Journal of Chromatography, 509 (1990) 283–286
Elsevier Science Publishers B.V., Amsterdam

CHROM. 22 206

Note

Utilization of capillary isotachophoresis in the determination of organic acids in food

J. KAROVIČOVÁ*, M. DRDÁK and J. POLONSKÝ
Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, 812 37 Bratislava (Czechoslovakia)

The contents of organic acids in various food products have been determined by traditional analytical methods[1], but such analyses are time consuming and suffer from errors due to the large number of operations necessary for the isolation and determination of individual components. In addition, the presence of other acids and/or materials can interfere and cause additional errors. Hence modern separation methods have been increasingly used[2–5].

The detection of individual acids in various food products is now carried out mostly by chromatographic methods, gas chromatography[6,7] and high-performance liquid chromatography[8,9] being the most widely used. However, the complete chromatographic separation of organic acids is not easily achieved, and the methods are sometimes time consuming and involve complex sample preparation organic acids in aqueous alcohols have been determined using zone electrophoresis[10,11].

Capillary isotachophoresis has been found suitable for the identification and determination of organic acids in foods[12–26]. Its main advantages are simple sample preparation and short analysis times. Separation can be performed in both aqueous and non-aqueous solutions [27,28].

We have used capillary isotachophoresis in the study of the formation of organic acids and phosphoric acid during lactic fermentation of cabbage and of changes in organic acid contents during the maturation of red wine.

EXPERIMENTAL

We used a CS ZKI 001 Isotachophoretic Analyser (Spišská N. Ves, Czechoslovakia) with a conductivity detector and a TZ 4200 double-line recorder (Laboratorní přístroje, Prague, Czechoslovakia), and also several electrolytic systems as shown in Table I. Samples diluted 1:50 with water were injected into the column using the four-way valve of the instrument. The duration of the analysis was 20–30 min, depending on the electrolytes used. Organic acids were identified by comparison of the isotachophoretic zones of the analytes in the samples and in standard solutions. Quantitative analysis was performed by calibration.

0021-9673/90/$03.50

TABLE I

COMPOSITION OF ELECTROLYTES AND CONDITIONS OF SEPARATION

Concentration of leading electrolyte (hydrochloric acid) (M)	10^{-2}	10^{-2}	10^{-2}
Counter ion	β-Alanine	ε-Aminocaproic acid	
pH	3.8	4.5	5.2
Additive polyvinylpyrrolidone (PVP) (%)	0.1	0.1	0.1
Terminating electrolyte	Caproic acid	Caproic acid	Caproic acid
Concentration (M)	$5 \cdot 10^{-3}$	10^{-2}	10^{-2}
Current of 200 μA in the preseparation column			
Current of 40 μA in the analytical column			

RESULTS AND DISCUSSION

The process of cabbage fermentation was followed by the determination of organic acids in the liquid phase. Lactic, acetic, phosphoric and ascorbic acid were determined after 4, 5, 6, 7, 10 and 11 days of fermentation. Fig. 1 shows the increase in organic acid contents. After 11 days of fermentation, the following acid contents were found in the liquid portion of fermented cabbage: ascorbic acid 0.16, lactic acid 6.13, acetic acid 1.52 and phosphoric acid 0.14 g l^{-1}. Lactic and acetic acid are the main

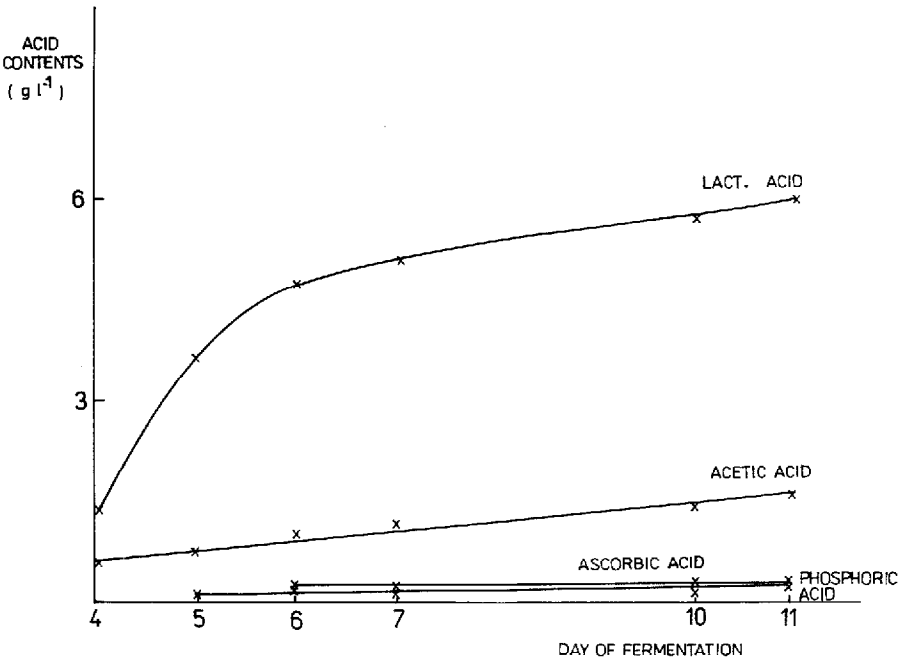

Fig. 1. Organic acids in fermented cabbage. LACT. = lactic.

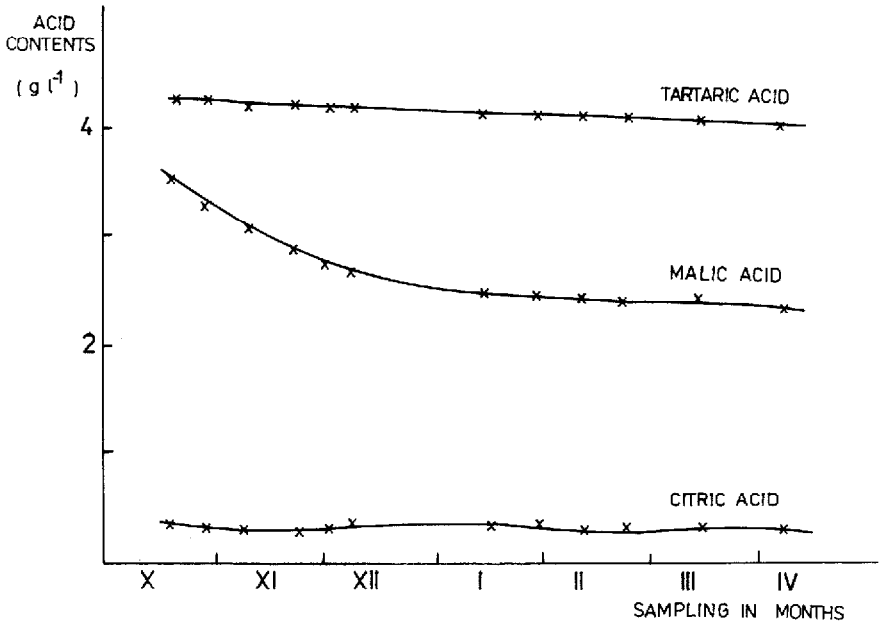

Fig. 2. Changes in tartaric, malic and citric acid contents in Svätovavrinecké red wine.

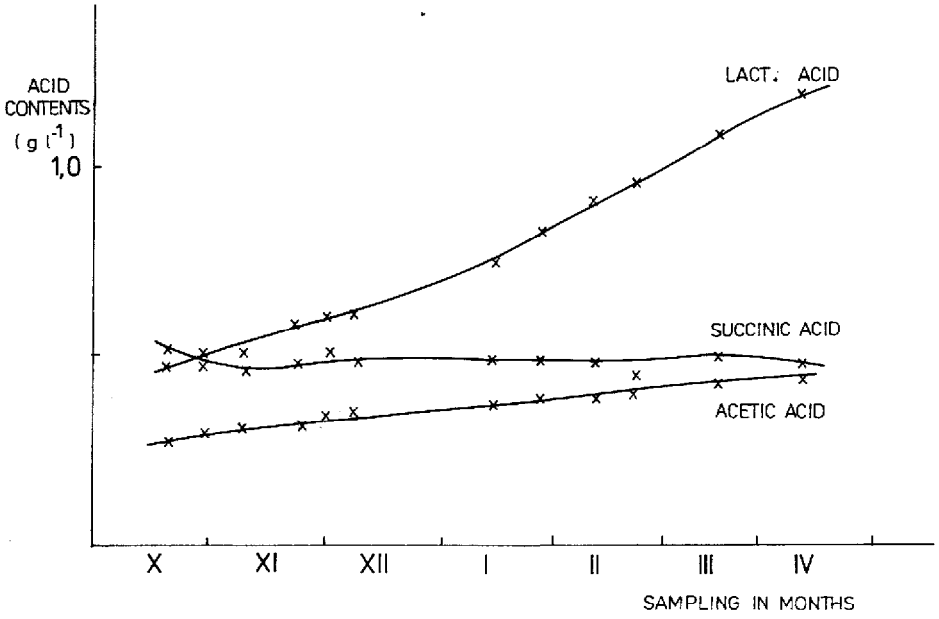

Fig. 3. Changes in lactic, succinic and acetic acid contents in Svätovavrinecké red wine.

products of the lactic fermentation process and are a significant parameter for the quality of fermented cabbage. By comparing the results obtained after 11 days of fermentation and those obtained by Klein[29] and Hamtschek[30], a further increase in the acid contents with prolonged fermentation could be assumed, with citric acid being formed in the final stage.

We also studied red wine formation from Svätovavrinecké (Saint Laures) grape mash. The wine was decanted from the dregs as the residual sugar content decreased to 4 g l^{-1}. The decanted wine was stored in glass containers and the changes in the contents of tartaric, malic, citric, lactic, succinic and acetic acid were followed by isotachophoresis. The organic acids formed influence the characteristic a stringent taste of the wine. Figs. 2 and 3 show the changes in the acid contents in the wine during maturation from October to April.

REFERENCES

1 J. Davídek, J. Hrdlička, M. Karvánek, J. Pokorný, J. Seifert and J. Velíšek, *Laboratorní Příručka Analýzy Potravín*, SNTL/Alfa, Prague, 1984, p. 718.
2 K. Suyama, K. Hori and S. Adachi, *J. Chromatogr.*, 174 (1979) 234.
3 H. G. Henkel, *J. Chromatogr.*, 58 (1971) 201.
4 A. Rapp and E. Zeigler, *Dtsch. Lebensm.-Rundsch.*, 75 (1979) 396.
5 E. M. Thurman, *J. Chromatogr.*, 185 (1979) 625.
6 M. J. Barcelona, H. M. Liljestrand and J. J. Morgan, *Anal. Chem.*, 52 (1980) 321.
7 J. F. T. Oldsfielde and M. Shore, *Sucr. Belge Sugar Ind. Abstr.*, 99 (1980) 143.
8 N. E. Bussele and R. A. Miller, *J. Liq. Chromatogr.*, 2 (1979) 697.
9 Y. Hashimoto, *Mikrochim. Acta*, Part II, (1978) 159.
10 V. Jokl, V. Dolejšová and M. Matušová, *J. Chromatogr.*, 172 (1979) 239.
11 H. L. Martelli and O. Pires, *J. Chromatogr.*, 8 (1962) 227.
12 F. M. Everaerts, J. L. Beckers and Th. P. E. M. Verheggen, *Isotachophoresis—Theory, Instrumentation and Applications* (*Journal of Chromatography Library*, Vol. 6), Elsevier, Amsterdam, 1976.
13 P. Boček, K. Leková, M. Deml and J. Janák, *J. Chromatogr.*, 117 (1976) 97.
14 A. Baldesten and S. G. Hjalmarson, *Fresenius Z. Anal. Chem.*, 290 (1978) 148.
15 Z. Stránsky, P. Peč, M. Kudlová, M. Mergl and J. Žabková, *Živočisna Výroba*, 33 (1988) 211.
16 K. Rubach, P. Offizorz and Z. Bremez, *Z. Lebensm.-Unters.-Forsch.*, 172 (1981) 351.
17 W. Arneth, *Fleischwirtschaft*, 864 (1984) 1098.
18 P. Offizorz, *Monatsschr. Brauwiss.*, 37 (1984) 168.
19 H. Klein and H. G. Stettler, *Wein-Wiss.*, 39 (1984) 51.
20 K. P. Kaiser and H. Hupf, *Dtsch. Lebensm.-Rundsch.*, 75 (1979) 346.
21 W. Nierle, *Getreide Mehl Brot*, 42 (1988) 138.
22 K. Pruša and O. Smejkal, *Kvasny Prům.*, 29 (1983) 7.
23 J. Farkaš, M. Koval' and J. Polonský, *Bulletin Potravinárskeho výskumu*, 21 (1982) 25.
24 J. Karovičová, J. Polonský and M. Drdák, *Kvasny. prům.*, 33 (1987) 292.
25 J. Karovičová, J. Polonský and M. Drdák, *Nahrung*, 32 (1988) 519.
26 J. Karovičová, J. Polonský and M. Drdák, *Potrav. Vědy*, 6 (1988) 105.
27 J. L. Beckers and F. M. Everaerts, *J. Chromatogr.*, 71 (1972) 380.
28 K. P. Kaiser and H. Hupf, *Dtsch. Lebensm.-Rundsch.*, 75 (1979) 300.
29 H. Klein, *Ind. Obst.-Gemüseverwert.*, 67 (1982) 31.
30 J. Hamtschek, *Ind. Obst.-Gemüseverwert.*, 71 (1986) 325.