Simultaneous Determination of Sweeteners and Preservatives in Preserved Fruits by Micellar Electrokinetic Capillary Chromatography

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Abstract

A micellar electrokinetic capillary method for the simultaneous determination of the sweeteners dulcin, aspartame, saccharin, and acesulfame-K and the preservatives sorbic acid; benzoic acid; sodium dehydroacetate; and methyl-, ethyl-, propyl-, isopropyl-, butyl-, and isobutyl-*p*-hydroxybenzoate in preserved fruits is developed. These additives are ion-paired and extracted using sonication followed by solid-phase extraction from the sample. Separation is achieved using a 57-cm fused-silica capillary with a buffer comprised of 0.05M sodium deoxycholate, 0.02M borate-phosphate buffer (pH 8.6), and 5% acetonitrile, and the wavelength for detection is 214 nm. The average recovery rate for all sweeteners and preservatives is approximately 90% with good reproducibility, and the detection limits range from 10 to 25 µg/g. Fifty preserved fruit samples are analyzed for the content of sweeteners and preservatives. The sweeteners found in 28 samples was aspartame (0.17–11.59 g/kg) or saccharin (0.09–5.64 g/kg). Benzoic acid (0.02–1.72 g/kg) and sorbic acid (0.27–1.15 g/kg) were found as preservatives in 29 samples.

Introduction

Artificial sweeteners and preservatives are often incorporated into preserved fruits in order to increase product quality and shelf life. Based on public concerns with the harm of food additives to human health, a method for the separation and identification of these compounds in preserved fruits is deemed necessary. The most common methods for determining sweeteners or preservatives in foods involve high-performance liquid chromatography using reversed-phase, gradient elution (1,2) or ion-paired chromatography (HPLC–PIC) (3–5). These methods depend highly on organic solvents as mobile phases and use buffers at low pH levels to suppress the ionization of the food additives for the separation (6). However, analysis times for these methods are usually long. Recently, capillary electrophoresis (CE) techniques, such as capillary zone electrophoresis (CZE) and micellar electrokinetic capillary chromatography (MEKC), have been used to analyze preservatives (6–9) and sweeteners (10) in foods. With the same order of reliability as HPLC–PIC, MEKC is rapidly gaining acceptance and has replaced HPLC–PIC as the analytical method of choice because of its greater resolution, faster analysis, and lower cost (9,11–12).

One notable characteristic of the many methods reported for the analysis of food additives by means of CZE or MEKC (6-12) is that most deal with either sweeteners or preservatives only. There are no reports of simultaneous analysis by CE of the considerable number of sweeteners and preservatives used. Additionally, preserved fruit samples consisting of high sugar and salt are not easily analyzed using CE, and the various pH levels of different samples are believed to influence the extraction recoveries of each additive. Therefore, developing an appropriate sample preparation method is necessary to minimize the influences of sugar, salt, and acid, which are used in the preservation process of fruit.

In this study, the 4 artificial sweeteners (dulcin, aspartame, saccharin, and acesulfame-K) and 9 preservatives (sorbic acid; benzoic acid; dehydroacetic acid; and methyl-, ethyl-, propyl-, isopropyl-, butyl-, and isobutyl-*p*-hydroxybenzoate) that are usually found in preserved fruits are separated simultaneously and quantitated using a rapid MEKC procedure. Many MEKC parameters are examined, and a solid-phase extraction (SPE) procedure for the preparation of the sample is described. The contents of these additives in 50 samples of preserved fruit are determined by the procedures mentioned.

Experimental

Chemicals and reagents

All solvents, sodium chloride, and sodium tetraborate were

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Table I. Variation of the Absorbance of Sweeteners and Preservatives Detected at Different Wavelengths

	Detected wavelength (nm)			
	200	214	254	280
Compounds*	Absorbance			
Dulcin	1.16849	0.43986	0.33461	0.05123
Aspartame	0.63569	0.31660	n.d.‡	n.d.
Methyl-PHBA	1.59146	0.97190	0.83059	0.84262
Ethyl-PHBA	1.68563	0.97824	0.84251	0.74559
Isopropyl-PHBA	1.56442	0.96483	0.91149	0.65955
Propyl-PHBA	1.58273	0.99486	0.93996	0.67136
DHA-Na	0.98980	0.95497	0.41181	0.62854
Sorbic acid	0.60134	0.79725	3.02439	0.50820
Isobutyl-PHBA	1.79174	1.14510	1.08335	0.61675
Butyl-PHBA	1.83797	1.19804	1.11094	0.62126
Benzoic acid	2.70701	1.00014	0.13443	0.02503
Saccharin	2.69283	1.99019	0.11507	0.09701
Acesulfame-K	0.30968	0.78775	0.42480	n.d.

*The concentration for each compound is 20 µg/mL.
†The average of 3 injections.
‡n.d., not detected.

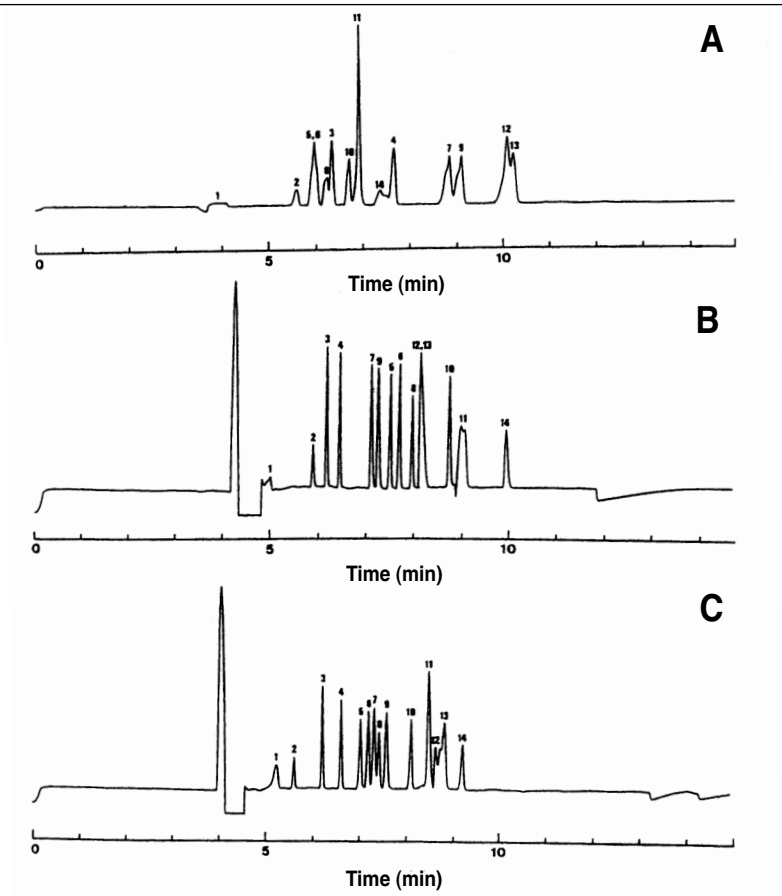

Figure 1. Comparison of electropherograms using different buffer systems for the simultaneous separation of sweeteners and preservatives. Buffers consisted of 0.05M SDS (A); SC (B); and 0.05M SDC, 0.01M potassium dihydrogenphosphate, and 0.01M sodium tetraborate at pH 8.6 (C). Peaks: 1, dulcin; 2, aspartame; 3, methyl-PHBA; 4, ethyl-PHBA; 5, dehydroacetic acid; 6, internal standard (4-ethylbenzoic acid); 7, isopropyl-PHBA; 8, sorbic acid; 9, propyl-PHBA; 10, benzoic acid; 11, saccharin; 12, isobutyl-PHBA; 13, butyl-PHBA; and 14, acesulfame-K.

obtained from E. Merck (Schuchardt, Germany). Sodium dehydroacetate (DHA-Na), potassium sorbate, sodium benzoate, the *p*-hydroxybenzoates (PHBAs) (methyl-, ethyl-, propyl-, isopropyl-, butyl-, and isobutyl-*p*-hydroxybenzoate), sodium saccharin, and 4-ethylbenzoic acid were obtained from TCI Chemical Co. (Tokyo, Japan). Dulcin and phosphoric acid were obtained from Sigma (St. Louis, MO). Aspartame, sodium deoxycholate (SDC), sodium cholate (SC), sodium dodecyl sulfate (SDS), and cetyltrimethylammonium chloride (CTA) were obtained from Nacalai (Koyto, Japan). Acesulfame potassium was obtained from Hoechst (Frankfurt, Germany). Water used in all experiments was purified by a Millipore Milli-QSP60 system (Bedford, MA). Sep-Pak C_{18} cartridges that contained 360 mg packing material were obtained from Waters Co. (Milford, MA).

Standards for ultraviolet spectrometry

Stock solutions of dulcin, aspartame, saccharin, and acesulfame-K and also sorbic acid, benzoic acid, dehydroacetic acid, and the PHBAs were prepared at a concentration of 1 µg/mL by dissolving them in 50% aqueous methanol. Standard solutions at a concentration of 2 µg/mL were prepared by diluting stock solutions with 20% aqueous methanol. Each standard solution was adjusted to pH 3 using 1M hydrochloric acid or 1M sodium bicarbonate.

Standards for MEKC

Working standards of select concentrations (5, 10, 20, 30, and 40 mg/mL) were prepared by diluting stock solutions with 20% aqueous methanol containing 0.5% sodium chloride. An internal standard of 4-ethylbenzoic acid was used at a concentration of 10 µg/mL for the quantitation of sweeteners and preservatives.

Buffers for MEKC

A CTA buffer, SDS buffer, SC buffer, SDC buffer, and SDC with acetonitrile modified buffer were used for parameter determination and preserved fruits analysis. The CTA buffer (5mM) was prepared by dissolving 0.8 g of CTA in 500 mL water. The SDS buffer (0.05M) was prepared by dissolving 1.44 g of SDS in 100 mL of a 0.02M sodium tetraborate–potassium dihydrogenphosphate solution. The pH level of the buffers was 8.6. The SC buffer, SDC buffer, and SDC with acetonitrile modified buffer were pre-

pared using the same method. All standards and buffers were filtered through a 0.45-µm cellulose acetate filter unit prior to use.

Sample preparation

Preserved fruits were obtained from local commercial outlets in Taipei. A method described by Chen and Fu (5) was modified to extract food additives from preserved fruits using SPE. Ten grams of preserved fruit was finely ground with a grinder. Two grams of the ground sample was mixed with 5 mL of a 0.2M potassium dihydrogenphosphate buffer at pH 3.0, 5 mL of a 5mM CTA ion-pairing solution, and 40 mL of water. The mixture was sonicated for 20 min and centrifuged at 3000 rpm for 10 min. Ten milliliters of the upper solution was poured into a Sep-Pak C_{18} cartridge, which had been previously activated with 10 mL of methanol, 10 mL of water, and 2 mL of the 5mM CTA ion-pairing solution. The cartridge was washed with 10 mL of water and then eluted with 2 mL of methanol. The eluate was collected into a 10-mL volumetric flask and diluted to volume with 1 mL of a 5% sodium chloride solution and water. The final solution was then filtered through a 0.45-µm cellulose acetate filter disk before analysis.

Apparatus

The ultraviolet (UV) spectra and the adsorption of sweeteners and preservatives at different wavelengths were determined using a Milton Roy Spectronic Array 3000 spectrophotometer (Spectronic Instrument Inc., Rochester, NY). MEKC analysis was performed with a 57-cm × 75-µm-i.d. fused-silica capillary (Beckman eCAP capillary tubing, Beckman Instruments, Fullerton, CA) with an effective length of 50 cm to the detector. A Beckman P/ACE system 5500 operated at 20 kV and 25°C was used for the analysis.

CE Conditioning

Before each analysis, the capillary was rinsed first with 0.1M sodium hydroxide for 4 min, followed by water and a running buffer solution for 2 min each.

Quantitation

The internal standard was added to the sample before extraction. The linear

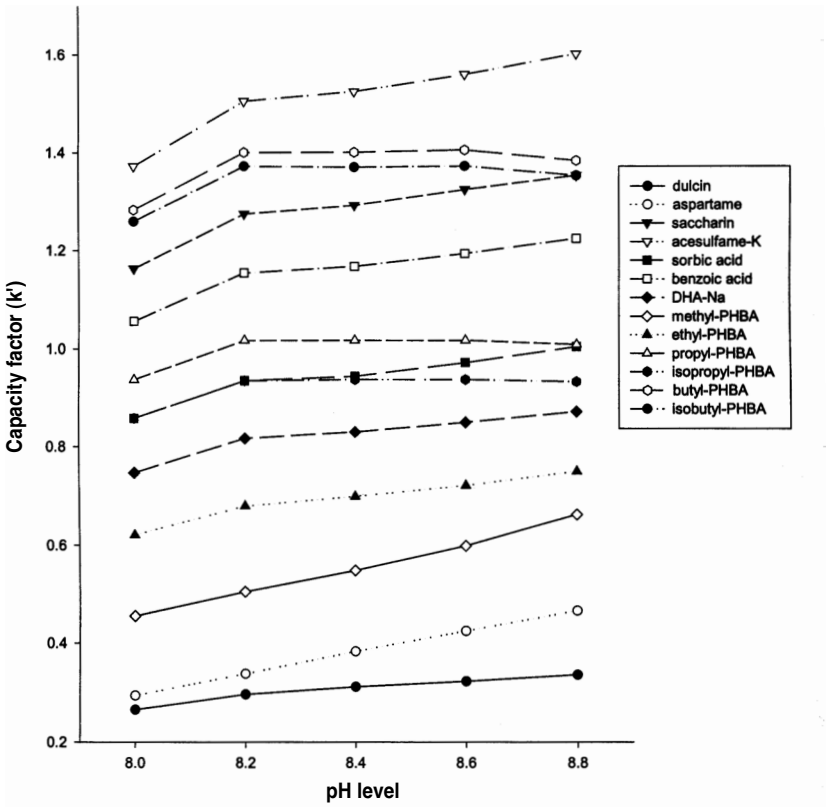

Figure 2. Effects of pH value of the buffer system on the k' values of sweeteners and preservatives. A buffer comprising of 0.05M sodium deoxycholate and 0.01M borate-phosphate was used for MEKC.

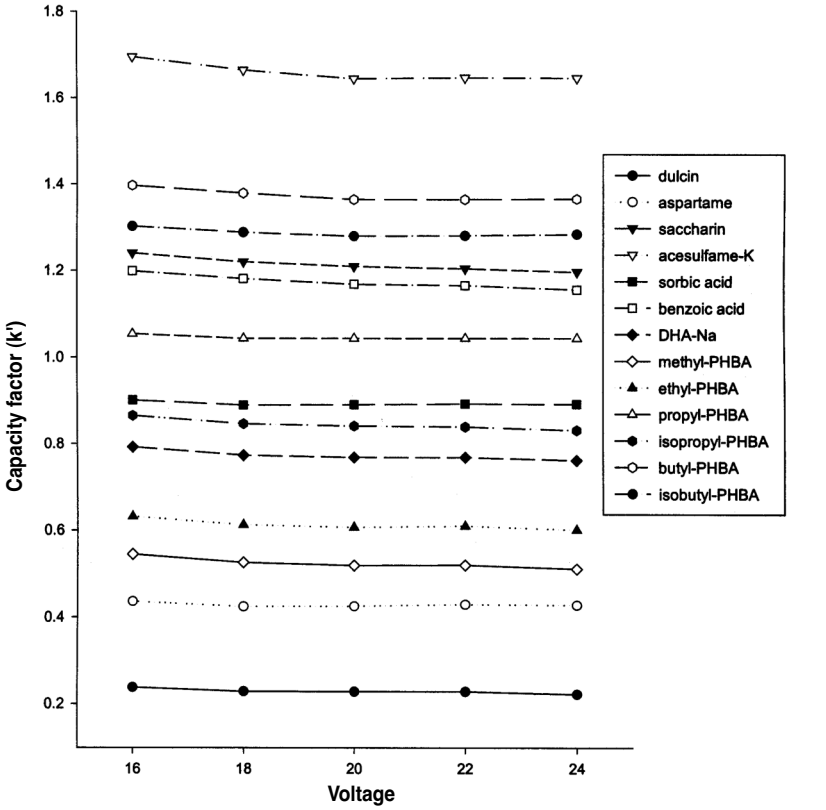

Figure 3. Effects of voltages on the k' values of sweeteners and preservatives. A buffer comprising of 0.05M

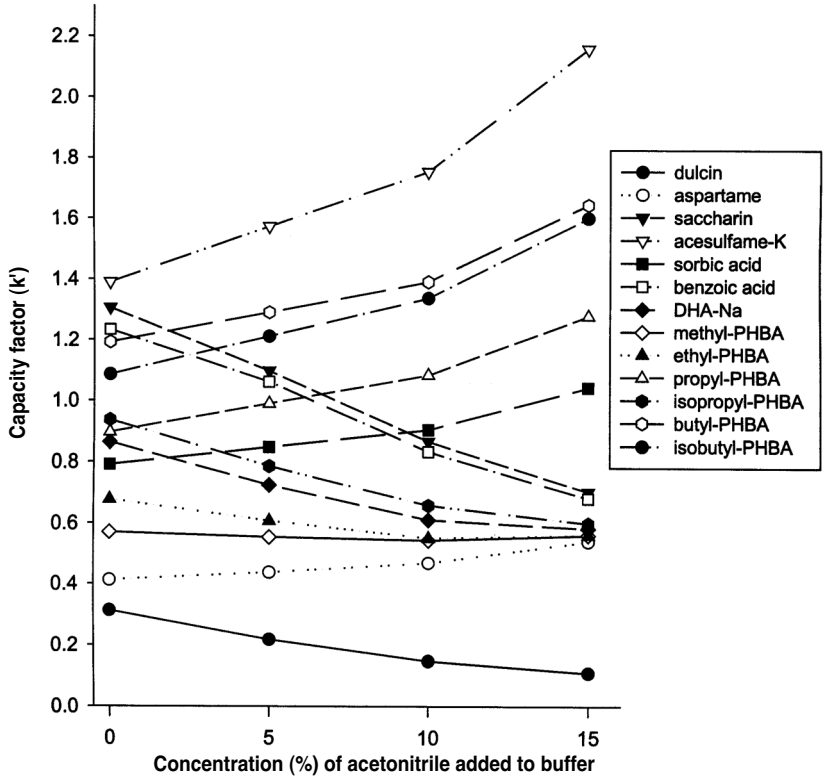

Figure 4. Effects of different concentrations of acetonitrile as the modifier was added to the buffer on the k' values of sweeteners and preservatives. A buffer comprising of 0.05M sodium deoxycholate and 0.01M borate-phosphate at pH 8.6 was used for MEKC.

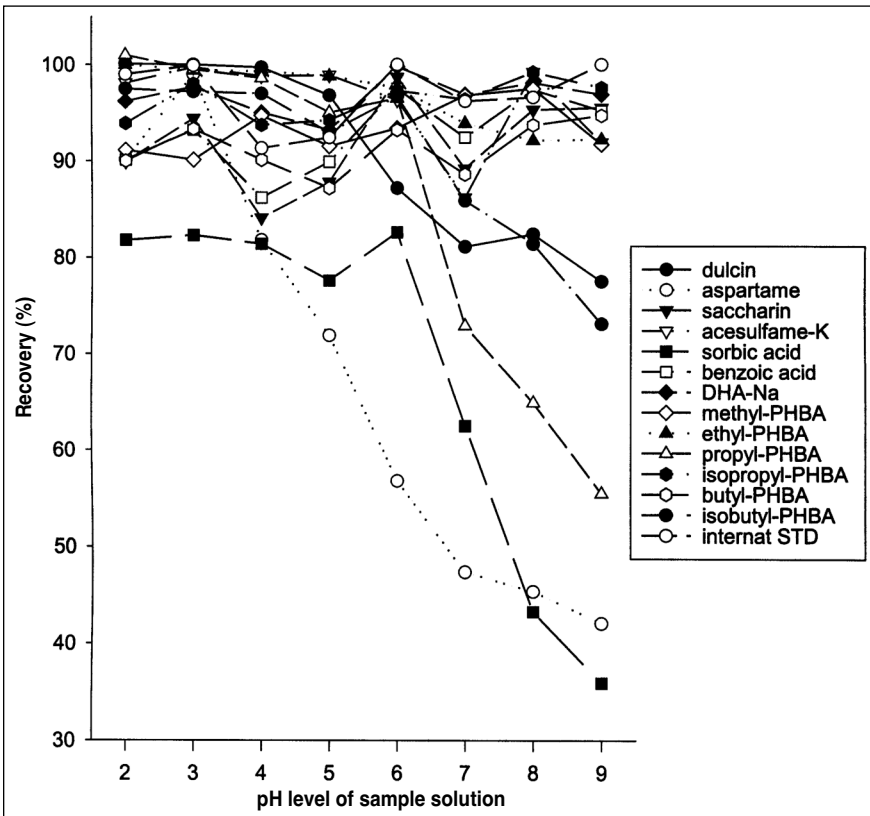

Figure 5. Effects of the pH value of the sample on the recoveries of sweeteners and preservatives. A buffer comprising of 0.05M sodium deoxycholate and 0.01M borate-phosphate was used for MEKC.

regression plot of the calibration curve for each additive was constructed for quantitation. Three analyses of each sample were made, and each experiment was carried out in triplicate (n = 3). Each additive was quantitated using the following formula:

$$WS = (AS/AIS) \times WIS \times RF;$$
$$C_{additive}\ (\mu g/g) = WS/W \qquad \text{Eq. 1}$$

where AS is the adsorption of additive at 214 nm, AIS is the adsorption of the internal standard at 214 nm, WS is the weight (µg) of the additive, WIS is the weight (µg) of the internal standard, RF is the response factor of the calibration curve, and W is the weight (g) of the sample.

Results and Discussion

In order to determine the optimal conditions for the simultaneous separation of the sweeteners and preservatives, the following parameters were carefully controlled and examined: (*a*) selection of an appropriate detection wavelength, (*b*) selection of an appropriate buffer, (*c*) the addition of the modifier, and (*d*) the preparation of the sample. The separation efficiency of each additive was evaluated based on the capacity factor (k').

$$k' = (t_m - t_0) / t_0 \qquad \text{Eq. 2}$$

where t_m is the migration time of the peak and t_0 is the migration time of the solvent front or nonmigration peak.

Selection of detection wavelength

It is important but difficult to select an appropriate wavelength for the simultaneous detection of these additives with various structures. In some HPLC reports, the detection wavelength for sweeteners was 205 or 233 nm, and it was 233 or 235 nm (3–5,13–15) for preservatives. As more additives were included in this study, the selection of an appropriate wavelength had to be reinvestigated. Both the UV spectra for sweeteners and preservatives were determined (data not shown) and the adsorption of each additive at different wavelengths was measured using a spectrophotometer (Table I). The ad-

sorptions of benzoic acid and saccharin were poor at 254 and 280 nm, and aspartame had no adsorption at these wavelengths. All additives adsorbed UV at 200 and 214 nm. Although the adsorption of each additive at 200 nm seemed to be a little higher than that at 214 nm, wavelength 214 nm was finally chosen to reduce the nonstability of the baseline during analysis.

Selection of an appropriate buffer

SDS, SC, and SDC are anionic surfactants. The structural differences of these surfactants affect not only the electrophoretic behavior of a mixture's components by their different micellar structures (16), but also the resolution of the electropherogram (12). The buffers of these 3 surfactants were examined in order to provide better separation of the additives during simultaneous analysis. As seen in the electropherogram in Figure 1C, all 13 additives gave baseline separation using an SDC buffer consisting of 0.05M SDC and 0.01M sodium tetraborate–potassium dihydrogenphosphate. The SDS buffer could not resolve butyl-PHBA and isobutyl-PHBA, as well as sorbic acid and methyl-PHBA (Figure 1A). In Figure 1B, butyl-PHBA and isobutyl-PHBA overlapped, and saccharin was split by an SC buffer. Also, dulcin coeluted with electro-osmotic flow when

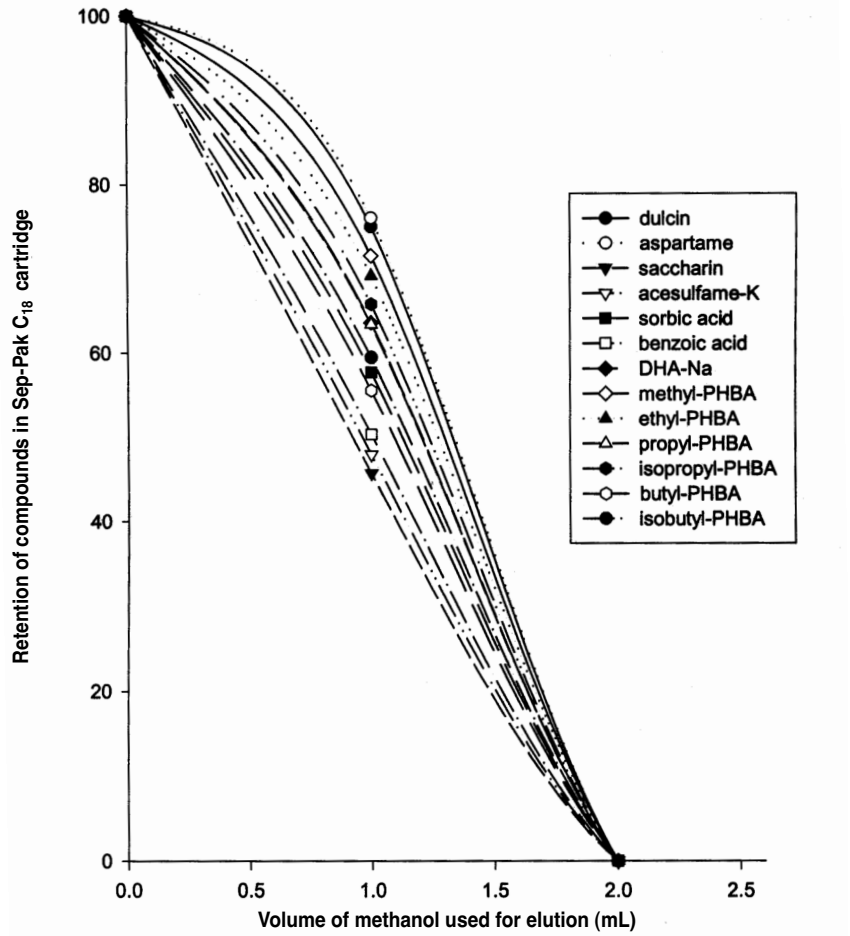

Figure 6. Elution curves of sweeteners and preservatives in a Sep-Pak C_{18} cartridge, which was measured as a fraction of its concentration in methanol effluent. $C_r = C_e / C_i$, where C_e and C_i are the amounts of the analyte in the effluent and influent.

Table II. Summary of the Analytical Characteristics of Each Additive by MEKC

Additives	Linearity (r^2)*	Reproducibility (%CV)	Recovery of spiked sample (%) Levels of standards spiked (µg/g) 250	500	1000	Detection Limit (µg/g)§
Dulcin	0.9843	0.54	107.0 ± 1.1	105.3 ± 1.7	107.1 ± 1.9	50
Aspartame	0.9995	0.71	100.2 ± 1.3	99.4 ± 4.7	92.8 ± 0.2	25
Saccharin	0.9997	1.27	97.6 ± 1.7	94.2 ± 2.6	93.8 ± 1.0	10
Acesulfame-K	0.9993	1.35	95.4 ± 0.8	90.8 ± 2.5	85.2 ± 1.6	25
Sorbic acid	0.9997	1.23	96.0 ± 0.3	94.8 ± 1.1	96.1 ± 1.2	25
Benzoic acid	0.9995	1.31	90.1 ± 1.2	96.8 ± 1.3	100.3 ± 2.4	25
DHA-Na	0.9998	1.11	92.5 ± 2.7	89.9 ± 2.0	90.2 ± 3.3	20
Methyl-PHBA	0.9976	0.61	103.6 ± 7.2	95.1 ± 4.8	100.6 ± 3.5	15
Ethyl-PHBA	0.9992	0.79	94.6 ± 0.2	93.1 ± 2.3	94.5 ± 1.4	15
Propyl-PHBA	0.9996	0.99	92.7 ± 0.5	91.8 ± 2.9	94.6 ± 2.0	15
Isopropyl-PHBA	0.9998	0.95	91.5 ± 1.2	91.2 ± 2.1	94.6 ± 1.6	15
Butyl-PHBA	0.9991	1.09	94.8 ± 1.6	98.6 ± 2.2	95.6 ± 0.7	20
Isobutyl-PHBA	0.9990	1.05	102.4 ± 2.0	94.6 ± 2.9	93.4 ± 1.0	20

* A 5-point regression of the standard curve was made for each compound.
† Obtained from 15 duplicate migrations.
‡ Average recovery percentage of the coefficients of variation obtained from triplicate injections.
§ Detection limit of additive added to the preserved fruit sample calculated as the minimum sample size that would produce a signal that is triple the short-term noise level.

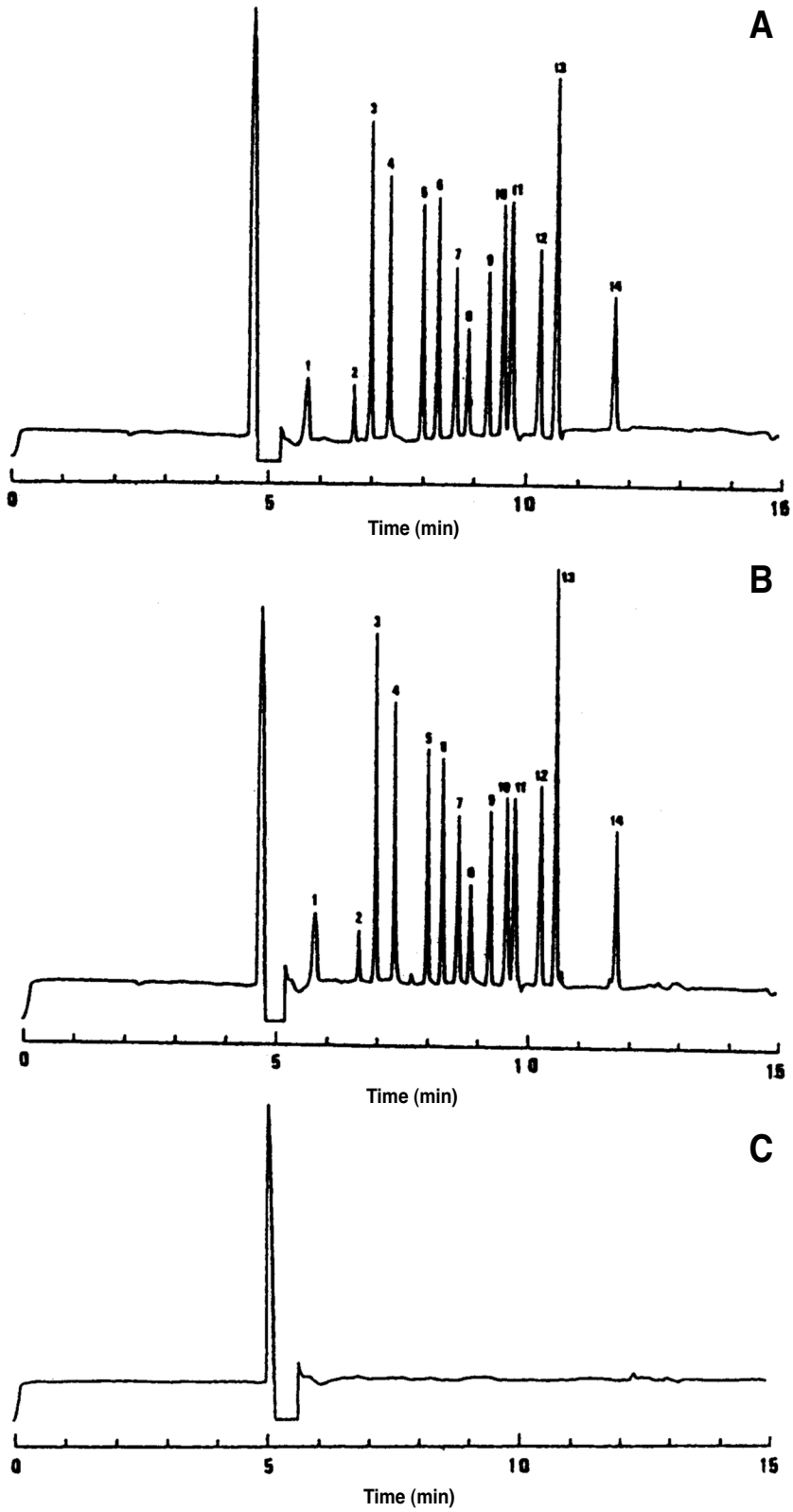

Figure 7. The recovery of electropherograms of sweeteners and preservatives: standard solution (A), preserved fruit fortified standards (B), and preserved fruit only (C). Peaks: 1, dulcin; 2, aspartame; 3, methyl-PHBA; 4, ethyl-PHBA; 5, isopropyl-PHBA; 6, propyl-PHBA; 7, dehydroacetic acid; 8, internal standard (4-ethylbenzoic acid); 9, sorbic acid; 10, isobutyl-PHBA; 11, butyl-PHBA; 12, benzoic acid; 13, saccharin; and 14, acesulfame-K.

using an SDS or SC buffer (Figure 1A and 1B). By comparing the separation performance of the 3 buffer systems, the SDC buffer was superior to the others in terms of the number of additives separated, which lead to its application in further experiments.

The appropriate pH value of the SDC buffer and the electrophoretic voltage of MEKC were examined. Figure 2 shows that the k' values for most additives were only slightly affected by increasing the pH level, and the best separation efficiency (13 additives) was achieved with a pH level of 8.6. The formation of the additives's micelle was believed to give stable k' values for the pH levels from 8.0 to 8.9. Also, the effect of electrophoretic voltage (16–24 kV) on the k' value was not significant (Figure 3). A regular voltage of 20 kV could provide an adequate separation of 13 additives.

Adding the modifier

Bretnall and Clarke (17) found that the addition of an organic modifier in buffers could decrease electro-osmotic flow and increase the resolution of peaks. Thus, various concentrations (5, 10, and 15%) of acetonitrile as the modifier was added to the SDC buffer in order to enhance the separation. Figure 4 shows that the modifier influenced the k' value for each additive. The k' values of the more hydrophilic additives including aspartame, sodium dehydroacetate, potassium sorbate, sodium benzoate, saccharin and acesulfame-K were increased, and the other more hydrophobic additives such as dulcin and 6 hydroxybenzoates showed decreased k' values. The appropriate concentration of acetonitrile that provided better separation when added to the 0.05M SDC buffer that was used as the modifier was determined to be 5% (Figure 4).

According to the results of the experiments of the MEKC parameters, all 13 additives can be well separated using an appropriate method of simultaneous analysis with a 57-cm × 75-µm fused-silica capillary column and a buffer consisting of 0.05M SDC, 0.01M potassium dihydrogen-phosphate, 0.01M sodium tetraborate, and 5% acetonitrile (pH 8.6).

Sample preparation

SPE (a rapid method of sample preparation) was used to extract the additives

from the preserved fruit samples. This SPE technique had previously been successfully applied to the analysis of food additives using HPLC (4–5,15) and also sorbic acid and benzoic acid using MEKC (9). Because more additives were included and a simultaneous analysis was desired, the parameters for preparation of the sample were also examined. Figure 5 shows the effect of pH on the SPE recoveries of the additives from the sample solutions. The recoveries of dulcin, aspartame, dehydroacetic acid, benzoic acid, and sorbic acid were found to significantly decrease as the pH level increased. A pH level of 3 to 5 seemed to be more suitable in order to provide higher recoveries of most additives (Figure 5). Therefore, a 0.2M potassium dihydrogenphosphate buffer at pH 3.0 and a 5mM CTA ion-pairing buffer (15) were added to the samples prior to extraction in order to stabilize the sample pH and to increase the retention of the additives in the C_{18} SPE cartridge. Moreover, the SPE elution curves of the additives (Figure 6) showed that 2 mL of methanol was an adequate volume to elute most of the additives from the Sep-Pak C_{18} cartridge.

Analytical characteristics of additives

A 5-point standard curve for each additive was individually plotted using the standard solutions. The mixed standard solution that consisted of 13 additives was run 15 times in order to examine its reproducibility by studying the drift in the migration times of the compounds. In addition, the mixed standard solutions were spiked to a blank preserved fruit sample in order for recovery examination. The detection limits (which were calculated as the minimum sample size to produce a signal-to-noise ratio of 3) were also studied. The analytical characteristics of each additive are summarized in Table II. The linearity of response for most additives was very high, and the coefficient of linear regression (r^2) with confidence better than 0.98 was observed for all solutes. There were no significant shifts in the migration times for any of the compounds over 15 replications of runs. The recoveries of all additives exceeded 90% in most levels spiked to the sample with the coefficient of variety under 3%. Detection limits ranged between 10 and 50 mg/g for 13 of the additives in the preserved fruit sample. The analytical characteristics indicated that using SPE and MEKC was an ideal method for the analysis of additives in preserved fruits. The recovery of the additives from the preserved fruit sample using SPE and MEKC is shown in Figure 7. The extension of this method to other foodstuffs was attempted in the same laboratory.

Table III. Investigation of the Content of Sweeteners and Preservatives in 50 Preserved Fruit Samples

Additive found	Number of samples positive	Content (g/kg)	CV%*
Sweetener			
aspartame	4	0.17–11.59	3.10
saccharin	24	0.09–5.64	1.64
Preservative			
benzoic acid	21	0.02–1.72	0.70
sorbic acid	5	0.27–0.65	0.46
benzoic acid and sorbic acid	3	0.9–1.32 0.69–1.15	1.11 0.92

* Obtained from triplicate injections.

Additive contents in preserved fruit samples

Fifty preserved fruit samples were analyzed, and the result of the investigation is shown in Table III. Saccharin seemed to be the most common sweetener in the samples (0.09–5.64 µg/kg was found in 19 samples), followed by aspartame (0.17–11.59 g/kg was found in 4 samples). Twenty-one samples were found to contain benzoic acid ranging from 0.02–1.72 g/kg, 5 samples contained sorbic acid ranging from 0.27–0.65 g/kg, and 3 samples contained both benzoic acid (ranging from 0.9–1.32 g/kg) and sorbic acid (ranging from 0.53–1.15 g/kg). No other sweetener or preservative was found in the samples examined.

Conclusion

In conclusion, a rapid SPE and MEKC procedure had been developed for the simultaneous extraction and determination of the sweeteners dulcin, aspartame, saccharin, and acesulfame-K and the preservatives sorbic acid, benzoic acid, dehydroacetic acid, and PHBAs in preserved fruit samples. The analytical characteristics revealed that the method presented in this study was very reproducible with good detection limits for all additives.

Acknowledgements

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