UI) measuring 30 m × 0.25mm i.d., with a 0.25 µm film. Helium was used as the carrier gas at a flow rate of 1.0 ml/min. The temperature program was set as follows: initial temperature at 60°C, with a ramp of 5°C min^{-1} up to 220°C. The injector temperature was maintained at 240°C, and the ion source temperature at 250°C. 0.3 µl of oil samples, diluted with 1 ml of hexane, were injected with a split ratio of 1:50 and a column pressure of 64.20 kPa. Mass Spectrometry was conducted using an ion capture detector with an impact energy of 70 eV. Constituent identification relied on comparing mass spectra with those in the equipment database (NIST11) and the library (Wiley Registry of Mass Spectral Data, 10th Edition, 2015). Retention indices for each identified component were determined using a C7-C30 saturated alkane standard (SUPELCO) at a concentration of 1000 µg/ml in hexane.

Analysis of Volatile Oils by Gas Chromatography – Flame Ion Detection

Each constituent's quantification was established through Gas Chromatographic analysis with Flame Ionization Detector (FID) using area normalization (%) method. The proportional concentrations of the identified oil components were derived through peak area normalization, where the total peak area served as the reference set at 100%. The proportion of each constituent was calculated based on the area of its respective peak. To prepare the samples for analysis, the oil samples underwent filtration using micro filters. Subsequently, 1 ml of each filtered sample was carefully transferred to glass vials using a micro pipette. The samples were then subjected to vortex for approximately 60 seconds before injection into the analytical system.

Refractive Index of Volatile Oils

The Refractive Index of the volatile oils obtained through hydro-distillation was assessed using an OGAWA SEIKI Refractometer, employing the methodology outlined in ISO 6320:2017.

Statistical Analysis

Every analysis was conducted in triplicate. The impact of the treatment was evaluated for significant differences at a significance level of $P<0.05$ using Minitab 14, employing ANOVA one-way analysis. Differences between means were assessed through a two-sample T-test at a 95% confidence level. Differences between means were further elucidated using Tukey's test at a significance level of $p < 0.05$.

RESULTS AND DISCUSSION

The amount of volatile oil extracted and their refractive indices obtained afterLow Pressure Cold Plasma (LPCP)and Gliding Arc Plasma Discharge(GAPD) treatments are depicted in Table 01. There is an increase in the volatile oils extracted from black pepper seeds when compared to the untreated sample in LPCP treatment.

The LPCP treatment was carried out in a low-pressure chamber where the pressure inside the chamber is 0.3 – 0.5 mbar. Moreover, more uniform treatment can be expected in the LPCP with the glow type uniform plasma discharge inside the LPCP chamber throughout the treatment. When black pepper seeds are exposed to plasma within the LPCP chamber, reactive species function to strip off or remove material from the surface more uniformly than in the GAPD treatment, creating channels that allow escaping of volatile oils. As depicted in Table 1, 5-minute treatment time had the highest volatile oil extraction yield confirming that 5-minute treatment was capable to facilitate the hydro distillation process increasing the volatile oil content by 24.1 %. Further treatments reduced the oil extraction yields and the very low pressure in the surrounding (LPCP chamber) might support the evacuation of volatile oils faster than in the GAPD which was carried out under atmospheric pressure conditions. Supporting to this, similar alteration of volatile oils were detected in treating lemon verbena and the research revealed that the alteration of the surface properties through cold plasma treatment made the development of porous surface, facilitating the easy extraction or release of volatiles from lemon verbena (Ebadi, 2019).

In GAPD treatment, the highest oil extraction yield was found after 10 minutes treatment and the sample treated for 15 min detected a reduced oil extraction when compared to10-minute treatment. It was observed that 10 min of GAPD treatment enhanced the volatile oil extraction content by 27.6 %.

Table 1: Volatile Oil Extraction and Refractive Index of Volatile Oils after Cold Plasma Treatments

Treatment	LPCP		GAPD	
	Oil Extraction (ml/g)	Refractive Index	Oil Extraction (ml/g)	Refractive Index
Control	0.029 ± 0.001^{a}	1.4851 ± 0.0029^{a}	0.029 ± 0.001^{a}	1.4851± 0.0029^{a}
5 min	0.036 ± 0.001^{b}	1.4836 ± 0.0008^{a}	0.032± 0.002^{a}	1.4849± 0.0006^{a}
10 min	0.029 ± 0.002^{a}	1.4839 ± 0.0007^{a}	0.037± 0.002^{a}	1.4870± 0.0008^{a}
15 min	0.028 ± 0.001^{a}	1.4840 ± 0.0009^{a}	0.032± 0.001^{a}	1.4829± 0.0010^{a}

Data is expressed as mean ± SEM, n = 3. Means followed by the different letters (a and b) indicate a significant difference at $p<0.05$ between the data within the same columns

These increases of the volatile oil extractions after GAPD treatment arises an argument that GAPD treatment can facilitate the hydro-distillation process allowing more trapped oils in the internal structures of the black pepper seeds to come out, assisting the hydro-distillation process. In GAPD, the reactive species generated (with nitrogen, oxygen and water vapors etc.) in the plasma interact with the black pepper seeds' surface along with the plasma flow which comes through the electrodes. This continuous bombardment of plasma species, throughout time, etched the surface of black pepper seeds, forming microchannels to facilitate the extraction of volatile oils. After this continuous interaction reach to a threshold point, the volatile oil tends to release from the black pepper seeds surface with the forced air flow (0.009 m^3/s) in the GAPD treatment. Therefore 10-minute treatment time was effective in obtaining the maximum volatile oil extraction. However, this optimum treatment time is specific to the amount of black pepper seeds used in this study and scaled up research are required for defining the batch sizes and treatment times to optimize the process. Complying with the increment of the volatile oils obtained, similar findings were obtained for lemon verbena and lemon peel resulting in loss of volatile oils (Ebadi, 2019). Moreover, the volatile oils extracted from citrus peels using Dielectric Barrier Discharge Plasma treatment of 30 kV, 50 Hz resulted in improving the extraction of oil yields confirming the results of this study (Kodama et al., n.d.).

In order to obtain the highest volatile oil extraction yield, LPCP was effective within a comparatively short time than GAPD. However, when proceeding with the scaled-up research the investment cost and the operating cost have to be considered for the industrial applications, since the investment and operating cost of LPCP is costlier than for GAPD.

The refractive index of volatile oils extracted using both plasma treatments did not affect significantly reflecting consistence of the quality of volatile oils as can be seen in Table 01. The refractive indices obtained in this study comply with the previously obtained values of refractive indices of black pepper volatile oils (Setiawan et al., 2019).

As Table 02 illustrates, 12 major components of volatile oils were identified and quantified from black pepper seeds. The identified volatile oils were, α-pinene, β-pinene, sabenine, δ-3-carene, β-myrcene, D-limonene, β-phellandrene, O-cymene, δ-elemene, α-copaene, β-caryophyllene and Caryophyllene Oxide. Each identified component is specific to a key feature in the aroma profile of the black pepper seeds. All the identified constituents except Caryophyllene and Caryophyllene Oxide were not changed statistically significantly ($P>0.05$) though there are changes in the composition in many of the identified oil components after GAPD treatment. Caryophyllene content in extracted volatile oils decreased significantly ($P<0.05$) after 15 min treatment, while Caryophyllene Oxide escalated after 15 min GAPD treatment which can lead to change the odor profile. Long exposure time together with the reactive oxygen species generated in plasma discharge can cause the oxidation of β-caryophyllene to Caryophyllene Oxide. This further confirmed that 10-minute treatment time of GAPD was the most appropriate time for facilitating the volatile oil extraction using hydro-distillation.

Similar to the GC-MS and GC-FID analysis carried out for the volatile oils extracted from the black pepper seeds after GAPD treatment, there was no statistical significance in the composition when twelve components of volatile oils are considered in LPCP treatment. However, as can be seen in Table 02, changes in composition were observed in volatile oils directing to change the quality of volatile oils. α-pinene, β-pinene, O-cymene, δ-3-carene and Caryophyllene Oxide were increased after LPCP treatments while, D-limonene, δ-elemene, α-copaene, and β-caryophyllene decreased after the treatments. Irrespective to the extracted volatile oils yield, slight changes of the composition with the LPCP treatment was observed.

The compositional analysis highlighted that, though the volatile oil extraction yield increased with the GAPD and LPCP treatments, no significant changes were detected composition wise confirming the applicability of the two methods for using as pretreatment methods.

Table 2: Variation of volatile oils with GAPD and LPCP Treatment

No:	Component	RI*	Control	GAPD			LPCP		
				5 min	10 min	15 min	5 min	10 min	15 min
1	α-pinene	932	21.34 ± 2.06	20.84 ± 1.22	21.56 ± 0.98	23.24 ± 0.12	24.05 ± 0.18	24.78 ± 0.09	22.79 ± 1.60
2	β-pinene	1049	0.37 ± 0.02	0.38± 0.04	0.42±0.04	0.41± 0.02	0.42± 0.01	0.41± 0.02	0.38± 0.02
3	Sabinene	1062	12.82± 0.66	12.66± 0.43	12.48± 0.73	13.02± 0.07	12.77± 0.14	13.03± 0.08	12.68± 0.19
4	δ-3-carene	1095	16.76± 0.63	16.68± 0.28	17.7± 2.41	17.45± 0.52	18.21± 0.39	18.41± 0.07	18.52± 0.61
5	β-myrcene	1106	8.65± 0.12	8.70± 0.23	8.01± 0.39	8.84± 0.12	8.05± 0.27	8.36± 0.13	7.91± 0.36
6	D-limonene	1141	2.41± 0.09	2.42± 0.05	1.98± 0.16	2.48± 0.02	2.17± 0.05	2.00± 0.05	2.15± 0.02
7	β-phellandrene	1152	15.04± 0.21	15.16± 0.11	14.46± 0.32	14.81± 0.33	14.15± 0.07	14.17± 0.14	14.26± 0.05
8	O-cymene	1214	0.58± 0.02	0.58± 0.04	0.61 ± 0.03	0.0.75 ± 0.05	0.63± 0.06	0.68± 0.06	0.66± 0.03
9	δ-elemene	1462	0.41± 0.02	0.44± 0.04	0.42± 0.12	0.45± 0.15	0.32± 0.06	0.30± 0.02	0.35± 0.03
10	α-copaene	1483	1.21± 0.16	1.15± 0.09	1.45± 0.31	0.93± 0.02	1.08± 0.01	0.99± 0.03	1.14± 0.13
11	β-caryophyllene	1585	13.75 ± 1.78	13.71± 1.00	16.11± 3.44	11.21± 0.11	12.00± 0.19	11.09± 0.24	12.62± 0.13
12	Caryophyllene Oxide	1969	0.47± 0.11	0.45± 0.05	0.15± 0.30	0.36± 0.03	0.55± 0.01	0.61± 0.09	0.52± 0.03

*** Retention Index** Data is expressed as mean ± SD, n = 3

Similar to the GC-MS and GC-FID analysis carried out for the volatile oils extracted from the black pepper seeds after GAPD treatment, there was no statistical significance in the composition when twelve components of volatile oils are considered in LPCP treatment. However, as can be seen in Table 02, changes in composition were observed in volatile oils directing to change the quality of volatile oils. α-pinene, β-pinene, O-cymene, δ-3-carene and Caryophyllene Oxide were increased after LPCP treatments while, D-limonene, δ-elemene, α-copaene, and β-caryophyllene decreased after the treatments. Irrespective to the extracted volatile oils yield, slight changes of the composition with the LPCP treatment was observed.

The compositional analysis highlighted that, though the volatile oil extraction yield increased with the GAPD and LPCP treatments, no significant changes were detected composition wise confirming the applicability of the two methods for using as pre-treatment methods.

Volatile oils of black pepper are predominantly composed of monoterpene hydrocarbons, constituting 30-70 % of the total. Examples include α-pinene, β-pinene, limonene, sabinene, myrcene, β-phellandrene, and δ-3-carene. Sesquiterpenes, particularly β-caryophyllene, contribute significantly to the volatile oil content, accounting for approximately 25-45 % of the total. Additionally, oxygen enriched sesquiterpenes like caryophyllene oxide are present in quantities ranging from 4-14 %(Schulz et al., 2005).

The oils identified in this study primarily consisted of monoterpene hydrocarbons, aligning with findings from prior research(Pepper et al., 2019). Figure 03 displays the monoterpenes, sesquiterpenes, and oxygenated compounds derived from volatile oils extracted from black pepper seeds pre- and post-treatment with GAPD and LPCP.

In GAPD treatment, there was no significant difference (P>0.05) in monoterpenes content of the volatile oils and the sesquiterpene content significantly decreased after 15 min treatment. Monoterpene content varied in between 60 – 66 % while sesquiterpenes ranged from 28 – 32 % by weight complying to the available literature (Pepper et al., 2019) even after exposing to the cold plasma treatment. Sesquiterpenes content decreased from 31.45 to 28.63 % and however the amount remaining after treatments was well within the typical ranges. These minor components of volatile oils create different and specific notes, contributing to the aroma profile of black pepper. As mentioned in the literature (Schulz et al., 2005), achieving an ideal pepper aroma quality involves a high concentration of monoterpenes excluding α-pinene and β-pinene, coupled with a low level of pinenes.

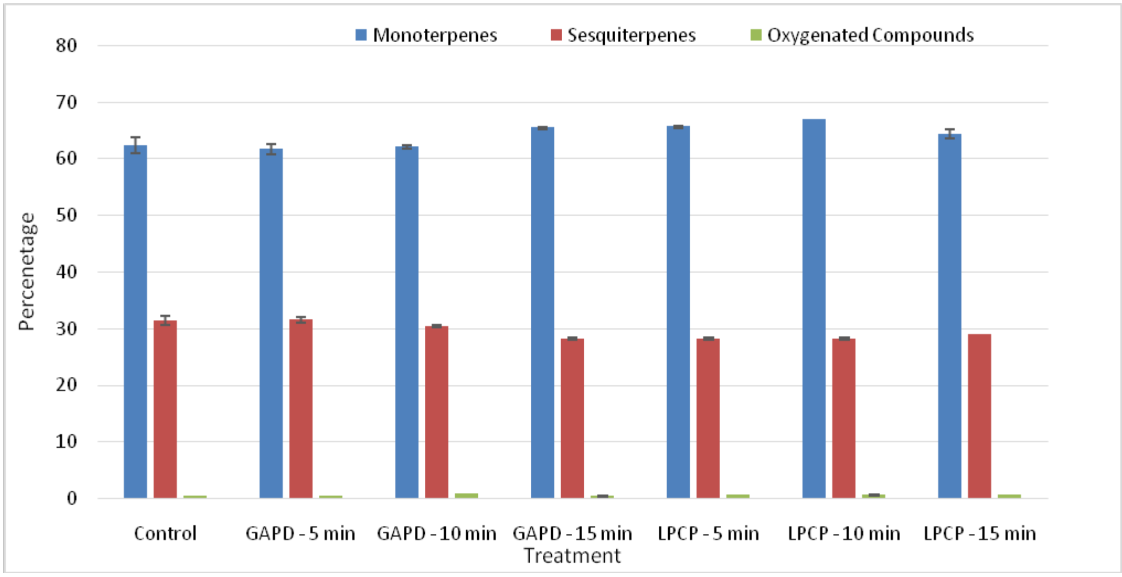


Figure 2: Variation of volatile oil components in GAPD and LPCP treatments

The term “top-peppery-note” is commonly associated with the monoterpene group, while the “pepper odor” is attributed to sesquiterpenes. Furthermore, the “body” of the pepper aroma is often linked to oxygen containing sesquiterpenes (Schulz et al., 2005). As per the results obtained from GC MS and GC FID, it was observed as a reduction of sesquiterpene content might have an effect on the odor of the black pepper seeds after GAPD treatment.

In LPCP treatment, there was no significant difference (P>0.05) in monoterpenes content of the volatile oils, sesquiterpene content significantly (P<0.05) decreased after 15 min treatment. Monoterpene content varied in between 62 – 67 % while sesquiterpenes ranged from 27 – 32 % by weight complying to the available literature even after exposing to the LPCP treatment. Sesquiterpenes content decreased from 31.00 to 28.22 % after 10 min treatment and however the amount again increased up to 29.02 % keeping the composition well within the typical ranges. These results further confirm that the 5-minute treated black pepper seeds are more with preserved quality than further treatment times confirming the suitability of 5-minute LPCP treatment as a pretreatment for hydro distillation process.

CONCLUSION

The study focused on the capability of Low-Pressure Cold Plasma (LPCP) and Gliding Arc Plasma Discharge (GAPD) treatment to be used as a pretreatment method for hydro-distillation. The ideal treatment times were 5 minutes and 10 minutes respectively for the LPCP and GAPD to extract maximum quantity of volatile oils. Results confirmed both LPCP and GAPD does not affect the quality of Volatile Oils extracted from black pepper seeds undesirably during the selected processing times revealing the applicability as a potential pretreatment technology. However, more scaled up research are required to confirm the process parameters prior to commercial level applications.

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