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Article

SPME Fiber Evaluation for Volatile Organic Compounds Extraction from Acerola

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Volatile organic compounds (VOCs) were isolated from acerola fruits (*Malpighia emarginata* D.C.), by means of different solid phase microextraction (SPME) fibers. For the extraction, the headspace SPME method was used, identifying the VOCs by gas chromatography (GC) with mass spectrometry (MS). The objective of this work was to evaluate the efficiency of SPME fibers and determine the best conditions for extracting VOCs from acerola fruit. The investigated conditions were: extraction time (20, 30 and 40 min), extraction temperature (25, 45 and 65 °C) and agitation (0, 50 and 100 rpm). Of the evaluated fibers, polydimethylsiloxane (PDMS)/divinylbenzene (DVB) extracted the highest number of VOCs, most belonging to terpene, carboxylic acids and hydrocarbons. According to the investigated conditions, most compounds were obtained with an extraction time of 20 min, extraction temperature 65 °C, and no agitation. Compounds cumene, *o*-xylene, thymol, *m*-cymene, *o*-cymene, 2-methyl-1,3-butadiene, anethol, 3-buten-2-one and methyl octadecanoic ester were responsible for the volatile profile of acerola.

Keywords: aroma, tropical fruits, *Malpighia emarginata*, GC-MS, solid phase microextraction, qualitative analysis

Introduction

Acerola (*Malpighia emarginata* D.C.) originates from the Antilles, northern South America and Central America, and is widely cultivated in Brazil, Puerto Rico, Cuba and the United States. Due to its capacity for industrial use and its high vitamin C content, it has attracted the interest of fruit growers and can be commercialized *in natura* or industrialized in the form of yogurts, sweets, biscuits, cake, ice cream and soft drinks.[1]

In Brazil, this fruit has been cultivated commercially since the mid-1980s, especially in Northeast Brazil, where the crop has adapted better due to the climate and soil conditions.[2,3]

The fruit is a fleshy drupe, of variable size, shape and weight; it is a climacteric fruit, presenting different tonalities, ranging from yellow to intense red or purple, which is the main criterion to characterize the ripening of the fruit.[4]

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The acerola is attractive for its pleasant taste, which varies from slightly acid to very acidic. The acceptance of the fruit is directly due to the flavor, a decisive factor between the sensations of taste and aroma, provided by the volatile and non-volatile compounds present in the foods.[2,5]

Non-volatile compounds, attributing to taste sensation, are classified into four basic categories: sweet, salty, sour and bitter. The volatile compounds are responsible for the aroma sensation, a much more complex class, since human olfaction can discriminate among a number of compounds.[5] The aroma is one of the most researched quality attributes, due to the innumerable olfactory sensations generated by the different fruit molecules.[5,6]

The typical aroma of acerola is the combination of a large number of volatile substances responsible for the scent emitted by the fruit, which are represented by various chemical classes with different physicochemical properties, such as esters, acids, ketones, aldehydes, alcohols and terpenes, which are used in minimal amounts, for the formation of food flavorings.[5,7]

Currently, many extraction methods are used for the analysis of these substances, highlighting solid phase microextraction (SPME). This is a technique that provides faster extraction of the compounds, as it requires a smaller sample volume and no organic solvent. SPME can be used in direct extraction and headspace (HS) extraction,[8] the latter being most commonly used for the analysis of fruit compounds.[9,10]

For the analysis of separation and identification of these compounds, gas chromatography (GC) instruments coupled to mass spectrometry (MS) are used, being preceded by the SPME technique, which consists of the extraction of the volatile compounds by the fiber and transfer of the fiber material to the injector of a chromatograph.[7,11]

The amount of analytes extracted by the fiber can be affected by the type of coating used, the extraction conditions (temperature and time), and sample agitation. The type of coating of the fiber is one of the main characteristics to choose if good analyte selectivity is desired, because different types of chemical compounds will be obtained depending on the coating.[12]

As such, the objective of this work was to evaluate the efficiency of different SPME fibers for the extraction of volatile compounds from acerola and to determine the best extraction conditions to define the volatile profile characteristic of acerola.

Experimental

Materials and methods

Five SPME fibers with different polarities were evaluated in the extraction of the volatile organic compounds (VOCs), two polar fibers: 85 μm polyacrylate (PA), and 65 μm carbowax (CW)/divinylbenzene (DVB); and three semipolar fibers: 65 μm polydimethylsiloxane (PDMS)/DVB, 50 μm DVB/carboxen (CAR)/PDMS, and 75 μm CAR/PMDS.

Equipment

The analyses were performed by a GC (Trace GC Ultra) coupled to an MS detector (Polaris Q) from Thermo Scientific, with an ion-trap type analyzer with a split/splitless injector, in splitless mode, installed in the Laboratory of Mass Spectrometry of the Department of Chemistry, Universidade Federal de Minas Gerais.

Helium was used as a drag gas at a constant flow of 1 mL min^{-1}. Chromatographic analysis conditions were: injector temperature 250 °C, desorption time 5 min, ion source temperature 200 °C, and interface temperature 275 °C.

An HP-5 MS capillary column (5% phenyl and 95% methylpolysiloxane) (30 m long, 0.25 mm i.d. and 0.25 μm film thickness) was used (Agilent Technologies). Column heating was programmed starting at 40 °C, remaining for 5 min, heating at 2.5 °C min^{-1} to 125 °C and then at 10 °C min^{-1} to 245 °C, at which temperature the isotherm was maintained for 3 min.[13]

Experimental design

A factorial 2^3 design with triplicate central point was used.[8] The effect of the dependent variables extraction time (t), extraction temperature (°C) and stirring (rpm) was used for the extraction of acerola VOCs (Table 1). For the factorial planning, the STATISTICA software version 10.0 was used.[14] The number of VOCs captured *per* test was used as response for the optimization of the evaluated factors.

Table 1. Factorial planning (2^3) with triplicate central point

Variable	Level		
	–	0	+
Extraction time / min	20	30	40
Extraction temperature / °C	25	45	65
Agitation / rpm	0	50	100

Preparation of the samples

The fruits used were of an unknown variety of acerola, located in the municipality of Sete Lagoas-MG in Brazil, at 761 m altitude, with geographical coordinates of 19°27'57"S latitude and 44°14'48"W longitude. Mature acerola fruits were collected during the 2014 crop season.

Approximately 100 fruits were selected at the red maturation stage.[15] After the collection, the fruits were carried in polyethylene bags to the Vegetable Production Laboratory of Universidade Federal de São João Del-Rei/Campus Sete Lagoas, where they were sanitized with running water for the elimination of impurities.

The fruits were then processed using a quick mixer and stored in a freezer at –18 °C until the analysis. For the preparation of the samples, the pulp previously obtained was used for the extraction of volatile compounds.

Extraction of VOCs

HS-SPME was used for the extraction of the VOCs, for which only 2.0 g of the acerola pulp samples were taken from the freezer. Samples were weighed and placed in HS flasks with 20 mL capacity, sealed with an aluminum seal and rubber septum.[13]

The HS flasks were subjected to different extraction times (20, 30 and 40 min), different extraction temperatures (25, 45 and 65 °C) and different agitations (0, 50 and 100 rpm). These variables were used in order to determine the optimum conditions to reach the partition equilibrium of the analytes between the sample and the fiber in the HS mode, which causes higher recovery of the VOCs after the chromatographic analysis.

After the flasks were subjected to the different conditions, the SPME fiber was introduced into the HS mode for absorption of the analytes and then exposed to the gas phase. After the extraction time, it was inserted in the injector of the chromatograph at 250 °C, for 5 min, for the desorption of the extracted VOCs.[13]

Identification of VOCs

The peaks present in the chromatograms were selected according to those that presented a signal-to-noise (S/N) ratio higher than 50, as well as compared to data obtained via the National Institute of Standards and Technology (NIST) library considering a similarity level (reversed search index, RSI) higher than 500.[16-19]

The VOCs were identified by the mass-load ratio (*m/z*) corresponding to each peak generated by the total ion chromatogram of each sample analyzed, and compared with the mass spectra obtained by electron impact ionization (EI), which uses an energy of 70 eV, with a full scan range of 50 to 650 *m/z*.[17] For VOC confirmation, a comparison of the compounds obtained with those already reported in the literature was performed.[19]

The S/N ratio values, as well as the peak intensity obtained, were taken from the Xcalibur 1.4 program (Thermo Scientific) and transferred to Microsoft Office Excel 2013, the program in which the peaks were selected, according to the S/N relation.[13]

Results and Discussion

Optimization of fiber extraction conditions by HS-SPME

The VOCs identified in the present study were extracted during different extraction times, at different extraction temperatures and under different agitations, as presented in Table 1, referring to the factorial planning used. For the separation and identification of these compounds, a GC-MS was used.

The results obtained for factorial design 2^3 with triplicate central point are presented in Table 2, where the response analyzed was the number of VOCs extracted for each of the fibers studied and the tests performed. The number of compounds extracted, according to the conditions proposed by the experimental design, varied between 13 and 37 VOCs.

The best conditions obtained for the efficiency of each of the SPME fibers was based on the maximum amount of volatile compounds isolated according to each of the conditions analyzed in Table 1, so, according to the greater number of volatile compounds extracted from each fiber, the obtained conditions are presented in Table 3. Note that of the five fibers evaluated, three presented similar extraction conditions (CW/DVB, DVB/CAR/PDMS and PA), while for the other two (CAR/PDMS and PDMS/DVB), the extraction conditions presented different behaviors for the acerola VOCs extraction.

According to the number of extracted compounds and experimental conditions studied, the fiber with the best VOC extraction efficiency was the polyacrylate (PA)

Table 2. Number of VOCs extracted by different SPME fibers

Assay	Variable			Response				
	A / min	B / °C	C / rpm	CAR/PDMS	CW/DVB	DVB/CAR/PDMS	PA	PDMS/DVB
01	20	25	0	24	22	20	22	25
02	40	25	0	26	22	23	26	26
03	20	65	0	23	16	22	35	33
04	40	65	0	18	17	18	29	24
05	20	25	100	21	18	16	23	17
06	40	25	100	21	21	27	34	29
07	20	65	100	15	20	28	34	24
08	40	65	100	24	18	13	29	24
09[a]	30	45	50	19	25	28	37	21
10[a]	30	45	50	19	20	24	32	21
11[a]	30	45	50	23	17	27	30	20

[a]Triplicate central point. A: extraction time; B: extraction temperature; C: agitation; CAR/PDMS: carboxen/polydimethylsiloxane; CW/DVB: carbowax/divinylbenzene; DVB/CAR/PDMS: divinylbenzene/carboxen/polydimethylsiloxane; PA: polyacrylate; PDMS/DVB: polydimethylsiloxane/divinylbenzene.

Table 3. Experimental conditions and responses obtained for the extraction of the VOCs of acerola, by HS-SPME and GC-MS

SPME fiber	Response		
	Extraction time / min	Extraction temperature / °C	Agitation / rpm
CAR/PDMS	40	25	0
CW/DVB	20	25	0
DVB/CAR/PDMS	20	25	0
PA	20	25	0
PDMS/DVB	20	65	0

SPME: solid phase microextraction; CAR/PDMS: carboxen/polydimethylsiloxane; CW/DVB: carbowax/divinylbenzene; DVB/CAR/PDMS: divinylbenzene/carboxen/polydimethylsiloxane; PA: polyacrylate; PDMS/DVB: polydimethylsiloxane/divinylbenzene.

fiber, since it was able to extract the most compounds in the following conditions: shorter extraction time (20 min), maximum extraction temperature (65 °C), and no need to expose sample to agitation (0 rpm).

The agitation does not influence the extraction capacity of the fiber, which minimizes the time necessary for the equilibrium of the analytes between the fiber and the sample and/or HS to be reached.[20] Thus, with the increase of the extraction temperature, the sensitivity can be improved in relation to the compounds with higher molecular weight, while at the same time it can hinder the extraction of the lower molecular weight compounds.

VOCs of acerola

In the present study, 51 VOCs were present in acerola fruits, 13 of which were reported for the first time in the literature (3-buten-2-one, ester methyl octadecanoic, isopropyl formate ester, ethyl vanillin, eucalyptol, *m*-cymene, *o*-cymene, citric acid, propanoic acid, tridecanoic acid, cumene, dodecane, and *o*-xylene), and 27 common among all the fibers, such as: 3-buten-2-one, acetophenone, 2-butanol, benzylic alcohol, ester methyl octadecanoic, anethole, eugenol, benzaldehyde, furfural, 2-methyl-1,3-butadiene, *m*-cymene, *o*-cymene, *p*-cymene, terpinen-4-ol, terpinolene, thymol, α-terpinene, decanoic acid, dodecanoic acid, heptanoic acid, hexadecanoic acid, nonanoic acid, tetradecanoic acid, cumene, ethylbenzene, *m*-xylene, and *o*-xylene.

The VOCs extracted were classified into eight different chemical classes: ketones, alcohols, esters, phenols, aldehydes, terpenes, carboxylic acids and hydrocarbons. For the extraction of these compounds, HS-SPME was used. SPME fibers with different polarities were used, which, for the most part, extracted compounds belonging to the classes of terpenes, carboxylic acids and hydrocarbons.

Efficiency of SPME fibers

A total of 33 VOCs were extracted using CAR/PMDS fiber, 39 using CW/DVB fiber, 43 with DVB/CAR/PDMS fiber, 41 with PA fiber and 44 with PDMS/DVB fiber, as shown in Table 4.

Among the SPME fibers evaluated, the PDMS/DVB semipolar fiber was better compared to the other fibers, as

Table 4. VOCs extracted from acerola, using fibers of different polarities, by HS-SPME/GC-MS

No.	Volatile organic compound	SPME fiber				
		CAR/PDMS	CW/DVB	DVB/CAR/PDMS	PA	PDMS/DVB
Ketone						
1	3-buten-2-one	×	×	×	×	×
2	acetophenone[15,17,21-23]	×	×	×	×	×
3	cyclohexanone[17,21,22]	–	×	×	–	×
Alcohol						
4	1-octadecanol[15,17,22]	–	×	–	–	×
5	1-tetradecanol[17,22]	–	–	×	×	×
6	2-butanol[17,21]	×	×	×	–	×
7	2-ethyl-1-hexanol[17]	×	–	×	×	×
8	2-methyl-3-buten-2-ol[17,21]	–	×	×	–	×
9	3-methyl-1-butanol[17,21,23,24]	–	×	–	×	–
10	benzylic alcohol[17,21,24]	×	×	×	×	×

Table 4. VOCs extracted from acerola, using fibers of different polarities, by HS-SPME/GC-MS (cont.)

No.	Volatile organic compound	SPME fiber				
		CAR/PDMS	CW/DVB	DVB/CAR/PDMS	PA	PDMS/DVB
Ester						
11	3-methyl-1-butanol acetate[21,24]	–	×	–	–	–
12	ester methyl octadecanoic	×	×	×	×	×
13	isopropyl formate ester	×	–	–	–	–
14	ethyl acetate[15,17,21,22,25]	×	×	×	–	×
15	isopropyl myristate[17]	–	×	×	×	×
16	isopropyl palmitate[17,22]	×	×	–	×	×
Phenylpropanoid						
17	anethole[17]	×	×	×	×	×
18	ethyl vanillin	–	–	×	–	–
19	eugenol[15,17,21]	×	×	×	×	×
20	vanillin[15,17,21]	–	×	×	×	×
Aldehyde						
21	benzaldehyde[21,22]	×	×	×	×	×
22	furfural[21-23]	×	×	×	×	×
Terpene						
23	2-methyl-1,3-butadiene[17]	×	×	×	×	×
24	eucalyptol	–	×	×	×	×
25	*m*-cymene	×	×	×	×	×
26	*o*-cymene	×	×	×	×	×
27	*p*-cymene[17,21,22]	×	×	×	×	×
28	terpinen-4-ol[17,21,22]	×	×	×	×	×
29	terpinolene[17,21,22]	×	×	×	×	×
30	thymol[26]	×	×	×	×	×
31	α-terpinene[17,21]	×	×	×	×	×
Carboxylic acid						
32	acetic acid[21]	–	–	×	–	×
33	citric acid	–	–	×	×	–
34	decanoic acid[21,22]	×	×	×	×	×
35	dodecanoic acid[15,17,21,22]	×	×	×	×	×
36	heptanoic acid[15,17,21,24]	×	×	×	×	×
37	hexadecanoic acid[15,17,21-23]	×	×	×	×	×
38	hexanoic acid[21,22,24]	–	×	–	–	×
39	nonanoic acid[17,21,22]	×	×	×	×	×
40	octanoic acid[21,22]	×	–	–	×	–
41	pentanoic acid[22]	–	–	×	×	×
42	propanoic acid	–	–	×	×	×
43	tetradecanoic acid[15,17,21,22]	×	×	×	×	×
44	tridecanoic acid	–	×	×	×	×
Hydrocarbon						
45	cumene	×	×	×	×	×
46	dodecane	–	–	×	×	×
47	ethylbenzene[17,22]	×	×	×	×	×
48	heptadecane[15,21]	×	–	–	×	–
49	hexadecane[17]	–	–	×	×	×
50	*m*-xylene[17,21,22]	×	×	×	×	×
51	*o*-xylene	×	×	×	×	×
Total compounds		33	39	43	41	44

SPME: solid phase microextraction; CAR/PDMS: carboxen/polydimethylsiloxane; CW/DVB: carbowax/divinylbenzene; DVB/CAR/PDMS: divinylbenzene/carboxen/polydimethylsiloxane; PA: polyacrylate; PDMS/DVB: polydimethylsiloxane/divinylbenzene. The superscript numbers refer to compounds that have been detected by other authors: Vendramini and Trugo;[15] García *et al.*;[17] Franco and Janzantti;[21] Pino and Marbot;[22] Bicas *et al.*;[23] Boulanger and Crouzet;[24] Carasek and Pawliszyn;[25] Polo *et al.*[26]

it extracted the most VOCs from the acerola fruit. However, the CAR/PDMS fiber extracted the fewest compounds among the classes, due to the low polarity that the fiber presents in relation to the other fibers.

However, the CAR/PDMS fiber was able to extract three of the compounds not extracted by the PDMS/DVB fiber belonging the chemical classes hydrocarbons (heptadecane), carboxylic acids (octanoic acid) and esters (isopropyl formate ester).

The fibers composed of liquid (PDMS) and solid components (DVB and/or CAR) present better efficiency in the extraction of the VOCs of tropical fruits, among them acerola.[25] The PDMS/DVB fiber presents a mixture of porous solid polymers of DVB with polymeric liquid PDMS, which presents efficiency for the extraction of molecules between 2 and 12 carbon atoms, because molecules larger that C_{12} present difficult desorption, although they have high adsorption capacity.[27]

The DVB coating shows uniform pore size, resulting in a lower adsorption discrimination of compounds with different molar weights.[10] The combination of these two polymers (PDMS and DVB) provides better retention of smaller analytes than with the use of the PDMS polymer. Furthermore, the DVB polymer has higher affinity for more polar analytes.[26]

The CW/DVB fiber is a mixture of divinylbenzene particles with the liquid carbowax phase.[27] In Table 4, it can be observed that the fiber presented higher affinity for polar compounds, compared to the PA fiber, which extracted the fewest compounds, although they had the same polarity.

The polar compounds cyclohexanone, 1-octadecanol, 2-butanol, 2-methyl-3-buten-2-ol, 3-methyl-1-butanol acetate, ethyl acetate and hexadecanoic acid were extracted by CW/DVB fiber; such compounds were not isolated by PA fiber. The CW polymer is relatively polar, but when it is combined with the DVB polymer, its polarity increases, so it is indicated as being the most suitable to extract polar analytes.[26]

A total of 37 volatile compounds present in the volatile fraction of the acerola fruit were identified, in three different stages of maturation. At the red maturation stage, compounds belonging to the classes of carboxylic acids, phenylpropanoids, alcohols, esters and ketones were detected.[15] According to the authors, these compounds are part of the flavor characteristics of acerola, especially esters, alcohols and ketones, which were also detected in the present study.

For this study, the results obtained in the literature show that the compounds obtained by two methods, solid phase extraction (SPE) and simultaneous distillation extraction (SDE), esters, alcohols and ketones, were the main classes of compounds detected in mature acerola fruits. Other studies consider ethyl acetate as a strongly important compound in relation to fruit flavor.[21] However, the compounds that were present during the three stages of maturation were hexadecanoic, octadecanoic and tetradecanoic acids, which were also detected in the present study.

In general, most of the scientific studies in which the VOCs of acerola were evaluated agree that alcohols and esters are the main chemical classes that characterize the fruits of the acerola tree, compounds which participate directly in the fresh and fruity aroma; therefore, the aroma is a quality that any fruit should have.[22,24]

However, other studies have reported monoisoprenoids and ketones as compounds strongly related to the taste of acerola fruits.[15,21] Nonetheless, alcohols, aldehydes, esters, phenylpropanoids and terpenoids have recently been reported as major chemical classes that characterize the volatile profile of acerola fruits.[17]

Evaluation of the extraction efficiency of SPME fibers

According to the response surface graph presented in Figure 1a, the interaction between the time and the extraction temperature had a positive influence by the use of PDMS/DVB fiber. Its positive effect is associated with the greater number of volatile compounds extracted, i.e., the higher the temperature increase and the extraction time, the greater the number of volatile compounds extracted. The extraction temperature was thus verified as the main condition for the extraction of the volatile compounds from acerola, where the number of VOCs was greater than 28, represented by the darker red color.

In Figure 1b, the response surface graph for the CW/DVB fiber is shown, and the interaction of the most significant variables shows that the extraction temperature influenced more than the extraction time and agitation, which tended toward lower extraction values. This indicates that the highest number of VOCs extracted is reached for the lowest temperature values, a result opposite to that obtained with the PDMS/DVB fiber, which required the highest temperature increase for the extraction of the highest amount of VOCs.

The PA and DVB/CAR/PDMS fibers presented the same conditions in response to the extraction of the volatile compounds (Tables 3 and 4). The response surface graphs presented in Figures 1c and 1d, respectively, showed that the interaction between the variables, extraction time and extraction temperature were the most relevant because, since the extraction values are lower, the number of volatile compounds extracted by each fiber was much higher.

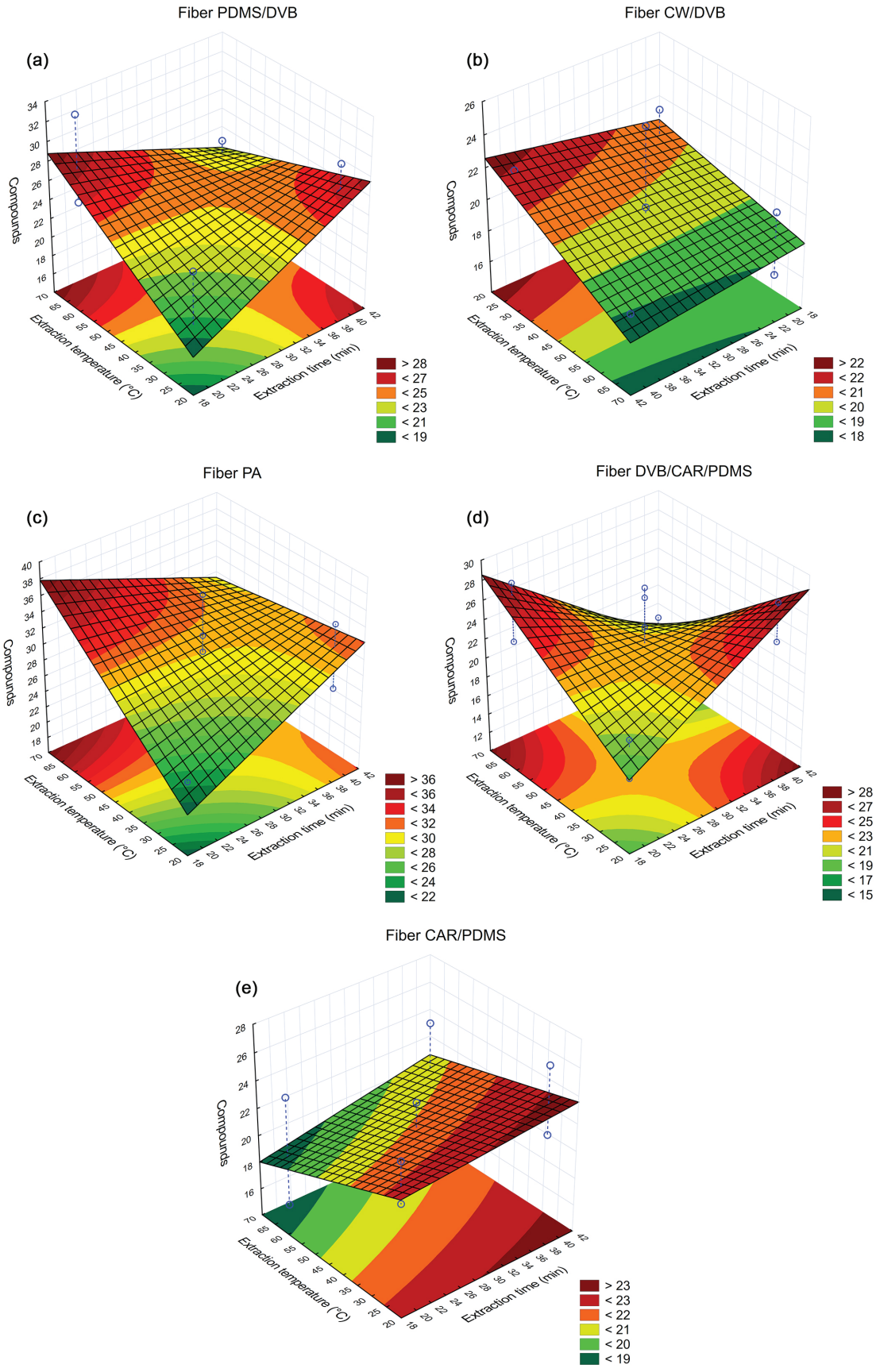

Figure 1. Response surface graphs for the fibers (a) PDMS/DVB; (b) CW/DVB; (c) PA; (d) DVB/CAR/PDMS; and (e) CAR/PDMS.

In the case of the variable agitation there was no influence on the volatile compounds extraction, therefore, none of these variables had a statistically significant effect for the extraction VOCs. However, the DVB/CAR/PDMS fiber extracted slightly more compounds with respect to the PA fiber, which is used for polar compounds in aqueous medium, because it is a more hydrophilic fiber, thus, the behavior of both fibers was similar (Table 2).

In the same way as in the previous fibers, for the CAR/PDMS fiber, the interaction variables were analyzed using the response surface graph, as shown in Figure 1e. The Figure shows the interaction of the variables that had the greatest effect for the extraction of the volatile compounds. As it can be seen, the extraction time had greater influence in comparison with the extraction temperature, since when there is increase of the temperature, the number of volatile compounds extracted is lower.

The time of extraction as the main condition for the extraction of volatiles from acerola was observed, since it had a greater effect, extracting more than 23 volatiles in comparison to the temperature of extraction and agitation. However, none of the variables investigated were significant for the extraction of volatile compounds, through the use of this fiber.

Conclusions

The use of HS-SPME and GC-MS techniques are efficient for the extraction of volatile compounds in acerola fruits.

The SPME fibers evaluated allowed to extract, together, a total of 51 volatile compounds, of which 26 were common to each other.

The compounds belonging to the classes terpenes, carboxylic acids and hydrocarbons were considered as the main constituents of acerola.

The temperature slightly influenced the extraction of the volatile compounds, since there was a temperature variation from one fiber to another, however, statistically none of the investigated variables were significant.

PDMS/DVB showed the best efficiency under extraction conditions: extraction temperature of 65 °C and extraction time of 20 min, without stirring.

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