



## ORIGINAL ARTICLE

# Chemical composition and antifungal activity of *Cinnamomum camphora* chvar. *Borneol* essential oil obtained using solvent-free microwave-assisted method



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## KEYWORDS

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Optimization;  
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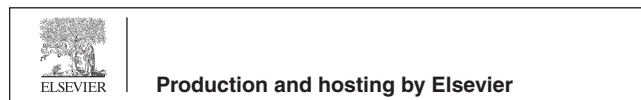
**Abstract** *Cinnamomum camphora* chvar. *Borneol* is an important commercial tree species in China as its essential oil contains abundant *endo*-borneol. Hydrodistillation (HD) is a mainstream technique for the separation of *C. camphora* chvar. *Borneol* essential oil. The shortcomings of HD include low extraction efficiency, high energy consumption, and long extraction time, emphasizing the need for the application of a fast and efficient procedure for recovering *C. camphora* chvar. *Borneol* essential oil. To determine the optimal conditions for the effective extraction, solvent-free microwave-assisted extraction (SFME) was used in conjunction with the Box-Behnken response surface design. The optimal conditions resulted in a maximum essential oil yield of  $42.03 \pm 1.56$  mg/g. These results showed that SFME is an efficient, green, and economical method for obtaining essential oil with a higher proportion of *endo*-borneol compared to HD. Furthermore, *C. camphora* chvar. *Borneol* essential oil exhibited remarkable inhibitory activity against the five *Fusarium* spp. species that cause potato dry rot. The results could provide useful information regarding the

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comprehensive utilization of *C. camphora* chvar. *Borneol* essential oil as a new botanical pesticide in agriculture and food production.

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## 1. Introduction

*Endo-borneol*, also known as natural borneol, is a highly sought-after compound in conventional Chinese medicine due to its antimicrobial activity, anti-inflammatory activity and neuroprotective effect (Cai et al., 2008; Chen et al., 2015; Chen et al., 2019). *Endo-borneol* can alleviate pain and swelling and is thus often applied to cure topical injuries, such as joint pain, burns, and muscle pain (Wang et al., 2017). *Endo-borneol* was once mainly extracted in the trunks of *Dipterocarpus turbinatus* (Li and He, 2017), which is now endangered due to overharvesting. *Cinnamomum camphora* chvar. *Borneol* was identified as a chemotype of *C. camphora* in Jian city (Jiangxi province) and Xinhuang town (Hunan province) in South China (Shi et al., 1989; Xie et al., 2019). *Endo-borneol* is the predominant component of *C. camphora* chvar. *Borneol* essential oil (Shi et al., 2013; Xie et al., 2019), and is considered to be a new source of *endo-borneol*. It is widely cultivated in Southern China as an important industrial crop. As of 2021, around 10,000 ha of land in the Jiangxi and Hunan provinces are used for *C. camphora* chvar. *Borneol* plantations (Mo et al., 2021).

*C. camphora* chvar. *Borneol* essential oil is primarily isolated using hydrodistillation (HD), which has been favored due to low costs and ease of operation, however, the extraction process generally involves a long extraction time, requires a high energy input, and generates a low yield (Chen et al., 2016). As this extraction method cannot keep up with the high demand for plant essential oils, alternative efficient and green extraction approaches are urgently needed. Neutral cellulase-assisted steam distillation has been employed to extract *C. camphora* chvar. *Borneol* essential oil and appeared to be a more effective method associating with higher yield and shorter processing time than HD (Yu et al., 2019). However, this method has the disadvantage of long enzymatic durations and enzymatic treatments generally incur high costs. Recently, solvent-free microwave-assisted extraction (SFME) has attracted more and more attention for obtaining plant essential oils. Araujo et al. (2021) reported that SFME is a feasible and environmentally-friendly extraction method that produces less waste, involves no extra solvent, and has low energy consumption, short extraction time, and high extraction efficiency. In the process of SFME, the directions of heat conduction and mass transfer were from inside to outside, which can greatly improve the extraction efficiency. As for HD, the direction of heat convection was in the opposite direction (namely from outside to inside). The *in situ* water contained within plant cells is directly heated by microwave radiation, which induces high temperatures and pressures in the inner plant cells, leading to the destruction of plant cytoderm and the release of essential oils (Chen et al., 2011; Benmoussa et al., 2019; Franco-Vega et al., 2019). Furthermore, the SFME process is environmentally friendly and the essential oil obtained is pure and does not require additional solvent (Khalili et al., 2018).

Potatoes, a staple food with high nutritional value, are one of the world's largest agricultural crops. Potatoes are easily infected by fungi, which are responsible for various plant fungal diseases. Potato dry rot (PDR) is a devastating post-harvest disease that occurs worldwide and is mainly caused by *Fusarium* spp. (Gachango et al., 2011). It was reported that potato tubers could be affected by PDR during storage or in the fields with the related losses ranging between 6 and 25%, and even as high as 60% (Wang et al., 2020a). Previous studies reported that there were frequent outbreaks of PDR in the northern regions of China, which are mainly caused by *Fusarium avenaceum*, *Fusarium sporotrioides*, *Fusarium culmorum*, *Fusarium trichothecioides*, and *Fusarium solani* (Min et al., 2010). It is worth noting that some

members of *Fusarium* spp. even generate toxins that are poisonous to humans and animals (Bojanowski et al., 2013; Choi et al., 2021; Zabka et al., 2014), such as fumonisin, which can cause brain damage in animals, liver toxicity, possesses carcinogenic effects, and may even be capable of triggering esophageal cancer (Reddy et al., 2010). Today, synthetic chemical fungicides are still the primary way in which PDR is controlled worldwide due to their effective inhibition of fungal activity, ease of operation, and low input (Hang and Woodams, 2003; Gachango et al., 2011; Wang et al., 2015). However, this method has led to severe problems, such as the increasing resistance of fungi, toxic residues, environmental pollution, and soil damage (Peters et al., 2008; Wang et al., 2020b). A safer and more effective way to control PDR is urgently needed. Plant essential oils have enormous potential as natural bacteriostatic agents against plant fungi pathogens (Mahmoud et al., 2010; Smeriglio et al., 2017). However, the antifungal bioactivity of *C. camphora* chvar. *Borneol* essential oil has not yet been investigated.

In this work, SFME was applied in the extraction of *C. camphora* chvar. *Borneol* essential oil and Box-Behnken response surface design was resorted to determine the optimal conditions that led to the maximum yield. HD was also conducted to compare the two methodologies. The chemical composition of the obtained essential oils was analyzed using gas chromatography-mass spectrometry (GC-MS). The *in vitro* antifungal activity of *C. camphora* chvar. *Borneol* essential oil against five species of *Fusarium* spp. was also studied.

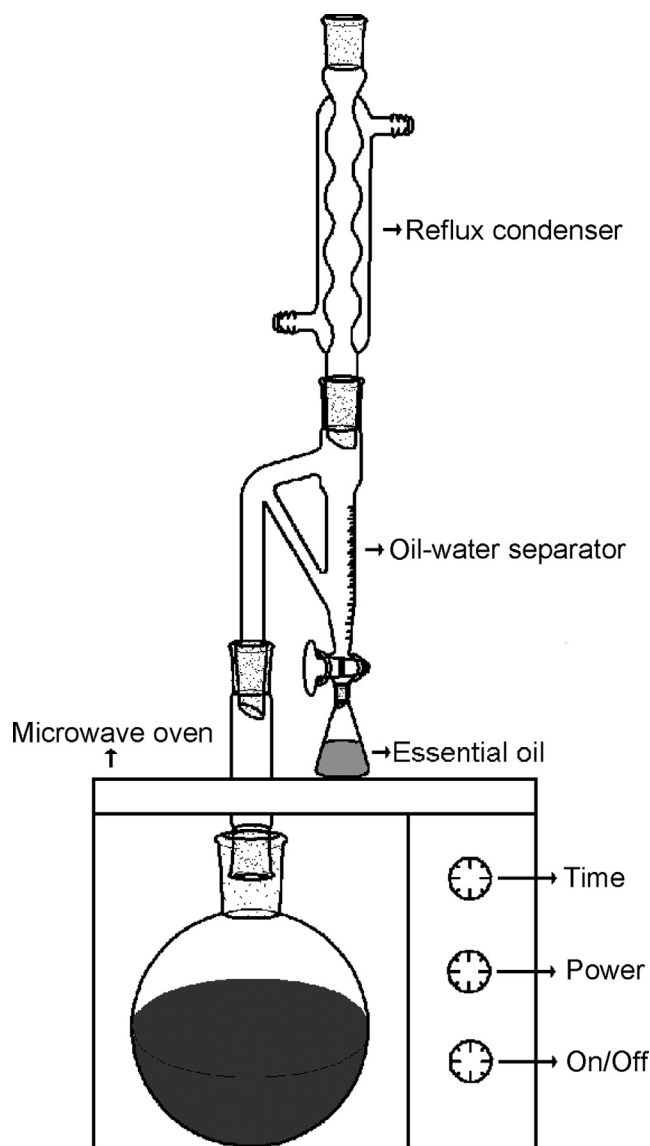
## 2. Materials and methods

### 2.1. Plant material and chemicals

The fresh leaves of *C. camphora* chvar. *Borneol* were bought from Jiangxi Tianxiang Incense Material Co., Ltd. (Fuzhou, China), and then cut into pieces (around 1 cm × 1 cm) before extraction. Potatoes were purchased from a supermarket. *F. avenaceum*, *F. sporotrioides*, and *F. culmorum* were bought from the CGMCC (Beijing, China). *F. trichothecioides* was acquired from Prof. Fenglan Li (Northeast Agricultural University, China), and *F. solani* was obtained from the CCTCC (Wuhan, China). Tween 80, D-Glucose, agar powder, and nystatin were bought from Shanghai Titan Technology Co., LTD (Shanghai, China).

### 2.2. Extraction of essential oil by SFME and HD

The SFME apparatus employed for *C. camphora* chvar. *Borneol* essential oil separation consisted of a microwave oven, an oil-water separator, and a reflux condenser (Fig. 1). Before *C. camphora* chvar. *Borneol* fresh leaves were immersed in distilled water to reach different moisture contents according to a previous study (Benmoussa et al., 2018). The moisture content was calculated by dividing the mass of water contained in leaves by the mass of leaves after immersing treatment. The dry weight of fresh leaves was measured by placing a certain mass of leaves into a drying oven and dried at 50 °C for 48 h. Microwave power levels and durations could be adjusted using the relevant button on the microwave oven. As the



**Fig. 1** A schematic diagram of the instrumental set up using SFME.

extraction system temperature come up to the water boiling point during the extraction process, the *in situ* water within the leaves containing the essential oil was evaporated, transferred, and condensed at the reflux condenser before finally being gathered in the oil–water separator. The bottom layer of water in the immiscible solution was discarded while the essential oil in the upper layer was collected in a brown sample bottle and placed at 4 °C for subsequent analysis and experimentation. The essential oil yield was calculated in terms of milligrams of essential oil per gram of sample (dry weight), and described in Eq. (1):

$$Y = \frac{M_1}{M_2} \quad (1)$$

where  $Y$  (mg/g) is the *C. camphora* chvar. *Borneol* essential oil yield;  $M_1$  (mg) is the quality of the *C. camphora* chvar. *Borneol* essential oil;  $M_2$  (mg) is the quality of *C. camphora* chvar. *Borneol* leaves (dry weight).

A Clevenger apparatus was used for the HD process. This was linked to an essential oil extractor. A total of 200 g of fresh *C. camphora* chvar. *Borneol* leaves were placed into a 2 L round-bottom flask and extracted for 4 h using an electric jacket at 1 kW of rating power with a corresponding temperature of 200 °C. All other steps were the same as described in the SFME process.

### 2.3. Experimental design

A Box-Behnken design (BBD) was performed to investigate the interactions between the independent variables, optimize SFME process conditions and achieve the greatest extraction yield. Three independent variables — moisture content ( $A$ ), microwave time ( $B$ ) and microwave power ( $C$ ) — were run at three levels by BBD as shown in Table 1. The independent variables were coded as  $-1$ ,  $0$ , and  $1$  to represent the low, central, and high levels, respectively. A total of 17 experimental runs involving 12 free combinations and 5 center points were generated through BBD. The quadratic polynomial equation model was used to depict the SFME process for extracting *C. camphora* chvar. *Borneol* essential oil and described in Eq. (2):

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i=1}^3 \sum_{j=i+1}^3 \beta_{ij} X_i X_j \quad (2)$$

where  $Y$  is the *C. camphora* chvar. *Borneol* essential oil yield;  $\beta_0$ ,  $\beta_i$ ,  $\beta_{ii}$ , and  $\beta_{ij}$  signify the relevant regression coefficients of the intercept, linearizer, quadric, and interactivity; and  $X_i$  and  $X_j$  are the independent variables, respectively.

### 2.4. Kinetic model

According to the theory proposed by Handayani et al. (2008), the extraction rate of essential oil is primarily controlled by the mass transfer of the target compounds from the raw materials to the bulk solvent. In accordance with Fick's law, a first-order kinetic model was used to describe the extraction process (Eq. (3)):

$$Y = Y_e [1 - \exp(-K_L \times \alpha \times t)] \quad (3)$$

where  $Y$  is the yield of *C. camphora* chvar. *Borneol* essential oil at any time in the bulk liquid;  $Y_e$  (mg/g) is the yields at equilibrium;  $K_L \times \alpha$  represent the volumetric mass transfer coefficient; and  $t$  refers to microwave time (min).

### 2.5. Chemical composition of essential oil analysis

The chemical composition of the obtained *C. camphora* chvar. *Borneol* essential oil was analyzed by a Thermo Trace-1300 ISQ-GC-MS spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA). It is coupled with an HP-5MS capillary column that has an internal diameter of 30 m  $\times$  0.25 mm and a film thickness of 0.2  $\mu$ m. The column oven was programmed with a beginning temperature of 60 °C (maintained for 5 min), then increased to 120 °C (maintained for 5 min) at a rate of 10 °C/min, before being subsequently increased to 200 °C (held for 5 min) at 10 °C/min, and ultimately rising to 280 °C (held for 15 min) at 10 °C/min. The temperatures for injector and detector were set to 230 °C and 280 °C, respec-

**Table 1** Coded and real levels of screened parameters and observed responses.

Run <sup>a</sup>	A	B	C	Yield (mg/g)	
				Actual	Predicted
1	60 (0)	20 (-1)	700 (+1)	38.97	38.25
2	60 (0)	20 (-1)	380 (-1)	28.39	28.70
3	60 (0)	30 (+1)	380 (-1)	39.79	40.52
4	70 (+1)	30 (+1)	540 (0)	39.72	39.29
5	50 (-1)	30 (+1)	540 (0)	33.53	33.55
6	70 (+1)	25 (0)	380 (-1)	35.07	34.78
7	70 (+1)	20 (-1)	540 (0)	32.12	32.10
8	70 (+1)	25 (0)	700 (+1)	32.14	32.88
9	50 (-1)	20 (-1)	540 (0)	31.57	32.00
10	60 (0)	25 (0)	540 (0)	39.19	40.02
11	50 (-1)	25 (0)	700 (+1)	33.59	33.89
12	60 (0)	25 (0)	540 (0)	40.68	40.02
13	60 (0)	25 (0)	540 (0)	38.51	40.02
14	60 (0)	25 (0)	540 (0)	40.35	40.02
15	60 (0)	25 (0)	540 (0)	41.39	40.02
16	60 (0)	30 (+1)	700 (+1)	35.24	34.92
17	50 (-1)	25 (0)	380 (-1)	28.78	28.04

<sup>a</sup> A: Moisture content, %; B: Microwave time, min; C: Microwave power, W.

tively. The flow rate of carrier gas (the helium) was 1 mL/min. Samples were injected at a 1:2 split ratio. The MS was performed at 70 eV with a scanning range of 50–500 amu in EI mode. The chemical composition of *C. camphora* chvar. *Borneol* essential oil was identified by comparison to the mass spectral patterns and retention indices in the NIST05 mass spectral library.

### 2.6. In vitro antifungal activity of *C. camphora* chvar. *Borneol* essential oil

Assays of mycelial growth and spore germination were performed to evaluate the antifungal activity of *C. camphora* chvar. *Borneol* essential oil against *Fusarium* spp. Minimum inhibitory concentration (MIC) refers to the lowest concentration that completely inhibited mycelial growth, while EC<sub>50</sub> refers to the concentration required for the inhibition of 50% mycelial growth as calculated by linear regression.

#### 2.6.1. Mycelial growth assay

The antifungal experiments were performed using the agar incorporation method with slight modifications (Yakhlef et al., 2020). Due to its hydrophobicity, the stock essential oil solution was prepared by dissolving essential oil in a 5% of tween-80 solution. A certain volume of the stock essential solution was added to the potato dextrose agar medium (PDA) before coagulation; the mixture was transferred into Petri dishes after homogenization. Some tween-80 solution and nystatin samples were added to the medium as negative and positive control, respectively. Fungal discs (approximate 5 mm in diameter) of each strain were moved from the peripheries of the seven-day-old cultures and inoculated aseptically in the center of Petri plates filled with the solidified medium. The inoculated Petri plates were covered with sealing film and placed in a biochemical incubator at 25 ± 2 °C for 7 days. After incubation, the diameter of each colony was measured to calculate their inhibition rates as described in Eq. (4):

$$\text{Inhibition rate(\%)} = \frac{d_c - d_t}{d_c} \times 100 \quad (4)$$

where  $d_c$  is the mean value of the diameter of the mycelial growth in the control set and  $d_t$  is the mean value the diameter of the mycelial growth in the tested sets.

#### 2.6.2. Spore germination assay

The effects of *C. camphora* chvar. *Borneol* essential oil on spore germination in the five *Fusarium* spp. were assessed in the potato dextrose broth as described by previous study (Li and Xue, 2014). The stock essential oil solution and the spore suspension of each strain were added to a 50 mL tube containing PDB, then placed in a shock water bath (60 rpm) held at a constant temperature of 25 ± 2 °C for 8 h. The samples were measured using an LW100B light microscope (Shanghai Baowei Photoelectric Technology Co., LTD). After incubating for 2 h, a minimum of 200 spores per replicate were examined microscopically to determine the germination rate.

### 2.7. Statistical analysis

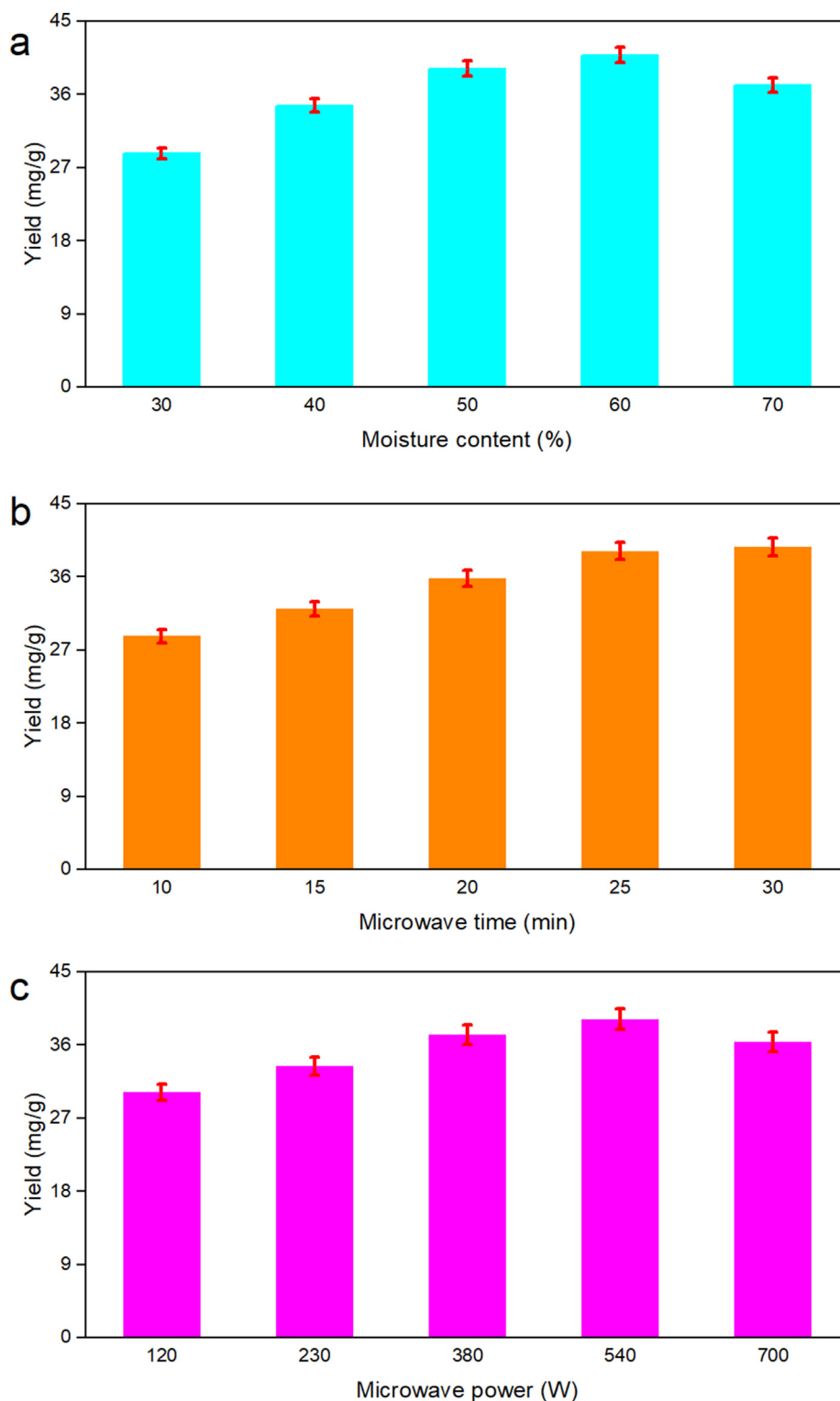
Design Expert 8.0 software (Stat Ease Inc., Minneapolis, MN, USA) was employed to perform BBD. An analysis of variance (ANOVA) was calculated using OriginPro 2018 software (OriginLab Corporation, Northampton, MA, USA). All assays were performed triple and the results were presented as the mean ± standard deviation except for data that was imported into the Design Expert 8.0 software without standard deviation.

## 3. Results and discussion

### 3.1. Single-factor experiment

#### 3.1.1. Influence of moisture content on essential oil yield

The influence of moisture content on *C. camphora* chvar. *Borneol* essential oil yield was evaluated in SFME by varying the



**Fig. 2** Effects of moisture content (a), microwave time (b), and microwave power (c) on the yield of *C. camphora* chvar. *Borneol* essential oil.

moisture content between 30% and 70% with the microwave time and power maintained at 20 min and 380 W, respectively. Fig. 2a, shows that the yield increased gradually as moisture content increased from 30 to 60%. However, the yield decreased as the moisture content steadily exceeded 60%.

The diffusion of essential oil was dependent on the evaporation speed of *in situ* water, and hence low moisture contents resulted in low essential oil yields (Filly et al., 2014). Based on previous studies, high moisture contents may hydrolyze some volatile constituents; however, low moisture contents



could decrease the evaporation velocity of the *in situ* water, leading to the insufficient extraction of plant essential oil (Mellouk et al., 2016; Cui et al., 2019). Thus, moisture contents between 50% and 70% were employed for further optimization.

### 3.1.2. Influence of microwave time on essential oil yield

Microwave irradiation time is a key variable in the isolation of essential oil. The influence of microwave time (10–30 min) on *C. camphora* chvar. *Borneol* essential oil yield was investigated with moisture content and microwave power held at 50% and 380 W, respectively. Fig. 2b, shows that yields improved gradually as microwave time enhanced from 10 to 25 min. However, further increase in microwave time did not result in any obvious increase in yield. To save on operating time and energy consumption, a microwave time ranging between 20 and 30 min was used for the subsequent optimization.

### 3.1.3. Influence of microwave power on essential oil yield

An appropriate level of microwave irradiation plays an important role in the essential oil extraction process. High microwave power may burn the biomass and pyrolyze the essential oil, while low microwave power will not be able to maximize the extraction of the essential oil (Chouhan et al., 2019). The influence of microwave power on yield in the SFME process was studied under the different microwave power as described in Fig. 2c. The results revealed that the highest microwave power did not give rise to the high yield; instead, the highest yield was observed at a microwave power of 540 W. When the plant materials are subjected to the high pressures and temperatures generated by microwave power, plant cell cytodermis

can be easily destroyed and consequently release essential oil (Mellouk et al., 2016). Thus, a range of microwave power between 380 and 700 W was used for follow-up experiments.

## 3.2. Optimization of SFME

### 3.2.1. Model fitting and regression coefficients

To study the significance and probable interactions between the three factors (*A* of moisture content, *B* of microwave power, and *C* of microwave time) in SFME, experiments were performed based on the experimental runs generated by BBD. The actual and predicted *C. camphora* chvar. *Borneol* essential oil yields obtained from 17 runs of the BBD were presented in Table 1. ANOVA was conducted to measure the significance of the factors on the extraction yield; these results are presented in Table 2. In accordance with the actual parameters, the quadratic polynomial regression equation obtained with the Design Expert 8.0 software is presented below (Eq. (5)):

$$Y(\text{mg/g}) = -277.09 + 5.50A + 3.93B + 0.34C + 0.03AB - 1.23 \times 10^{-3}AC - 4.81 \times 10^{-3}BC - 0.04A^2 - 0.05B^2 - 1.27 \times 10^{-4}C^2 \quad (5)$$

The factors significance was assessed by their *F*-value and *p*-value, where a high *F*-value coupled with a low *p*-value indicated a significant effect (Pongsumpun et al., 2020). The classification of the extremely significant ( $p < 0.001$ ) factors was:  $A^2 > BC > C^2 > B$ . The linear term of *A* and the interactive term of *AC* was highly significant ( $p$ -value  $< 0.01$ ); while other coefficients of the model (*C*, *AB*, and  $B^2$ ) were significant ( $p$ -value  $< 0.05$ ). On the basis of the ANOVA results

**Table 2** Analysis of variance (ANOVA) for the experimental results<sup>a</sup>.

Source	Sum of squares	Degree of freedom	Mean square	<i>F</i> value	<i>P</i> value		
Model <sup>b</sup>	287.55	9	31.95	27.02	0.0001***		
<i>A</i>	17.12	1	17.12	14.48	0.0067**		
<i>B</i>	38.15	1	38.15	32.26	0.0008***		
<i>C</i>	9.46	1	9.46	8.00	0.0255*		
<i>AB</i>	7.95	1	7.95	6.72	0.0358*		
<i>AC</i>	15.03	1	15.03	12.71	0.0092**		
<i>BC</i>	57.34	1	57.34	48.48	0.0002***		
$A^2$	85.10	1	85.10	71.96	$< 0.0001$ ***		
$B^2$	7.04	1	7.04	5.95	0.0447*		
$C^2$	41.75	1	41.75	35.30	0.0006***		
Residual	8.28	7	1.18				
Lack of fit	2.89	3	0.96	0.71	0.5927		
Pure error	5.39	4	1.35				
Cor total <sup>c</sup>	295.83	16					
Credibility analysis of the regression equations							
Std. Dev. <sup>d</sup>	Mean	C.V. <sup>e</sup> %	Press	$R^2$	Adjust $R^2$	Predicted $R^2$	Adequacy precision
1.09	35.83	3.04	54.65	0.9720	0.9360	0.8153	14.9660

\*  $p < 0.05$ , significant; \*\*  $p < 0.01$ , highly significant; \*\*\*  $p < 0.001$ , extremely significant.

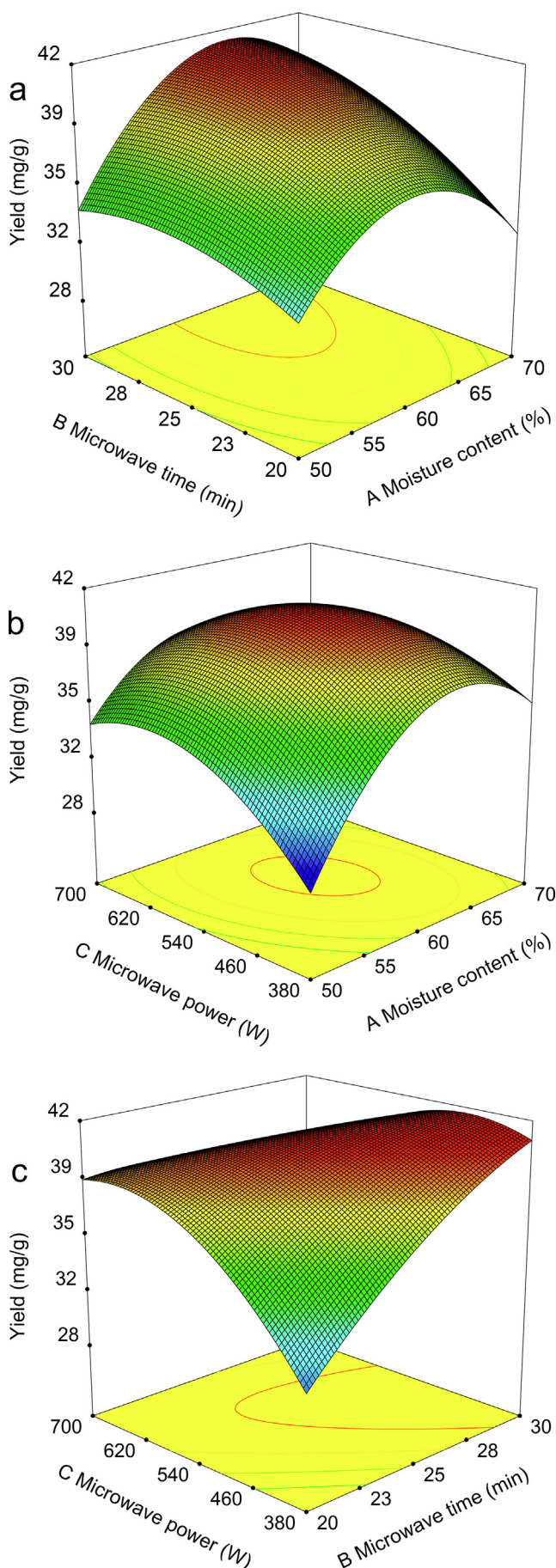
<sup>a</sup> The results were obtained with the Design Expert 8.0 software.

<sup>b</sup> *A*: Moisture content, %; *B*: Microwave time, min; *C*: Microwave power, W.

<sup>c</sup> Totals of all information corrected for the mean.

<sup>d</sup> Standard deviation.

<sup>e</sup> Coefficient of variation.



(Table 2), the developed model was accurate and satisfactory, possessing a  $R^2$  of 0.9720, an adjusted  $R^2$  of 0.9360, and a  $p$ -value of 0.5746 for the lack-of-fit (Belhachat et al., 2018).

### 3.2.2. Response surface analysis

To clarify the interactions between the three factors on *C. camphora* chvar. *Borneol* essential oil yield, three-dimensional response surface methodology (RSM) graphs were produced (Fig. 3). Fig. 3a, shows that increases in both moisture content and microwave time resulted in improvements in yield. The highest yields were obtained at a microwave time of 28 min and a moisture content of 63%. As shown in Fig. 3b, the essential oil yield fluctuated with varying of microwave power and moisture content, with the maximum yield obtained at a microwave power of 460 W and a moisture content of 45%. To enhance mass transfer velocity and improve the essential oil yield without destroying the composition of the essential oils, middling levels of microwave power (540 W) and moisture content (60%) are preferable as the polar properties of the *in situ* water could facilitate its connection to the microwave irradiation (Kapadiya et al., 2018). However, extreme microwave power could result in the hydrolysis and fractional condensation of biomass and consequently result in a decline in the yield and quality of the essential oil (Cui et al., 2019). As exhibited in Fig. 3c, microwave time and power exhibited quadratic influences on yield as these two variables were increased. Microwave power exhibited both positive and negative effects on yields. Microwave irradiation accelerated the evaporation velocity of the *in situ* water to induce the diffusion of the essential oil (Peng et al., 2021). However, a low microwave power could not completely extract all essential oil from the plant materials, while a high microwave power led to the decomposition or the combustion of the plant's essential oils.

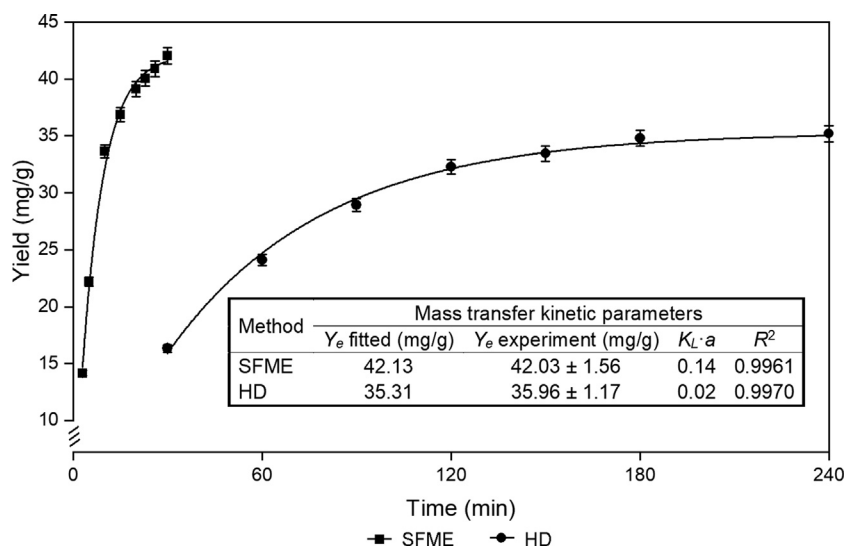
### 3.2.3. Method validation

RSM was carried out on the quadratic regression equation that described the process optimization of SFME for *C. camphora* chvar. *Borneol* essential oil extraction. In a predicted maximum extraction yield of 42.22 mg/g, the optimal conditions of SFME for essential oil isolation were determined to be: 65% moisture content, 30 min microwave time, and 451 W microwave power. The validation assays were performed in triplicate while slightly modifying the microwave power (450 W of microwave power) to validate the precision and acceptability of the SFME procedure for essential oil extraction. A mean yield of  $42.03 \pm 1.56$  mg/g was obtained, which indicated that the optimal conditions provided by BBD were reliable.

### 3.3. Extraction kinetic studies of SFME and HD

The mass transfer kinetic model was employed to describe the experimental data and the results of the *C. camphora* chvar.

Fig. 3 The three-dimensional and contour surface plots from BBD. (a) The interactive effects of moisture content and microwave time on extraction; (b) the interactive effects of moisture content and microwave power on extraction; and (c) the interactive effects of microwave time and power on extraction.



**Fig. 4** Mass transfer kinetics curves and temperature profile as a function of time of SFME and HD for extraction of *C. camphora* chvar. *Borneol* essential oil. Insert: experimental data, fitting of mass transfer kinetic model, and the parameters of  $Y_e$  and  $K_L \cdot a$ .

*Borneol* essential oil yield evolution varying from extraction time are presented in Fig. 4. The high  $R^2$  value indicates that the mass transfer kinetic model appropriately described the entirety of both the SFME and HD processes. The  $K_L \cdot a$  value of SFME, which represents the extraction rate, was clearly greater than that of HD. The main reason was that the diffusion of essential oil was influenced by the inner temperature of the extraction system. The primary benefit of SFME is that the extraction system temperature could be quickly achieved by microwave irradiation. Only 30 min of microwave time was needed to reach the extraction equilibrium point of *C. camphora* chvar. *Borneol* essential oil in the process of SFME, which is significantly shorter than the time required for HD (180 min). This was similar to the SFME process to extract the essential oil of Tunisian cumin seed (Benmoussa et al., 2018).

#### 3.4. Chemical compositions analysis of *C. camphora* chvar. *Borneol* essential oil

The GC-MS analysis of *C. camphora* chvar. *Borneol* essential oil allowed for the identification of 25 compounds; these are presented in order of elution from the column (retention times), and account for  $99.79 \pm 0.79\%$  of the total essential oil (Table 3 and Fig. 5). Volatile compounds were ranked based on their relative abundance in terms of their percentage of the total peak area. The *C. camphora* chvar. *Borneol* essential oil extracted by SFME contained 23 volatile compounds with an overall percentage of 99.79%, while the essential oil extracted by HD contained 21 compounds with a total percentage of 99.82%. *Endo*-borneol,  $\alpha$ -linalool and camphor were the three main components contained within the *C. camphora* chvar. *Borneol* essential oil extracted by SFME and HD. Specifically, the *C. camphora* chvar. *Borneol* essential oil extracted by SFME contained a higher proportion of *endo*-borneol (80.08%) compared to the essential oil extracted by HD (72.01%). In contrast, neutral cellulase

assisted-steam distillation just yielded 10 mg/g of *C. camphora* chvar. *Borneol* essential oil with a relative percentage of *endo*-borneol of around 10% (Yu et al., 2019); these values were much lower than those obtained in this study. The differences in the yield and the major constituent content of *C. camphora* chvar. *Borneol* essential oil are likely due to the geographic area, harvest season, climate, and even the characteristics of the plant soil (Brahmi et al., 2016; Yakhlef et al., 2020). Furthermore, it was observed that the *C. camphora* chvar. *Borneol* essential oil was rich in oxygenated compounds and contained small amounts of terpene hydrocarbons. Particularly, the compounds (bornyl acetate, caryophyllene oxide, humulene epoxide and globulol) detected under SFME but not in HD were all oxygenated compounds, and the *C. camphora* chvar. *Borneol* essential oil that was isolated by SFME possessed a much higher percentage of oxygenated components than when isolated by HD. This phenomenon may be due to HD having a longer extraction time than SFME, in which oxygenated compounds were thermally decomposed and hydrolyzed to generate terpenes hydrocarbons (Ferhat et al., 2006).

#### 3.5. Environmental impact

The environmental impact of the separation of *C. camphora* chvar. *Borneol* essential oil by the SFME process was evaluated and compared to HD in the aspect of electricity consumption and the amount of  $\text{CO}_2$  emission. According to Ma et al. (2012), the progress for producing 1 kW-h electricity by burning fuels will discharge 800 g of  $\text{CO}_2$  into the atmosphere. Meanwhile, the electricity consumption was calculated by multiplying the electricity power by time. Table 3 shows that the SFME process was more environmentally friendly than HD as evidenced by the lower electricity energy consumption (0.23 kW-h compared to 4 kW-h) and carbon emissions (184 g versus 3200 g), which were ten times higher in HD than in SFME. This indicates that SFME is an economic, green,



**Table 3** Chemical composition of essential oil from *C. camphora* chvar. *Borneol* leaves by GC–MS analysis.

No. <sup>a</sup>	Retention time (min)	Compounds	CAS number	Molecular formula	Relative peak area (%)		RI <sup>b</sup>	Identification
					SFME	HD		
1	6.79	$\alpha$ -Pinene	02437-95-8	C <sub>10</sub> H <sub>16</sub>	0.19	0.27	917	RI <sup>c</sup> , MS <sup>d</sup>
2	6.98	L- $\alpha$ -Pinene	07785-26-4	C <sub>10</sub> H <sub>16</sub>	ND <sup>e</sup>	4.73	934	RI, MS
3	7.36	Camphene	00079-92-5	C <sub>10</sub> H <sub>16</sub>	0.37	3.23	936	RI, MS
4	7.97	$\alpha$ -Phellandrene	00099-83-2	C <sub>10</sub> H <sub>16</sub>	0.42	0.59	966	RI, MS
5	8.05	4-thujene	03387-41-5	C <sub>10</sub> H <sub>16</sub>	0.29	1.68	973	RI, MS
6	8.36	$\alpha$ -Myrcene	01686-30-2	C <sub>10</sub> H <sub>16</sub>	0.15	1.38	986	RI, MS
7	9.12	p-Cymene	00099-87-6	C <sub>10</sub> H <sub>14</sub>	ND	0.54	992	RI, MS
8	9.22	Limonene	00138-86-3	C <sub>10</sub> H <sub>16</sub>	0.39	3.62	995	RI, MS
9	9.28	Eucalyptol	00470-82-6	C <sub>10</sub> H <sub>18</sub> O	0.78	1.29	1028	RI, MS
10	10.45	$\alpha$ -Terpinene	00099-86-5	C <sub>10</sub> H <sub>16</sub>	0.48	0.32	1057	RI, MS
11	10.64	$\alpha$ -Linalool	00078-70-6	C <sub>10</sub> H <sub>18</sub> O	5.40	3.93	1062	RI, MS
12	11.55	Camphor	00076-22-2	C <sub>10</sub> H <sub>16</sub> O	3.21	3.58	1110	RI, MS
13	12.08	<i>endo</i> -Borneol	00507-70-0	C <sub>10</sub> H <sub>18</sub> O	80.08	72.01	1134	RI, MS
14	12.25	Terpinen-4-ol	00562-74-3	C <sub>10</sub> H <sub>18</sub> O	1.37	0.55	1140	RI, MS
15	12.53	$\alpha$ -Terpineol	00098-55-5	C <sub>10</sub> H <sub>18</sub> O	2.57	0.75	1170	RI, MS
16	14.08	Geraniol	00106-24-1	C <sub>10</sub> H <sub>18</sub> O	0.53	0.23	1224	RI, MS
17	15.18	Bornyl acetate	00076-49-3	C <sub>12</sub> H <sub>20</sub> O <sub>2</sub>	0.21	ND	1261	RI, MS
18	19.22	Caryophyllene	00087-44-5	C <sub>15</sub> H <sub>24</sub>	0.38	0.25	1390	RI, MS
19	19.94	Humulene	06753-98-6	C <sub>15</sub> H <sub>24</sub>	0.52	0.34	1420	RI, MS
20	20.48	Germacrene D	23986-74-5	C <sub>15</sub> H <sub>24</sub>	0.23	0.19	1442	RI, MS
21	20.76	Elixene	15423-57-1	C <sub>15</sub> H <sub>24</sub>	0.38	0.10	1535	RI, MS
22	22.24	Caryophyllene oxide	01139-30-6	C <sub>15</sub> H <sub>24</sub> O	0.60	ND	1548	RI, MS
23	22.41	Guaiol	00489-86-1	C <sub>15</sub> H <sub>26</sub> O	0.53	0.24	1593	RI, MS
24	22.63	Humulene epoxide	19888-34-7	C <sub>15</sub> H <sub>24</sub> O	0.34	ND	1577	RI, MS
25	23.27	Globulol	51371-47-2	C <sub>15</sub> H <sub>26</sub> O	0.37	ND	1598	RI, MS
Total (%)					99.79	99.82		
Oxygenated compounds (%)					95.99	82.9		
Terpene hydrocarbons (%)					3.8	16.38		
Others (%)					0	0.54		
Extraction time (min)					30	240		
Electric consumption (kW·h)					0.23	4		
CO <sub>2</sub> produced (g)					184	3200		

<sup>a</sup> Compounds listed in order of elution from HP-5MS capillary column.

<sup>b</sup> Retention indices relative to C11–C21 *n*-alkanes on HP-5MS capillary column.

<sup>c</sup> Tentative identification by comparison with RI on HP-5MS capillary column with literature data.

<sup>d</sup> Confirmed by comparison with mass data obtained from NIST05 mass spectra library.

<sup>e</sup> Not detected.

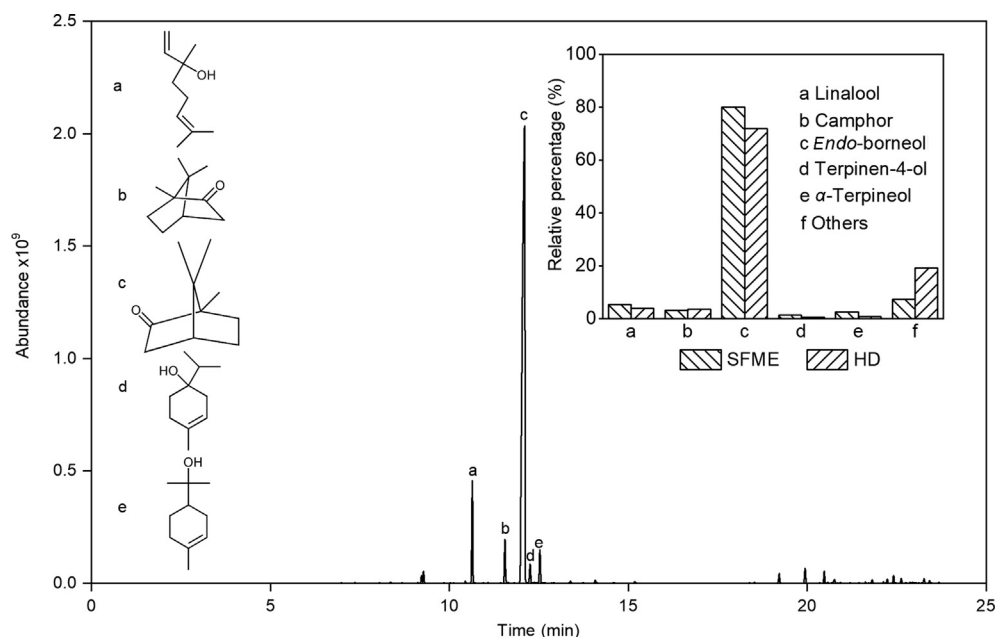
and efficient technique for the extraction of *C. camphora* chvar. *Borneol* essential oil.

### 3.6. *In vitro* antifungal activity of *C. camphora* chvar. *Borneol* essential oil

The antifungal activity of *C. camphora* chvar. *Borneol* essential oil on the mycelial growth and spore germination rates of five pathogenic fungi was investigated—the results are given in Table 4 and Fig. 6. The MIC values of *C. camphora* chvar. *Borneol* essential oil for the complete inhibition of mycelial growth and spore germination of the pathogens were found to be 1–2 mg/mL and 0.75–1.25 mg/mL, respectively. In particular, *C. camphora* chvar. *Borneol* essential oil showed excellent inhibitory activity against the mycelial growths and spore ger-

minations of the five pathogenic fungi as evidenced by their low EC<sub>50</sub> values (< 1 mg/mL). The inhibition patterns of the five tested pathogenic fungi after being treated with essential oil at EC<sub>50</sub> values (Fig. 6A2–E2) also proved that *C. camphora* chvar. *Borneol* essential oil exhibited remarkable antifungal activity against *F. culmorum*, *F. solani*, *F. trichothecioides*, *F. sporotrioides* and *F. avenaceum* compared to the negative (Fig. 6A1–E1) and the positive control sets (Fig. 6A3–E3). Table 4, shows that *C. camphora* chvar. *Borneol* essential oil exhibited relatively higher inhibitory activity against spore germinations than mycelial growths for the five tested pathogenic fungi.

The antifungal activity of *C. camphora* chvar. *Borneol* essential oil against the five pathogenic fungi is likely a consequence of its high *endo*-borneol content, an oxygenated



**Fig. 5** Total ion chromatograms and the major constituents of *C. camphora* chvar. *Borneol* essential oil extracted by SFME and HD. Insert: chemical structural formulas of the main compounds and their corresponding relative percentages in *C. camphora* chvar. *Borneol* essential oil extracted by SFME and HD.

**Table 4** Antifungal activity of *C. camphora* chvar. *Borneol* leaf essential oil against five fungi.

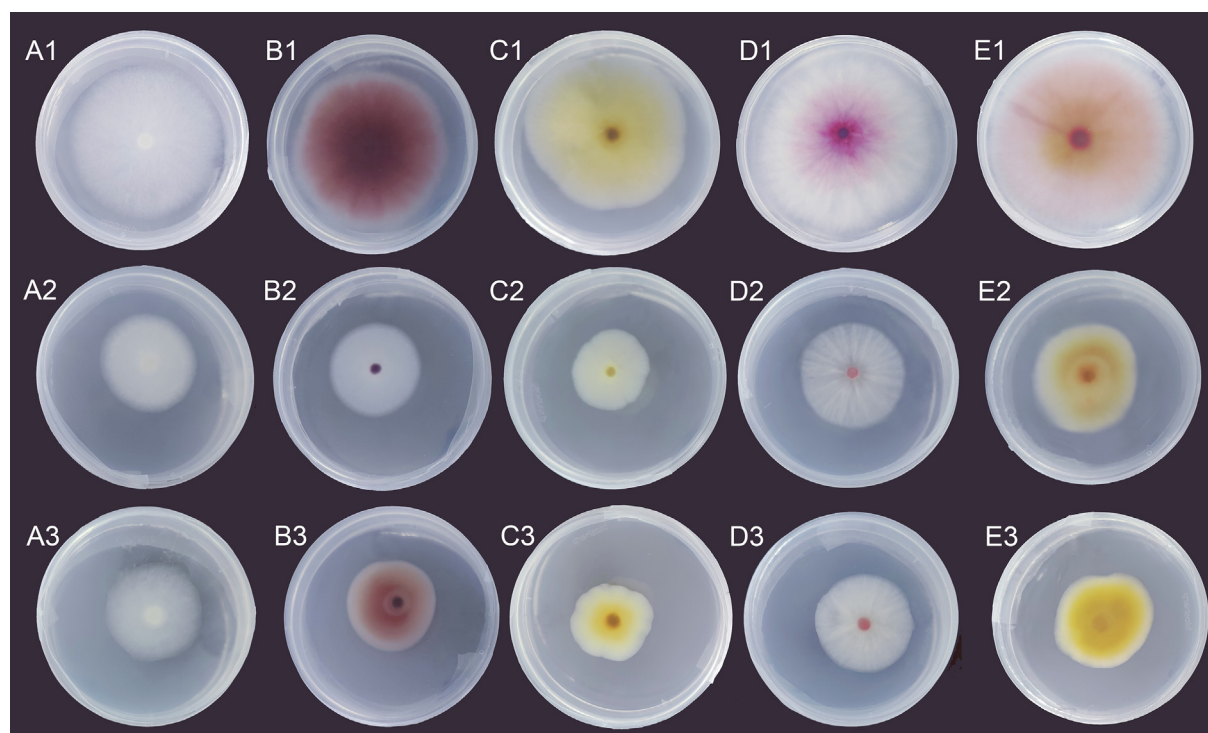
Fungal strain	Mycelial growth		Spore germination	
	MIC <sup>a</sup> (mg/mL)	EC <sub>50</sub> <sup>b</sup> (mg/mL)	MIC (mg/mL)	EC <sub>50</sub> (mg/mL)
<i>F. culmorum</i>	1.00	0.44	0.75	0.28
<i>F. solani</i>	2.00	0.87	1.25	0.38
<i>F. trichothecioides</i>	1.25	0.52	1.00	0.28
<i>F. sporotrioides</i>	1.25	0.66	1.00	0.35
<i>F. avenaceum</i>	1.00	0.56	0.75	0.32

<sup>a</sup> Minimum inhibitory concentration resulting in complete inhibition of mycelial growth.

<sup>b</sup> Effective concentration resulting in 50% inhibition of mycelial growth or spore germination as determined by linear regression.

monoterpene (Cosentino et al., 1999; Castilho et al., 2012; Kasrati et al., 2014). Marei and Abdelgalei (2018) reported that six monoterpenes resulted in the inhibition of the mycelial growth of eight phytopathogenic fungal species and noted that antifungal activity largely depends on the specific compounds and the species of the fungus. Meincken et al. (2005) pointed out that *Mentha piperita* oil demonstrated a high inhibitory effect against microorganisms, because oxygenated monoterpenes constituents can destroy their cell membrane, cell permeability and interrupting cell proliferation. Research by Gao et al. (2021) showed that *endo*-borneol has remarkable antifungal activity against plant and animal pathogenic fungi. Furthermore, Chang and Li (2000) reported that *endo*-borneol exhibited good inhibitory

activity against *Aspergillus niger* by disrupting its cell structure and causing the fungal dissolution. Generally speaking, the dominant components of the plant essential oils are good reflections of their biological activities, and the intensity of their effects is primarily dependent on their concentration as well as whether they were assayed alone or contained in essential oils (Hajlaoui et al., 2009). In contrast, the proportions of the other compounds are likely to modulate the activity of the main constituents. Previous studies have shown that other oxygenated monoterpenes, such as  $\alpha$ -linalool, camphor,  $\alpha$ -terpineol, and caryophyllene oxide, can also facilitate the antimicrobial activity of *C. camphora* chvar. *Borneol* essential oil (Sonboli et al., 2006; Kasrati et al., 2014; Stević et al., 2014; Yakhlef et al., 2020).



**Fig. 6** The inhibition patterns for the mycelial growths of the five tested pathogenic fungi and after treated with *C. camphora* chvar. *Borneol* essential oil (A2-E2) and nystatin (C3-E3).

#### 4. Conclusion

In this study, SFME was optimized for the separation of *C. camphora* chvar. *Borneol* essential oil using single-factor experiments and BBD-based RSM. The optimal SFME conditions yielded  $42.03 \pm 1.56$  mg/g at 65% moisture content, 30 min microwave time, and 450 W microwave power. Compared to HD, SFME is a faster, more efficient and greener method of *C. camphora* chvar. *Borneol* essential oil extraction with a higher extraction efficiency and less harmful impacts on the environment. GC-MS results indicated that the essential oil extracted by SFME contained a higher percentage of oxygenated compounds than HD. The results of in vitro antifungal assays suggested that *C. camphora* chvar. *Borneol* essential oil has the potential to be used as preparatory material for a safe, eco-benign fungistatic agent for the management of fungal plant diseases caused by *Fusarium* spp.

#### 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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