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Review

Determination of organic acids in food samples by capillary zone electrophoresis

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Abstract

A comprehensive survey of the use of capillary zone electrophoresis for the determination of organic acids in food and beverage samples is presented. The analytes discussed in this paper include low-molecular-mass organic acids, amino acids, vitamin related compounds and free fatty acids.

Keywords: Reviews; Food analysis; Organic acids; Vitamins; Amino acids; Fatty acids; Phenolic compounds

Contents

1. Introduction

Within the last decade capillary zone electrophoresis (CZE) has become a versatile analytical technique employed for the determination of different types of solutes in a variety of matrices including such important fields of application as food chemistry [1–5]. Focusing on the analysis of organic acids like amino acids, low-molecular-mass carboxylic acids or fatty acids in beverage and food samples, these solutes are usually determined by a number of chromatographic methods including gas chromatog-

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raphy (GC), high-performance liquid chromatography (HPLC), and ion chromatography (IC). However, there is still a need for alternative methods of analysis that overcome some of the drawbacks of chromatographic methods, such as the derivatisation steps often necessary in GC, or insufficient separation selectivity and efficiency sometimes encountered in HPLC or IC. In this regard, CZE offers a number of promising features including short analysis times, high numbers of theoretical plates and separation selectivities which are often completely different from those encountered in chromatographic methods. For these reasons, CZE is becoming increasingly accepted for routine analytical work, although its introduction into regulated methods might still take some time [6].

This paper aims to give an overview regarding the increasing number of reports published within recent years dealing with the determination of organic acids in various food samples such as vegetables, fish and meat products as well as beverages by CZE.

2. Determination of organic acids by capillary zone electrophoresis

2.1. Choice of separation mode

Most of the solutes discussed in this paper are generally analysed in their anionic form. Therefore when fused-silica capillaries and background electrolytes (BGEs) with a pH above 3 are used, these analytes are separated in a counter-electroosmotic mode (i.e., the electroosmotic and electrophoretic mobilities are in opposite directions) unless co-electroosmotic conditions (same directions for electroosmotic and electrophoretic mobility) are established by reversal of the electroosmotic flow (EOF). The latter method is better suited for solutes showing higher electrophoretic mobilities (e.g., short-chain fatty acids or certain polycarboxylic and hydroxycarboxylic acids). Solutes showing lower mobilities and at the same time only small differences in mobility between each other (e.g., like long-chain fatty acids) are favourably separated employing counter-electroosmotic conditions to obtain improved resolution. In the case of amino acids a somewhat different situation occurs because of their zwitterionic nature. Therefore these solutes can be separated in either their anionic or cationic forms [6].

2.2. Detection methods

Most CZE separations of low-molecular-mass organic acids are carried out using indirect UV detection because of the low UV absorptivities of these analytes in the region above 220 nm. Direct UV detection is only appropriate for aromatic carboxylic acids in combination with UV transparent BGEs. When the instrument being used is suitable for detection at wavelengths below 200 nm, even aliphatic acids may be analysed in the direct detection mode, but it should be taken into account that in this case the choice of suitable BGEs and additives is also restricted to substances which are UV transparent at the wavelength selected for detection. Although only a few CZE separations of organic acids using conductivity detectors have been described up to now, this detection method can be regarded as an interesting alternative to methods based on UV absorbance [2–6]. The benefits of both methods of detection can be combined by using indirect UV detection and direct conductivity detection simultaneously, as has been demonstrated recently [7,8].

3. Analysis of food samples

Table 1 lists details of published methods for the determination of low-molecular-mass organic acids, amino acids, vitamins and related compounds, phenolic acids, fatty acids and other analytes in food samples. Some features of these methods are discussed in more detail below.

3.1. Low-molecular-mass organic acids

The determination of low-molecular-mass organic acids in food and beverage samples has been of interest right from the very early days of CZE. Kenney described the separation of a number of organic acids in different juices and soy sauce, employing a chromate-based BGE and indirect UV detection at 254 nm [9]. Propionic acid in various types of bread has been determined by Ackermans et

Table 1
Determination of organic acids in food samples by CZE[a]

Sample	Analytes	Separation conditions	Ref.
Low-molecular-mass organic acids			
Fruit juices and soy sauce	Acetic acid, lactic acid, citric acid, butyric acid and tartaric acid	5 m*M* potassium phthalate, 0.5 m*M* OFM (pH 7.0)	[9]
Bread	Propionic acid	(a) 5 m*M* Tris (pH 4.18–5.39) (b) 10 m*M* His (pH 5.84–6.67) (c) 10 m*M* Tris (pH 7.05–8.01) pH adjusted with benzoic acid	[10]
Chicory root thick juice and beet sugar juice	Formic acid, tartaric acid, malic acid, citric acid, succinic acid, glycolic acid, acetate, lactic acid and inorganic anions	(a) 5 m*M* phthalate, 2–2.5% OFM (pH 5.6–7) (b) 5 m*M* phthalate, 0.2–0.6 m*M* Ca^{2+}, 2% OFM (pH 5.6)	[11]
Fruit juices, wine, margarine and marmalade	Sorbic acid	100 m*M* MES, 10 m*M* Bis–Tris, 0.2% PEG (pH 5.2)	[12]
Wines and fruit juices	Oxalic acid, tartaric acid, malic acid, succinic acid, citric acid, acetic acid, lactic acid and inorganic anions	3 m*M* DMA, 3 m*M* DETA (pH 7.5)	[13]
Fruit juice, sport drink, nutrient added drink and tea	Tartaric acid, malic acid, citric acid, acetic acid, lactic acid, aspartic acid, glutamic acid, ascorbic acid and gluconic acid	(a) 5 m*M* TMA, 1 m*M* TTAB (pH 5.5) (b) 5 m*M* TMA, 1 m*M* TTAB (pH 9.0)	[15]
Various vegetables	Oxalic acid, succinic acid, citric acid, formic acid, acetic acid, propionic acid, butyric acid and inorganic anions	10 m*M* chromate, 2.30 m*M* CTAB (pH 11.5)	[16]
Vegetables	Oxalic acid	10 m*M* chromate, 4 m*M* OFM, 10% methanol (pH 8.7–9.2)	[17]
Wines and apple juice	Oxalic acid, tartaric acid, malic acid, succinic acid, citric acid, acetic acid, lactic acid and inorganic anions	3 m*M* PMA, 3 m*M* DETA (pH 7.5)	[14]
Beer	Oxalic acid, formic acid, malic acid, citric acid, succinic acid, pyruvic acid, acetic acid, lactic acid, pyroglutamic acid and inorganic anions	5 m*M* 2,6-pyridinedicarboxylic acid, 0.5 m*M* CTAB (pH 5.6)	[18]
Beer	Oxalic acid, formic acid, malic acid, citric acid, succinic acid, pyruvic acid, acetic acid, lactic acid, pyroglutamic acid and inorganic anions	5 m*M* 2,6-pyridinedicarboxylic acid, 0.5 m*M* CTAB (pH 5.6)	[19]
Orange juice and milk	Citric acid, malic acid, succinic acid, benzoic acid, butyric acid and inorganic anions	6 m*M* chromate, 3 m*M* borate and 0.032 m*M* CTAB, 5% acetonitrile (pH 8.0)	[21]
Tea infusions	Oxalic acid, citric acid, malic acid, aspartic acid, glutamic acid and quinic acid	10 m*M* chromate, 0.5 m*M* TTAB, 0.1 m*M* Na_2EDTA	[22]
Wine	Oxalic acid, tartaric acid, malic acid, succinic acid, adipic acid, glutaric acid, acetic acid, lactic acid, butyric acid, valeric acid and shikimic acid	7.5 m*M* *p*-AB, 10.5 m*M* Tris, 0.1 m*M* TTAB (pH 7.0) with LiOH	[7]
Beer	Oxalic acid, formic acid, malic acid, citric acid, succinic acid, pyruvic acid, acetic acid, lactic acid, pyroglutamic acid	7.5 m*M* *p*-AB, 0.12 m*M* TTAB (pH 5.75) with His	[8]
Beer	Oxalic acid, formic acid, malic acid, citric acid, succinic acid, pyruvic acid, acetic acid, lactic acid, pyroglutamic acid and amino acids	(a) 7.5 m*M* *p*-AB, 0.12 m*M* TTAB (pH 5.75) with His (b) 10 m*M* phosphate, 30 m*M* octanesulfonic acid (pH 2.36)	[23]
Wine	Tartaric acid	12 m*M* benzoic acid, 10 m*M* His and 1 m*M* TTAB (pH 5.0) with NaOH	[24]
Fruit juices and soy sauce	Oxalic acid, formic acid, malic acid, citric acid, acetic acid, lactic acid, amino acids and inorganic anions	20 m*M* 2,6-pyridinedicarboxylic acid, 0.5 m*M* CTAOH (pH 12.1)	[20]
Wines and grape juices	Tartaric acid, malic acid, citric acid, lactic acid, acetic acid and succinic acid	5 m*M* 2,6-pyridinedicarboxylic acid, 0.5 m*M* CTAB (pH 5.0)	[25]
Amino acids			
Citrus juices	Tryptophan, phenylalanine, tyrosine and ascorbic acid	(a) 50 m*M* phosphate (pH 6.8) (b) 35 m*M* sodium borate (pH 9.3) (c) 50 m*M* sodium borate (pH 7.6–9.2)	[26]
Citrus juices	Tryptophan, phenylalanine, tyrosine, sorbic acid, benzoic acid and ascorbic acid	35 m*M* sodium borate (pH 9.3)	[27]

Table 1. Continued

Sample	Analytes	Separation conditions	Ref.
Orange juice	Phenylalanine and ascorbic acid	35 m*M* sodium tetraborate, 5% acetonitrile	[28]
Citrus juices	Tryptophan, phenylalanine, tyrosine, ferulic acid and ascorbic acid	35 m*M* sodium borate (pH 9.3)	[29]
Beer and orange juice	Amino acids	(a) 10 m*M* phosphate, 30 m*M* octanesulfonic acid (pH 2.36)	
		(b) 10 m*M* phosphate, 30 m*M* octanesulfonic acid, 5% acetonitrile (pH 2.36)	[30]
Vitamins and related compounds			
Orange juice	Ascorbic acid	20 m*M* phosphate (pH 7.0)	[31]
Orange juice	Ascorbic acid and isoascorbic acid	100 m*M* tricine (pH 8.8)	[32]
Fruit juices	Biotin, ascorbic acid and nicotinic acid	20 m*M* phosphate (pH 8.0)	[33]
Fruit juices	Ascorbic acid	100 m*M* sodium borate (pH 8.0)	[34]
Bread, cereal, canned ham and yeast	Nicotinic acid	20 m*M* KH_2PO_4–Na_2HPO_4 (1:1)–acetonitrile (19:1)	[35]
Various meat, vegetables, fruit, nuts and milk	Nicotinic acid	20 m*M* KH_2PO_4–Na_2HPO_4 (1:1) (pH 7.0)–acetonitrile (1:13)	[36]
Yeast spread	Nicotinic acid	20 m*M* KH_2PO_4–Na_2HPO_4 (1:1) (pH 9.2)	[37]
Fruit juices	Ascorbic acid and isoascorbic acid	0.2 m*M* HIQSA, 1 m*M* phosphate, 20% isopropanol and 40% acetonitrile (pH 5.0)	[38]
Phenolic acids			
Wines	Syringic acid, *p*-coumaric acid, vanillic acid, caffeic acid, 3,4-dihydroxybenzoic acid and gallic acid	50 m*M* hydrogencarbonate (pH8.3)	[39]
Brandy extracts	Phenolic acids	10 m*M* sodium tetraborate, 5% ethanol (pH 9.2)	[40]
Green tea	Gallic acid, caffeic acid	150 m*M* borate (pH 8.5)	[41]
Beer	Phenolic acids	25 m*M* sodium phosphate (pH 7.2)	[42]
Fatty acids			
Coco oil extract	Fatty acids (C_2–C_{14})	20 m*M* Tris, 10 m*M* *p*-anisate, 1 m*M* trimethyl-β-CD, 50% MeOH (pH 8.2)	[46]
Butter sample	Fatty acids (C_4–C_{24})	5 m*M* diethylbarbiturate, 0.5 *M* Z1-Methyl (Waters), 70% ethyleneglycolmonomethyl ether	[44]
Vegetable oils	Fatty acids (C_{14}–C_{18})	10 m*M* Tris, 5 m*M* *p*-anisate, 1 m*M* dimethyl-β-CD (pH 8.1) in methanol–water (1:1)	[47]
Hydrogenated fish oil	Fatty acids (C_{14}–C_{22})	4 m*M* Tris, 2.5 m*M* antraquinone-2-carboxylic acid in NMF–dioxane (3:1)	[45]
Butter extracts	Fatty acids (C_8–C_{20})	10 m*M* sodium dodecyl benzenesulfonate, 30 m*M* Brij 35, 50% acetonitrile	[48]
Plant margarine and salad oil	Fatty acids (C_{12}–C_{18})	10 m*M* *p*-hydroxybenzoate, 5 m*M* Tris, 40 m*M* Brij 35, 50% acetonitrile	[50]
Other solutes			
Whey	Hippuric acid and orotic acid	40 m*M* AMPD (pH 8.8) with BICINE	[52]
Seafood	Domoic acid and isomers	20 m*M* sodium borate (pH 9.0)–acetonitrile (9:1)	[53]
Seafood	Domoic acid	10–50 m*M* sodium borate	[54]
Soybeans	Phytic acid	50 m*M* benzoic acid (pH 6.2) adjusted with His	[55]
Soy bean extract and tempeh	Phytic acid	50 m*M* benzoic acid (pH 6.2) adjusted with His	[56]

[a] Abbreviations: OFM: OFM Anion-BT proprietary name of Waters, Z1-Methyl, propriety of Waters, TTAB: tetradecytrimethylammonium bromide, CD: cyclodextrin, CTAB: cetyltrimethylammonium bromide, CTAOH: cetyltrimethylammonium hydroxide, DTAB: dodecyltrimethylammonium bromide, *p*-AB: 4-aminobenzoic acid, Tris: trishydroxymethylaminomethane, His: histidine, MES: 2-(*N*-morpholino)-ethanesulfonic acid, Bis–Tris: bis(2-hydroxyethyl)amino–tris(hydroxymethyl)methane, PEG: polyethylene glycol, NMF: *N*-methylformamide, tricine: *N*-[tris(hydroxymethyl)-methyl]glycine, AMPD: amino-2-methyl-1,3-propanediol, BICINE: *N*,*N*-bis(2-hydroxyethyl) glycine, DETA: bis(2-aminoethyl) amine, PMA: pyromellitic acid, TMA: trimellitic acid, HIQSA: 8-hydroxy-7-iodoquinoline.

al. using BGEs within a pH range from 4.2 to 8.0 and including benzoate as electrolyte co-ion [10]. Lalljie et al. investigated the effect of the addition of alkaline earth ions to the carrier electrolyte on the mobility of organic acids using sugar refinery juices as real samples [11]. Kaniansky et al. determined sorbic acid in a variety of food products including fruit juices, wine margarine and marmalade by CZE in a hydrodynamically closed separation compartment employing capillaries made from fluorinated ethylene–propylene copolymer [12]. Procedures for method development and validation to obtain simultaneous separations of inorganic anions and organic acids have been reported by Arellano and co-workers using different beverages to demonstrate the suitability of their approach for real samples [13,14]. Wu et al. investigated a number of BGEs including different UV absorbing probes and EOF-modifying agents with respect to their suitability for the determination of organic acids using CZE with indirect UV detection at wavelengths between 220 and 265 nm. The applicability of these BGEs for the analysis of real samples has been demonstrated for the case of beverages [15]. A comprehensive study mainly dealing with the nitrate and nitrite content but also the amount of formate, citrate and oxalate in various types of vegetables has been performed by Jimidar et al. [16]. Different vegetables have been analysed by Trevaskis et al. using an acidic extraction procedure and a modified chromate BGE including 10% methanol to determine the amount of oxalic acid [17]. Recently, Soga and co-workers introduced 2,6-pyridinedicarboxylic acid as a new electrolyte co-ion for the rapid and sensitive analysis of inorganic and organic anions as well as amino acids, and demonstrated the applicability of this BGE for the analysis of beer [18,19] and fruit juices [20]. An interesting approach combining on-line dialysis performed in a flow-injection analysis system with CZE allowing high-speed determination of organic acids and inorganic anions in complex matrices has been described by Kuban and Karlberg [21]. Using this system, milk and orange juice containing fruit pulp could be analysed without any sample pretreatment. The major organic acids in tea infusions have been determined by Horie et al. employing CZE with a chromate-based BGE containing ethylenediaminetetraacetic acid (EDTA), which was added to reduce the effect of metal ions present in the tea samples on the separation efficiency for the solutes under investigation [22]. Recently, a system allowing the simultaneous use of indirect UV detection and direct conductivity detection which provides improved sensitivity over a wide range of analyte mobilities has been reported by Klampfl and co-workers [7,8,23]. Inorganic and organic anions in beer and wine samples have been determined using this technique. Very recently Mallet et al. reported the analysis of tartaric acid in solid wine residues using a simple CZE method [24]. Using a very similar BGE composition to that of Soga and co-workers [18–20], Kandl and Kupina analysed a series of wine samples and grape juices to determine the content of several organic acids. The variety of different wines included in this paper provides some interesting aspects, although the separation system employed showed severe restrictions regarding the linearity of the detector response for several analytes [25].

3.2. Amino acids

A series of papers has been published reporting the analysis of native amino acids in different types of food and beverage samples by CZE. Because of their zwitterionic nature two different approaches are possible regarding the separation of these analytes, namely the use of counter-electroosmotic conditions employing highly basic BGEs so the amino acids are present in their anionic form, or the use of BGEs with pH values in the range of 2–3 and separating the amino acids as cations. Cancalon et al. were the first to determine phenylalanine, tryptophan and tyrosine in citrus juices, as well as other organic acids, biogenic amines, flavonoids and polyphenols using basic borate BGEs and direct UV detection at 200 nm [26–29]. The second of the above approaches has been taken by Klampfl et al. who compared amino acid distributions in orange juices and different beer samples using highly acidic phosphate BGEs including up to 35 m*M* of an alkylsulfonic acid, and direct UV detection at 185 nm [23,30]. A simple chromate-based BGE and indirect detection at 254 nm was used by Horie et al. for the separation of glutamic acid and aspartic acid in tea infusions, as well as the low-molecular-mass organic anions mentioned above [22].

3.3. Vitamins and vitamin related compounds

The analysis of vitamins and vitamin related compounds has always been an important field of application for CZE. Some of these compounds can also be regarded as organic acids and therefore they have been included in this review. Vitamin C has been determined in fruits employing a coated capillary and a phosphate-based BGE using direct UV detection at 254 nm [31]. Using an unmodified fused-silica capillary, Koh et al. determined vitamin C in beverage samples and biological fluids after spiking the samples with isoascorbic acid as internal standard [32]. Together with biotin and nicotinic acid, vitamin C has been quantified in fruit beverages and multi-vitamin preparations adding high levels of cysteine to the ascorbic acid standards to prevent decay of this easily oxidised compound [33]. Several vegetables, fruits and different fruit juices have been investigated for their vitamin C level using CZE, as well as spectrophotometric methods and HPLC for comparative purposes, by Choi and Jo [34]. A number of papers have described sample pretreatment as well as separation procedures for the determination of niacin in a number of different food samples. Using CZE this solute has been determined in meat products, vegetables, fruit and cereals [35,36] as well as concentrated yeast spreads [37]. An unusual approach for the analysis of ascorbic acid in fruit juices employing a non-aqueous BGE and fluorescence detection has been reported by Chen et al. [38].

3.4. Phenolic acids

A series of different phenolic acids can generally be found in wines and other alcoholic drinks and their determination by CZE using counter-electroosmotic conditions has been described in a number of papers. The levels of syringic acid, *p*-coumaric acid and vanillic acid in Italian chianti wine have been determined by Cartoni et al. using a hydrogencarbonate BGE [39]. Employing a BGE based on sodium tetraborate, Bronze et al. investigated oak wood extracts as well as brandy samples with respect to their levels of phenolic acids and a series of related compounds [40]. Some of these solutes can also be found in green tea, as has been demonstrated by Arce et al. [41]. CZE with amperometric detection was used by Moane et al. [42] for the determination of phenolic acids in beer samples and by Zhong et al. [43] for the determination of phenolic acids in tea samples.

3.5. Fatty acids

The potential of CZE for the separation of saturated and unsaturated fatty acids in their free form has been demonstrated in several reports. BGEs suitable for this purpose are either based on non-aqueous systems or contain at least a significant amount of organic solvents to obtain sufficient analyte solubility. Detection is generally performed by indirect UV detection. Buchberger and Winna [44] were able to determine free fatty acids in butter using a diethylbarbiturate carrier electrolyte containing a high concentration of a zwitterionic reagent to inactivate the surface of the fused-silica capillary. Separation of fatty acids in hydrogenated fish oil was achieved by Drange and Lundanes [45] in a separation medium based on anthraquinone-2-carboxylic acid in *N*-methylformamide–dioxane. Gareil and coworkers [46,47] demonstrated improved separation selectivity and enhanced analyte solubility in carrier electrolytes containing cyclodextrins. A somewhat different approach was introduced by Erim et al. [48] and optimised by others [49,50] who added micelle-forming reagents to the BGE. Such CZE conditions, known under the name micellar electrokinetic chromatography (MEKC), were applied to the analysis of various saponified fat samples. It is also worth mentioning that more sophisticated capillary electrophoretic techniques like capillary electrochromatography (CEC) have recently been employed by Dermaux et al. [51] for the separation of free and derivatized fatty acids and applied to the analysis of fish oil.

3.6. Other solutes

Milk products contain small amounts of hippuric acid and orotic acid. Tienstra et al. determined these analytes in rennet whey using CZE [52]. Domoic acid, one of the major toxic agents present in seafood, has been extracted from mussels and analysed using a basic tetraborate BGE or a borate BGE

containing acetonitrile to increase the solubility of the crude extract [53,54]. Nardi et al. [55] and Tawain et al. [56] determined the amount of phytic acid in soybean extracts and tempeh (a product made from soybeans by fermentation) using a benzoic acid based BGE.

4. Conclusions

As can be seen from the number of applications reviewed in this paper, CZE has undoubtedly become an attractive alternative to chromatographic techniques for the determination of organic acids in food samples. It offers high separation efficiency, simple sample pretreatment procedures, and short analysis times. Early problems encountered with low reproducibility of migration times, overloading effects or the lack of detection sensitivity have become less severe within the last few years mainly due to the availability of improved instrumentation. As its features are complementary to the chromatographic methods normally employed in the field of food analysis, CZE will not displace GC, HPLC or IC but offers an additional tool for problem solving and ultimately will find its way into approved official methods of analysis.

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