



## Research article

Optimization of extraction conditions of phenolic compounds from *Cymbopogon citratus* and evaluation of phenolics and aroma profiles of extractWiyeh Claudette Bakisu Muala<sup>a</sup>, Zangué Steve Carly Desobgo<sup>b,\*</sup>, Nso Emmanuel Jong<sup>a</sup><sup>a</sup> Department of Process Engineering, National School of Agro-Industrial Sciences (ENSAI), University of Ngaoundere, P.O. Box 455, Adamaoua, Cameroon<sup>b</sup> Department of Food Processing and Quality Control of University Institute of Technology (UIT) of The University of Ngaoundere, P.O. Box 455, Cameroon

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## ABSTRACT

Decoction extraction procedure was implemented to regain phenolic compounds from *C. citratus* leaves. The extraction variables, solid/liquid ratio (2–5 g/100 mL), temperature (85–95 °C), and time (5–10 min) were assessed by central composite design for process optimization. Antioxidant activity (DPPH) and total polyphenol content (TPC) were monitored as responses. The TPC and DPPH were 71.98 ± 0.33 mg GAE/100 mL extract and 80.63 ± 0.49 mg TE/100mL extract respectively under optimal conditions (solid/liquid ratio = 5, temperature = 93.8 °C and time 11.3 min). The evaluation of phenolic compounds and volatile compounds of *C. citratus* extract at conditions for optimum extraction revealed that caffeic (20.81 ± 0.003 mg/100mL) and syringic acids (18.63 ± 7.390 mg/100mL) were the main phenolic compounds while citral and geraniol were the primary volatile compounds. The results achieved herein suits the potential use of *C. citratus* extract as natural source of antioxidant and aroma compounds that can be employed in different industrial sectors.

**Practical application:** Lemongrass obtained at the optimal extraction conditions is a good source of antioxidants and the extract has organic acids and a lemon scent due to the presence of citral. This extract can thereby be incorporated in the production of beverages which can help aromatize the beverage and also contribute in the addition of the antioxidant property of the beverage. It is also rich in organic acids, the main being propionic acid, which is known to have antimicrobial activity primarily against bacteria and mold. The lemongrass extract can therefore, extend the shelf life of the beverage they are incorporated in and also the citral present in lemongrass has antimicrobial properties.

## 1. Introduction

*Cymbopogon citratus* (lemongrass) is a tall perennial grass of the family Poaceae commonly cultivated in humid subtropical and tropical regions of the world (Olorunnisola et al., 2014). Lemongrass is rich in minerals, vitamins, and macronutrients (carbohydrate, protein, and small amounts of fat). These leaves also are good source of various bioactive compounds including alkaloids, terpenoids, flavanoids, phenols, saponins and tannins that confer *C. citratus* leaves pharmacological properties such as anti-cancer, antihypertensive, anti-mutagenicity, anti-diabetic, anti-oxidant, anxiolytic, anti-nociceptive and anti-fungi (Balakrishnan et al., 2014; Olorunnisola et al., 2014). In this new era, the search of food ingredients rich in bioactive components is increasing due to the outbreak of COVID-19 caused by the SARS-CoV-2 virus. Foods rich in bioactive compounds are advantageous because they boost the immune

system and natural polyphenols have exhibited properties as inhibitors of COVID-19 main protease (Galanakis, 2020).

Lemongrass has been used either as fresh leaves, dried powdered concentrated extract, or essential oil depending on the application. Several conventional and non-conventional methods are used in the extraction of bioactive components from plants (Zinoviadou et al., 2015). Non-conventional methods like ultrasound, microwave-assisted extraction, high pressure combined with thermal processing, supercritical carbon dioxide (SC-CO<sub>2</sub>), pulsed electric fields assisted processing (Deng et al., 2014; Roselló-Soto et al., 2015a,b; Siewe et al., 2019, 2021) have been employed in the extraction of polyphenols from different plant sources. These emerging separation techniques are advantageous because they use limited extraction time and solvent, polyphenol yield is high (Jovanovic et al., 2017) and they also have minimal impact on sensorial and nutritional properties (Zinoviadou et al., 2015; Siewe et al., 2019). However, the extraction of these bioactive components depends

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on the matrix and solvent used and the emerging techniques are used in combination with other techniques for better yield (Jovanovic et al., 2017; Siewe et al., 2019). Up to date, the lemongrass extract from infusion method remains the widely employed methods owing to its lower cost and simplicity. Some process parameters, like, process time, water/substrate ratio, and temperature has been declared as the principal factors for efficient extraction of bioactive components from plants (Oboh et al., 2010; Uma et al., 2010; Roseiro et al., 2013; Thangam et al., 2014). The application of unsuitable conditions during the extraction procedure could lead to the degradation of target compounds and reduce extraction efficiency. Therefore, optimizing the extraction process can aid in choosing suitable process conditions for improving the extraction yield of bioactive compounds. Response surface methodology (RSM), an assembly of statistical and mathematical methods, is widely used in all sectors to optimize operating conditions of processes. With RSM, there is a limited number of experimental runs, which facilitates its application to the development, improvement, and optimization of operating conditions for a process or product (Dean et al., 2017). Thangam et al. (2014) used Box-Behnken design, a three-level factorial design, to survey the extraction of hot water-soluble polysaccharides (HWSPs) from *Cymbopogon citratus* via decoction and at the end did not evaluate the various polyphenols in the extract. The Box-Behnken design is efficient and economical but lacks accuracy (Lundstedt et al., 1998). However, central composite design is a full factorial or fractional factorial design with axial points in which experimental points are at a distance  $\alpha$  from its center, the design is studied at five levels and has additional center points (Bezerra et al., 2008). Enough replication of center points, allow for a test for model lack of fit (Dean et al., 2017). Hence, the objective of this present study was to optimize the polyphenol and antioxidant extraction from lemongrass by decoction method using central composite design. In addition, the aroma profile and organic acid content of the lemongrass extract at optimum conditions were determined.

## 2. Materials and methods

### 2.1. Materials

#### 2.1.1. Chemicals

HPLC grade phenolic standards, were obtained from Sigma-Aldrich. HPLC grade acetonitrile, acetic acid, methanol were from Merck, HCl (hydrochloric acid) and Folin-ciocalteu reagent were purchased from Merck. HPLC grade organic acid standards were from Sigma-Aldrich. Sodium carbonate, DPPH, Trolox (6-hydroxy-2, 5, 7, 8-tetramethylchroman-2-carboxylic acid) were supplied from Sigma-Aldrich (St. Louis, MO, USA). All chemicals utilised were of analytical grade.

#### 2.1.2. Biological materials

*C. citratus* leaves were collected from a plantation in Bini-dang, Ngaoundere, Adamawa region, Cameroon in the month of October, 2018. They were washed with running tap water, cut into 2 cm cuts and oven-dried at 60 °C for 3h. Dried leaves were ground, sieved with a 1 mm sieve, packaged and stored at -40 °C in order to preserve its quality until use.

### 2.2. Methods

#### 2.2.1. Determination of proximate and bioactive composition of lemongrass leaves

Moisture, ash, crude fiber, protein, and fat contents were estimated by procedures provided by the Association of Official Analytical Chemists (AOAC, 2005). The phytochemical content of *C. citratus* leaves was extracted in 80% methanol, and TPC, FRAP, and DPPH analyzed in the extract.

#### 2.2.2. Decoction method

The lemongrass powder was extracted with distilled water at lemongrass to water ratio (2–5 g/100mL), temperature between (85–95 °C) and time (5–10 min). In brief, the water bath (Julabo TW8, Germany)

was set at the temperature, as imposed by the design of experiments. The conical flask containing distilled water was deposited in the water bath until it reached the set temperature. The lemongrass powder was then introduced in the conical flask and agitated at 150 rpm. After extraction, samples were cooled immediately in ice water to reach room temperature (28–30 °C). The slurry was further filtrated using Whatman paper N° 4. The filtrate was rinsed three times with distilled water, and all three extracts were mixed. The volume of the total extract was adjusted to 100 mL with distilled water and referred to as lemongrass extract.

#### 2.2.3. Experimental design, modelling, validation of model, and optimization

The orthogonal quadratic central composite design (CCD) was utilized to scrutinize the decoction process. Table 2 presents the factors and their coded levels utilized for the CCD. The independent factors studied were lemongrass powder to water ratio ( $x_1$ ), decoction temperature ( $x_2$ ), and extraction time ( $x_3$ ). The range of factors were;  $x_1$ , 2–5g/100 mL;  $x_2$ , 85 to 95 °C, and  $x_3$ , 5–10 min. The CCD consisted of 20 trials, and each trial was done in triplicate, and the average responses (TPC and DPPH) were reported. The mathematical model employed was a second-degree polynomial model with linear, quadratic, and interaction terms (equation 1).

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j \quad (1)$$

Where Y is the response,  $\beta_0$  is the constant term,  $\beta_i$  are the linear coefficient terms,  $\beta_{ii}$  are the quadratic coefficient terms,  $\beta_{ij}$  are the interaction coefficient terms and  $x_i$  and  $x_j$  the factors.

From the coded variables, Eq. (2) was used to transform them into real values to realize experiments in the laboratory. The equation is as follows:

$$X_i = X_{0i} + x_i \Delta X_i \quad (2)$$

The value of  $\alpha$  and the number of experiment N (Eqs. (3) and (4) respectively) were calculated in order to respect the orthogonality criterion and using the formulas:

$$\alpha = \left( \frac{2^k (\sqrt{2^k + 2k + n_0} + \sqrt{2^k})^2}{4} \right)^{\frac{1}{2}} \quad (3)$$

$$N = k^2 + 2k + n_0 \quad (4)$$

Where: k is the number of variables,  $n_0$  is the number of trials in the centre.

The CCD matrix was obtained by Minitab 19.2 (2019 Minitab, LLC, USA). R-square ( $R^2$ ), R-square adjusted ( $adj-R^2$ ), absolute average deviation (AAD) (equation 5), the bias factor ( $B_f$ ) (equation 6), and the accuracy factor ( $A_f$ ) (equation 7) were utilized to validate the models.

$$AAD = \frac{\left[ \sum_{i=1}^N \left( \frac{|Y_{i,exp} - Y_{i,cal}|}{Y_{i,exp}} \right) \right]}{N} \quad (5)$$

$$B_f = 10^{\frac{1}{N} \sum_{i=1}^N \log \left( \frac{Y_{i,cal}}{Y_{i,exp}} \right)} \quad (6)$$

$$A_f = 10^{\frac{1}{N} \sum_{i=1}^N \left| \log \left( \frac{Y_{i,cal}}{Y_{i,exp}} \right) \right|} \quad (7)$$

Where  $Y_{i,exp}$  is the responses,  $Y_{i,cal}$  the calculated responses, and N is the number of experiments used in the calculation.

The statistical analyses of the experimental design data and plotting of surface plot were realized using the softwares, Minitab 19.2 and OriginPro 2019b (9.6.5.169, OriginLab Corporation). ANOVA test was utilized to obtain the statistical significance of the regression coefficient on the level of significance declared at  $p \leq 0.05$ .

Lastly, optimization was executed in one hand using Minitab 19.2 (2019 Minitab, LLC, USA). The conditions fixed were to maximize both total polyphenol content and DPPH Radical Scavenging Activity. A composite optimal was considered for the two responses and the responses at the composite optimal verified.

### 2.3. Physicochemical analyses

#### 2.3.1. Determination of total polyphenol content

The TPC of the lemongrass extract was analyzed according to the method mentioned by Marigo (1973) with modifications. 20  $\mu\text{L}$  of the extract was mixed with 680  $\mu\text{L}$  of distilled water and 100  $\mu\text{L}$  of 1N folin-ciocalteu reagent (FCR). The mixture was kept for 5 min, and 200  $\mu\text{L}$  of 20%  $\text{Na}_2\text{CO}_3$  was introduced in the test tube. The whole mixture was agitated and incubated at 40  $^\circ\text{C}$  for 20 min in the dark. The blue complex absorbance was read at 725 nm using TECAN microplate reader (Infinite M200 PRO, Tecan Austria). Standard gallic acid (0.001–0.008 g/L) was used to construct the calibration curve and the results were expressed as mg of gallic acid equivalent (GAE)/100 mL extract. All samples were analyzed in triplicate and an average taken.

#### 2.3.2. Estimation of DPPH radical scavenging activity

The DPPH radical scavenging activity was performed as mentioned by Shimada et al. (1992) with some modifications. Briefly, 30  $\mu\text{L}$  of lemongrass extract was reacted with 150  $\mu\text{L}$  of 0.1 mM DPPH in 96-well microplate. The mixture was homogenized and incubated at 37  $^\circ\text{C}$  for 30 min. The absorbance was then measured at 517 nm and a standard curve was constructed using standard Trolox in the range of 0–50  $\mu\text{g}/\text{mL}$ . The activity was expressed as mg Trolox equivalents (mg TE)/100 mL of extract.

#### 2.3.3. Determination of ferric reducing antioxidant power (FRAP)

The FRAP assay was assessed with respect to the method of Benzie and Strain (1996) with slight modifications. FRAP solution was prepared fresh daily by mixing 0.3 M acetate buffer (pH 3.6), 0.01 M TPTZ in 40 mM HCl, and  $\text{FeCl}_3$  (20 mM) in the ratio 10:1:1 respectively. Then, 30  $\mu\text{L}$  of the extract was mixed with 200  $\mu\text{L}$  of FRAP solution (freshly prepared). The mixture was homogenized properly and incubated for 30 min at 37  $^\circ\text{C}$  in dark conditions. The ferrous tripyridyltriazine complex formed was measured at 595 nm against a blank prepared in the same manner using distilled water instead of the sample. A standard curve was prepared using Trolox in the range of 0–50  $\mu\text{g}/\text{mL}$ , and the activity was expressed as mg TE/100 mL extract.

#### 2.3.4. Analysis of phenolic compounds

HPLC analyses for polyphenol compounds of lemongrass extract was performed on LC-10AS (Shimadzu, Kyoto, Japan) system equipped with Shimadzu LC-10AT pumps, and CBM-20A communication bus module. Chromatography was carried out in a gradient system using 250  $\times$  4.60 mm, 5 $\mu\text{m}$  C18 column (Gemini, Phenomenex, California, USA). A flow rate of 1 mL/min and the injection volume of 10 $\mu\text{L}$  was employed. The mobile phases consisted of A (99.9% acetonitrile and 0.1% acetic acid) and B (0.1% acetic acid in MQ water). The gradient elution was 0–15min (8% A and 92% B), 30min (22% A and 78% B), 45min (78% A and 22% B), 55min (8% A and 92%B), and 60min (8% A and 92% B). A UV-Visible DAD detector was used, and the wavelengths detected at 280 and 320nm. Pure standards were injected at different concentrations and the calibration curves obtained. Polyphenols were identified by comparing their retention times with those of pure standards and the polyphenol concentration of each compound calculated with respect to the calibration curves of the standards (Sonmezdag et al., 2017).

#### 2.3.5. Analysis of organic acids

The organic acid profile was obtained following the method of Sharma and Devi (2018) with slight modifications. Organic acids were determined using LC-8A HPLC (Shimadzu, Kyoto, Japan) equipped with

SPD-M10A VP diode array detector. A C18 (250  $\times$  4.60 mm, 5 $\mu\text{m}$ ) column (Gemini, Phenomenex, California, USA) was used, and the mobile phase was 8 mM  $\text{H}_2\text{SO}_4$ . Ten microliters of the extract were injected, and the separation of organic acids was carried out at a flow rate of 1 mL  $\text{min}^{-1}$  for 30 min. Standards organic acids were run for identification and quantification of individual organic acids.

#### 2.3.6. Volatile analysis

Extraction and analysis of volatile compounds in lemongrass extract was done according to the method described by Siewe et al. (2020) with some modifications. The solid-phase microextraction (SPME) method was used in extraction of volatile compounds from lemongrass extract. 5 mL of lemongrass extract in well-sealed glass vials (20 mL) were heated at 50  $^\circ\text{C}$  for 20 min in a water bath. The aroma compounds trapped in the headspace of the vials were adsorbed for 30 min with the 2 cm, 50/35  $\mu\text{m}$  Carboxen/polydimethylsiloxane/divinylbenzene (CAR/PDMS/DVB) fiber (Supelco Inc., Bellefonte, USA). The adsorbed fiber was directly introduced into a 7890B Agilent GC injector port (Agilent Technologies, Santa Clara, California, USA) at 250  $^\circ\text{C}$  for 3 min to desorb the volatile compounds. The RT-WAX capillary column (60  $\times$  0.25 mm, 0.25  $\mu\text{m}$ ; J & W Scientific, Folsom, CA) assisted in separating the volatile compounds. The program of the GC column was set at 40  $^\circ\text{C}$  for 3 min, then the temperature was increased with an increment of 5  $^\circ\text{C}/\text{min}$  to 235  $^\circ\text{C}$ , and maintained for 10 min. The carrier gas, helium was used at a flow rate of 1.8 mL/min. The temperature and electron voltage of the mass spectrometric detector was operated at 230  $^\circ\text{C}$  and 70 eV respectively with the transfer line temperature of 250  $^\circ\text{C}$ . The chromatogram was recorded in the range of 40–450 amu of the total ion current.

The mass spectra of the volatile compounds obtained were identified by comparing with the mass spectra database of the US National Institute of Standards and Technology (NIST). Calculations of the relative percentage (% area) were based on the ratio between the peak area of each compound and the sum of areas of all compounds (Pino and Barzola-Miranda, 2020).

## 3. Results and discussion

### 3.1. Proximate composition, polyphenol content and DPPH activity of *C. citratus* extract

The proximate and phytochemical composition of lemongrass is presented on Table 1. Lemongrass leaves contain nutrients (proteins, carbohydrate and fibers) which explains its use. The moisture content of dried lemongrass, 10.09  $\pm$  0.06%, lower than 11.35% determined by Uraku et al. (2016) shows it is desirable for longer periods of storage and less attack of microorganisms.

The value of ash content 8.06  $\pm$  0.05% indicates the presence of minerals in lemongrass leaves Asaolu et al. (2009). The protein content

**Table 1.** Proximate composition of *C. citratus* L. extract.

Proximate	
Component	Content (g/100 g DW sample)
Moisture content	10.09 $\pm$ 0.06
Ash	8.06 $\pm$ 0.05
Fat	4.45 $\pm$ 0.1
Proteins	8.40 $\pm$ 0.05
Crude fibre	30.32 $\pm$ 0.65
Total carbohydrate	44.16 $\pm$ 0.77
Polyphenol content (mg/g DW sample)	
TPC	16.56 $\pm$ 0.05
Antioxidant activities (mg TE/100 g DW sample)	
DPPH	1261.30 $\pm$ 3.28
FRAP	1920.92 $\pm$ 2.59

**Table 2.** Central composite design coded values, real values and experimental responses.

Run	Coded variables			Real variables			Experimental responses	
	Solid/liquid ratio (w/v)	Decoction temperature (°C)	Extraction time (min)	Solid/liquid ratio (w/v)	Decoction temperature (°C)	Extraction time (min)	DPPH (mg TE/100 mL extract)	TPC (mg GAE/100 mL extract)
19	0	0	0	3.5	90	7.5	58.73 ± 0.18	52.62 ± 0.24
10	1.52	0	0	5.78	90	7.5	85.26 ± 0.15	65.92 ± 0.03
2	1	-1	-1	5	85	5	77.13 ± 0.49	59.39 ± 0.16
8	1	1	1	5	95	10	77.01 ± 0.01	68.09 ± 0.07
1	-1	-1	-1	2	85	5	38.09 ± 0.05	32.29 ± 0.62
9	-1.52	0	0	1.22	90	7.5	19.10 ± 0.06	23.32 ± 0.41
13	0	0	-1.52	3.5	90	3.7	63.69 ± 0.17	43.78 ± 0.30
11	0	-1.52	0	3.5	82.4	7.5	51.09 ± 0.51	44.15 ± 0.11
14	0	0	1.52	3.5	90	11.3	55.93 ± 0.01	55.04 ± 0.05
18	0	0	0	3.5	90	7.5	59.08 ± 0.04	56.17 ± 0.10
3	-1	1	-1	2	95	5	36.92 ± 0.03	29.57 ± 0.11
17	0	0	0	3.5	90	7.5	61.01 ± 0.01	55.08 ± 0.55
12	0	1.52	0	3.5	97.6	7.5	54.04 ± 0.53	54.33 ± 0.51
4	1	1	-1	5	95	5	79.95 ± 0.04	60.05 ± 0.65
6	1	-1	1	5	85	10	73.40 ± 0.38	64.96 ± 0.23
5	-1	-1	1	2	85	10	25.50 ± 0.33	28.80 ± 0.49
7	-1	1	1	2	95	10	34.43 ± 0.17	38.46 ± 1.13
15	0	0	0	3.5	90	7.5	55.93 ± 0.24	55.92 ± 0.85
16	0	0	0	3.5	90	7.5	58.49 ± 0.10	56.41 ± 0.57
20	0	0	0	3.5	90	7.5	58.12 ± 0.53	53.70 ± 0.85

(8.40 ± 0.05%) was higher compared to 4.56% reported by Asaolu et al. (2009) and in agreement with 8.51% reported by Ojo (2017). The crude fiber content (30.32 ± 0.65%) indicates lemongrass leaves are an adequate source of crude fiber compared to other conventional plant leaves (Asaolu et al., 2009; Nambiar and Matela, 2012). The carbohydrate content of 44.16 g/100 g sample indicates it is a good energy source. The variations in the composition of lemongrass leaves with literature could be due to differences in maturity stage and geographical location of the plant (Ranjah et al., 2019).

Polyphenol compounds are one of the vital group of compounds acting as main antioxidants which contributes to the medicinal value of various plants (Kouassi et al., 2017). The total polyphenol content of the methanol extract of *C. citratus* was found to be 16.56 ± 0.05 mg GAE/g DW. The antioxidant activities, DPPH and FRAP activities were found to be 1261.30 ± 3.28 and 1920.92 ± 2.59 mg TE/100 g DW, respectively. The total

polyphenol content and antioxidant activities of *C. citratus* leaves were different from those stated in literature (Kouassi et al., 2017; Nambiar and Matela, 2012; Sah et al., 2012). The values of 118.14 ± 1.05 mg GAE/g and 178.069 ± 1.57 mM TE/mL for TPC and antioxidant was observed by Kouassi et al. (2017); 1324.9 ± 31.06 mg %, 15.96 ± 0.53 and 23.40 ± 1.19 µmol TE/g DW for TPC, DPPH and FRAP respectively was realized by Nambiar and Matela (2012). The variations in the polyphenol content and antioxidant activities of *C. citratus* leaves compared to that of literature could be as a result of the maturity stage, geographical location and extraction method (Adeyemo et al., 2018; Ranjah et al., 2019).

### 3.2. Mathematical modelling

The results of the 20 experimental runs with their responses are presented in Table 2, while the ANOVA results are put forward in Table 3.

**Table 3.** Analysis of variance for the regression models of TPC and DPPH.

Source	TPC (mg GAE/100 mL extract)			DPPH (mg TE/100 mL extract)		
	Sum of squares	F-value	P-value	Sum of squares	F-value	P-value
Model	3239.34	62.97	<0.0001	6169.45	213.63	<0.0001
X <sub>1</sub>	2804.15	1210.94	<0.0001	5909.96	2203.64	<0.0001
X <sub>2</sub>	54.35	23.47	0.0047	27.65	10.31	0.0237
X <sub>3</sub>	103.35	44.63	0.0011	89.13	33.24	0.0022
X <sub>1</sub> X <sub>2</sub>	1.25	0.54	0.4960	0.22	0.081	0.7878
X <sub>1</sub> X <sub>3</sub>	8.44	3.65	0.1145	8.85	3.298	0.1290
X <sub>2</sub> X <sub>3</sub>	27.57	11.91	0.0182	14.77	5.507	0.0658
X <sub>1</sub> <sup>2</sup>	163.71	70.698	0.0004	58.40	21.774	0.0055
X <sub>2</sub> <sup>2</sup>	39.07	16.87	0.0093	50.38	18.786	0.0075
X <sub>3</sub> <sup>2</sup>	36.14	15.61	0.0108	10.04	3.74	0.1107
Residual	57.16			32.09		
Lack of fit	45.58	3.94	0.0794	18.68	1.39	0.3625
Pure error	11.58			13.41		
Cor total	3296.50			6201.54		

X<sub>1</sub>, X<sub>2</sub>, and X<sub>3</sub> represents the linear effects (solid/liquid ratio, temperature and time respectively); X<sub>12</sub>, X<sub>13</sub> and X<sub>23</sub> are the different interactions and X<sub>1</sub><sup>2</sup>, X<sub>2</sub><sup>2</sup>, and X<sub>3</sub><sup>2</sup> the quadratic effects.

**Table 4.** Model validation parameters.

Model	R <sup>2</sup>	R <sub>adj</sub>	AAD	B <sub>f</sub>	A <sub>f</sub>
TPC	0.9827	0.9671	0.0011	1.0004	1.0034
DPPH	0.9948	0.9902	0.0016	1.0012	1.0218

The significance of the model terms and model equations were validated with respect to the  $p$ -value ( $p \leq 0.05$ ). The models of both responses were highly significant ( $p < 0.0001$ ). The linear coefficients ( $x_1$ ,  $x_2$ ,  $x_3$ ), the interaction ( $x_2x_3$ ) and the quadratic terms ( $x_1^2$ ,  $x_2^2$ ,  $x_3^2$ ) were the significant model terms ( $p \leq 0.05$ ) for TPC while for DPPH scavenging activity, the linear terms ( $x_1$ ,  $x_2$ ,  $x_3$ ), and the quadratic terms ( $x_1^2$ ,  $x_2^2$ ) were significant ( $p \leq 0.05$ ). The model validation terms, lack of fit, coefficient of determination ( $R^2$ ), adjusted  $R^2$  (adj- $R^2$ ), AAD,  $B_f$  and  $A_f$  are presented in Table 4. The lacks of fit for both models were not significant ( $p > 0.05$ ), revealed that no considerable improvement was achieved by the inclusion of the statistically parametric values.

The coefficient of determination  $R^2$  were 0.9829 and 0.9948 for TPC and DPPH, respectively; indicating that both mathematical models can explain 98.29% and 99.48% (respectively for TPC and DPPH) experimental observations as a function of independent variables. Besides, adj- $R^2$  of both models (0.9674 and 0.9900 for TPC and DPPH, respectively) were within close range to their respective coefficient of determination indicating that the variability of each response can be explained by the independent variables involved in the process. Joglekar and May (1987) suggested that  $R^2$  should at least be 80% for model fit; therefore, the empirical models of TPC and DPPH fits the actual data models. Baranyi et al. (1999) and Ross (1996) stated, in addition to  $R^2$ , other validation model terms, AAD, bias and accuracy factors are of great interest to be considered. They measure the relative average deviation of predicted and observed responses. An AAD of 0 and a bias factor and accuracy factor of 1 indicate model adequacy. In this study, all the validation terms fell within the accepted range of model validity which affirms the validity of the model (Table 4). The empirical equations developed for TPC (equation 8) and DPPH (equation 9) activity are as follows:

$$TPC = 54.735 + 9.806x_1 + 1.365x_2 + 1.883x_3 - 1.692x_1^2 - 0.827x_2^2 - 0.795x_3^2 - 0.171x_1x_2 + 0.445x_1x_3 + 0.803x_2x_3 \quad (8)$$

$$DPPH = 58.377 + 14.237x_1 + 0.974x_2 - 1.748x_3 - 1.011x_1^2 - 0.939x_2^2 + 0.419x_3^2 - 0.071x_1x_2 + 0.455x_1x_3 + 0.588x_2x_3 \quad (9)$$

### 3.2.1. Singular and quadratic effects on TPC and DPPH

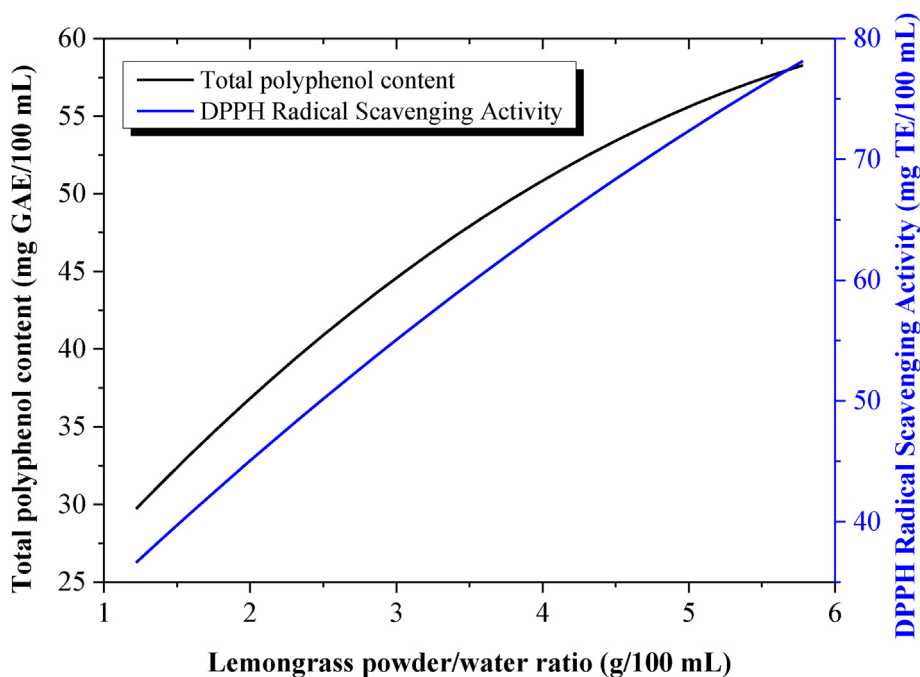
In this case, to view the effect of a singular factor, the other factors were fixed at their minimal value to lessen their contribution. That means for lemongrass powder/water ratio ( $x_1$ ), for decoction temperature ( $x_2$ ) and extraction time ( $x_3$ ), the respective fixed values were: 1.22; 82.4 °C and 3.7 min.

**3.2.1.1. Effect of lemongrass powder/water ratio ( $x_1$ ).** The lemongrass powder/water ratio ( $x_1$ ) has a significant impact on TPC and DPPH (Table 3). It is observed from Figure 1 that, at an initial ratio value of 1.22 g/100 mL, the values of 29.72 mg GAE/100 mL and 36.62 mg TE/100 mL were obtained for TPC and DPPH respectively.

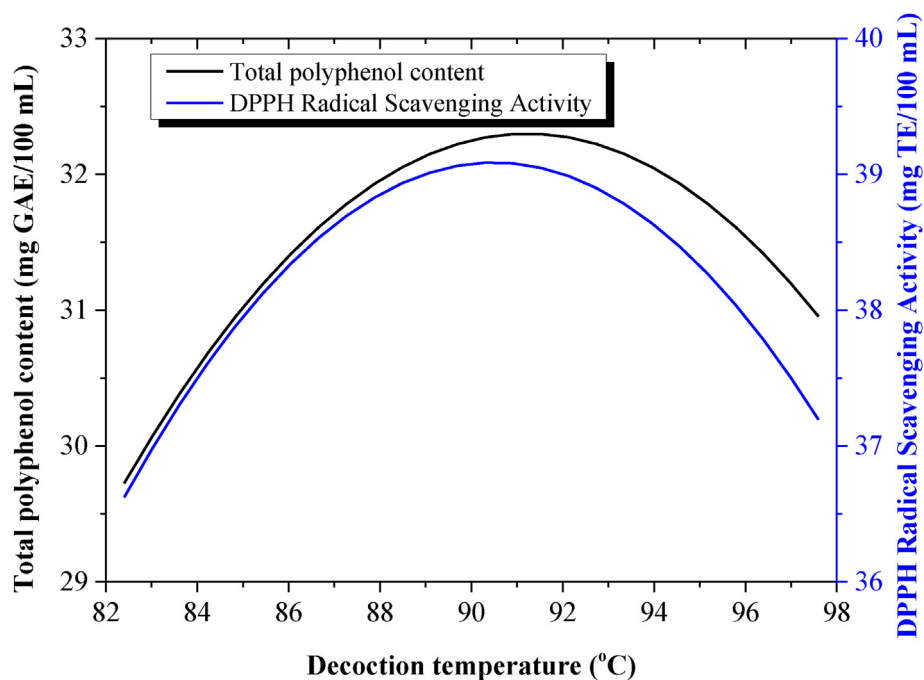
An increase of that ratio until 5.78 g/100 mL, generated a significant increase of TPC and DPPH respectively to 58.27 mg GAE/100 mL and 78.13 mg TE/100 mL. At a fixed decoction temperature of 82.4 °C and a fixed extraction time of 3.7 min, an increase in ratio led to increase in the amounts of TPC and DPPH activity in the extract.

The solid/liquid ratio was found to be the essential factor ( $p \leq 0.0001$ ) that affected the yield of TPC and DPPH activity of the lemongrass leaves extract. As the solid/liquid ratio increased, the TPC and DPPH activity increased significantly. However, when the solid/liquid ratio was extended beyond the critical limit (quadratic effect), a marked decline in TPC was observed while the DPPH activity was constant. Indeed, increasing the mass with a constant solvent volume caused molecule congestion which decreased mass transfer hence, decrease in the extraction of total polyphenols.

**3.2.1.2. Effect of decoction temperature ( $x_2$ ).** The decoction temperature has a significant positive and negative impact on TPC and DPPH (Table 3). Firstly, it is observed (Figure 2) that, at an initial decoction



**Figure 1.** Evolution of Total polyphenol content and DPPH radical scavenging activity as a function of lemongrass powder/water ratio. Decoction temperature and extraction time respectively fixed at 82.4 °C and 3.7 min.

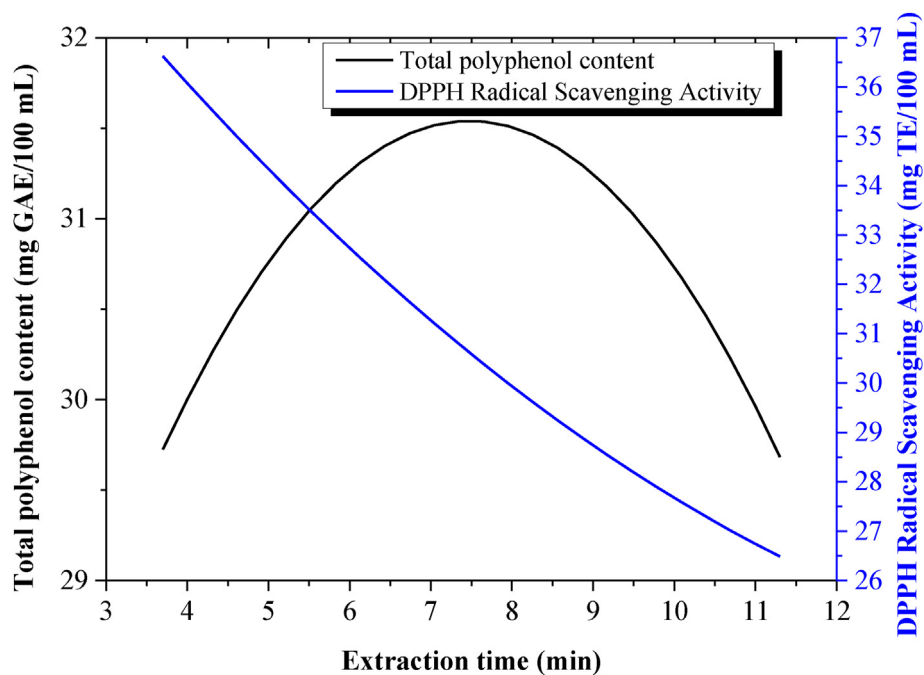


**Figure 2.** Evolution of Total polyphenol content and DPPH radical scavenging activity as a function of decoction temperature. Lemongrass powder/water ratio and extraction time respectively fixed at 1.22 and 3.7 min.

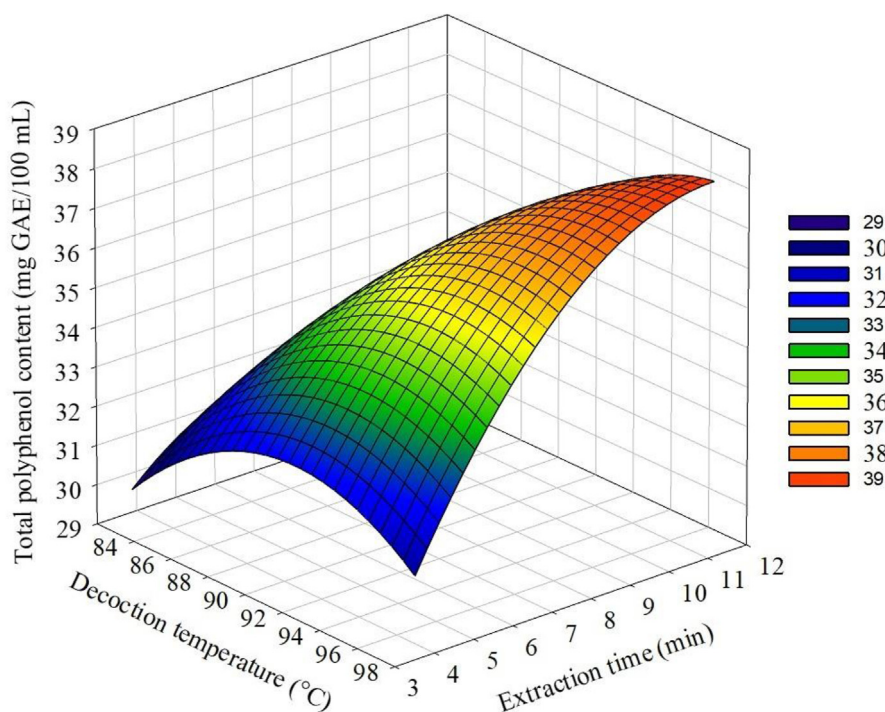
temperature of 82.4 °C, the values of 29.72 mg GAE/100 mL and 36.62 mg TE/100 mL were obtained for TPC and DPPH respectively. These values increased to reach a maximum of 32.29 mg GAE/100 mL for TPC at 91.2 °C and, 39.08 mg TE/100 mL for DPPH at 90.5 °C. After that, a significant decrease was obtained up to 30.95 mg GAE/100 mL for TPC and 37.19 mg TE/100 mL for DPPH, when increasing the decoction temperature to 97.6 °C.

The TPC and DPPH activity increased with the rise of extraction temperature and then level off at high temperature. The increment in TPC and DPPH activity is due to the fact that high temperatures soften

cell wall tissue and hydrolyse the phenolic compounds present thereby enhancing the solubility of polyphenols into the solvent (Cacace and Mazza, 2003; Irakli et al., 2018). The solvent thereby penetrates the plant matrix and results in the mass transfer of compounds from the matrix into the solvent (Jovanovic et al., 2017). However, a further increase of the temperature led to the decrease of both TPC and DPPH activity suggesting that high temperature may have caused the degradation of phenolic compounds resulting in a decrease in DPPH activity.



**Figure 3.** Evolution of Total polyphenol content and DPPH radical scavenging activity as a function of extraction time. Lemongrass powder/water ratio and decoction temperature respectively fixed at 1.22 and 82.4 °C.



**Figure 4.** Mesh plot of the evolution of Total polyphenol content as a function of decoction temperature and extraction time. Lemongrass powder/water ratio fixed at 1.22.

**3.2.1.3. Effect of extraction time ( $x_3$ ).** The extraction time has a negative significant impact on DPPH while, it is noted in the first hand a positive significant and after a significant negative impact on TPC (Figure 3). For TPC, the value of 29.72 mg GAE/100 mL was obtained at 3.7 min extraction time and increased with a rise in extraction time to reach a max of 31.54 mg GAE/100 mL at 7.47 min extraction time. After that, TPC decreased to 29.68 mg GAE/100 mL at 11.3 min extraction time.

This could be explained by that, as the lemongrass get in to contact with the hot liquid, there is immediate dissolution of the phenolic compounds, and diffusion of target analytes from materials to outside solvent. A relatively lengthy extraction time contributed to a positive influence on the TPC. However, extended extraction time led to degradation of polyphenols and lowering of DPPH activity due to the thermolabile feature of phenolic compounds. This finding is in accord with that of Jeszka-Skowron and Zgoła-Grześkowiak (2014) who realized that extended extraction time of *Camellia sinensis* caused a decrease in rutin and chlorogenic acid content.

### 3.2.2. Effect of interaction decoction temperature/extraction time ( $x_2x_3$ ) on TPC

The synergistic effect of temperature and time generated a positive impact (the increase) on the TPC. With increase in temperature at a short time (Figure 4), the plant matrix is fragilized and, the solvent enters the cell leading to a mass transfer of soluble compounds from the matrix into the solvent (Jovanovic et al., 2017).

### 3.2.3. Determination of optimal conditions

Composite desirability was effectuated to find the composite optimum by maximizing the TPC and DPPH (Table 5). Lemongrass was

therefore extracted at the composite optimum (solid/liquid ratio; 5g/100 mL, temperature: 93.8 °C and time: 11.3 min) and analyzed. The experimental values of TPC and DPPH obtained at the optimal conditions were  $71.98 \pm 0.33$  mg GAE/100 mL of extract and  $80.63 \pm 0.49$  mg TE/100 mL of extract, respectively. Compared to the predicted response of 73.00 mg GAE/100 mL extract for polyphenol and 87.75 mg TE/100mL extract for DPPH, the experimental data was in conformity.

Comparing to the results of Oboh et al. (2010), they obtained a total polyphenol content of 0.5 mg GAE/g and DPPH radical scavenging activity of 70% for hot water extracts of lemongrass at conditions (lemongrass concentration 10 g/100 mL, temperature 100 °C and time 10 min). The differences could be as a result of the different extraction conditions used and the temperature of extraction has a major effect on the polyphenol content as described above. Due to the importance of natural polyphenols, many works are done in recovering polyphenols from different sources and with different solvents (Galanakis et al., 2013; Rahmanian et al., 2014) for their applications in food and cosmetics.

### 3.2.4. Characterisation of *C. citratus* extract obtained at optimum conditions

**3.2.4.1. Phenolic compounds of *C. citratus* extract.** Phenolic acid compound content of the lemongrass leaf extract is presented in Table 6. Caffeic ( $20.816 \pm 0.003$  mg/100mL) and syringic ( $18.635 \pm 7.390$  mg/100mL) acids were the prominent phenolic acid compounds in the lemongrass extract. Other phenolic compounds (Gallic acid, dihydroxybenzoic acid, catechin, vanillic acid, epicatechin, *p*-coumaric acid, trans-ferulic acid, quercetin, and trans-cinnamic acid) were present in small amounts. Caffeic acid was also reported the main phenolic acid compound present in lemongrass infusion (Coelho et al., 2016). Other

**Table 5.** Composite desirability of total polyphenol and DPPH activity of *Cymbopogon citratus* extract.

Solid/liquid ratio	Temperature (°C)	Time (min)	Predicted TPC (mg GAE/100 mL)	Experimental TPC (mg GAE/100 mL)	Predicted DPPH (mg TE/100 mL)	Experimental DPPH (mg TE/100 mL)	Desirability value
5	93.8	11.3	73.00	71.98	87.75	80.63	0.92

**Table 6.** Phenolic acid content of *C. citratus* L. extract.

Compound	Concentration (mg/100 mL)
Gallic acid	3.932 ± 0.515
Dihydroxybenzoic acid	3.411 ± 0.121
Catechin	9.433 ± 5.493
Vanillic acid	5.979 ± 2.172
Caffeic acid	20.816 ± 0.003
Syringic acid	18.635 ± 7.390
Epicatechin	3.765 ± 1.243
<i>p</i> -coumaric acid	0.881 ± 0.394
Trans ferulic acid	0.582 ± 0.173
Quercetin	6.068 ± 0.326
Trans cinnamic acid	0.251 ± 0.010

phenolic acids (chlorogenic acid, *p*-coumaric acid, ferulic acid, quercetin, rosmarinic acid) that can arouse the antioxidant activities of lemongrass extract were also present in small amounts.

Otherwise, [Rodrigues et al. \(2015\)](#) instead detected a high quantity of chlorogenic acid in hot and cold extracts of lemongrass. [Kouassi et al. \(2017\)](#) also detected the presence of protocatechuic acid, caffeic acid, rutin, *p*-coumaric acid, ferulic acid, quercetin, kaempferol in ethanol and methanol extracts of lemongrass. Harvest region and seasons might account for the differences in profile and content in phenolic compounds ([Costa et al., 2016](#)). Likewise, the difference in profile and content might be the main factors that drive the difference in antioxidant activities of lemongrass extract.

Due to the antioxidant properties of natural polyphenols, they have gained applications in several fields. They have been applied in chicken patties ([Ibrahim and Abu Salem, 2013](#)), in herbal cookies ([Thorat et al., 2017](#)), in meat products ([Siewe et al., 2015](#); [Galanakis, 2018](#)), chicken sausage ([Boeira et al., 2018](#)) ([Boeira et al., 2018](#)), and as UV booster in cosmetics ([Galanakis et al., 2018](#)).

**3.2.4.2. Organic acids of lemongrass extract.** An organic acid is an organic compound with acidic properties known to affect particularly taste formation and many physiological functions ([Theron and Lues, 2010](#)). Organic acids content of lemongrass extract are displayed in [Table 7](#).

Propionic acid was the principal organic acid with a concentration of 20.137 ± 0.163 mg/mL, followed by glutaric acid, succinic acid, citric acid, tartaric acid, citric acid, malic acid, oxalic acid in descending order. Organic acids are traditionally employed as food preservatives because they exhibit antimicrobial inhibitory activities and also act as acidulants. Propionic acid, the main acid found in lemongrass is known to have antimicrobial activity primarily against molds and bacteria ([Theron and Lues, 2010](#)), this explains the antibacterial effect of lemongrass ([Balakrishnan et al., 2014](#); [Ekpenyong et al., 2015](#)). Succinic acid, citric acid, malic acid and tartaric acid are employed in industry as acidulants to modulate the taste of juice. Citric acid is the primal acid found in fruits and it possess a fresh acidic flavor and a pleasant taste. Malic acid has a smooth lingering taste, tart taste but not as sharp as that of citric acid ([Theron and Lues, 2010](#)).

**Table 7.** Organic acid content of *C. citratus* extract.

Organic acid	Concentration (mg/mL)
Oxalic	0.009 ± 0.002
DL-Tartaric	0.131 ± 0.022
L-malic	0.038 ± 0.009
Isocitric acid	0.217 ± 0.025
Citric	0.059 ± 0.008
Succinic	0.259 ± 0.007
propionic	20.137 ± 0.163
Glutaric	0.459 ± 0.106

**Table 8.** Volatile compounds content (%) of *C. citratus* extract.

Composition	RT	% composition
<b>Hydrocarbons</b>		4.13 ± 0.31
Undecane	5.431	2.38 ± 0.73
Naphthalene, decahydro-1,6-dimethyl-	25.664	1.75 ± 0.37
<b>Esters</b>		11.35 ± 3.44
Oxalic acid, allyl ethyl ester	3.64	0.92 ± 0.21
Acetic acid, butyl ester	5.14	1.54 ± 0.44
Linalyl acetate	24.058	8.74 ± 2.21
Methyl 2-undecynoate	39.794	0.15 ± 0.00
<b>Alcohols</b>		10.70 ± 0.71
2-Nonanol	5.641	1.46 ± 0.51
6-methyl-5-Hepten-2-ol	20.367	1.04 ± 0.08
Verbenol	24.493	0.88 ± 0.04
cis-Verbenol	24.678	3.12 ± 0.03
5,8,10-Undecatrien-3-ol	26.944	0.73 ± 0.12
cis-p-mentha-1(7),8-dien-2-ol	31.091	1.02 ± 0.50
3,7-dimethyl-6-Octen-1-ol	32.116	1.45 ± 0.38
Selina-6-en-4-ol	37.924	1.01 ± 0.17
<b>Aldehydes</b>		38.33 ± 14.06
3,7-dimethyl-2,6-Octadienal	22.502	0.61 ± 0.06
2-Decenal, (E)	27.119	0.38 ± 0.20
Citral	30.306	35.77 ± 4.21
2-Undecenal	30.986	0.30 ± 0.13
2,4-Decadienal	32.496	1.26 ± 1.11
<b>Acids</b>		5.48 ± 0.34
Acetic acid	19.481	0.49 ± 0.14
Hexanoic acid	33.391	1.11 ± 0.20
Pentanoic acid, 2,2,4-trimethyl-3-carboxyisopropyl,	34.162	1.14 ± 0.90
Octanoic acid	36.698	1.00 ± 0.36
Nonanoic acid	37.928	0.66 ± 0.16
n-Decanoic acid	39.044	0.19 ± 0.04
Benzoic acid	40.439	0.28 ± 0.11
Dodecanoic acid	41.025	0.60 ± 0.18
<b>Terpenes and terpenoids</b>		19.74 ± 7.17
Citronellol	31.861	2.38 ± 0.45
2,6,10-trimethyl-Dodecane	32.852	0.69 ± 0.09
Geraniol	33.632	16.67 ± 2.83
<b>Ketone</b>		7.51 ± 3.19
6-methyl-5-Hepten-2-one	13.974	6.95 ± 3.70
6-methyl-3,5-Heptadien-2-one	25.133	0.57 ± 0.26
<b>Others</b>		2.76 ± 0.57
Tetrahydro-3-Furanol	4.815	0.33 ± 0.05
1,1'-oxybis- Octane	31.491	1.69 ± 0.30
4-Acetyl-cycloheptanone	39.399	0.75 ± 0.19

**3.2.4.3. Aroma profile of lemongrass extract.** Lemongrass extract is widely used in perfumery and beverages owing to its desirable aroma ([Haque et al., 2018](#)). A total of 35 aroma compounds were identified in the *C. citratus* extract ([Table 8](#)). Amongst the group, aldehydes were the most prominent that made up to 38.33 ± 14.06%. The principal aldehyde identified was citral (35.77 ± 4.21%), reporting to be the main compound that accounts for scent and antimicrobial properties of lemongrass ([Fattah et al., 2010](#); [Li et al., 2018](#)). Terpenes and terpenoids were also present in appreciable amounts (19.74 ± 7.17%). In this group, geraniol (16.67 ± 2.83%) was the main component, followed by citronellol (2.38 ± 0.45%) and 2,6,10-trimethyl-dodecane (0.69 ± 0.09%), respectively. [Coelho et al. \(2016\)](#) also stated that citral and geraniol were the major volatile compounds of lemongrass extract. Through biological activities and flavouring properties of lemongrass, it has been used in beverages like yoghurt ([Fattah et al., 2010](#)), soy ice cream ([Natisri et al., 2014](#)), and ice cream ([Chamchan et al., 2017](#)). The essential oils of lemongrass are applied in perfumes and cosmetics ([Wifek et al., 2016](#)).



#### 4. Conclusion

Nowadays, consumers are increasingly choosing food products formulated with natural additives due to the comprehension of the relationship between health and diet. Therefore, it is important for the industrial food sector to find novel sources and efficient extraction methods of bio-based ingredients, including natural antioxidant and aroma. In this study, the RSM was successfully used to optimize the decoction conditions of lemongrass leaves. The optimal conditions generated were: solid/liquid ratio (5g/100 mL), temperature (93.8 °C) and time (11.3 min). This yielded a TPC of  $71.98 \pm 0.33$  mg GAE/100 mL of extract and  $80.63 \pm 0.49$  mg TE/100 mL for TPC and DPPH, respectively. The achieved experimental data were successfully fitted to the theoretical models used to determine the optimal extraction conditions. Caffeic ( $20.816 \pm 0.003$  mg/100mL) and syringic ( $18.635 \pm 7.390$  mg/100mL) acids were the most abundant phenolic acid compounds found in lemongrass extract. In addition, citral and geraniol were detected as the essential volatile compounds of lemongrass extract. This extract could therefore be employed in beverages.

#### Declarations

##### Author contribution statement

Wiyeh Claudette Bakisu Muala: Performed the experiments; Wrote the paper.

Zangué Steve Carly Desobgo: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Nso Emmanuel Jong: Contributed reagents, materials, analysis tools or data.

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##### Data availability statement

Data included in article/supplementary material/referenced in article.

##### Declaration of interests statement

The authors declare no conflict of interest.

##### Additional information

No additional information is available for this paper.

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