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Impacts of industrial microwave and infrared drying approaches on hemp (*Cannabis sativa* L.) quality and chemical components

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ABSTRACT

The rapid increase in industrial hemp cultivation requires the use of high throughput drying approaches to prevent fresh hemp spoilage and quality degradation. The aim of this study was to understand the impact of hot air (HA), 915 MHz microwave (MW) and infrared (IR) drying techniques on the color, cannabinoids, and volatile contents of dried hemp. Fresh hemp was dried to a moisture content of 10% wet basis with HA, IR heat, and MW heat (at 2- and 3-kW power levels). Afterward, the color, cannabinoid and volatile compounds in the hemp samples were determined. There was a high total color difference (4.12) between hemp dried with HA and IR heat. Hemp dried with HA and IR had higher cannabidiol (CBD), caryophyllene, and humulene concentrations than fresh hemp. However, the total cannabinoids and volatile of hemp dried with IR, MW-2 kW, and MW-3 kW were lower than that of the fresh hemp.

1. Introduction

Industrial hemp (*Cannabis sativa L.*) is an annual herbaceous flowering plant that has found uses in different industries due to its high fiber and pharmaceutically important chemical compounds (Russo et al., 2008; Vonapartis et al., 2015). Terpenes and cannabinoids are the main chemical compounds found in industrial hemp. Terpenes are volatile hydrocarbons with small isoprene units in chains and are responsible for the aroma of hemp. Both cannabinoids and terpenes have a variety of uses in treating several medical conditions in humans (Garcia-Tejero et al., 2019; White, 2019). Industrial hemp produces cannabinoids such as cannabidiol (CBD), cannabichromene (CBC), cannabinol (CBN), cannabigerol (CBG), cannabidivarin (CBDV), but has low levels (< 0.3%) of tetrahydrocannabinol (THC). Industrial hemp has, therefore, gained popularity and market potential due to its rich chemicals and uses in pharmaceutical, cosmetical, and food applications.

Like other herbs, fresh hemp is harvested at a high moisture content (MC) of about 80% wet basis (w.b.) and is highly perishable. In order to prevent spoilage due to microbial growth and other chemical reactions, fresh hemp needs to be rapidly dried to a safe MC of approximately 10% w.b. before storage (Kwasnica et al., 2020). In addition, drying

Abbreviation: ΔE, Total color change; CBC, Cannabichromene; CBD, Cannabidiol; CBDA, Cannabidiolic acid; CBG, Cannabigerol; CBGA, Cannabigerolic acid; CBN, Cannabinol; CBDV, Cannabidivarin; CBDVA, Cannabidivarinic acid; FID, Flame ionization detector; GC, Gas chromatography; HPLC, High performance liquid chromatography; IR, Infrared; MC, Moisture content; MS, Mass spectrometry; MW, Microwave; THC, Tetrahydrocannabinol; w.b, Wet basis; Rcf, Relative centrifugal field

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of herbs leads to a huge reduction in weight, which invariably help reduce costs for storage, packaging, and transportation. It is known that dehydrating methods and other processing parameters can have major impacts on the physical and chemical components, especially volatiles compounds, of herbs including hemp (Calin-Sanchez et al., 2015; Chen et al., 2021); (Figiel et al., 2010); (Xing et al., 2017). The chemical compounds in hemp are sensitive to heat and light, therefore, the dehydration process may have effects on the composition and concentration of the volatile substance and may lead to the formation of new chemical compounds (Chua et al., 2019).

The conventional method of drying hemp is by leaving them on the field to dry completely before being packed. An improved conventional method of drying involves hanging hemp to dry in a dark room at 15.6 °C. This latter method, however, takes about 7–10 days or even longer to attain a storage-safe MC. The conventional dehydration approaches are time consuming and may lead to microbial contamination and degradation of physical and chemical quality in hemp before the target MC is reached (Challa et al., 2020). Due to the high yield of hemp plant and its continual demand, a high throughput drying technology is required to rapidly dry a large amount of freshly harvested hemp to a safe MC within a short duration to prevent spoilage and ensure its availability for end-use purposes.

Microwave (MW) and infrared (IR) radiation are part of the electromagnetic spectrum and they have been widely used to rapidly dry food material to a MC level that permits long-term storage. For instance, (Orphanides et al., 2013) explored the use of MW in dehydrating spearmint, while (Nozad et al., 2016) used IR technology to dehydrate spearmint. Other herbs and leaves such as coriander, oregano, parsley, basil, peppermint, and sage have been dried using both MW and IR heating technology (Divya et al., 2012; Figiel et al., 2010; Hamrouni-Sellami et al., 2012; Salarikia et al., 2016; Soysal, 2004; Yousif et al., 1999). As opposed to domestic MW with 2450 MHz, an industrial MW with 915 MHz has higher power and deeper penetration (Smith et al., 2021). However, to the best of our knowledge, there is a little information in the literature on the exploration of an industrial 915 MHz MW and IR technologies to dry industrial hemp.

Therefore, the aim of this study was to understand the impact of hot air (HA), industrial 915 MHz MW and IR drying techniques on the color, cannabinoids, and volatile content of dried industrial hemp.

2. Materials and methods

2.1. Sample collection and preparation

In this study, the industrial hemp (var. Janets G.) used was grown in Fair Play, South Carolina, USA. The hemp was harvested, in the Fall of 2020, by The Triminator Company and shipped to the Department of Food Science, University of Arkansas, Fayetteville, AR, USA. The sample was stored in a walk-in refrigerator set at 4 °C immediately it was received. Prior to the drying experiment, the sample was retrieved from the refrigerator and allowed to equilibrate to room temperature (25 °C). The initial harvest MC of the sample was measured to be 77% w.b. The MC of the sample was determined by drying the sample in a convective oven set at 100 °C for 8 h.

2.2. IR heating equipment

The catalytic IR heating system (Catalytic Drying Technologies LLC, Independence, KS) assembled in-house at the University of Arkansas, Food Science Department Lab, was used for this study. The pilot-scale IR system, which was previously described by Mohammadi Shad et al. (Mohammadi Shad et al., 2021), consist of a control panel, a conveyor belt equipped with a vibrator and a variable-rate belt speed controller, a heating chamber (where samples are placed for drying), and propane gas-powered catalytic IR emitter.

2.3. Industrial 915MHz MW heating equipment

The industrial 915 MHz MW heating system (AMTek Microwaves, Cedar Rapids, IA) used in this study was previously described by Olatunde, Atungulu, & Smith (Olatunde et al., 2017). Briefly, the system consists of a MW generator, control panel, waveguide, and heating zone. The MW generator in this system is a high-powered vacuum tube that worked as a self-excited MW oscillator that convert high-voltage electric energy into MW radiation. Also, the waveguide has a rectangular pipe that allows for the propagation of the electromagnetic field – the pipe conveys the generated MW from the magnetron into the heating zone where samples are placed for drying.

2.4. Drying experiment

For HA drying, hemp flower was arranged, in a single layer, on a mesh tray and gently dried in a controlled environment (Conviron, G1000, USA) set at 30 °C and 65% relative humidity for 26 hr to achieve a final MC of 10% w.b.

For the IR heating drying approach, the IR equipment was set at 5.55 kW/m^2 intensity and the belt speed was set at 0.0192 m/s. A 7 kg sample of fresh hemp was loaded (single-layered – thickness of about 5 cm) onto the moving belt of the IR heating equipment. The moving belt then continuously carried the sample into the heating chamber for drying and exit the heating chamber after 3 min (referred to as 'one pass' under IR heat). This process was then repeated 8 times (passes uner the IR heat) to achieve a final MC of approximately 10% w.b. The total IR drying time was 24 min

For the the industrial 915 MHz MW drying approach, similar to the IR drying approach described, a 8 kg sample of fresh hemp was loaded onto the conveyor belt of the MW equipment and then exposed to MW heat using a power of 2 kW. Six passes under MW heat was done to achieve a final MC of approximately 10% w.b. The total drying duration using 915 MHz MW at 2 kW was 45 min. A second experiment was conducted by drying a 8 kg sample of fresh hemp using MW at a power of 3 kW. In this experiment, a total of 4 passes was done to achieve a final MC of approximately 10% w.b. – the total duration was 30 min

Subsequently, all dried samples were analyzed for color; all samples, including fresh hemp, were analyzed for cannabinoids and volatile contents.

2.5. Color analysis

Three replicates, representative sample, were collected from all the dried samples. The dried samples were then homogenized by grinding into a fine powder using a coffee grinder

(Hamilton Beach, 80393, Virginia, USA) for 1 min. Afterward, the color of the dried powder sample was measured using a Konica Minolta colorimeter (CR-400 Chroma Meter, Tokyo, Japan). This instrument measures color indices (L^*, a^*, and b^*) as specified by the International Commission on Illumination (CIE). The parameter L^* describes the lightness from 100 (light) to 0 (dark), parameter a^* describes red-green color with $+a^*$ (redness) to $-a^*$ (greenness), and parameter b^* describes yellow-blue color with $+b^*$ (yellowness) to $-b^*$ (blueness). The total change in color (ΔE) between the HA-dried sample, and IR- and MW-dried sample was calculated using Eq. 1.

$$\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2} \quad (1)$$

Where the subscripts 1 and 2 (on the L^*, a^*, and b^* parameters) represent sample dried with HA and IR-dried (or MW-dried) samples, respectively.

2.6. Cannabinoids and volatile extraction and analyses

2.6.1. Chemicals

Cannabinoid mixture - acids (C-218–1 mL), cannabis terpene mix A (CRM40755) and cannabinoid mixture - neutrals (C-219–1 mL) were obtained from Sigma-Aldrich, St. Louis, MO. Ethanol (Koptec, 190 proof, V1101), acetonitrile (Supelco, HPLC grade, AX0145–1) and formic acid (Millipore-Sigma, 98%, FX0440–6) were obtained from VWR, Radnor, PA.

2.6.2. Extraction of cannabinoids

Prior to extraction, dried hemp was ground into a powder using a coffee grinder. Following the method described by (Brighenti et al., 2017) and (Pellati et al., 2018), with slight modifications, a 250 mg of the sample was weighed into a 50 mL centrifuge tube, and 10 mL of ethanol was added. The slurry was stirred on a magnetic stir plate for 30 min, followed by sonication (VWR B2500A-MT sonicator) for 15 min. The slurry was then centrifuged at 10,000 rcf for 5 min and the supernatant was decanted through Miracloth (Millipore-Sigma, St. Louis, MO) into a 25 mL volumetric flask. To the remaining pellet, 10 mL of ethanol was added, and the stirring, sonicating, centrifuging, and filtering steps were repeated. The process was repeated a third time using 5 mL ethanol for the final extraction. All the supernatants were pulled and assured of a final volume of 25 mL. For the fresh hemp sample, 1 g was homogenized (Ultra Turrax, T18 IKA Works Wilmington, NC) with 20 mL ethanol, centrifuged at 10,000 rcf, and filtered through Miracloth into a 100 mL volumetric flask. The remaining pellet was extracted 2 more times using this method and the supernatant was pooled and brought to a final volume of 100 mL with ethanol.

2.6.3. Extraction of volatiles

Volatiles extraction was performed using solid phase micro extraction (SPME) following the method previously described by Pellati et al. (Pellati et al., 2018) with a slight modification. The fiber used in this analysis was the 85 µm, CAR/PDMS, Stableflex, 24 Ga, Manual Supelco (Bellefonte, PA). A 20 mL headspace vial containing 5 mg of ground hemp was placed in a heat block on a stir plate with heating capability at 65 °C and equilibrated at this temperature for 30 min. The SPME fiber was inserted into the headspace above the sample and adsorbed for 20 min. For the fresh hemp sample, 2 g was homogenized with 20 mL water, then 0.5 mL was placed into a 20 mL headspace vial and allowed to equilibrate at 65 °C for 30 min and adsorbed for 20 min in the same manner as ground hemp. Samples were desorbed into the injection at 250 °C for 3 min

2.6.4. HPLC analysis for the cannabinoids

A method previously described by Giese, Lewis, Giese, & Smith (Giese et al., 2015) was used with little modifications. Briefly, samples (10 µL) were analyzed using a Waters HPLC system equipped with a model 600 pump, a model 717 Plus autosampler and a model 996 photodiode array detector. Separation was carried out using a 4.6 mm × 250 mm Symmetry® C18 column (Waters Corp, Milford, MA, USA) with a 3.9 mm × 20 mm Symmetry® C18 guard column. The mobile phase was isocratic for 30 min at a ratio of 30% using mobile phases of 0.1% formic acid (A) and 70% acetonitrile (B). A linear gradient followed from 70% B to 90% B for 10 min at 1 mL/min. The system was equilibrated for 10 min at the initial gradient prior to each injection. Detection wavelength was 215 nm. Peaks were identified comparing retention times and UV spectra to that of the authentic standard. Calibration curves were performed for each cannabinoid standard 10–200 µg/g, and compounds concentrations were calculated from the linear regression lines. After the quantification of all cannabinoids, the total CBD was calculated using Eq. 2.

$$Total \quad CBD = CBD + (0.877 * CBDA) \quad (2)$$

Where 0.877 represents the compensatory mass loss during the decarboxylation process from CBDA to CBD.

2.6.5. GC-MS-FID analysis of volatile compounds

With a slight modification, the method described by (Giese et al., 2015) was used for hemp volatile compounds analysis. Gas chromatography analysis was performed using a Shimadzu GC-2010 Plus Gas Chromatograph equipped with a Flame Ionization Detector (GC-FID) and a GCMS-QP2010 SE Mass Spectrometer (GC-MS). Samples were analyzed by both GC-FID and GC-MS and separation was performed on each using a HP-5 (30 m × 0.25 mm inner diameter, 5% phenylmethylpolysiloxane, 1.0 µm film thickness) capillary column (Agilent, Santa Clara, CA). For both GC-MS and GC-FID analysis, the injector temperature was 250 °C. Helium was used as the carrier gas and column flow rate was 1.0 mL/min. The oven temperature was programmed from 45° to 100°C at 2 °C/min, then from 100° to 250°C at 5 °C/min, and with a 5 min hold at 280 °C. The GC-FID detector temperature was 300 °C and the interface temperature for the GC-MS had an ion source temperature of 230 °C and an interface temperature of 250 °C. GC-MS was performed in full scan mode, with a scan range of 20–300 *m/z*. The volatiles were identified by comparison of their mass spectra with the National Institute of Standards and Technology NIST17 spectral library, literature data, and retention indices. The retention indices were performed after running alkane standards of 5–20 carbons and online searches of similar work with HP5 or comparable columns. Calibration curves were performed for several standards, and compounds concentrations were calculated from the linear regression lines from authentic standards or quantified as equivalents of related compounds where standard was not available.

All cannabinoid and volatile concentrations of dried and fresh hemp samples were corrected for moisture differences and are, therefore, reported per gram of the dry weight.

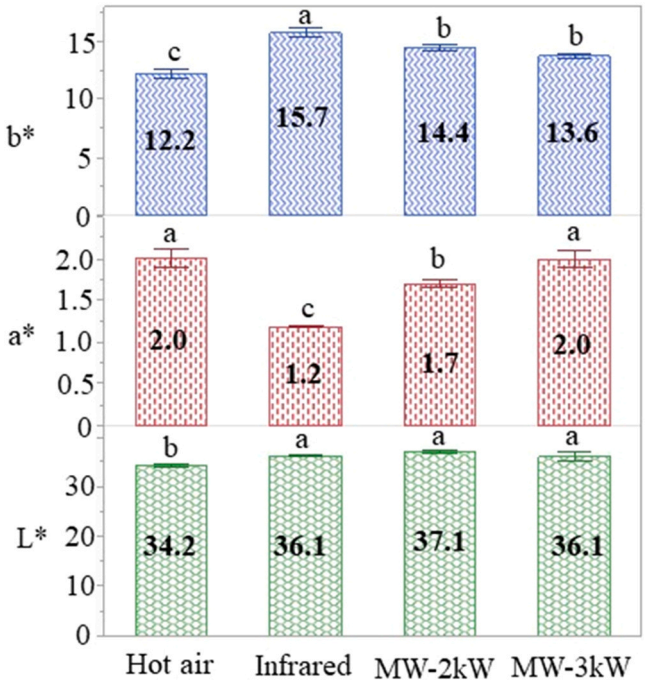

Fig. 1 – the color profiles of hemp dried using different drying approaches; MW means microwave.

2.7. Statistical analysis

A statistical software (JMP version 16.0.0, SAS Institute) was used to carry out an analysis of variance (ANOVA) and Tukey's honest significant difference (HSD) test to determine significant differences within and among samples. All tests were considered to be significant when p-value < 0.05.

3.0. Results and discussion

3.1. Effects of different drying approaches on dried hemp color

Fig. 1 shows the color profile of hemp dried using different drying approaches. The L^* values of hemp dried using IR, MW at 2 kW, and MW at 3 kW were significantly greater than that of hemp dried with HA. This means that hemp dried with HA had a darker color than hemp dried using other drying approaches tested in this study. However, there was no significant difference between the L^* values of hemp dried with IR, MW-2 kW, and MW-3 kW. Out of all the dried hemp samples, IR-dried hemp had the least redness (a^*) value (1.2). On the other hand, hemp dried with HA and MW-3 kW did not have significant difference in their redness. Hemp dried with IR had the greatest value of yellowness (b^*) when compared to hemp dried using HA and MW. The total color change (ΔE) value can be used as an estimate of color change between samples.. All ΔE calculated in this study are greater than 1, which means the color differences can be perceived by human eye. The highest ΔE (4.12) was observed between hemp dried with HA and IR heat. This indicated that IR heat had the greatest impact on the color change of the dried hemp. On the other hand, the least ΔE was observed between hemp dried with HA and MW-3 kW (2.42). (Chen et al., 2021) and (Selvi, 2020) also reported significant changes in the color of linden (*Tilia platyphyllos Scop.*) and hemp leaves after drying with IR and MW drying techniques.

3.2. Impact of different drying approaches on the cannabinoid content of dried hemp

Table 1 show the cannabinoid contents of fresh hemp and hemp dried using different drying approaches. The major cannabinoid compounds found in the hemp sample, used in this study, were CBDA and CBGA. The CBDA of the fresh hemp was 215.0 mg/g while that of hemp dried with HA, IR, MW-2 kW, and MW-3 kW were 93.4, 70.6, 93.0, and 89.6 mg/g,

Table 1 – Concentrations of cannabinoid compounds present in fresh hemp and hemp dried with different drying approaches; MW means microwave. In each row, values not connected by same letters are significantly different (p < 0.05).

Compound (mg/g of dry weight)	Drying approaches				
	Fresh	Hot air	Infrared	MW-2 kW	MW-3 kW
CBDVA	0.2 ± 0.0^{a}	0.1 ± 0.0^{b}	0.1 ± 0.0^{ab}	0.1 ± 0.0^{b}	0.1 ± 0.0^{b}
CBDV	0.0 ± 0.0^{c}	0.2 ± 0.0^{b}	0.3 ± 0.1^{a}	0.1 ± 0.0^{b}	0.1 ± 0.0^{b}
CBDA	215.0 ± 4.0^{a}	93.4 ± 4.2^{b}	70.6 ± 2.1^{c}	93.0 ± 2.3^{b}	89.6 ± 0.5^{b}
CBGA	3.5 ± 0.4^{a}	1.9 ± 0.2^{b}	1.5 ± 0.0^{b}	1.8 ± 0.2^{b}	1.9 ± 0.0^{b}
CBG	1.4 ± 0.0^{b}	0.7 ± 0.0^{c}	2.0 ± 0.0^{a}	0.7 ± 0.1^{c}	0.6 ± 0.0^{c}
CBD	15.6 ± 0.4^{d}	27.1 ± 0.6^{b}	51.4 ± 0.8^{a}	22.2 ± 0.4^{c}	22.6 ± 0.7^{c}
Total CBD	204.2 ± 3.7^{a}	109.1 ± 4.2^{bc}	113.3 ± 1.2^{b}	103.7 ± 2.0^{cd}	101.1 ± 0.7^{d}
Total Cannabinoid	235.7 ± 5.8^{a}	123.4 ± 5.3^{b}	125.9 ± 2.1^{b}	117.8 ± 2.4^{b}	114.8 ± 0.6^{b}

Table 2 – Concentrations of volatile compounds present in fresh hemp and hemp dried with different drying approaches; MW means microwave. In each row (for total volatile), values not connected by same letters are significantly different ($p < 0.05$).

S/N	Compound (μg/g dry weight of hemp)	Retention index	Drying approaches				
			Fresh	Hot air	Infrared	MW-2 kW	MW-3 kW
1.	Acetic acid	553	0.4 ± 0.2	13.3 ± 4.1	36.6 ± 6.8	7.8 ± 0.7	24.3 ± 4.1
1.	Hexanal	798	3.6 ± 1.3	0.9 ± 0.2	0.7 ± 0.1	0.8 ± 0.1	1.1 ± 1.1
2.	2-hexenal	849	3.7 ± 0.9	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
3.	3-hexen-1-ol	852	0.8 ± 0.1	0.8 ± 0.0	1.4 ± 0.2	0.0 ± 0.0	0.0 ± 0.0
4.	1-hexanol	864	4.6 ± 0.3	8.4 ± 0.5	5.6 ± 0.3	2.3 ± 0.4	5.4 ± 0.2
5.	2-heptanone	888	0.6 ± 0.1	1.0 ± 0.1	1.4 ± 0.1	0.0 ± 0.0	0.0 ± 0.0
6.	Heptanal	900	0.9 ± 0.3	1.2 ± 0.0	1.7 ± 0.1	2.2 ± 0.1	2.6 ± 0.2
7.	α-thujene	928	6.4 ± 0.5	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
8.	α-pinene	937	1551.5 ± 321.9	959.3 ± 393.0	158.4 ± 28.9	157.7 ± 36.3	66.7 ± 7.8
9.	Camphene	952	24.0 ± 4.3	16.2 ± 5.3	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
10.	Benzaldehyde	961	3.8 ± 1.2	1.1 ± 0.2	1.5 ± 0.1	0.0 ± 0.0	2.7 ± 3.8
11.	1-heptanol	966	1.0 ± 0.2	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
12.	Hexanoic acid	968	0.0 ± 0.0	3.3 ± 0.7	1.7 ± 0.1	0.8 ± 0.2	1.5 ± 0.4
13.	β-pinene	981	5105.8 ± 1083.4	289.8 ± 106.5	52.7 ± 8.2	37.1 ± 7.7	19.2 ± 2.3
14.	6-methyl-5-Hepten-2-one	985	1.2 ± 0.1	6.0 ± 0.5	4.2 ± 0.1	2.2 ± 0.3	4.6 ± 1.4
15.	β-myrcene	991	368.9 ± 46.6	191.4 ± 39.5	131.9 ± 11.8	20.1 ± 1.7	18.8 ± 3.1
16.	Ethyl hexanoate	997	0.7 ± 0.5	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
17.	α-terpinene	1022	1.9 ± 0.1	3.9 ± 0.6	3.7 ± 0.3	3.2 ± 0.5	4.3 ± 0.4
18.	Cymene	1032	0.8 ± 0.1	1.6 ± 0.1	1.1 ± 0.1	3.9 ± 4.6	1.3 ± 0.0
19.	D-Limonene	1037	726.1 ± 101.8	354.9 ± 85.2	214.3 ± 24.0	36.1 ± 18.4	49.1 ± 1.4
20.	Eucalyptol	1041	3.4 ± 0.4	5.7 ± 1.2	0.0 ± 0.0	1.9 ± 1.7	1.9 ± 0.1
21.	trans-β-ocimene	1042	3.9 ± 0.2	2.7 ± 0.4	1.0 ± 0.1	0.1 ± 0.2	0.4 ± 0.4
22.	β-ocimene	1054	112.5 ± 17.2	55.7 ± 10.5	26.2 ± 0.4	2.4 ± 0.3	2.9 ± 0.3
23.	γ-terpinene	1067	0.9 ± 0.1	1.5 ± 0.4	0.7 ± 0.1	1.1 ± 0.3	1.0 ± 0.1
24.	Terpinolene	1094	5.0 ± 0.5	6.2 ± 0.8	0.0 ± 0.0	1.4 ± 0.1	1.7 ± 0.1
25.	Fenchone	1095	13.3 ± 1.3	5.6 ± 9.7	28.5 ± 2.2	11.5 ± 1.6	9.7 ± 1.1
26.	Linalool	1100	4.1 ± 0.2	7.2 ± 0.7	6.8 ± 0.6	6.4 ± 0.1	8.1 ± 0.1
27.	Perillene	1103	1.8 ± 0.3	2.7 ± 0.1	0.5 ± 0.4	1.1 ± 0.3	1.3 ± 0.2
28.	Phenylethyl Alcohol	1118	1.2 ± 0.1	2.6 ± 0.3	2.5 ± 0.1	0.6 ± 0.0	2.7 ± 0.3
29.	Fenchol	1122	26.4 ± 1.2	40.6 ± 7.5	25.6 ± 5.2	29.3 ± 4.9	28.0 ± 2.3
30.	trans-2-Pinanol	1132	21.9 ± 1.7	41.3 ± 5.3	27.1 ± 3.9	26.8 ± 3.9	25.7 ± 2.6
31.	cis-p-Menth-2,8-dien-1-ol	1136	37.4 ± 2.0	24.7 ± 1.6	10.8 ± 9.5	3.1 ± 0.2	3.9 ± 0.4
32.	4-Acetyl-1-methylcyclohexene	1138	0.0 ± 0.0	0.5 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.4 ± 0.1
33.	cis-p-Mentha-2,8-dien-1-ol	1143	7.0 ± 2.8	7.3 ± 0.2	5.1 ± 1.8	5.3 ± 0.7	6.1 ± 1.0
34.	4E,6Z-allo-Ocimene	1146	2.3 ± 0.1	2.6 ± 0.3	1.8 ± 0.3	0.0 ± 0.0	0.2 ± 0.2
35.	Myrcenone	1153	0.4 ± 0.0	0.6 ± 0.0	0.6 ± 0.0	0.4 ± 0.1	0.6 ± 0.6
36.	3-Nonen-1-ol	1157	0.2 ± 0.0	0.3 ± 0.0	0.2 ± 0.1	0.4 ± 0.0	0.5 ± 0.0
37.	2,6-dimethyl-1,5,7-Octatrien-3-ol	1160	2.5 ± 0.3	4.2 ± 0.6	2.9 ± 0.4	2.0 ± 0.1	2.8 ± 0.6
38.	endo-borneol	1178	12.1 ± 1.0	15.2 ± 2.9	9.2 ± 2.2	13.0 ± 1.8	13.8 ± 0.6
39.	Terpinen-4-ol	1187	0.6 ± 0.1	1.8 ± 0.3	1.1 ± 0.1	2.1 ± 0.3	2.2 ± 0.2
40.	Hexyl butanoate	1191	0.0 ± 0.0	1.2 ± 0.1	1.6 ± 0.1	0.1 ± 0.1	0.4 ± 0.0
41.	L-α-terpineol	1198	4.5 ± 0.3	14.9 ± 1.6	11.1 ± 0.4	14.8 ± 0.7	16.6 ± 1.3
42.	Myrtenol	1206	0.2 ± 0.0	0.4 ± 0.0	0.0 ± 0.0	0.2 ± 0.4	0.4 ± 0.4
43.	Carveol	1209	0.2 ± 0.0	0.3 ± 0.0	0.0 ± 0.0	0.1 ± 0.1	0.3 ± 0.1
44.	Citronellol	1229	3.8 ± 0.4	11.4 ± 1.0	7.9 ± 0.9	9.1 ± 0.7	9.5 ± 1.1
45.	Verbenone	1237	0.3 ± 0.0	0.7 ± 0.1	0.2 ± 0.1	0.2 ± 0.0	0.4 ± 0.0
46.	(-)-Carvone	1256	0.0 ± 0.0	0.2 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
47.	Linalool oxide	1273	0.0 ± 0.0	3.3 ± 1.0	1.9 ± 0.1	0.9 ± 1.1	2.4 ± 0.6
48.	Perillic alcohol	1309	0.1 ± 0.0	0.5 ± 0.1	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.2
49.	Hexyl hexanoate	1385	0.1 ± 0.0	0.3 ± 0.1	0.0 ± 0.0	0.2 ± 0.1	0.0 ± 0.0
50.	Ylangene	1394	0.5 ± 0.1	2.8 ± 0.2	1.0 ± 0.1	1.2 ± 0.2	1.4 ± 0.1
51.	Copaene	1398	0.6 ± 0.1	2.8 ± 0.3	0.9 ± 0.1	1.0 ± 0.1	1.6 ± 0.1
52.	cis-α-bergamotene or Cedr-8-ene or 7-epi-Sesquithujene	1401	0.3 ± 0.1	1.9 ± 0.1	0.6 ± 0.0	0.6 ± 0.1	0.6 ± 0.1
53.	γ-gurjunene or γ-muurolene	1411	0.2 ± 0.0	0.3 ± 0.5	0.3 ± 0.0	0.1 ± 0.2	0.2 ± 0.2
54.	7-epi-Sesquithujene or Zingiberene	1417	0.4 ± 0.1	3.3 ± 0.4	1.0 ± 0.0	0.9 ± 0.1	1.3 ± 0.3
55.	Eremophilene or Selina-5,11-diene	1423	2.1 ± 0.5	8.8 ± 1.4	3.4 ± 0.3	3.8 ± 0.1	4.8 ± 0.4
56.	cis-α-bergamotene	1433	3.4 ± 1.3	13.5 ± 2.3	4.3 ± 0.4	4.4 ± 1.4	5.2 ± 0.6
57.	α-santalol or santalene	1440	2.1 ± 0.6	8.3 ± 0.6	3.0 ± 0.2	3.1 ± 0.4	3.3 ± 0.4
58.	Caryophyllene	1452	343.2 ± 72.4	1111.4 ± 176.3	464.4 ± 29.9	396.1 ± 30.6	468.5 ± 40.8
59.	Isocaryophyllene	1458	1.9 ± 0.3	3.7 ± 3.2	0.8 ± 0.7	2.3 ± 0.1	2.6 ± 0.1

(continued on next page)

– Table 2 (*Continued*)

S/N	Compound (μg/g dry weight of hemp)	Retention index	Drying approaches				
			Fresh	Hot air	Infrared	MW-2 kW	MW-3 kW
60.	β-famesene	1462	95.1 ± 21.8	370.5 ± 75.3	99.9 ± 9.5	87.8 ± 13.7	108.5 ± 10.4
61.	α-himachalene	1470	1.6 ± 0.1	6.7 ± 1.2	0.8 ± 0.7	2.5 ± 0.2	2.4 ± 0.3
62.	(+)– 9-epi-β-Caryophyllene or Alloaromadendrene	1478	2.1 ± 0.4	6.8 ± 1.2	2.0 ± 0.2	2.7 ± 0.2	2.9 ± 0.2
63.	Humulene	1486	186.2 ± 34.6	585.0 ± 95.5	225.3 ± 12.7	216.3 ± 25.1	250.6 ± 22.6
64.	Eremophilene or (+)– 9-epi-β-Caryophyllene or Alloaromadendrene	1494	7.1 ± 0.9	31.2 ± 6.2	7.0 ± 0.4	11.8 ± 2.5	10.7 ± 1.3
65.	Curcumene	1497	3.1 ± 0.4	10.5 ± 1.7	4.1 ± 0.4	4.7 ± 0.5	4.6 ± 0.3
66.	Valencene	1501	3.1 ± 0.7	11.2 ± 2.0	4.3 ± 0.3	4.0 ± 0.3	4.6 ± 0.6
67.	(+)– 9-epi-β-Caryophyllene or Alloaromadendrene or Valencene	1505	3.4 ± 0.9	12.8 ± 2.5	5.0 ± 0.4	3.5 ± 0.3	4.3 ± 0.8
68.	α-farnesene or α-chamigrene	1514	62.8 ± 20.3	160.1 ± 38.9	51.3 ± 4.2	31.7 ± 5.0	38.6 ± 2.6
69.	β-eudesmene or selinene	1520	5.7 ± 1.2	19.0 ± 3.4	7.5 ± 0.4	6.7 ± 0.7	7.7 ± 1.1
70.	β-bisabolene	1524	24.7 ± 5.1	96.8 ± 21.1	27.1 ± 2.6	31.6 ± 4.8	33.0 ± 3.1
71.	α-selinene	1527	7.0 ± 1.6	24.0 ± 4.8	9.4 ± 0.9	8.6 ± 1.0	9.9 ± 1.1
72.	α-bulnesene or δ-guaiene	1534	3.7 ± 0.7	10.2 ± 2.0	4.5 ± 0.6	3.8 ± 0.9	4.1 ± 0.2
73.	(-)-β-sesquiphellandrene or cis-Sesquisabinene hydrate	1542	7.6 ± 1.7	23.3 ± 4.8	9.0 ± 0.7	9.1 ± 1.4	9.4 ± 0.6
74.	Guaia-3,9-diene or β-guaiene or γ-selinene	1548	5.6 ± 1.5	21.4 ± 3.7	7.6 ± 1.3	8.2 ± 2.0	8.6 ± 0.9
75.	α-bisabolene	1556	22.7 ± 5.4	92.5 ± 21.2	26.4 ± 2.6	33.2 ± 6.2	33.1 ± 3.3
76.	3,7(11)-Selinadiene	1570	51.9 ± 15.9	222.0 ± 42.7	71.1 ± 10.4	86.3 ± 21.2	91.0 ± 7.9
77.	Selina-3,7(11)-diene	1578	63.4 ± 20.2	286.6 ± 53.7	87.4 ± 15.3	106.7 ± 30.5	111.4 ± 13.9
78.	β-guaiene	1595	1.6 ± 0.3	6.4 ± 3.0	1.6 ± 1.5	2.0 ± 0.5	2.1 ± 0.2
79.	Guaiol	1628	165.8 ± 28.8	471.1 ± 127.1	186.5 ± 13.6	203.6 ± 79.4	242.1 ± 33.1
80.	α-epi-7-epi-5-Eudesmol or α-eudesmol	1639	1.1 ± 0.2	3.0 ± 1.0	1.2 ± 0.1	1.4 ± 0.4	1.6 ± 0.3
81.	γ-eudesmole	1662	132.5 ± 23.7	372.3 ± 97.5	160.0 ± 13.2	194.2 ± 49.6	210.5 ± 12.1
82.	Agarospirol	1668	18.9 ± 2.7	58.0 ± 15.8	25.8 ± 1.8	28.0 ± 10.1	33.9 ± 3.4
83.	Agarospirol 2	1672	17.4 ± 3.7	13.6 ± 23.6	16.1 ± 1.5	19.2 ± 5.9	23.3 ± 5.3
84.	α-eudesmol or 7-epi-α-eudesmol	1693	80.2 ± 27.5	264.5 ± 81.4	109.4 ± 8.6	111.5 ± 52.0	165.2 ± 28.1
85.	Bulnesol	1702	43.0 ± 3.2	170.7 ± 46.9	62.4 ± 11.2	61.8 ± 40.8	79.0 ± 16.0
86.	α-bisabolol	1706	25.9 ± 21.1	95.7 ± 35.0	31.2 ± 3.1	2.7 ± 2.6	55.1 ± 8.4
87.	Eudesm-7(11)-en-4-ol; Juniper camphor	1740	1.4 ± 0.5	3.8 ± 1.4	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0
	Total volatile		9471.9 ± 1736.0[a]	6722.0 ± 1588.3[a]	2514.1 ± 152.3[b]	2105.9 ± 328.0[b]	2383.4 ± 112.5[b]

respectively. On the other hand, the CBD (decarboxylated version of CBDA) of all dried hemp samples were higher than that of the fresh hemp; hemp dried with IR had the highest CBD value (51.4 mg/g). This result can be explained because the heat supplied to hemp during the drying process contributed to the conversion of CBDA and CBGA to CBD and CBG, respectively. In the presence of light and heat, CBDA and CBGA are decarboxylated and converted to CBD and CBG, respectively (Brighenti et al., 2017; Pellati et al., 2018). Chen et al. (Chen et al., 2021) reported an increase in the CBD content of hemp exposed to IR heating. CBD is a valuable compound for pharmaceutical industry due to its high antibiotic, anticonvulsant, antioxidant, and anti-inflammatory properties (Alexander, 2016; Appendino et al., 2011). The total CBDs of hemp dried with MW-2 kW and MW-3 kW were significantly lower than that of hemp dried with HA and IR. From this result, if the end goal in a commercial process is the conversion of CBDA to CBD, it may be beneficial to employ IR heating to simultaneously dry hemp and decarboxylate CBDA to CBD. Unlike CBDA, all the drying approaches (HA, IR, and MW) tested in this study did not significantly contribute to the decarboxylation of CBGA to CBG. Compared with fresh hemp, all the dried hemp samples had significantly lower total cannabinoid.

3.3. Impact of different drying approaches on volatile compounds of dried hemp

Analysis of the volatiles present in both fresh and dried hemp samples showed 84 volatile compounds (Table 2). Some of the major identified volatile compounds include α-pinene, β-pinene, β-myrcene, D-limonene, β-ocimene, caryophyllene, β-famesene, humulene, α-farnesene, guaiol, γ-eudesmole, and α-eudesmol; other volatiles were present in low quantities. Similarly, (Kwasnica et al., 2020) also found myrcene, β-(E)-caryophyllene, limonene, α-humulene, β-pinene, α-pinene as the predominant volatiles present in hemp. The α-pinene value of the fresh hemp was 1551.5 μg/g, while that of hemp dried with HA was 959.3 μg/g (about 38.2% reduction); the other drying approaches (IR and MW) significantly reduced the α-pinene of the dried hemp. Also, the β-pinene value of the fresh hemp (5105.8 μg/g) was greater than the values for hemp dried with HA, IR, MW-2 kW, and MW-3 kW (289.8, 52.7, 37.1, and 19.2 μg/g respectively). In agreement with this finding, (Kwasnica et al., 2020) also found a reduction in the α-pinene value of hemp dried using a vacuum MW. In addition, (Chen et al., 2021) reported an 80% decrease in the α-pinene content of hemp dried using IR. Interestingly, compounds such as caryophyllene, humulene,

guaiol, γ-eudesmole, and α-eudesmol were present in higher concentrations in all dried hemp than fresh hemp. For instance, fresh hemp had a caryophyllene value of 343.2 µg/g, while hemp dried with HA, IR, MW-2 kW, and MW-3 kW had caryophyllene values of 1111.4, 464.4, 396.1, and 468.5 µg/g, respectively. Also, γ-eudesmole concentration in fresh hemp was 132.5 µg/g, while that of hemp dried with HA, IR, MW-2 kW, and MW-3 kW were 372.3, 160.0, 194.2, and 210.5 µg/g, respectively. It is expected that volatiles compounds react differently to thermal treatment since there exist differences in the ability of compounds to breakdown and volatilize. For instance, monoterpene compounds including α-pinene, β-pinene, myrcene, limonene, ocimene relatively have low molecular weight with 2 isoprene units and thus are highly volatile. On the contrary, sesquiterpenes including caryophyllene, guaiol, and humulene have larger molecular weight and are less volatile (Bueno et al., 2020; Pellati et al., 2018).

The total concentration of volatile compounds present in the fresh hemp sample was 9471.9 µg/g. Hemp dried with IR, MW-2 kW, and MW-3 kW had significant reduction in the total concentration of volatile compounds. Although HA drying of hemp resulted in a 29.0% decrease in the total concentration of volatile compounds, the difference was not statistically significant. IR, MW-2 kW, and MW-3 kW drying of hemp resulted in significant decreases (73.5%, 77.8%, and 74.8%, respectively) in the total concentrations of volatile compounds. In agreement with this result, (Chen et al., 2021) found that IR drying of different hemp varieties resulted in a 51.9 – 72.3% decrease in hemp total concentration of volatile compounds. It is important to note that in this study, however, drying hemp with IR and MW resulted in an increase in some pharmaceutically important volatile compound such as caryophyllene, humulene, guaiol, etc. For example, caryophyllene has anti-inflammatory properties, while humulene has antiallergy and antibacterial properties.

Conclusion

The results showed that when compared with HA drying, IR and MW (at both 2- and 3-kW power levels) heating had impacts on the color of the dried hemp sample. In addition, drying hemp using IR and MW resulted in both an increase and a decrease in some medically important compounds. For instance, when compared with the fresh hemp and hemp dried with HA, hemp dried with IR heat had higher concentrations of CBD, CBG, caryophyllene, humulene, etc. On the other hand, the total cannabinoids and volatile of the hemp samples dried with IR, MW-2 kW, and MW-3 kW were lower than that of the fresh hemp. This study provided an insight into the impact of HA and electromagnetic energy driven drying approaches on hemp quality. With parameters optimization and further studies, the use of both IR and 915 MHz MW can be employed as effective drying alternatives to dry hemp for specific end-use application.

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.

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