



Received: 07-02-2014
Accepted: 03-03-2014

ISSN: 2321-4902
Volume 1 Issue 6



Online Available at www.chemijournal.com

International Journal of Chemical Studies

Determination of Citric acid in Soft drinks, Juice drinks and Energy drinks using Titration

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Citric acid is used as an additive in different drinks to improve flavour and taste. However; higher concentration may cause damage to tooth enamel. The objective of this study was to determine the citric acid level in different drinks by using titration. Citric acid was determined in soft drinks, juice drinks and energy drinks from Najran city in the Kingdom of Saudi Arabia (KSA), using normal titration. In total 27 samples were analysed. The highest concentrations were recorded for energy drinks and the lowest values for soft drinks. In conclusion the concentrations (mean \pm SD) of citric acid in energy drinks (n = 2) were 7.3 ± 0.06 g/L, in juice drinks (n = 11) were 2.79 ± 0.04 g/L and in soft drinks (n = 14) were 1.76 ± 0.04 g/L. This study reveals new data of citric acid levels in different drinks in the KSA.

Keyword: Citric acid; Energy drinks; Juice drink; Soft, drinks, Titration; KSA

1. Introduction

Citric acid is a weak organic acid occurring naturally in many fruits, especially in citrus fruits, also found in animal fluids and tissue. It is very soluble and used as an additive in many drinks. The role of citric acid in drinks is to improve taste and flavour, antioxidant and to maintain stability (preservative enhancement) [1-2]. Citric acid contains three carboxylic acid groups; 2-hydroxy-1,2,3-propanetricar-boxylic acid. In human physiologic blood pH and urine, it found mainly as the trivalent anion. Citrate salts are used to deliver minerals in biologically available forms these include dietary supplements and medications. In lemons and limes; citric acid is the most concentrated comparing with other fruits [3].

The production of ATP in the citric acid cycle is the major source of citric acid in vivo from endogenous metabolism in the mitochondria [4].

The modest increase in urinary citrate excretion has been associated with gastrointestinal absorption of citric acid from dietary sources, citrate is the most abundant organic ion found in urine [5, 6]. Many studies showed that intake of citric acid products leads to citrate excretion in urine. This may prevent stone formation by inhibiting the calcium oxalate nucleation process and the growth of both calcium oxalate and calcium phosphate stones. It is also result in reduction of the free calcium concentration in urine. Therefore, the increase of urinary citrate was a primary focus in the medical management of urolithiasis [7-9]. In addition, a study was carried out on diabetic rats and was concluded that citric acid inhibits development of cataracts [10].

On the other hand, other studies showed that higher concentrations of citric acid can cause softening, erosion and damage to tooth enamel. In

addition, the hardness of enamel can be reduced by 84% with high levels of citric acid in orange juice [3, 11]. Results from a recent study showed that the enamel erosive potential of fruit juice drinks was more than other soft drinks. The acidulant in fruit juice drinks is citric acid while acidulant in other drinks is phosphoric acid [12]. In addition, it was concluded that dietary citric acid caused more enamel erosion than phosphoric acid [13].

A study carried out on lemon Juice, lime Juice, and commercially-available fruit Juice to assess citric acid, was found its level in lemon juice and lemon juice concentrate 1.44 g/Oz (48.7g/L) and 1.10 g/oz (37.2 g/L), respectively. While its level in other commercially available juice products was ranging from 0.03 to 0.22 g/oz (1.01-7.44g/L) [3].

Worldwide, there has been a continuous increase in the number of people consuming soft drinks energy drinks and juice drinks. It is noteworthy these drinks contain citric acid. Different studies suggested that the total acid level is considered as more important than pH level in assessing the erosive potential of these drinks; they argued that total acid will give the actual hydrogen ion available to interact with teeth enamel [14-15].

Therefore, the aim of this study was to provide information about the citric acid concentration in soft drinks, juice drinks and energy drinks which commonly consumed in the KSA. The second purpose was to raise the awareness of the public health.

2. Material and Methods

2.1. Reagents and solutions

Deionised water ($18\Omega\text{ cm}^{-1}$) from a Millipore Elix5 (France) was used throughout the study to prepare all stock and diluted solutions. The solutions were prepared with chemicals of analytical grade.

2.1.1. Potassium iodate

Potassium iodate (KIO_3) extra Pure (Loba Chemie Pvt Ltd.; India) was used to standardized sodium hydroxide solution.

2.1.2. Sodium hydroxide solutions

Sodium hydroxide pellets extra pure (Loba Chemie Pvt Ltd.; India) was standardized against KIO_3 and daily working solution (0.1M) was prepared by appropriate dilution.

2.1.3. Citric acid standard solutions

Citric acid anhydrous (NTL Nentech Ltd.; Brixworth, UK) was used and standardized against NaOH. Standard solutions were prepared and used as spikes to check the recovery percentage and accuracy of the measurement.

2.1.4. Phenolphthalein solution

Phenolphthalein (Loba Chemie Pvt Ltd.; India) was used to prepare the indicator solution (0.05 g in 50 ml ethanol, then add 50 ml of H_2O) [16]. The indicator solution (3- 4 drops) was used through the study in each titration experiment.

2.2. Titration

Acid-base titration was used to determine the citric acid concentration in all samples. Potentiometric titration was also used to check the accuracy of acid-base titration in the case of coloured samples. Lovibond sensodirect pH 200; potentiometer was used for this purpose.

2.3. Samples collection and preparation

In total 27 samples were collected from supermarkets and local markets in the Najran city in the KSA. The majority (74%) of the samples were produced in the KSA the rest were imported. Fourteen samples were soft drinks, eleven samples were juice drinks and two samples were energy drinks. All samples didn't contain phosphoric acid.

All samples have been opened for two days in the laboratory to assure their decarbonation. A volume of 10 ml of each sample was used to carry out a titration experiment against 0.1M NaOH for total citric acid determination

3. Results and discussion

Titration is a very easy, cost effective and reliable method to be used in daily and routine laboratory work [17]. These are the reasons why we used such

method to carry out this very important study. For quality control purpose potentiometric titration was used for one coloured sample and the result was 1.52 ± 0.0 compared to 1.49 ± 0.04 g/L (mean \pm SD) result of the normal titration. Spiked samples were also prepared with low citric acid concentration (1.54 g/L) and high citric acid concentration (19.21 g/L); recoveries were

97.0%, and 98.1% (n = 3), respectively.

Results of total citric acid determined in all 27 samples are shown in Table 1. The samples were labeled as soft drinks (DS) juice drinks (DJ) and energy drinks (DE) as shown Table1. The citric acid content of all measured samples has been ranging from 0.49 to 8.41 g/L.

Table 1: Citric acid concentrations in soft drinks, Juice drinks and energy drinks.

Sample name	Mean (g/L)	SD (n=3)	%RSD	Sample name	Mean (g/L)	SD (n=3)	%RSD
<i>Soft Drinks (n = 14)</i>				<i>Juice Drinks (11)</i>			
DS2	2.03	0.04	1.82	D J 1	2.73	0.10	3.58
DS3	2.05	0.06	3.12	D J 4	2.90	0.04	1.27
DS5	2.71	0.04	1.36	D J 6	2.75	0.06	2.32
DS10	1.17	0.04	3.15	D J 8	1.60	0.00	0.00
DS14	1.81	0.04	2.04	D J 9	3.89	0.04	0.95
DS15	1.71	0.04	2.17	D J 11	5.04	0.07	1.47
DS16	1.96	0.04	1.88	D J 13	3.07	0.00	0.00
DS17	1.99	0.06	3.22	D J 22	2.48	0.04	1.49
DS18	1.71	0.04	2.17	D J 23	1.39	0.04	2.67
DS19	2.84	0.04	1.30	D J 24	2.33	0.04	1.59
DS20	0.49	0.04	7.54	D J 26	2.48	0.04	1.49
DS21	2.86	0.04	1.29	<i>Energy Drinks (n = 2)</i>			
DS25	3.74	0.10	2.62	D E 7	6.19	0.04	0.60
DS27	1.49	0.04	2.48	D E 12	8.41	0.07	0.88

The average concentrations (mean \pm SD) of citric acid in energy drinks DE (n = 2) were 7.3 ± 0.06 g/L, in juice drinks DJ (n = 11) were 2.79 ± 0.04 g/L and in soft drinks DS (n = 14) were 1.76 ± 0.04 g/L, as shown in Fig.1. The small standard deviation shows consistency of citric acid concentration in each drinks type.

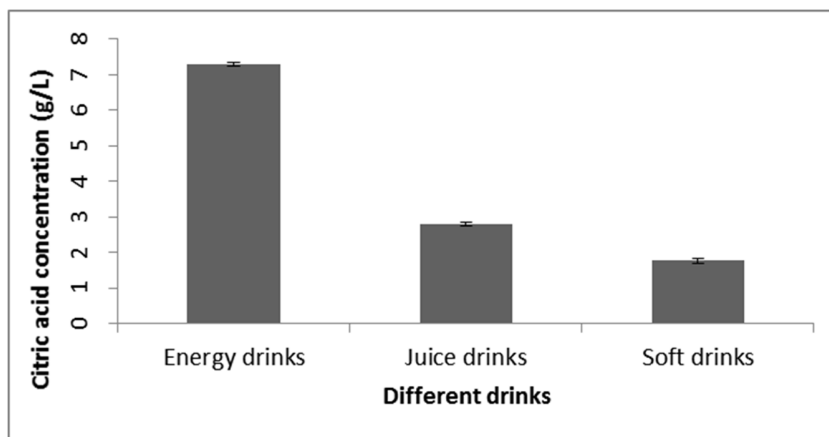


Fig 1: Mean concentrations of citric acid with SD (n = 3) in samples of soft drinks, Juice drinks and energy drinks.

These results are complied with previously published results, which carried out for assessment of citric acid in commercially available juice products, in local supermarkets in Madison, Wisconsin and Winston-Salem, North Carolina, USA. The content of the citric acid concentration in commercially available juice products, excluding juice concentrate and fresh juice, was ranging from 1.01 to 7.44g/L³.

There are no recommended levels of citric acid in drinks. However, USA soft and fruit drinks contain varying quantities of 0.131- 0.350 % and 0.6-6%, respectively [18]. Citric acid with E330 code is permitted as a food additive in EU. It is also considered as GRAS (Generally Recognized as Safe) by the USFDA (US Food and Drug Administration). Citric acid is given a Quantum Satis status (QS: the amount which is needed to achieve the desired result) and its use is limited only by good manufacturing practice (GMP) [1]. Generally the use of citric acid in beverage and soft drinks reflects levels commonly found in natural fruits (Orange 1%, grapefruit 1.5% and lemon 2.5%). Actually, typical additions of 0.25 to 0.4% of the citric acid are used to enhance flavor and as a preservative agent [1].

Our results in this study showed citric acid percentage range from 0.05-0.84% including soft drinks range 0.05-0.37%, juice drinks range 0.14-0.5% and two energy drinks range 0.62-0.84%. These percentages are yet lower than levels in natural fruits. This data can be used as a guideline for formulating health policy.

4. Conclusions

Titration method is simple, accurate, precise, and rapid method to determine citric acid content of commercially available soft drinks and juice drinks. Our results showed a range of concentrations in different sample types (juice drinks, soft drinks and energy drinks). 22% of the samples were shown citric acid concentration higher than 3 g/L. Higher concentrations were reported in energy drinks compared to other two types. The data of this study can be used to formulate public health awareness. More studies are needed to evaluate the availability of citric

acid from different sources and to assess its effect on public health.

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