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An investigation into the determination of oxalic acid in vegetables by capillary electrophoresis

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The presence of natural levels of oxalate in foods may have important dietary implications through interference with calcium availability as well as an effect on renal stone formation. Recently, capillary electrophoresis (CE) has been used to separate a number of commonly occurring organic and inorganic anions, including oxalate. Initial attempts to determine oxalic acid by CE using an electrolyte consisting of 5 mM sodium chromate and 0.05 mM OFM Anion-BT reagent were unsuccessful in the presence of the large chloride concentration from the extract solution due to co-migration of oxalate, molybdate (chosen as the most suitable compound for use as an internal standard) and naturally occurring nitrate. Changing the composition of the electrolyte to 10 mM sodium chromate, 4 mM Anion-BT reagent and adding 10% methanol produced baseline separation of oxalate, nitrate and molybdate in vegetable extracts, despite the presence of a huge chloride peak. The levels of oxalic acid in a variety of vegetables were determined by CE and compared favourably with the levels determined by both the high-performance liquid chromatographic and the enzymatic procedure and levels reported in an Australian food composition table. The CE procedure is more convenient and quicker than the traditional titrimetric and chromatographic methods. Copyright © 1996 Elsevier Science Ltd

INTRODUCTION

The oxalic acid content of foods has traditionally been determined by extraction of oxalic acid and oxalate salts with 1 M hydrochloric acid followed by precipitation of calcium oxalate (Association of Official Analytical Chemists, 1980). The precipitate is isolated from the extract solution by a complex series of centrifuging, decanting and washing, repeated twice before it is dissolved in dilute sulphuric acid. Following dissolution of the precipitate the oxalate is determined directly by titration with 0.01 N potassium permanganate or indirectly by analysis of calcium using atomic absorption spectrophotometry or some other method.

More recently a number of high-performance liquid chromatography (HPLC) techniques have been reported for the determination of oxalic acid in various matrices. These include the use of a C8 column (Libert, 1981), a porous graphitic carbon column (Dutton *et al.*, 1991) and an ion exclusion column (Levi *et al.*, 1993) to perform the separation of oxalate from other extract solution components. Ion chromatography (IC) has been reported for the analysis of oxalate in urine (Von Schnakenburg *et al.*, 1994) and oxalic acid in vegetables (Ishii, 1991). Ion exclusion/ligand exchange chromatography was used for the determination of oxalic acid in taro cultivars (Wills *et al.*, 1983*a*) and of other organic acids in stone fruit cultivars (Wills *et al.*, 1983*b*). A number of enzymatic determinations of oxalate have also been reported involving either oxalate oxidase or oxalate decarboxylase (Hallson & Rose, 1974; Kasidas & Rose, 1980).

During the last few years capillary electrophoresis (CE) has been demonstrated to be a successful technique for separations of inorganic and organic anions (Romano *et al.*, 1991). CE has advantages over HPLC and IC for these determinations. CE separations typically have very high resolution, they are fast, sample size is very small and the cost of consumables (capillaries and electrolytes) is low. The technique of CE is based upon the application of an electric potential (typically 10–30 kV) across a fused silica capillary filled with an electrolyte solution into which a sample may be introduced. Ions are separated according to their respective mobilities in the electrolyte rather than by interaction with a stationary phase. High efficiency separations of ions are possible in under 5 min with plate numbers greater than 250 000 (Romano *et al.*, 1991).

Because it is the anion of a small, easily ionised diprotic acid ($pK_{a1} = 1.3$, $pK_{a2} = 4.3$) oxalate has a

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relatively high mobility and behaves in a similar manner to inorganic anions in CE. Consequently, techniques used to analyse oxalate by CE are similar to those used in the analysis of inorganic anions (Romano *et al.*, 1991; Wildman *et al.*, 1991; Kelly & Nelson, 1993). Typically this involves the use of a reagent to reduce or reverse the electro-osmotic flow (EOF) of the electrolyte solution.

The EOF is a bulk liquid flow which occurs in addition to the movement of ions according to their respective electrical charges and mobilities and is determined by the charge on the inner wall of the capillary. The walls of fused silica capillaries are negatively charged at most pH values, causing the direction of the EOF to be toward the negative electrode. In conventional CE, sample introduction is carried out at the positive electrode and detection near the negative electrode. Upon application of the electric potential, anions will migrate towards the positive electrode according to their respective mobilities and they will be transported towards the negative electrode by the EOF. Only anions with a mobility less than that of the EOF can be determined using this approach and even they have relatively long migration times (Romano *et al.*, 1991).

This problem can be overcome by simultaneously reversing the polarity of the electrodes at either end of the capillary and introducing a reagent to reduce or reverse the EOF. The sample is introduced at the negative electrode and analytes are detected near the positive electrode, towards which the anions migrate under the influence of the applied potential. The EOF modifier reagent either reduces the opposing EOF or, more commonly, reverses it to aid in the transportation of anions towards the detector and hence reduces considerably the migration times of the anions. EOF modifiers are typically cationic surfactants such as alkyltrimethylammonium compounds, which shield the negatively charged silanol groups on the inner wall of the capillary and expose the positively charged quaternary groups to the electrolyte, creating a nett positive wall charge and hence reversing the direction of the EOF (Jones & Jandik, 1991).

In CE, detection of analytes takes place within the capillary and is usually accomplished by the absorption of ultraviolet (UV) or visible light directed through a 'window' in the capillary near the end opposite that to which the sample is introduced. Most inorganic anions do not absorb UV or visible light or only absorb at wavelengths at which CE electrolyte anions often also absorb (Jandik *et al.*, 1992). However, they can be detected by the use of indirect detection with electrolyte anions such as chromate or phthalate, which are strongly absorbing at wavelengths at which almost all other anions do not absorb light.

In indirect detection it is the absorption of the electrolyte anion that is monitored by the detector, not that of the analytes. Analyte anions displace electrolyte anions as they migrate through the capillary and produce a reduction in absorption when they pass through the detector. The extent of the reduction is dependent on a number of factors including the concentration of the analyte anions, the concentration of the electrolyte and the difference in molar absorptivity between them (Yeung & Kuhr, 1991; Kelly & Nelson, 1993). With indirect photometric detection the absorption signal is inverted so that reductions in absorption appear as peaks.

The CE separation of 30 organic and inorganic anions in 3 min using an electrolyte containing a UV absorbing anion and an EOF modifier has been reported (Jones & Jandik, 1991). It was achieved using a 5 mM sodium chromate electrolyte containing 0.5 mM OFM Anion-BT (a proprietary EOF modifier solution reported by the authors to contain an 'alkylammonium compound') and with a pH of 8.0. Indirect photometric detection at 254 nm was used and sample introduction occurred at the negative electrode end of the capillary. Oxalate was not amoung the group of anions studied.

A sodium chromate electrolyte containing OFM Anion-BT and indirect detection at 254 nm was also used to analyse a mixture of inorganic and organic anions (including oxalate) in a sample of Kraft black liquor (Romano *et al.*, 1991) and in urine samples (Wildman *et al.*, 1991). The oxalate peak was well separated from all other peaks produced by the Kraft black liquor sample but was not fully separated from the nitrate peak in electropherograms of urine samples.

A mixture of anions containing chloride, sulphate, nitrate, nitrite, chlorate, bromide and oxalate has been separated by CE using an electrolyte containing 3 mM pyromellitic acid, 0.02% diethylenetriamine and 1% methanol at a pH of 9.6 (Kelly & Nelson, 1993). The authors also report the CE separation of a mixture of organic anions containing oxalate using a buffer containing 5 mM potassium hydrogen phthalate, 0.5 mM tetradecyltrimethylammonium bromide and 50 mM 2-(*N*-morpholino)-ethanesulphonic acid.

The use of an electrolyte containing 5 mM potassium hydrogen phthalate and 0.5 mM OFM Anion-BT with indirect photometric detection at 254 nm has been reported for the CE determination of organic acids in food samples (Kenney, 1991), sugar refinary juices (Lalljie *et al.*, 1993) and dental plaque and human saliva (Romano *et al.*, 1991). The lower mobility phthalate electrolyte is used in order to optimise CE conditions for lower mobility anions such as citrate, tartrate, malate, acetate and lactate. Analyte anions with a higher mobility than the electrolyte anion exhibit 'fronting', while anions with a lower mobility than the electrolyte anion show tailing (Romano *et al.*, 1991).

The influence of different electrolyte salts, EOF modifiers and organic solvents on separation selectivity in the CE of inorganic anions has been investigated (Buchberger & Haddad, 1992). Sodium chromate was considered to be the best choice for the main electrolyte salt. The effect of four different alkyltrimethylammonium halides as EOF modifiers was described, as was the effect of the addition of between 10 and 30% methanol, acetonitrile, tetrahydrofuran, acetone or ethylene glycol.

The aim of this investigation is to determine the optimum conditions for the extraction of oxalic acid

from a range of vegetables and for the subsequent CE separation and quantitation of oxalate in the extract solution.

EXPERIMENTAL

Reagents

AR grade oxalic acid dihydrate was obtained from Ajax Chemicals (Sydney, NSW, Australia). OFM Anion-BT is an EOF modifier solution obtained from Waters Chromatography (Milford, MA, USA). Concentrations of OFM Anion-BT used in CE electrolyte solutions are reported here in terms of the reagent concentration on the basis that the supplied solution contains 20 mM EOF modifier reagent (Jandik & Jones, 1991). All other reagents were AR grade or HPLC grade (solvents) and used without further purification.

Apparatus

Capillary electrophoresis

Analyses were performed using an Isco Model 3140 Electropherograph (Isco Inc., Lincoln, NE, USA) operating at −20 kV and at 28°C with a 75 cm×75 μm i.d. fused silica capillary (Polymicro Technologies, Phoenix, AZ, USA) with an effective length of 50 cm to the detector. Sample and standard solutions were loaded onto the column under vacuum (level 2, 20 kPa s^{-1}) and analytes detected by indirect absorption at 254 nm and 0.01 absorbance unit full scale (AUFS). Between each run the capillary was flushed for 2 min with electrolyte, which was an aqueous solution containing 10% methanol, 10 mM sodium chromate tetrahydrate and 4 mM OFM Anion-BT. Electropherograms were recorded with the ICE Data and Management and Control Software supplied with the electropherograph. Peak areas were used in the calculations. The limit of quantification for the procedure is 0.01 g per 100 g.

Liquid chromatography

This method was based on the HPLC procedure reported by Wills *et al.* (1983*a*).

Analyses were performed at ambient temperature using a Waters Model 501 pump, WISP 710 sample injector and a Waters Model 490 UV/visible detector (Waters) with an Aminex HPX-87H ion exclusion column (300 mm×7.8 mm) and an Aminex 125-0129 guard column (Bio-Rad Laboratories, Hercules, CA, USA). The mobile phase used was 0.005 M sulphuric acid at a flow rate of 0.6 ml min^{-1}, and analytes were detected by absorption at 214 nm and 0.1 AUFS. Peak areas were obtained using a Hewlett-Packard 3350 laboratory data system (Hewlett-Packard, Palo Alto, CA, USA) and were used in the calculations.

Enzymatic analysis

Analyses were performed using an enzymatic bioanalysis kit for oxalic acid (Boehringer Mannheim, Mannheim, Germany) and a GBC 911A UV/visible spectrophotometer (GBH, Dandenong, Victoria, Australia).

Samples and standards

Samples of fresh vegetables were purchased from local food outlets and homogenised in a food blender. The homogenised samples were analysed either immediately or after being frozen (sometimes both). All solutions were prepared using 18 Mohm Milli-Q deionised water (Millipore, Milford, MA, USA).

Approximately 10 g of sample was weighed into a 250-ml beaker and heated in a hot water bath to 60 or 90°C for 30 min with 50 ml of 0.1 M hydrochloric acid or heated to 90°C for 30 min with 50 ml of 1.0 M hydrochloric acid. The resulting solutions containing vegetable fragments were allowed to cool to room temperature, transferred to 100-ml volumetric flasks and made up to volume with deionised water. They were then filtered through Whatman 540 filter papers and passed through activated C18 Sep-Pak cartridges to remove potentially interfering organic compounds. The C18 Sep-Pak cartridges were preconditioned with 10 ml of methanol followed by 10 ml of water before use. After filtration the solutions were diluted with deionised water and an appropriate amount of molybdate internal standard (IS) solution. The diluted solutions were filtered through a 0.8-μm membrane filter before CE and HPLC analysis.

Stock standard solutions of oxalic acid (200 μg ml^{-1}) and molybdate (1000 μg ml^{-1}) were prepared by dissolving oxalic acid dihydrate crystals and ammonium molybdate tetrahydrate in deionised water. These solutions were used to produce standard oxalic acid solutions for use in CE and HPLC, and molybdate IS spikes for the oxalic acid standard solutions and for sample solutions. The standard solutions were also filtered through a 0.8-μm membrane filter before use in CE and HPLC. A typical standard solution contained 40 μg ml^{-1} oxalic acid and 100 μg ml^{-1} molybdate IS.

RESULTS AND DISCUSSION

Optimisation of CE electrolyte composition

The CE separation of oxalate from other anions was first attempted using the 5 mM sodium chromate and 0.5 mM OFM Anion-BT electrolyte previously reported (Jones & Jandik, 1991). Oxalate was clearly separated from other organic anions such as malate, citrate and tartrate, which were not well separated from each other, and from inorganic anions such as chloride and sulphate, which have shorter migration times than oxalate. Formic acid was initially considered for use as an internal standard but the formate peak could not be resolved from the malate, citrate and tartrate peaks. The electropherogram produced by Jones & Jandik (1991) suggests that molybdate can be separated from other anions likely to be found in vegetables. However, the

molybdate peak could not be resolved from the oxalate peak using this electrolyte. Changing the electrophoresis voltage or the pH of the electrolyte does not help to separate the two anions.

Changing the concentration of OFM Anion-BT in the chromate electrolyte has been shown to affect CE separations (Jones & Jandik, 1991). Increasing the concentration of OFM Anion-BT increased the separation between oxalate and molybdate. Baseline separation of the two peaks was produced using an electrolyte containing 10 mM sodium chromate and 4 mM OFM Anion-BT. Figure 1 shows electropherograms of a standard solution containing oxalate (40 μg ml^{-1}) and molybdate produced with three different electrolytes.

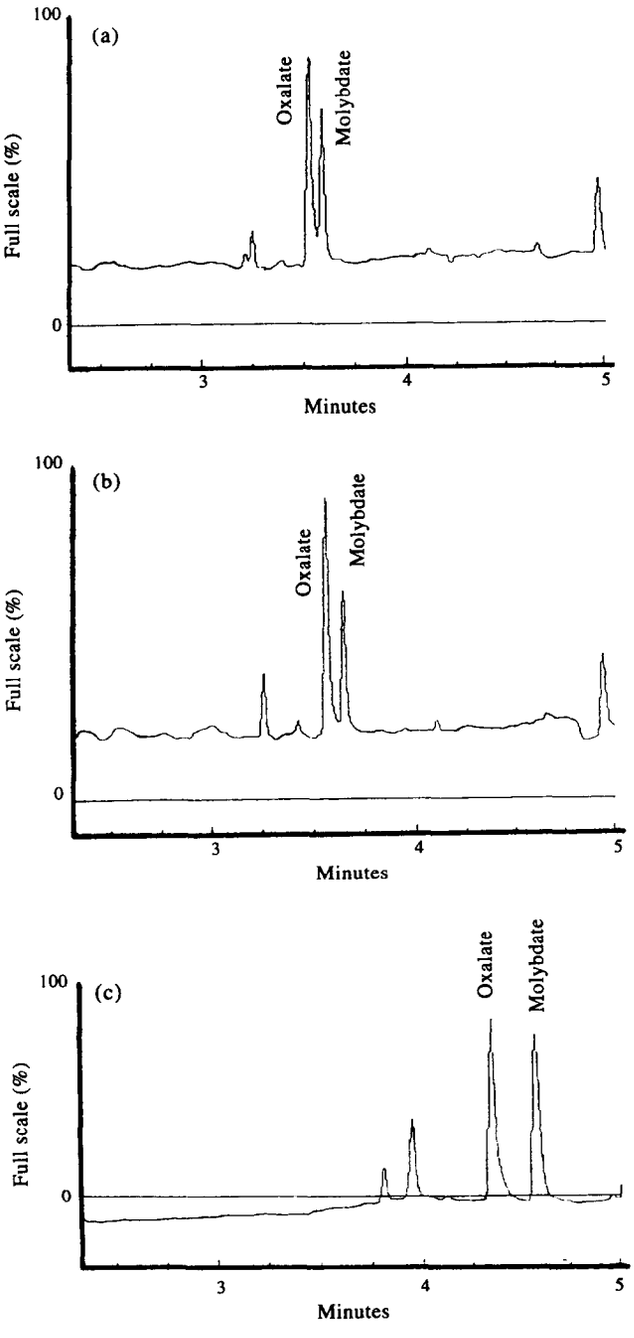

Fig. 1. Electropherograms of a solution containing oxalic acid (40 μg ml^{-1}) and molybdate IS (a, b: 40 μg ml^{-1}; c: 80 μg ml^{-1}) produced using the following electrolyte solutions: (a) 5 mM sodium chromate, 0.5 mM OFM Anion-BT, pH 8.0; (b) 10 mM sodium chromate, 1 mM OFM Anion-BT, pH 8.0; (c) 10 mM sodium chromate, 4 mM OFM Anion-BT, pH 8.0.

Figure 2 shows electropherograms of extract solutions of beetroot and rhubarb produced using the electrolyte containing 10 mM sodium chromate and 4 mM OFM Anion-BT. In the electropherograms of the pH 1 hydrochloric acid extract solutions, oxalate and nitrate virtually co-migrate and the two peaks appear almost as one. The interference is particularly bad in the case of beetroot because of the relatively high concentration of nitrate. The existence of the separate oxalate and nitrate peaks can be seen more clearly in the electropherogram of the aqueous extract of beetroot without hydrochloric acid. The lack of a high chloride concentration in the sample solution appears to allow oxalate and nitrate to have slightly different migration times, hence the two peaks are distinct although unresolved.

Organic solvents added to the electrolyte have been reported to affect the relative migration times of many inorganic anions in chromate electrolytes (Buchberger & Haddad, 1992). The addition of 10% methanol to the electrolyte was sufficient to separate the oxalate and nitrate peaks in the electropherogram of a solution containing a mixture of anions (Fig. 3). There is a good separation between the nitrate, oxalate and molybdate peaks, which is maintained in electropherograms of 0.1 M and 1.0 M hydrochloric acid extract solutions of vegetables (Fig. 4). The slight change in the migration times of the oxalic acid and molybdate internal standard peaks in Figs 3 and 4 was probably due to minor variations in the condition of the column and the buffer composition. Running standard solutions before and after the sample solutions enables the correct assignment of the peaks to be made.

The chromate electrolytes reported usually have a pH of 8.0 (Romano *et al.*, 1991; Wildman *et al.*, 1991; Jones & Jandik, 1991), which requires lowering the pH of a solution containing sodium chromate. At this pH the electrolytes containing methanol slowly produced a precipitate which caused severe interference with the electropherograms. The formation of this precipitate was virtually eliminated when the pH of the electrolyte was left unadjusted (pH approximately 9). Migration times of the more strongly acidic anions with a pK_a below 8 are unaffected by electrolyte pH changes between 8 and 10 (Jones & Jandik, 1991). As predicted, the change to the electrolyte pH produced no noticeable difference to the CE separation of nitrate, oxalate and molybdate.

It was subsequently noticed that the pH of the electrolyte containing methanol slowly fell to approximately 8 while it was being used for CE and while it remained unused. An electrolyte containing 10 mM sodium chromate, 4 mM OFM Anion-BT and 10% methanol with unadjusted pH was used to produce the analytical results discussed below. Using this electrolyte, oxalate and molybdate separations can be produced within 5 min. Any sample components remaining in the capillary after

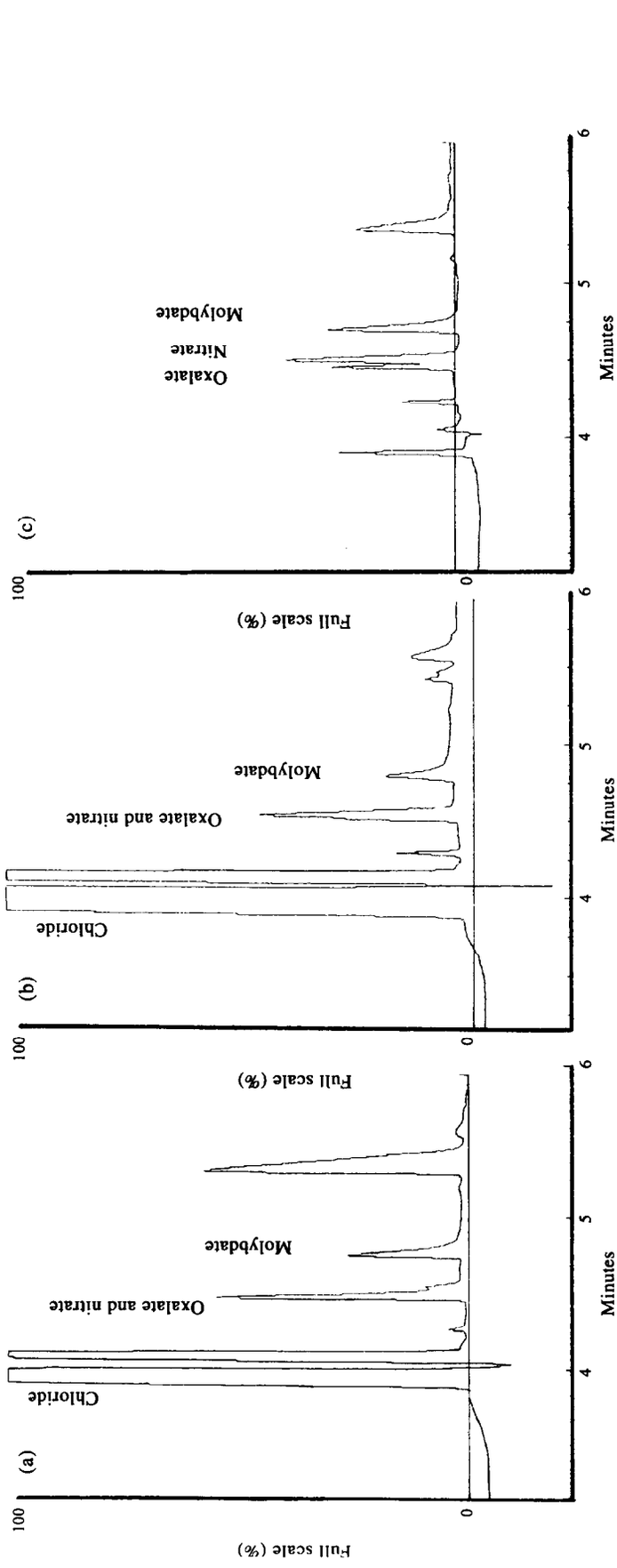

Fig. 2. Electropherograms of: (a) pH 1 hydrochloric acid extract of rhubarb, ten-fold dilution, containing molybdate IS (80 μg ml^{-1}); (b) pH 1 hydrochloric acid extract of beetroot, five-fold dilution, containing molybdate IS (80 μg ml^{-1}); (c) aqueous extract of beetroot, five-fold dilution, containing molybdate IS (80 μg ml^{-1}). The electrolyte solution used was 10 mM sodium chromate, 4 mM OFM Anion-BT, pH 8.0.

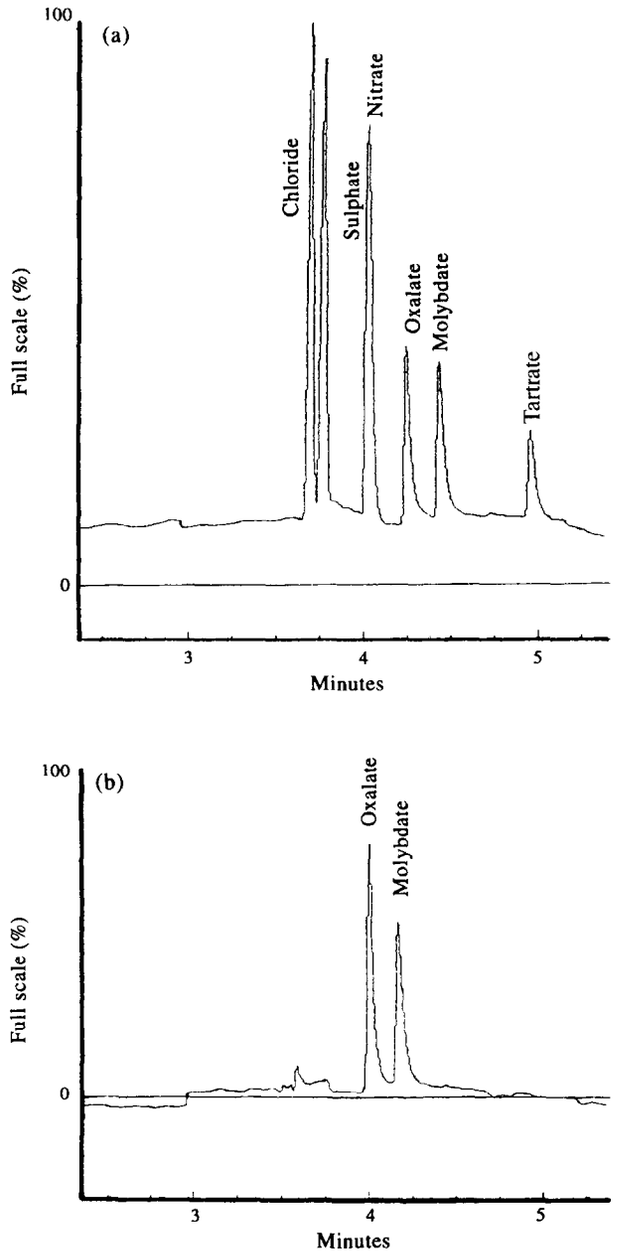

Fig. 3. Electropherograms of a solution containing: (a) hydrochloric acid (70 μg ml^{-1}), sulphuric acid (40 μg ml^{-1}), nitric acid (40 μg ml^{-1}), oxalic acid (40 μg ml^{-1}), molybdate IS (100 μg ml^{-1}) and tartaric acid (40 μg ml^{-1}); (b) oxalic acid (40 μg ml^{-1}) and molybdate IS (100 μg ml^{-1}). The electrolyte solution used was 10 mM sodium chromate, 4 mM OFM Anion-BT, 10% methanol, pH 9.2.

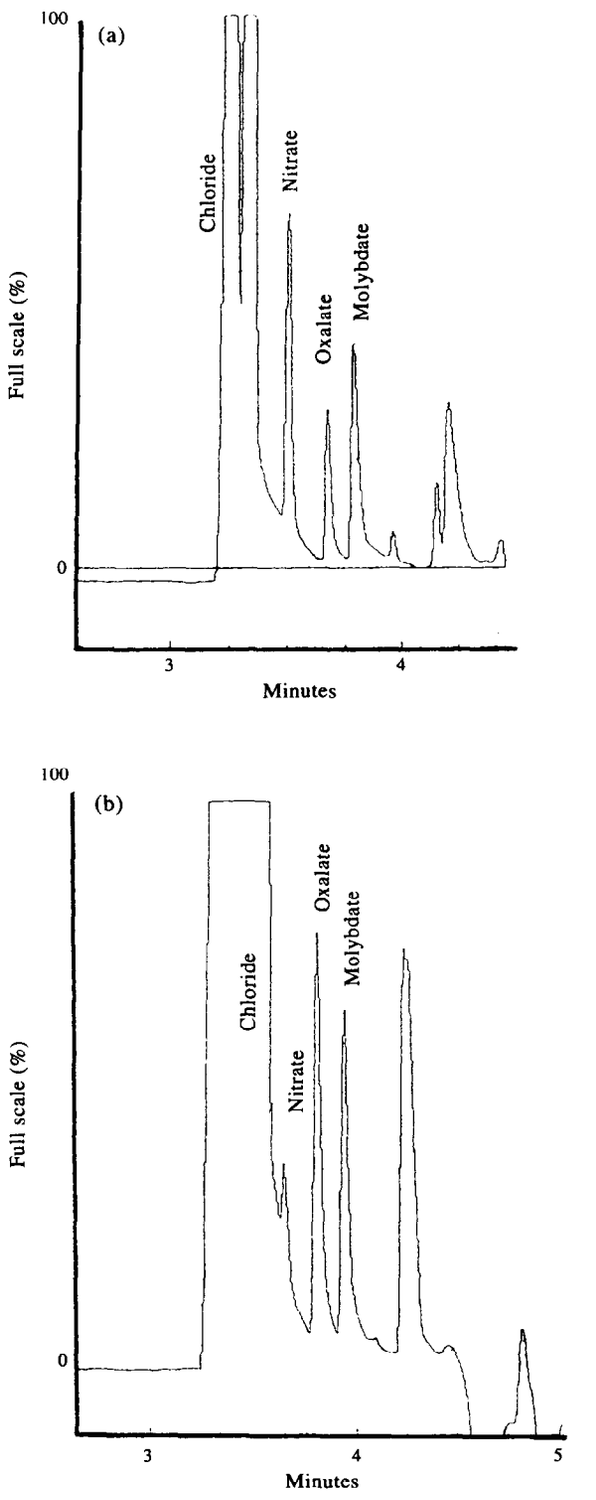

Fig. 4. Electropherograms of: (a) 0.1 M hydrochloric acid extract of beetroot, five-fold dilution, containing molybdate IS (100 μg ml^{-1}); (b) 1.0 M hydrochloric acid extract of rhubarb, ten-fold dilution, containing molybdate IS (100 μg ml^{-1}). The electrolyte solution used was 10 mM sodium chromate, 4 mM OFM Anion-BT, 10% methanol, pH 8.7.

this time can be flushed away in 2 min with fresh electrolyte solution. In contrast, the HPLC determination of oxalic acid required up to 4 h between injections due to the long elution times of the components of some samples.

Oxalic acid extraction

Reported methods for the extraction of oxalic acid from vegetables describe digestion in 1 M hydrochloric acid at 100°C for 15 min (Association of Official Analytical Chemists, 1980; Libert, 1981) or hydrochloric acid at a pH of 2 at 50°C for 15 min (Boehringer Mannheim, 1992). Preliminary tests to extract oxalic acid from beetroot and rhubarb using hydrochloric acid with a pH of 1 (approximately 0.1 M) and a pH of 2 (approximately 0.01 M) at 60°C for 30 min showed a considerable increase in the oxalic acid extracted using the pH 1

Table 1. Results of CE, HPLC and enzymatic determination of oxalic acid in beetroot and rhubarb following extraction using hydrochloric acid with a pH of 1 and 2 at 60°C for 30 min

Vegetable	pH of HCl	Molarity of HCl (approx.)	Oxalic acid (g per 100 g)		
			CE	HPLC	Enzymatic
Beetroot	1	0.1	0.10	0.10	0.08
	2	0.01	0.04		
Rhubarb	1	0.1	0.42	0.48	0.45
	2	0.01	0.22		

hydrochloric acid and good agreement between the results obtained by the CE, HPLC and enzymatic analysis of the pH 1 extract solutions (Table 1).

No increase in the amount of oxalic acid extracted could be found when the extraction temperature was increased to 90°C and the hydrochloric acid concentration was increased from 0.1 to 1.0 M. Results obtained from CE and HPLC determinations when oxalic acid was extracted from six different vegetables using 1.0 M hydrochloric acid at 90°C and 0.1 M hydrochloric acid at 60°C are shown in Table 2. Extraction using 1.0 M hydrochloric acid at 90°C for 30 min approximates the conditions specified by the Association of Official Analytical Chemists (1980) and produced results similar to those reported in an Australian food composition table (Wills, 1987; Table 2), indicating that all the oxalic acid had been extracted from the vegetables.

Repeatability and accuracy of oxalic acid determinations

The coefficient of variation (%CV) for values of the ratio oxalate peak area/IS peak area, determined for each of three oxalic acid standard solutions (10, 20 and 40 μg ml^{-1}) from seven separate electropherograms, varied between 2.7 and 4.9%. Linear regression analysis produced a correlation coefficient of 0.9994 for the relationship between the average ratio, oxalate peak area/IS peak area, and the concentration of oxalic acid in each standard solution. The ratio, oxalate peak area/IS peak area, was used for repeatability and linearity studies because it is this ratio, determined for any unknown solution, which is compared to the same ratio for a standard solution to calculate the concentration of oxalic acid in the unknown solution. The correlation coefficient value for oxalic acid standard solution concentrations suggests that this calculation is suitable for solutions with oxalic acid concentrations no greater than 40 μg ml^{-1}.

Table 2. Results of CE and HPLC determination of oxalic acid in vegetables following extraction using 0.1 M hydrochloric acid at 60°C or 1.0 M hydrochloric acid at 90°C for 30 min

Vegetable	Molarity of HCl	Temperature (°C)	Oxalic acid (g per 100 g)		
			CE	HPLC	Wills (1987)[a]
Beetroot	0.1	60	0.10	0.09	0.14
	1.0	90	0.08	0.07	
Rhubarb	0.1	60	0.40	0.40	0.43
	1.0	90	0.37	0.40	
Broccoli	0.1	60	<0.01	0.01	0.05
	1.0	90	<0.01	<0.01	
Carrot	0.1	60	0.02	0.02	<0.01
	1.0	90	0.02	0.02	
Silverbeet	0.1	60	0.42	0.43	0.46
	1.0	90	0.42	0.39	
Celery	0.1	60	0.02	0.01	<0.01

[a]Results in the final column are from an Australian food composition table compiled by Wills (1987).

Table 3. Results of CE, HPLC and enzymatic analysis of beetroot and rhubarb following extraction using 0.1 M and 1.0 M hydrochloric acid at 90°C for 30 min

Vegetable	Molarity of HCl	%CV (oxalate/IS)	Oxalic acid (g per 100 g)		
			CE	HPLC	Enzymatic
Beetroot	0.1	6.1	0.10	0.07	0.07
	1.0	4.4	0.10	0.08	0.07
Rhubarb	0.1	3.0	0.37	0.35	0.38
	1.0	2.8	0.38	0.41	0.37

The CE results were calculated from the average ratio, oxalate peak area/IS peak area, determined from seven electropherograms of each extract solution. Also shown is the %CV for values of the ratio for each extract solution.

Oxalic acid was extracted from beetroot and rhubarb using 0.1 M and 1.0 M hydrochloric acid at 90°C for 30 min. The results of oxalic acid determination by CE are in good agreement with HPLC and enzymatic analyses of the same extract solutions and with the results from the previous set of extractions shown in Table 2. The CE determinations of oxalic acid were calculated by comparing the average ratio, oxalate peak area/IS peak area, determined for each of the four extract solutions from seven electropherograms, with the average ratio determined for one of the oxalic acid standard solutions. %CV for values of the ratio for each extract solution and the results of the CE, HPLC and enzymatic analyses are shown in Table 3. The %CV values are similar to those determined from the oxalic acid standard solution results. Calculation by comparison of average ratios for sample extract and standard solutions produces oxalic acid determinations with %CV values between 6 and 10.

The enzymatic analysis of the 200 μg ml^{-1} oxalic acid stock standard solution produced a result of 206 μg ml^{-1}, in good agreement with the result of 205 μg ml^{-1} obtained from the 0.203 g $litre^{-1}$ standard solution supplied with the enzymatic analysis kit. The enzymatic analysis of oxalic acid in both sample extract and standard solutions was particularly useful because it provided a completely independent means of determining oxalic acid, without reference to standard solutions. Calculations of oxalic acid concentrations in enzymatic determinations are based on the 340 nm absorption and extinction coefficient of a product of the reaction between oxalate and certain enzymes.

CONCLUSION

Capillary electrophoresis presents an alternative technique for the determination of oxalic acid in vegetables. %CV for values of the ratio oxalate peak area/IS peak area, determined for each of four beetroot and rhubarb extract solutions from seven separate electropherograms, indicate an acceptable instrument reproducibility. The levels of oxalic acid determined by CE were in good agreement with those determined by HPLC and enzymatic analysis and with those reported in a composition table of Australian foods. The CE procedure is also quicker or less costly than other methods.

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