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Analysis of anions and cations in drinking water samples by Capillary Ion Analysis

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The use of capillary ion electrophoresis (CIE, Waters' tradename: Capillary Ion Analysis, CIA) for the analysis of four anions and four cations in drinking water samples available on the Malaysian market, such as natural mineral water, bottled drinking water and tap water, was investigated. In addition, *zam-zam* water (an underground water, much sought-after by Muslims and only available in Mekah, Saudi Arabia) was also analyzed. The anions analyzed were chloride, sulphate, nitrate and fluoride while the cations analyzed were potassium, calcium, sodium and magnesium. Results of this determination generally show a low content of anions and high content of calcium and magnesium in natural mineral water and non-detectable amounts of anions and cations in bottled drinking water. Out of the 15 mineral waters of different brands that were analyzed, four brands show anionic and cationic levels almost similar to that of tap water. With the exception of fluoride, an abnormally high level of both anions and cations were detected in all the *zam-zam* water samples analyzed, as compared to the other drinking waters.

INTRODUCTION

In the past ten years, the drinking water industry has developed very rapidly in Malaysia and, today, it is estimated that there are about 100 brands of natural mineral waters as well as bottled drinking waters available on the Malaysian market (Barbosa, 1990; Fernandez *et al.*, 1990). Diverse factors such as the belief that our bodies are being replenished with minerals after strenuous activities, and the belief that mineral waters are of medicinal value, fuelled by the impression that mineral waters are always associated with nature as portrayed in advertisements, has contributed to this popularity. In fact, in certain circles, it has become a status symbol to drink mineral water. However, this bustling business was slowed down in the early 1990s due to the 'Great Mineral Water Scam', as publicised by the local media, of unscrupulous activities of a number of manufacturers who were just bottling tap water, often at their backyards, and selling it as mineral water (Cheah, 1990; Fernandez & Azman, 1990; Fernandez *et al.* 1990; Hamid & Hussain, 1990). The authorities have adopted the Codex guideline in an effort to regulate the industry. Despite the high consumption of bottled drinking waters in Malaysia, reports on their analysis and their mineral contents are scattered and not well documented. However, a number of studies have been carried out in other countries and the analysis results can be found in the literature (Li & Li, 1994, Rhemrev-Boom, 1994; Blanco *et al.*, 1995; Papadoyannis *et al.*, 1995; Van Holderbeke *et al.*, 1995).

For the determination of inorganics in solutions, a number of analytical methods have been utilised, such as amperometry (Carpenter & Pletcher, 1995), potentiometry (Pasquini & Cunha, 1995), voltammetry (Neuhold *et al.*, 1994), flow-injection approaches (Porter *et al.*, 1995; Haj-Hussein, 1996; Hernandez *et al.*, 1996; Nagy *et al.*, 1996) and chromatography (Shpigun & Zolotov, 1988; Edgell *et al.*, 1994; Gros & Gorenc, 1994, 1995*b*; Albazi *et al.*, 1995; Chu *et al.*, 1995; Hong *et al.*, 1995; Jun *et al.*, 1995). Of these techniques, ion chromatography is the most widely used technique and has been recommended by the Environmental Protection Agency as the official method for analysis of drinking water samples in the USA (Romano & Krol, 1992). However, such recommendation is not stipulated in the Malaysian Food Act 1983.

Capillary ion electrophoresis has emerged as a valuable alternative that offers considerable advantages. Some of these advantages include fast analysis and

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conditioning time, few moving parts, small compact design and cost-effective as compared to the conventional ion chromatography (Romano *et al*., 1991; Jones *et al*., 1992; Romano, 1993; Oehrle, 1994, 1996). CIE is a branch of capillary electrophoresis which is optimized for the rapid separation and quantitation of low-molecular-mass anions and cations. Ions are separated based on their mobility in electrolytic solutions (Jones & Jandik, 1991; Jandik *et al*., 1992; Weston *et al*., 1992). This technology has attracted the interest of numerous researchers and has been used in the determination of inorganics in drinking water samples available in USA (Romano & Krol, 1993; Oehrle, 1996), France (Morin *et al*., 1994) and Slovenia (Gros & Gorenc, 1995*a*).

A survey of the literature reveals that most of the earlier studies on the analysis of drinking waters were performed mainly on anions only. In this report, a more thorough analysis covering not only anions, but also cations, was performed. *Zam-zam* water is a natural unprocessed underground water that is in high demand by Muslims due to its medicinal value. This water, only available at specific locations in Mekah, Saudi Arabia, had been consumed by Muslim pilgrims since time immemorial. For the first time, an anionic and cationic analysis of this nature was conducted on *zam-zam* water. As will be shown later, CIA seems to be well suited to perform this task.

MATERIALS AND METHODS

Materials and reagents

All solutions, electrolytes and standards were prepared using 18.2 MΩ-cm Milli-Q water generated by Milli-Q Plus Water System (Millipore, Bedford, MA, USA). Sodium chromate was obtained from Fluka (Buchs, Switzerland), guanidine hydrochloride from BDH (Poole, UK), the complexants, tropolone and 18-crown-6-ether from Aldrich (Milwaukee, WI, USA), potassium hydroxide from Merck (Darmstadt, Germany) while the electro-osmotic flow modifier (CIA-Pak OFM Anion-BT Solution) was supplied by Waters (Milford, MA, USA). The working electrolyte for anion analysis consisted of 4.6 mM sodium chromate, 0.46 mM OFM Anion-BT and 0.003 mM sulfuric acid (RDH, Seelze, Germany) while the working electrolyte for cation analysis consisted of 2.5 mM tropolone, 6.3 mM guanidine hydrochloride and 2.0 mM 18-crown-6-ether. Standard concentrates of anions and cations were prepared in 1000 mg litre^{-1}, in which the anions were prepared using their sodium salts while the cations were either from their chloride (K^+ and Na^+) or nitrate (Ca^{2+} and Mg^{2+}) salts. All these chemicals were purchased from Fluka (Buchs, Switzerland). Fresh working electrolytes and working standards were prepared daily, vacuum-filtered and degassed prior to use.

Samples

With the exception of one mineral water sample which was obtained from Thailand, all the mineral waters and drinking waters were purchased from sundry shops in Penang. Tap waters were collected at 10.30 am every morning directly from the tap in the School of Chemical Sciences, Universiti Sains Malaysia, for a period of one week while the seven batches of *zam-zam* waters were obtained from Muslim pilgrims who had just returned from their pilgrimage to Mekah, Saudi Arabia. The well water was collected from a village in Kedah, Malaysia.

Apparatus and methodology

The analysis was carried out on a Waters Capillary Ion Analyzer (Milford, MA, USA) which was interfaced to a Waters PC 800 Workstation. The capillary (75 μm × 60 cm) used was constructed of fused silica and was supplied by Waters. Indirect UV detection was achieved at 254 nm with a mercury lamp and a 254-nm optical filter. The samples were introduced into the capillary using 30-s hydrostatic injections, from a height of 10 cm. The separations were carried out by using a negative power supply for anion and positive power supply for cation determination with the applied voltage set at 20.0 kV, temperature controlled at 25°C. Isomigration time mode was used for the anion determination.

Each day before starting analysis, the capillary was conditioned by purging with 100 mM potassium hydroxide solution and Milli-Q water each for 5 min. Between each run the capillary was purged with electrolyte for 2 min. Before powering down, the capillary was flushed and cleaned with Milli-Q water for 5 min. These steps are crucial for the good effectiveness and reproducibility of the capillary electrophoretic separation.

Anion analysis with indirect UV detection was achieved using high-mobility chromate electrolyte and an added osmotic flow modifier to reverse the direction of the osmotic flow. This will generate a net direction of the anions to migrate towards the anode at the detection side. The isomigration time mode was performed to ensure the reproducibility of the migration times which shifted due to the variability in sample conductivity. For the cation analysis, the UV absorbing coion, guanidine salt, was employed to provide UV background. Some complexants were also introduced to partially complex the cations to increase the differences in effective mobilities and, thus, the cations could be well separated and quantitated.

RESULTS AND DISCUSSION

In USA, the Food and Drug Administration (FDA) has categorized bottled water as a food and a minimum standard has been established. FDA classifies the bottled waters as distilled water, fluorinated water, hard water, mineral water, natural water, public water, soft water and spring water which are explained in the Glossary of Water Terms of FDA. However, in Malaysia, bottled waters are only classified into two categories; 'natural mineral water' and 'packaged drinking water'. As defined in Malaysian Food Act 1983 (anonymous, 1995), natural mineral water shall be ground water which is obtained for human consumption from subterranean water-bearing strata through a spring, well, bore or other exit, with or without the addition of carbon dioxide, while packaged drinking water shall be potable water or treated potable water, other than natural mineral water, that is hermetically sealed in bottles or other packages and is intended for human consumption.

Analysis of natural mineral waters

Figures 1 and 2 show the electropherograms of the anion and the cation analysis of a natural mineral water by CIA. The CIA procedure obviously allows fast analysis time in which the separation can be accomplished in less than 5 min. For quantitation, a plot of absorbance vs concentration of anions or cations yield r^2 values of at least 0.99. Table 1 shows the results of the anion and cation analysis of fifteen locally available natural mineral waters including samples of French and Thailand origin (M14 and M15, respectively). On the whole, the amount of anions found were of slightly lower concentration than the values claimed by the companies. In brand M10, we found that it contains 19.2 mg litre^{-1} of nitrate but the concentration of this anion was not stated on the bottle while, in brand M13, the magnesium content was found to be about five times the value stated on the bottle. Of the eight anions and cations determined here, only fluoride and nitrate are regulated by Malaysian Food Act 1983. As stipulated in the Act, the standard for natural mineral water permits the maximum concentrations of fluoride and nitrate up to 2 mg litre^{-1} and 45 mg litre^{-1}, respectively. Based on this loose standard, all the locally available mineral waters chosen for this study meet the chemical standard as a natural mineral water.

For the cation analysis, the samples were found to contain a relatively high concentration of calcium and magnesium compared to potassium and sodium. Generally, the calcium content is the highest among the cations analyzed, ranging from 20 to 90 mg litre^{-1}. On the other hand, potassium content is the lowest, most of them below 5 mg litre^{-1}. In some cases (M1, M6 and M10), a relatively low concentration of cations compared to the other mineral water samples was observed. The mineral water from Thailand (M15) shows an abnormally high content of chloride and sodium. Both the chloride and sodium concentrations were found to be at least ten times higher than the concentrations found in the Malaysian natural mineral waters. It is clear that this sample is of different quality from the natural mineral waters in Malaysia.

The reproducibility of the CIA technique was found to be satisfactory as exemplified by the relative standard deviation, RSD of 6.2, 5.4, 4.3, 8.5, 1.6 and 10.9% for five repeated determinations of potassium, calcium, sodium, magnesium, sulphate and fluoride, respectively. The relatively high value of RSD for magnesium and fluoride is due to the small values of the mean.

Analysis of drinking waters and well water

Results of the analysis of four different brands of packaged drinking waters reveal an interesting result—there were no peaks detected for all their electropherograms

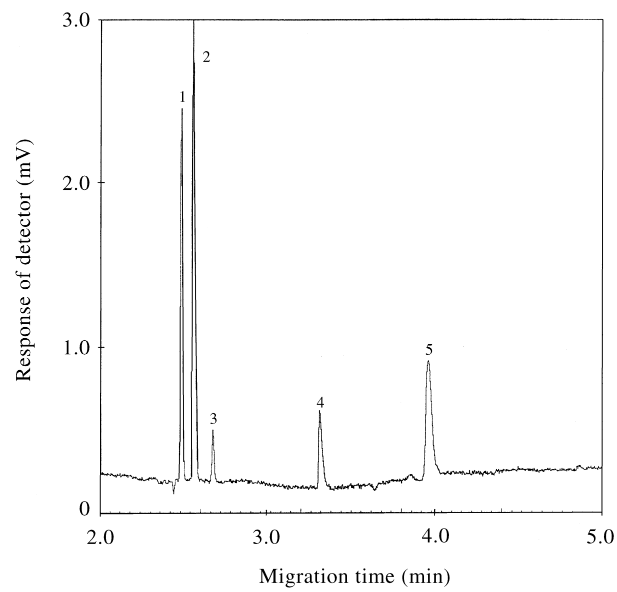

Fig. 1. Electropherogram of a typical mineral water sample, showing the anions analyzed. (1) chloride, (2) sulphate, (3) nitrate, (4) fluoride, (5) carbonate

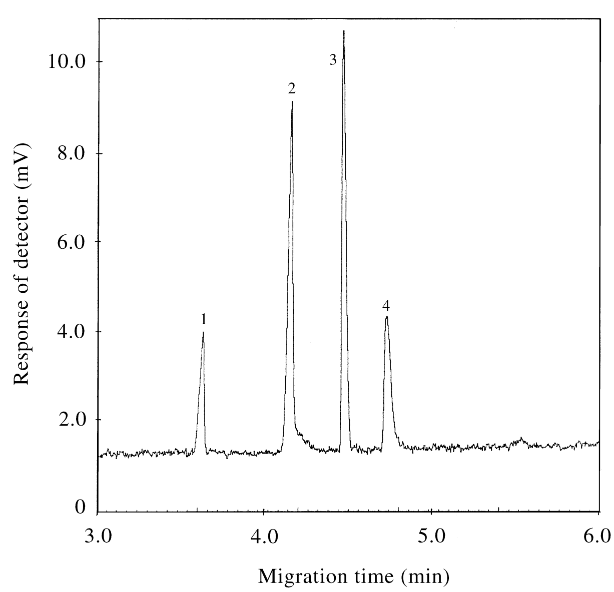

Fig. 2. Electropherogram of a typical mineral water sample, showing the cations analyzed. (1) potassium, (2) calcium, (3) sodium, (4) magnesium

and indeed all the electropherograms are identical to that of an 18.2 MΩ-cm Milli-Q water which was used as a blank. From this evidence, it seems that the packaged drinking waters sold in the Malaysian market are actually distilled deionized water which probably has been sterilized. In an analysis of a well water, it was found that its mineral content was very low (most of them even lower than the minerals in tap water), which therefore should not be qualified and sold as a natural mineral water in the market. The results are summarized in Table 2 .

Analysis of tap waters

In this study, we found that the mineral content in Penang tap water is consistent over the period of our studies. For most of the analysis, with the exception of

Table 1. Results for the determination of anions and cations in some mineral waters available in Malaysia*

Mineral water sample	Concentration (mg $litre^{-1}$) of mineral							
	Anion				Cation			
	Cl^-	SO_4^{2-}	NO_3^-	F^-	K^+	Ca^{2+}	Na^+	Mg^{2+}
M1	2.7 (ns)	< 1.0 (ns)	1.4 (ns)	< 1.0 (ns)	2.2 (0.09)	nd (0.14)	5.2 (4.12)	nd (ns)
M2	4.6 (8.00)	< 1.0 (1.33)	1.8 (ns)	nd (ns)	3.6 (4.98)	96.9 (20.0)	1.9 (1.47)	54.6 (16.1)
M3	< 1.0 (10)	< 1.0 (1)	nd (ns)	< 1.0 (< 1)	2.1 (2)	21.0 (29)	7.5 (4)	2.6 (1)
M4	1.6 (12.0)	< 1.0 (< 5.0)	< 1.0 (ns)	nd (ns)	4.7 (4.2)	27.3 (20.0)	5.8 (5.0)	4.9 (16.0)
M5	1.0 (10)	< 1.0 (1)	nd (ns)	< 1.0 (< 1)	2.3 (2)	21.7 (29)	7.6 (4)	2.6 (1)
M6	2.9 (ns)	3.4 (ns)	< 1.0 (ns)	< 1.0 (ns)	nd (1.44)	nd (0.69)	10.0 (2.10)	nd (0.34)
M7	< 1.0 (< 1)	nd (1)	nd (ns)	< 1.0 (1)	2.0 (2)	5.9 (29)	3.7 (4)	5.9 (1)
M8	7.4 (12.0)	nd (< 5.0)	nd (ns)	nd (ns)	5.2 (4.2)	28.6 (20.0)	4.9 (5.0)	9.9 (16.0)
M9	< 1.0 (< 1)	1.0 (< 3)	nd (ns)	< 1.0 (ns)	4.7 (4.6)	12.4 (7.5)	5.2 (6.0)	9.1 (1.8)
M10	6.7 (4.97)	< 1.0 (ns)	19.2 (ns)	nd (ns)	1.0 (0.33)	1.3 (9.19)	11.8 (4.94)	6.3 (0.26)
M11	4.6 (8.00)	< 1.0 (1.33)	1.8 (ns)	nd (ns)	3.4 (4.98)	60.8 (20.1)	2.1 (1.47)	37.1 (16.1)
M12	1.3 (10)	1.0 (1)	nd (ns)	1.0 (< 1)	2.6 (2)	52.3 (29)	14.6 (4)	3.1 (1)
M13	10.1 (8.4)	6.9 (6.9)	6.3 (6.3)	< 1.0 (ns)	6.3 (5.7)	34.0 (9.9)	14.3 (9.4)	31.1 (6.1)
M14 (imported from France)	5.2 (4.5)	10.6 (10)	3.4 (1)	nd (ns)	< 1.0 (1)	37.4 (78)	42.7 (5)	2.6 (24)
M15 (purchased from Thailand)	> 100 (ns)	14.1 (ns)	< 1.0 (ns)	< 1.0 (ns)	< 1.0 (ns)	41.8 (ns)	> 100 (ns)	27.9 (ns)

nd = not detected, ns = not stated;
* number in parentheses denotes concentration value stated on the bottle as claimed by company;

Table 2. Concentration profiles of the common anions and cations in several water samples

Water sample	Concentration (mg $litre^{-1}$) of mineral							
	Anion				Cation			
	Cl^-	SO_4^{2-}	NO_3^-	F^-	K^+	Ca^{2+}	Na^+	Mg^{2+}
Drinking water								
Brand D1	nd	nd	nd	nd	nd	nd	nd	nd
Brand D2	nd	nd	nd	nd	nd	nd	nd	nd
Brand D3	nd	nd	nd	nd	nd	nd	nd	nd
Well water								
Sample W1	3.3	15.9	< 1.0	< 1.0	1.2	1.2	< 1.0	nd
Tap water								
Day 1	5.2	8.0	1.3	1.9	2.5	8.2	3.3	3.7
Day 2	5.4	8.2	1.7	2.1	2.5	9.1	3.1	3.7
Day 3	5.5	8.4	1.7	2.2	2.6	8.5	3.2	3.8
Day 4	5.6	8.5	1.7	2.4	2.6	8.8	3.2	3.7
Day 5	5.5	8.4	1.7	2.4	2.6	8.7	3.2	3.7
Day 6	5.7	8.3	1.7	2.4	2.7	7.7	3.2	3.4
Day 7	5.8	8.0	1.8	2.4	2.5	8.1	3.2	3.5
Zam-zam water								
Batch 1	159	162	170	1.4	77.9	72.6	136	20.5
Batch 2	167	148	175	nd	70.8	63.7	122	22.6
Batch 3	154	153	163	nd	75.5	61.4	125	19.9
Batch 4	158	163	166	nd	79.9	57.4	151	21.7
Batch 5	172	152	164	nd	77.4	66.8	146	22.5
Batch 6	165	153	162	nd	76.6	68.4	140	22.0
Batch 7	159	148	165	nd	77.2	68.1	129	21.6

nd = not detected

sulphate (RSD of 8%), the RSD achieved were below 4%. It is interesting to note that the mineral content of some of the mineral waters (M1, M6, M7 and M10) are comparable or, in certain instances, inferior compared to tap water. In terms of mineral content only, consumers may just consume tap water as the price is only a fraction of that of these mineral waters. Unethical practices by these companies should not be ruled out.

Analysis of *zam-zam* waters

Zam-zam water is obtained from an underground source in Mekah, Saudi Arabia. The water is strongly believed among the Muslims to have extraordinary medicinal values. The water is very popular among the Muslims, especially during the Haj seasons when it is consumed extensively to replace the mineral lost from the body due to the high perspiration rate under the scorching desert conditions. In view of its popularity, we are keen to know the mineral contents of this water. As summarized in Table 2, the *zam-zam* waters contain an abnormally high level of anions and cations compared to the natural mineral waters and the tap waters that were analyzed. It is obvious from this result that this water is of totally different quality than the available natural mineral water and tap water. Its medicinal value is probably partially attributed to its abnormally high mineral content. Electropherograms are shown in Figs 3 and 4.

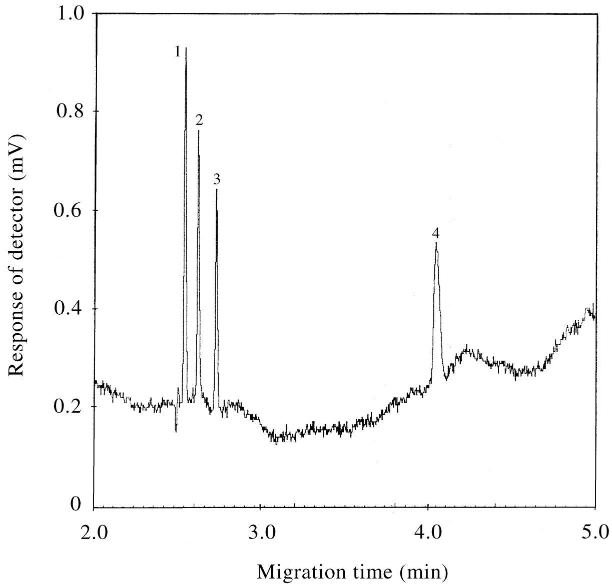

Fig. 3. Electropherogram of a *zam-zam* water sample (after 100 times dilution), showing the anions analyzed. (1) chloride, (2) sulphate, (3) nitrate, (4) carbonate.

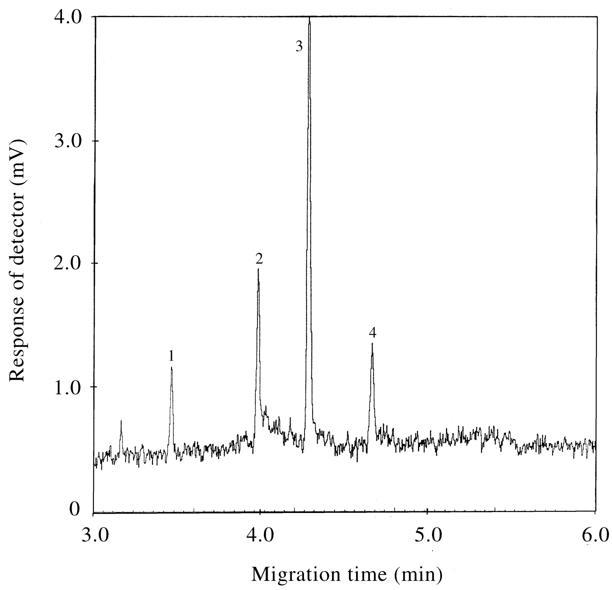

Fig. 4. Electropherogram of a *zam-zam* water sample (after 100 times dilution), showing the anions analyzed. (1) potassium, (2) calcium, (3) sodium, (4) magnesium.

CONCLUSION

Analysis of anions (Cl^-, SO_4^{2-}, NO_3^- and F^-) and cations (K^+, Ca^{2+}, Na^+ and Mg^{2+}) in drinking waters such as mineral waters, tap waters, *zam-zam* waters using CIA were conducted. Results of the analysis suggest that the mineral water industry in Malaysia needs more stringent regulations in view of the findings that the mineral contents of four of the mineral waters analyzed are comparable and sometimes even inferior in quality compared to tap water. With the exception of these four suspected samples, the mineral waters of Malaysian origin are characterised by a mean calcium and magnesium content of 39.5 and 16.9 g mg $litre^{-1}$ respectively. Water samples that are labelled as drinking water are actually just deionized water. The medicinal value of *zam-zam* waters is probably attributed to the fact that it contains an abnormally high content of anions and cations among the drinking waters analyzed.

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