Talanta 109 (2013) 46–51

Contents lists available at SciVerse ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Dispersive liquid–liquid microextraction followed by high-performance liquid chromatography for determination of benzoate and sorbate in yogurt drinks and method optimization by central composite design

Marzieh Kamankesh [a,b], Abdorreza Mohammadi [a,*], Zohreh Modarres Tehrani [b], Roohallah Ferdowsi [a], Hedayat Hosseini [a]

[a] Department of Food Science and Technology, Faculty of Nutrition Science, Food science and Technology/National Nutrition and Food Technology Research Institute, Shahid Beheshti University of Medical Sciences, Tehran, Iran
[b] Department of Chemistry, Faculty of Science, Alzahra University, Tehran, Iran

ARTICLE INFO

Article history:
Received 28 October 2012
Received in revised form
23 January 2013
Accepted 25 January 2013
Available online 13 February 2013

Keywords:
Dispersive liquid–liquid microextraction
High-performance liquid chromatography
Preservatives
Benzoate and sorbate
Central composite design

ABSTRACT

A new method based on dispersive liquid–liquid microextraction (DLLME) followed by high-performance liquid chromatography (HPLC) for determination of benzoate and sorbate salts in yogurt drinks was developed. The effective parameters in DLLME process, including volume of extraction and disperser solvents, pH and salt effect, were optimized using response surface methodology (RSM) based on central composite design. The yogurt drink samples were extracted using NaOH and Carrez solutions (potassium hexaferrocyanide and zinc acetate) were used for sedimentation of proteins. For DLLME, a mixture of extraction solvent (1-octanol) and disperser solvent (ethanol) was rapidly injected into the sample solution by syringe and cloudy solution is formed. Subsequently, the upper 1-octanol layer was analyzed by HPLC. The detection limits for benzoate and sorbate were 0.06 ng mL^{-1} and 0.15 ng mL^{-1}, respectively. The relative standard deviations (RSD) for seven analyses were 4.96% for benzoate and 4.58% for sorbate. The proposed method demonstrated good linearity and high enrichment factor. A clean separation and good chromatogram is readily achieved without the presence of matrix interference. A comparison of this method with previous methods demonstrated that the proposed method is an accurate, rapid and reliable sample-pretreatment method that gives very good enrichment factors and detection limits for extracting and determining sorbate and benzoate in yogurt drink samples.

1. Introduction

Many water-based products, especially yogurt drinks contain antimicrobial preservatives such as benzoate and sorbate salts. Although these additives impart many advantages to these drinks including extending their shelf life, they also have disadvantages [1]. Analysis of benzoate and sorbate is of great importance, because these widely-used preservatives have adverse effects on human health. Up to now, methods such as gas chromatography (GC) [2–4], HPLC [5–12], thin layer chromatography (TLC) [13], capillary electrophoresis [14–17] and spectroscopic methods [18–20] have been used to analyze benzoate and sorbate. Among these techniques, some of them including GC may require lengthy extraction, preparation and derivatization of the analytes. In contrast, HPLC requires minimal preparation and does not require derivatization; therefore, this method has been widely used to analyze benzoate and sorbate. Sample preparation involves precipitation of fat and proteins, dilution, filtration and direct injection of the extraction solution into HPLC. Conventional liquid–liquid extraction (LLE) [21] and solid-phase extraction (SPE) [3,22–24] have been commonly used as sample preparation techniques. Microextraction techniques have been characterized as a promising basis for a new generation of sample preparation techniques and have recently received a great deal of attention. These techniques are distinguished by their negligible extractant phase consumption, high potential to pre-concentrate target analytes and ease of use. Moreover, the generated waste could be reduced in volume by several orders of magnitude [25]. Microextraction techniques have several advantages such as miniaturization (minimized volume of extraction solvent to even solvent-free extraction), automation, high-throughput performance, on-line coupling with analytical instruments and no solvent consumption [26–30]. Microextraction techniques including solid phase microextraction (SPME) and liquid phase

* Corresponding author. Tel.: +98 21 22376426; fax: +98 21 22360660.
E-mail address: ab.mohammadi@sbmu.ac.ir (A. Mohammadi).

0039-9140/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.talanta.2013.01.052

microextraction (LPME) have been used in a number of studies to analyze preservatives [31–34].

In 2006, Assadi et al. proposed DLLME as a novel microextraction technique which provides excellent enrichment factors, good repeatability and high recovery within a much short time [35].

This modified solvent microextraction method offers greatly reduced acceptor-to-donor phase ratio as compared to other preconcentration methods and is based on a ternary solvent system analogous to a combination of LLE and cloud point extraction [36]. The phase combination in DLLME is accomplished instantly by rapidly adding the mixture of disperser and extraction solvent (organic phase) into the sample solution (aqueous phase) using a syringe. The cloudy phase appears, and the phase separation is performed by centrifuging. Acetonitrile, acetone, isopropanol, methanol and ethanol are commonly selected as disperser solvents. High-density solvents, such as chloroform, chlorobenzene, dichloromethane, tetrachloroethylene and carbon tetrachloride, and low-density solvents, including cyclohexane, hexanol, di-isobutyl ketone (DBIK) and 1-octanol, are conventionally utilized as extraction solvents. The main advantages of DLLME include its simplicity, requirement of very small volumes of extraction solvents, and the presence of very large surface area between the extraction solvent and the aqueous sample which rapidly reaches a state of equilibrium between the organic and aqueous phases. Other advantages include high enrichment factor, high speed, low cost, and high recovery.

In the present study, a novel analytical method (DLLME) followed by HPLC with UV detection was applied to extract benzoate and sorbate from yogurt drinks. The extraction was carried out in a binary system composed of extractant solvents (1-octanol and ethanol) and the sample solution. Several experimental parameters influencing the extraction performance of the proposed method were investigated and optimized by CCD. The proposed method was successfully applied to the analysis of trace amounts of benzoate and sorbate in yogurt drink samples.

2. Experimental

2.1. Reagent, material and standards

Sodium benzoate and potassium sorbate were obtained from Dae Jung (South Korea). 1-octanol, ethanol, sulphuric acid, acetic acid, sodium di-hydrophosphate and sodium chloride (analytical grade), sodium acetate, glacial acetic acid, HPLC solvents, acetonitrile and water were obtained from Merck (Darmstadt, Germany). Sodium hydroxide was obtained from Acros (Belzhike). Carrez solution containing potassium hexaferrocyanide 0.25 mol L^{-1} (Carrez solution I) and zinc acetate 0.4 mol L^{-1} (Carrez solution II) was purchased from Panreac (Belzhike).

Stock standard solutions of benzoate and sorbate were prepared together at a concentration of 2000 μg mL^{-1} in double-distilled water. To obtain a working solution (10 μg mL^{-1}), the upper standard solution was diluted with distilled water. The model solutions containing the required amount of each analyte (0.05–500 μg mL^{-1}) were prepared by diluting stock and mixed standard solutions with double-distilled water to evaluate the extraction performance under different conditions. Stock and working solutions were refrigerated at 4 °C. All solvents were of analytical reagent grade or HPLC grade.

2.2. Instrumentation

The chromatographic analysis was carried out with a Cecil CE-4900 and HPLC (Cambridge, England) equipped as follows: two CE-4100 pumps, multiple solvent delivery unit, vacuum degasser, mixing chamber, six-port valve (Rheodyne, USA), and CE-4200 UV–vis detector (Cambridge, England). An ODS column (250 mm × 4 I.D., 5 μm) was used for the separation of analytes. The injection volume was 20 μL, and the column temperature was 25 °C (ambient temperature). Separation of benzoate and sorbate was achieved using an acetate buffer (0.2 mol L^{-1}; pH 4.4): acetonitrile (50:50) acted as the mobile phase at a flow rate of 1 mL min^{-1}. The effluent was monitored at 225 and 255 nm for benzoate and sorbate, respectively.

2.3. Procedures

2.3.1. Preparation of model sample

To prepare the model sample, 104.7 g powdered milk and 20 g NaCl were added to 2 L distilled water. After shaking, the closed vessel was placed in a water bath at 85 °C for 30 min, after which the temperature was decreased to 45 °C. Starter (Thermophilic Yogurt Culture YC-X11) was added to 1 L low-fat milk, and 10 mL of this mixture was added to the closed vessel at 45 °C; this was then placed in an incubator at 45 °C for 6 h. At this stage, the pH was modified to 4.2, and the container was placed in a water bath at 15 °C. The prepared solution was refrigerated for 1 day. To optimize and calculate using the figure of merit, specified amounts of the mixed solution of benzoate and sorbate were spiked in the model sample.

2.3.2. Sample preparation

2 mL of spiked yogurt drink (100 ng mL^{-1}) was placed in a 15 mL screw-cap, glass test tube. After adding 8 mL of NaOH (0.1 mol L^{-1}), the container was closed and shaken for 1 min, then centrifuged and the upper phase was separated. In this step, 1.5 mL H_2SO_4 (0.5 mol L^{-1}), 1 mL potassium hexaferrocyanide (Carrez solution I) and 1 mL zinc acetate (Carrez solution II) were added to the precipitated protein phase, and the closed container was again shaken for 1 min. After shaking, the closed container was centrifuged at 4000 rpm for 10 min. The aqueous phase was filtered and 10 mL was transferred to another conical flask containing NaCl where the DLLME process was performed.

2.3.3. Dispersive liquid–liquid microextraction (DLLME)

A solution consisting of 450 μL of ethanol (disperser solvent) and 60 μL 1-octanol (extraction solvent) was rapidly injected into the extraction device containing 10 mL of sample solution (after adjusting it to pH 3 and adding 2 g NaCl). The mixture was thoroughly shaken using a flat shaker for 2 min. In this step, benzoate and sorbate were extracted into fine droplets of 1-octanol. Subsequently, to separate the organic phase, the liquid mixture was centrifuged for 10 min at 4000 rpm. After this process, the dispersed fine droplets of 1-octanol floated on the aqueous sample. The lower-aqueous phase was separated using a syringe and 20 μL of the floated phase was injected directly into the HPLC using a microsyringe.

2.4. Experimental design

The most effective parameters on the performance of DLLME process, including the volume of disperser solvent (A), volume of extraction solvent (B), amount of salt (C) and sample pH (D), were chosen based on literature and preliminary experiments. In order to optimize the values of these factors and to reach the best response, CCD was used. CCD, which is one of the most frequently used RSM, is affected by a combination of several factors. RSM plays an important role in designing, formulating, developing and analyzing new scientific research, as well as improving existing studies and products.

Table 1
The experimental range and levels of the variables in the CCD.

Variable parameter		Variable		Levels		
		−α (low)	−1	0	+1	+α (high)
A	Disperser solvent volume (μL)	300	475	650	825	1000
B	Extraction solvent volume (μL)	60	82.5	105	127	150
C	Salt (g)	o	0.5	1	1.5	2
D	PH	3	4.5	6	7.5	9

Table 2
Analysis of variance (ANOVA) for response surface quadratic model (benzoate and sorbate).

Source	Sum of squares	d.f.[a]	Mean square	*F*-value[b]	*p*-value[c] prob > *F*
Model	4.281E+7	14	3.058E+6	169.64	< 0.0001 (significant)
A	211.7	1	211.7	0.012	0.9151
B	2.335E+5	1	2.335E+5	12.95	0.0026
C	9.251E+5	1	9.251E+5	51.32	< 0.0001
D	3.285E+7	1	3.285E+7	1822.5	< 0.0001
AB	4.95	1	4.95	2.746E−4	0.9870
AC	25688.08	1	25,688.08	1.43	0.2511
AD	1389.43	1	1389.43	0.077	0.7851
BC	94.6	1	94.6	5.247E−3	0.9432
BD	2.691E+5	1	2.691E+5	14.93	0.0015
CD	9.589E+5	1	9.589E+5	53.19	< 0.0001
A^2	22,522.8	1	22,522.8	1.25	0.2812
B^2	3083.3	1	3083.3	0.17	0.6850
C^2	13,792.3	1	13,792.3	0.77	0.3955
D^2	3.950E+5	1	3.950E+5	21.92	0.0003
Residual	2.704E+5	15	18,026.10		
Lack of fit	2.002E+5	10	20,016.75	1.43	0.3651 (not significant)
Pure error	70,224.05	5	14,044.81		
Cor total	4.308E+7	29			

[a] Degrees of freedom.
[b] Test for comparing model variance with residual (error) variance.
[c] Probability of seeing the observed *F*-value if the null hypothesis is true.

According to the design, each of the four factors (A, B, C and D) was studied at five levels (Table 1). For each of the five studied variables, high and low set points were selected to construct an orthogonal design (Table 1). The design included six replicates of the central point. Hence, the employed design consisted of 30 randomly performed experiments. Three replicate extractions and quantitative determinations were performed for each experiment. One type of yogurt drink was used for the optimization process, and the sum of the peak areas was assumed as the HPLC response to evaluate the extraction efficiency of sorbate and benzoate. The response surface plots describing the design and the modeled CCD data were obtained through statistical analysis. The RSM illustrates the relationship between the parameters and responses graphically, and is used to obtain an exact optimum (Fig. 3). To analyze the data and the design of the experiment, the software package Design-Expert 8.0.5 (Minneapolis, USA) was employed.

3. Results and discussion

The optimum separation and detection of benzoate and sorbate was achieved by the optimization of four variables which could affect the extraction yield in the DLLME procedure: the volumes of extraction and disperser solvent, salt and pH effect. The response surface model was used for response optimization of the four factors. The extraction solvent may have a higher or lower density than water, and should have a high efficiency for extracting the desired compounds and low solubility in water. 1-octanol was selected as the extraction solvent because extraction solvents with density higher than water add additional steps to the extraction procedure (evaporation and dissolution of the residue analyte into a polar solvent) since they have a non-polar nature and therefore cannot be injected into the reverse HPLC. The disperser solvent should be miscible with both water and the extraction solvent. Three organic solvents, methanol, ethanol and acetone, were tested to select the best disperser solvent for DLLME of benzoate and sorbate from yogurt drink samples. The findings showed that among the three tested organic solvents, ethanol was the most efficient compared to others under the same experimental conditions.

3.1. Optimization of DLLME method

This step finds the optimal values for the significant variables to obtain the maximum response. After performing some

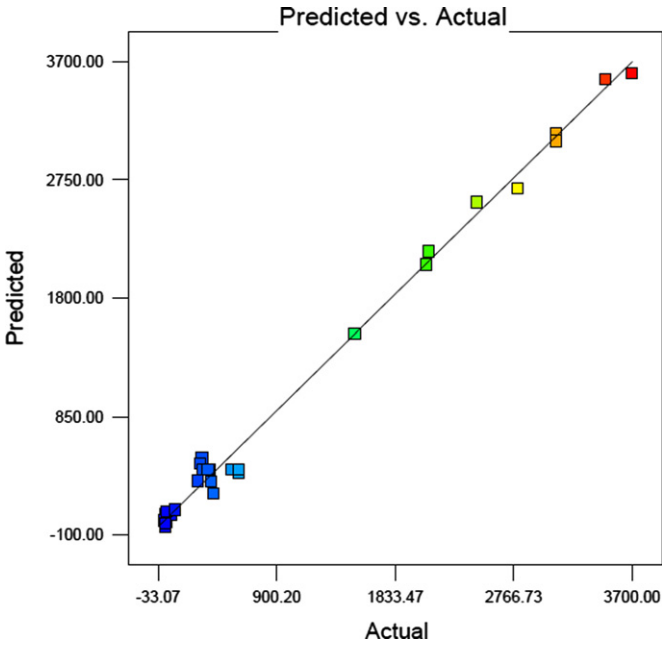

Fig. 1. The predicted responses versus the observed responses.

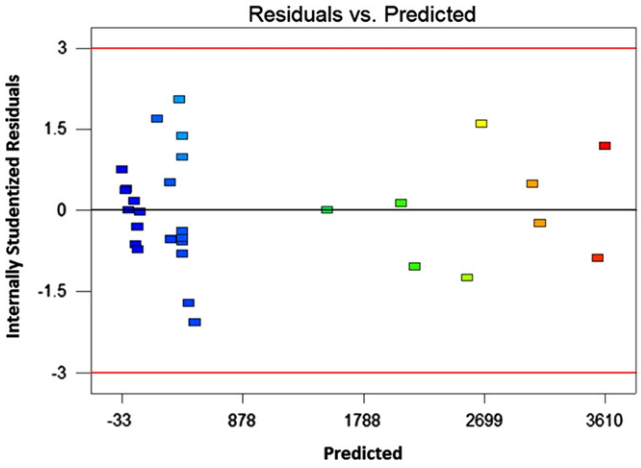

Fig. 2. A plot of the internally studentized residuals versus the predicted response.

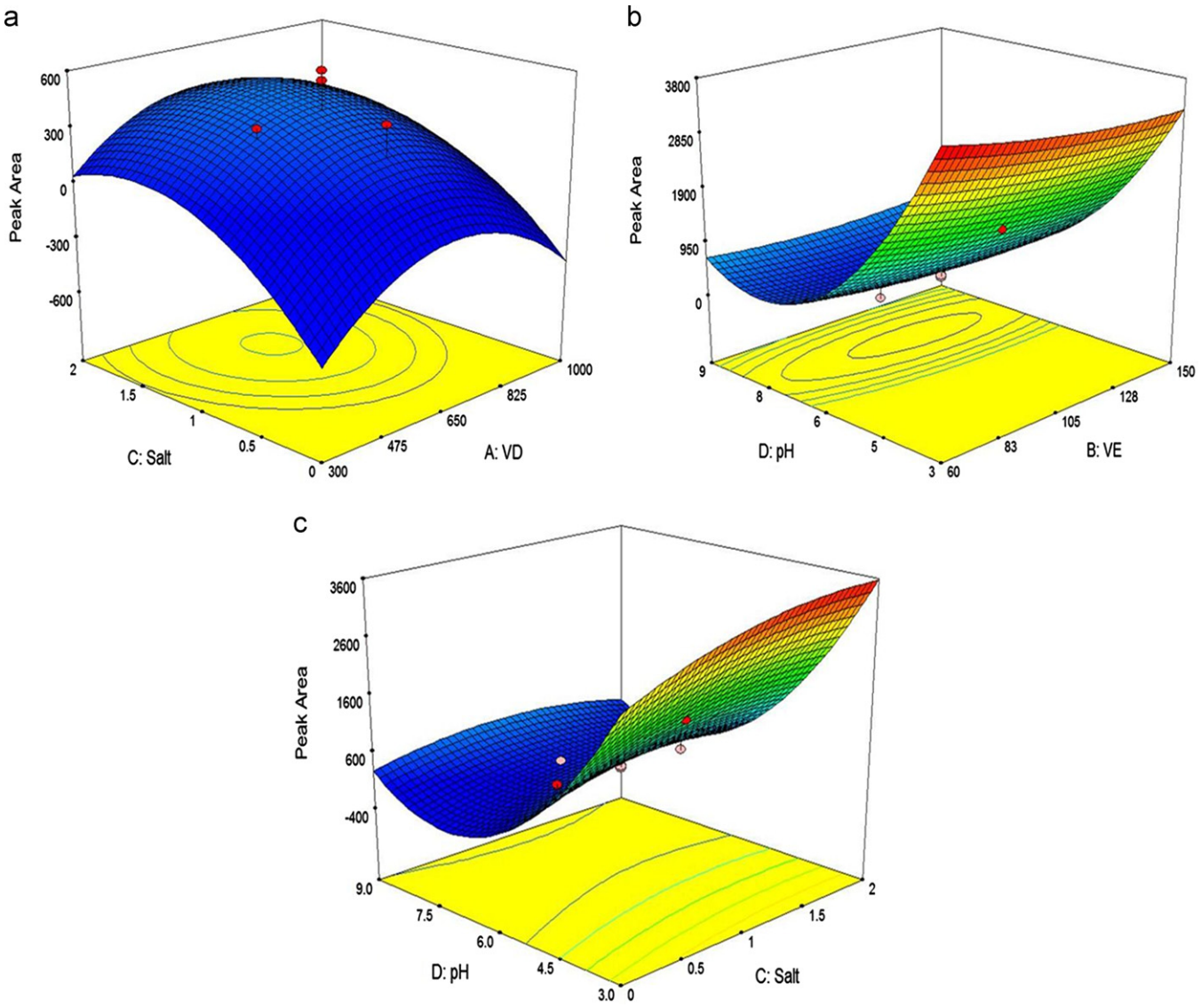

Fig. 3. Response using the central composite design obtained by plotting: (a) salt versus volume of dispersive solvent; (b) pH versus volume of extraction solvent and (c) pH versus salt.

Table 3
Figures of merit for yogurt drink using the DLLME method.

Analyte	R^2	RSD (%)	Recovery (%)	LOD (ng mL^{-1})	LOQ (ng mL^{-1})	Enrichment factor
Benzoate	0.9920	4.96	91.25	0.2	0.06	162
Sorbate	0.9907	4.58	105	0.5	0.15	181

preliminary experiments, the extraction conditions were optimized using the CCD. Effective parameters such as disperser solvent volume (ethanol), organic solvent volume (1-octanol), salt amount and pH of the aqueous phase were included in the design (Table 1). A quadratic model was fitted to the obtained data. This model was used to predict the response at any point, even those not included in the design. Eq. (1) shows the RSM model in terms of coded values for the sum of benzoate and sorbate:

$$R = +423.34 + 3.58A - 118.96B + 236.78C - 1411.05D - 0.56AB - 40.07AC - 9.32AD + 2.43BC + 129.68BD - 244.81CD - 367.88A^2 + 136.12B^2 - 287.88C^2 + 1540.72D^2 \quad (1)$$

where R is the peak area for benzoate and sorbate as a function of disperser solvent volume (A), extraction solvent volume (B), salt amount (C) and pH (D). In addition to describing the linear effects of each factor on the response, CCD explains the interaction and quadratic effects of the variables. To evaluate the significance of each factor and interaction terms, analysis of variance (ANOVA) was used (Table 2). The quality of model fitting was represented by the coefficient of determination (R^2 and adjusted-R^2). R^2 of 0.9937 and an adjusted R^2 of 0.9879 for benzoate and sorbate showed good agreement between the experimental data and the fitted model, and indicated that the model had a high potential for predicting the response. The ANOVA summary showed that the model was significant, with a p-value less than 0.0001 and F-value of 169.64. A lack-of-fit P value of 0.3651 implies that the lack of fit is not significantly associated to the pure error. The parameters of disperser solvent volume (A), extraction solvent volume (B), salt amount (C) and pH (D) had significant linear effects on the response. The effects of the amount of salt and pH factors on the response were also significant. The interactive effects of disperser solvent volume and salt amount (AC term), volume of extraction solvents and pH (BD term) and salt and pH (CD term) were significant. Fig. 1 depicts the predicted versus

Table 4
Dynamic linear range (DLR), correlation coefficient (R^2), repeatability (RSD %), recovery and limit of detections (LOD) for the analysis of benzoate and sorbate in food samples with other methods and comparison with proposed methods.

Method	Sample type	Compounds	DLR (μg mL^{-1})	R^2	RSD (%)	Recovery (%)	LOD (ng mL^{-1})
HS/SPME/GC-FID [31][a]	Food dressing	Benzoate	0.02–40	0.9994	8.63	94.10	1.22
		Sorbate	0.02–40	0.9986	1.70	103.0	2
HS/LPME/CE [15][b]	Soy sauce and soft drinks	Benzoate	3–1000	0.9994	2.5	105	30
		Sorbate	3–1000	0.9973	3.2	94.7	70
LLE/CE [14][c]	Beverage	Benzoate	4–45	0.9992	2.84	97.9	900
		Sorbate	2–20	0.9994	2.57	98.2	300
LLE/HPLC [12][d]	Foodstuffs	Benzoate	5–120	–	2.76	112	500
		Sorbate	1–75	–	2.90	105	100
DLLME/HPLC[e]	Yogurt drinks	Benzoate	0.001–10	0.9920	4.96	91.25	0.06
		Sorbate	0.001–10	0.9907	4.58	105	0.15

[a] Headspace/solid phase microextraction/gas chromatography-flame ionization detector.
[b] Headspace/liquid phase microextraction/capillary electrophoresis.
[c] Liquid–liquid extraction/capillary electrophoresis.
[d] Liquid–liquid extraction/High performance liquid chromatography.
[e] The proposed method.

Table 5
Analytical results of benzoate and sorbate in Iranian yogurt drinks by DLLME–HPLC method.

Sample	Benzoate (μg mL^{-1})	Sorbate(μg mL^{-1})
1	11.4 ± 0.5[a]	45 ± 2
2	40 ± 1.6	ND[b]
3	11.6 ± 0.5	ND
4	8.1 ± 0.4	ND
5	7.6 ± 0.3	12.5 ± 0.4
6	17.2 ± 0.8	ND
7	13.7 ± 0.7	ND
8	2.4 ± 0.1	ND
9	8.1 ± 0.4	3.4 ± 0.1
10	18.2 ± 0.8	ND

[a] Mean value ± standard deviation (n=3).
[b] ND: not detected.

actual responses. Most plots were scattered in close proximity with the line, which indicates a good correlation between predicted and actual responses and in turn, the good fit of the proposed quadratic model. Fig. 2 shows the residuals versus the predicted responses. The residual plots were scattered randomly, indicating that the variance of the experimental measurements is constant for all values of Y.

The next step was to find the optimum value for each factor to achieve the maximum response. The response-surface plots show the results of the extraction recovery modeling for some of the significant factors. The curvatures of the plots indicate the interaction between the factors. Three-dimensional graphs were used to evaluate the interactive effect of the two variables on the response. Fig. 3a depicts the response surface obtained by plotting salt versus disperser solvent volume with the volume of aqueous sample fixed at 10 mL. The extraction efficiency improved with the increase of the amount of salt while increasing the disperser solvent until it reaches 450 μL. Thus, the salt amount has a positive effect on the response. The maximum response was obtained at 2 g NaCl and 450 μL disperser solvent.

Fig. 3b shows that concurrently decreasing pH and extraction solvent volume enhances the performance of the microextraction process. The maximum response was obtained at a pH of 3 and a 60 μL volume of extraction solvent. The increased efficiency was due to the reduction in the volume of the extraction solvent to 60 μL leading to an increase in the concentration of the analytes (benzoate and sorbate). The pH and salt terms in the model equation had a larger coefficient as seen in Fig. 3c. Their interaction was significant and simultaneously changing them (decreasing the pH to 3 and increasing the salt concentration to 2 g) leads

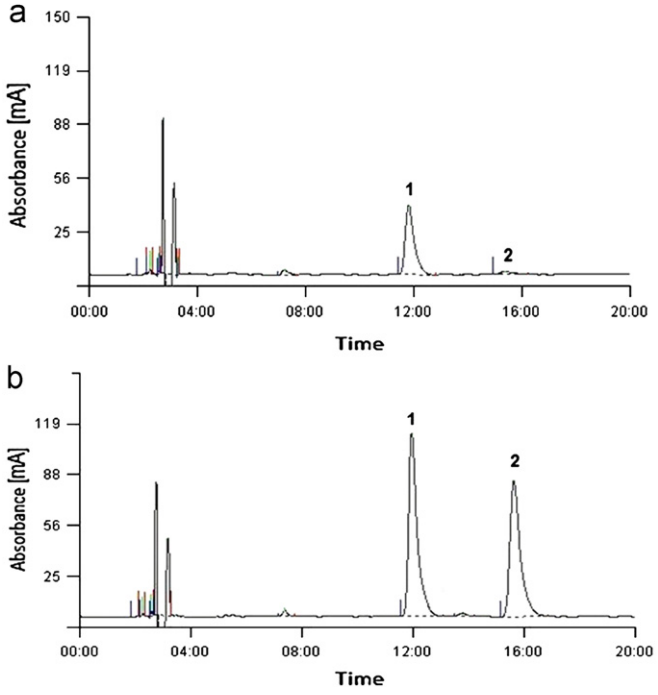

Fig. 4. The chromatogram obtained by DLLME–HPLC for a yogurt drink under optimum conditions: (a) non-spiked and (b) spiked with 0.1 μg mL^{-1} of benzoate (1) and sorbate (2).

to enhanced extraction performance. A decrease of the extraction efficiency associated with a decrease in pH is evident. The negative effect of pH may be because benzoate and sorbate formed an ionic structure with the decrease in pH, and in these conditions, extraction of the analyte was more effective in an organic solvent.

3.2. Quantitative analysis of benzoate and sorbate

To evaluate the analytical performance of DLLME–HPLC method, linearity, repeatability, recovery, limit of detection (LOD) and limit of quantification (LOQ) were investigated under optimal conditions (Table 3). Calibration curves of benzoate and sorbate (standard solution) were linear over the range of 0.001–10 μg mL^{-1}. The curves were constructed by the peak area of benzoate and sorbate versus their concentrations for nine points. The square of the correlation coefficient (R^2) was 0.9920 for

benzoate and 0.9907 for sorbate. The comparative peak area, calculated from six replicate extractions from one yogurt drink sample, was employed to estimate the repeatability, and was shown as the relative standard deviation percentage (RSD %). As Table 3 shows, the RSD % was 4.96 and 4.58 for benzoate and sorbate, respectively ($n=7$). The enrichment factor was expressed as the ratio of the final concentration of the analyte in the organic solvent to its concentration in the original solution. The enrichment factors of the proposed method were 162 and 181 for benzoate and sorbate, respectively. The recovery for each compound was determined for the DLLME procedure by comparing the amount of analyte added to a yogurt drink sample with the concentration left after the procedure. The values of recovery using the proposed method were 91.25% for benzoate and 105% for sorbate. The LOD and LOQ (based on signal-to-noise ratios of 3 and 10, respectively) when using the optimized conditions in DLLME–HPLC were respectively 0.06 and 0.21 ng mL^{-1} for benzoate and 0.15 and 0.5 ng mL^{-1} for sorbate (Table 3). The DLLME–HPLC results for extraction and determination of benzoate and sorbate compared with the corresponding literature data using other methods show that the proposed method has a better detection limit, satisfactory linear behavior with correlation coefficients higher than 0.99, comparable repeatability and good recovery in yogurt drink samples (Table 4).

3.3. Application to real samples

To evaluate the reliability of the proposed method, ten Iranian yogurt drinks (Doogh) were purchased from different shops in Tehran (Iran) city market and tested with the optimized method. The concentrations of the analytes were calculated from the linear regression equations of the standard curves going through the DLLME–HPLC process. The results are summarized in Table 5. Fig. 4 shows the chromatograms obtained by DLLME–HPLC for a yogurt drink sample when (a) non-spiked and (b) spiked with sorbate and benzoate at a level of 0.1 μg mL^{-1}. A clean separation and good chromatogram is readily achieved without the presence of matrix interference.

4. Conclusion

In this study, efficient, fast and reliable extraction of sorbate and benzoate from yogurt drink was achieved using the DLLME procedure. The HPLC system allowed an efficient analysis with a considerable gain in the speed of chromatographic analysis, because HPLC has minimal preparation requirement and does not require derivatization. RSM based on central composite design was used to optimize effective parameters on the performance of microextraction process which determines the interaction and quadratic effects of variables. Thus, it elucidates the interactive effects of variables on each other and the efficiency of the microextraction process and allows to select the best experimental conditions with the minimum number of experiments. Low consumption of the solvent (20 μL per analysis), high enrichment factor, low cost, high recovery ($>90\%$ efficiency), repeatability ($<5\%$ RSD), short extraction time, good precision, no matrix interference and good merit figures compared to other methods are clear advantages of the proposed method. The developed method was successfully used for the routine monitoring of benzoate and sorbate in various types of real yogurt drinks.

Acknowledgment

This study has been supported by the National Nutrition & Food Technology Research Institute of Iran. We gratefully acknowledge their assistance.

References

[1] D. Barbano, K. Wojciechowski, J. Lynch, J. Dairy Sci. 93 (2010) 6000–6011.
[2] P. Jüngel, S. de Koning, U.A.T. Brinkman, E. Melcher, J. Chromatogr. A 953 (2002) 199–205.
[3] L. Wang, X. Zhang, Y. Wang, W. Wang, Anal. Chim. Acta 577 (2006) 62–67.
[4] G. Shanmugam, B.R. Ramaswamy, V. Radhakrishnan, H. Tao, Microchem. J. 96 (2010) 391–396.
[5] K.M. Ma, C.M. Chan, S.W.C. Chung, Y.Y. Ho, Y. Xiao, Food Addit. Contam. 26 (2009) 12–16.
[6] M. Mroueh, D. Issa, J. Khawand, B. Haraty, A. Malek, Z. Kassaify, I. Toufeili, J. Food Agr. Environ. 6 (2008) 62–66.
[7] F.J.M. Mota, I.M. Ferreira, S.C. Cunha, M. Beatriz, P. Oliveira, Food Chem. 82 (2003) 469–473.
[8] H.M. Pylypiw, M.T. Grether, J. Chromatogr. A 883 (2000) 299–304.
[9] B.R. Ramaswamy, J.W. Kim, T. Isobe, K.H. Chang, A. Amano, T.W. Miller, F.P. Siringan, S. Tanabe, J. Hazardous Mater. 192 (2011) 1739–1745.
[10] V.A. Lozano, J.M. Camiña, M.S. Boeris, E.J. Marchevsky, Talanta 73 (2007) 282–286.
[11] C. Lino, A. Pena, Food Chem. 121 (2010) 503–508.
[12] B. Saad, M.F. Bari, M.I. Saleh, K. Ahmad, M.K.M. Talib, J. Chromatogr. A 1073 (2005) 393–397.
[13] M. Thomassin, E. Cavalli, Y. Guillaume, C. Guinchard, J. Pharm. Biomed. 15 (1997) 831–838.
[14] A.C.O. Costa, L. da Silva Perfeito, M.F.M. Tavares, G.A. Micke, J. Chromatogr. A 1204 (2008) 123–127.
[15] D.S. Ling, H.Y. Xie, Y.Z. He, W.E. Gan, Y. Gao, J. Chromatogr. A 1217 (2010) 7807–7811.
[16] R.A. Frazier, E.L. Inns, N. Dossi, J.M. Ames, H.E. Nursten, J. Chromatogr. A 876 (2000) 213–220.
[17] R. Wei, W. Li, L. Yang, Y. Jiang, T. Xie, Talanta 83 (2011) 1487–1490.
[18] C. Campos, L.I.A.N. Gerschenson, S.M. Alzamora, J. Chirife, J. Food Sci. 56 (1991) 863 -863.
[19] M.Z. Martin, N. Labbé, T.G. Rials, S.D. Wullschleger, Spectrochim. Acta B: At. Spectrosc. 60 (2005) 1179–1185.
[20] T. Ohtsuki, K. Sato, N. Sugimoto, H. Akiyama, Y. Kawamura, Talanta 734 (2012) 54–61.
[21] E. Ibrahim, R. Lippincott, L. Brenner, I. Suffet, R. Hannah, J. Chromatogr. A 393 (1987) 237–253.
[22] I. Techakriengkrai, R. Surakarnkul, J. Food Comp. Anal. 20 (2007) 220–225.
[23] M. Gonzalez, M. Gallego, M. Valcarcel, J. Chromatogr. A 823 (1998) 321–329.
[24] M. Moors, C. Teixeira, M. Jimidar, D. Massart, Anal. Chim. Acta 255 (1991) 177–186.
[25] F. Pena-Pereira, R.M.B.O. Duarte, A.C. Duarte, Cent. Eur. J. Chem. 10 (2012) 1–17.
[26] H. Kataoka, A. Ishizaki, Y. Nonaka, K. Saito, Anal. Chim. Acta 655 (2009) 8–29.
[27] S. Risticevic, V.H. Niri, D. Vuckovic, J. Pawliszyn, Anal. Bioanal. Chem. 393 (2009) 781–795.
[28] S. Risticevic, H. Lord, T. Górecki, C.L. Arthur, J. Pawliszyn, Nat. Protocols 5 (2010) 122–139.
[29] M. Dehghani Mohammad Abadi, N. Ashraf, M. Chamsaz, F. Shemirani, Talanta 99 (2012) 1–12.
[30] A. Spietelun, M. Pilarczyk, A. Kloskowski, J. Namieśnik, Talanta 87 (2011) 1–7.
[31] C. Dong, Y. Mei, L. Chen, J. Chromatogr. A 1117 (2006) 109–114.
[32] T.A. Kokya, K. Farhadi, A.A.M. Kalhori, Food Anal. Method 5 (2012) 351–358.
[33] T.J. Yang, F.J. Tsai, C.Y. Chen, T.C.C. Yang, M.R. Lee, Anal. Chim. Acta 668 (2010) 188–194.
[34] N. Alizadeh, N. Saburi, S.E. Hosseini, Food Control 28 (2012) 315–320.
[35] M. Rezaee, Y. Assadi, M.R. Milani Hosseini, E. Aghaee, F. Ahmadi, S. Berijani, J. Chromatogr. A 1116 (2006) 1–9.
[36] A.N. Anthemidis, K.I.G. Ioannou, Talanta 80 (2009) 413–421.