NF7016 - Food Chemistry and Analysis

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Determination of Salt Content in Food/Beverages (AES)

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Abstract

Salt content in a food product is a very important aspect required to be analyzed from a nutritional point of view. Excessive salt consumption is a big concern for individuals suffering from hypertension. A Microwave Plasma based Atomic Emission Spectroscopy (MP-AES) method is used to determine the salt content in two variants of Tomato juice, one was produced "From Concentrate" and one is claimed to be "100% Pressed Juice" bought from a supermarket chain named Sainsbury's. Accuracy of the standards were satisfactory (0.998), and the results showed that salt content was more in pressed juice (0.51g/100ml) as compared to juice made from concentrate (0.33g/100ml). When compared to the nutritional information on the labels of the product the results for Pressed juice corresponded closely to the label but in case of From concentrate juice the result came to be 0.10g/100ml lower than what is mentioned. This comparison is an important aspect for verifying Food authenticity.

Keywords: Salt, Sodium, Tomato juice, MP-AES.

Abbreviations: MP-AES – Microwave Plasma Atomic Emission Spectroscopy.

1. Introduction

Fresh Fruits and vegetable juices are rich in different essential components and nutrients, but these fresh juices have a limitation of shelf life as they are prone to contamination and spoilage. These spoilages contribute to economic losses in the food industry. This is the reason why packed juices are very convenient as they go through processing procedures and packaging process to extend the shelf life of the product without adding high amounts of preservatives (Khandpur and Gogote, 2016). The limited availability of fresh fruits and vegetables to certain vulnerable groups like elderly people also contributes to the preference of packed juices. Tomatoes with stand thermal processes well hence there is a wide range of convenient tomato products available in the market. Tomato juice has a wide range of uses other than simple beverage like it acts as an ingredient for many cooking recipes and is also considered healthy because of the high concentration of carotenoids and phenolic compounds (Wu, Yu and Pehersson, 2022).

Juice concentrates are rehydrated to produce packed juices which mention Form Concentrate in the label. These concentrates can be made by removing certain amount of free water from the food system and can be processed using various techniques like spray drying, thermal vapor recompression, vacuum pan evaporators and cryoconcentration (Adnan, Mushtaq and ul Islam, 2018).

In recent years 100% pressed juice or not from concentrate juice is marketed as high value product and always compared to From concentrate juices (Tian et al., 2018 and Yasunaga, 2020). This has created an issue of validating the authenticity of contents of 100% pressed juice and from concentrate juices because the potential of food fraud increases in case of these claims. There are numerous methods to check the contents of these juices like gas chromatography and spectroscopy but according to (Xu et al., 2020) there hasn't been a significant method to differentiate between them on the basis of metabolomics. Food authenticity is a very big concern because it prevents the manufacturers from committing fraud and helps the general population to be aware of what they are consuming because every individual has different preferences and dietary restrictions for example in case of salt content determination, problem of high blood pressure or hypertension is very commonly detected in adults and hence they reduce their daily salt intake to keep their blood pressure in check. Tomato juice is considered to be rich source of antioxidants and good for preventing coronary heart conditions (Sánchez-Moreno et al., 2006). If the label doesn't show the accurate salt content present in that particular product it can affect the daily salt intake quantity and can cause adverse effects on health.

Salt has the chemical name Sodium Chloride (NaCl) in which cation Sodium is very important to regulate osmotic pressure in the body. The balance of excretion and intake is very necessary for cellular structure and is regulated by the kidney. Kidney can regulate the sodium level according to the response created by heart as the blood plasma volume increases. If sodium intake is high the functioning of kidney will be affected and hence it cannot regulate the sodium content which will indirectly affect blood pressure in future creating cardiovascular problems. Although low sodium levels also have adverse effects on body as it increases the aldosterone levels in the body. Hence monitoring balanced salt intake is very crucial to prevent long term problems in body (O'Donnell, Mente and Yusuf, 2015).

Sodium chloride has two important roles in a food system, first is adding taste to the product and second acting as a class 1 preservative. Salt changes the osmotic pressure of environment near spoilage causing microbial organisms which execute forced osmosis in the cells and destroying them. Salt has been used as a preservative for ages and is mentioned in many traditional recipes. It also affects oxygen solubility and making it scarce for to the aerobic microorganisms (Dwivedi et al., 2017). Hence it is a very common practice to add salt in packed juices.

Microwave plasma atomic emission spectroscopy (MP- AES) is widely used for food analysis especially mineral content like sodium potassium magnesium and other elements. Brzezińska-Rojek et al., 2023 used MP-AES method for analysis of 19 mineral elements present in beetroot based dietary supplements. MP-AES came in use in recent years research labs and food industries as it is time efficient and is capable of detecting even trace concentration of element in the food system. There are many methods for elemental analysis like atomic absorption spectroscopy and inductively coupled plasma mass spectrometry which work on different variations of spectroscopy-based principles. AES works on the principle of quantifying emissions of radiations coming from atoms in excited states. This method has widely replaced the traditional wet chemistry method for mineral analysis due to its simple and hassle-free approach. Individual elements release a characteristic wavelength when because of an energy source, the electrons in the atom move from ground state to low energy state and is analysed by the program using Maxwell- Boltzemann Equation. In the case of MP-AES the energy source is microwave plasma. (Yeung, Miller and Rutzke, 2017).

2. Materials and Methods

2.1 Materials and Reagents

Common table salt for the standards and juice samples were obtained by ordering from website of Sainsbury's supermarket. Distilled water was produced by a lab scale distilling unit. MP-AES equipment used is manufactured by Agilent technologies, Model number 4100 as shown in Figure 2.1.

Glassware and equipment used- 50ml Volumetric flasks, 100ml volumetric flasks, 500ml beakers and Auto-pipettes.

For the procedure instructions were followed as mentioned in the *Food analysis laboratory manual* by (Nielsen, 2010). For preparing sodium standards for dilution, 1g of Common salt was used in distilled water to plot the sodium standard calibration curve.

Fig. 2.1 MP-AES equipment 4100 Agilent Technologies

2.2 Sample Preparation

For sample preparation sealed Tetra packs of two variants of tomato juices were used. One is not from concentrate 100% pressed juice (Fig 2.2.1) and other from concentrate (Fig 2.2.2). For dilution of this sample 0.1ml of sample was taken using pipette and injected into 100ml volumetric flask and distilled water was added till the mark to make the solution up to 100ml. So 0.1% juice sample were prepared.

There are two hashing methods used for sample preparation, wet ashing and dry ashing. In dry ashing method vacuum oven is used to dry the sample and then HCL added for dilution. In wet ashing method nitric acid is used for digestion of the sample.

Tomato juice is a low viscosity liquid sample there was no need for ashing and direct dilution was done.

Figure 2.2.1 100% Pressed Juice

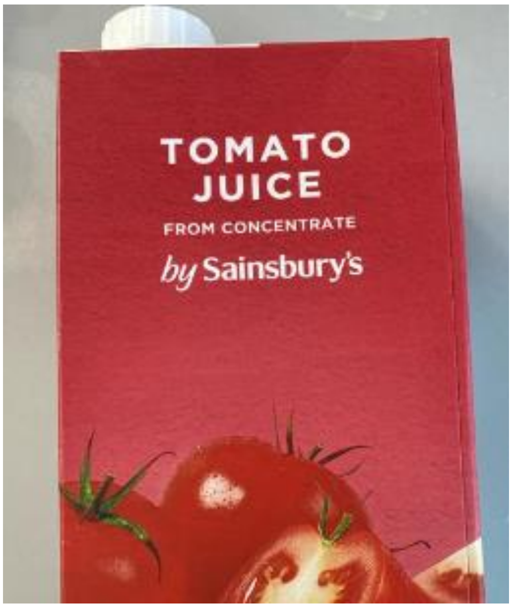

Figure 2.2.1 From Concentrate Juice

2.3 Sodium Standards Preparation

Calibration curve is prepared using sodium standards and 8 standards were prepared using common salt in 50ml volumetric flasks. Figure 2.3 has some standards which were prepared. The standards were made in ppm Parts per million unit (1mg/litre).

Blank- Only distilled water.

1000 ppm standard- 1g common salt in 1000ml water.

100 ppm standard solution- 5 ml from 1000 ppm standard and add up to 50ml distilled water in volumetric flask.

25 ppm standard solution- 12.5ml from 100 ppm standard and add up to 50ml distilled water in volumetric flask.

12.5 ppm standard solution- 25ml from 25 ppm standard and add up to 50ml distilled water in volumetric flask.

6.25 ppm standard solution- 25ml from 12.5 ppm standard and add up to 50ml distilled water in volumetric flask.

3.13 ppm standard solution- 25ml from 6.25 ppm standard and add up to 50ml distilled water in volumetric flask.

1.56 ppm standard solution- 25ml from 3.13 ppm standard and add up to 50ml distilled water in volumetric flask.

0.78 ppm standard solution- 25ml from 1.56 ppm standard and add up to 50ml distilled water in volumetric flask.

Due to limitations of MP-AES equipment, only standards from 0.78 to 6.25 were used for calibration as it was showing reading error out of this range.

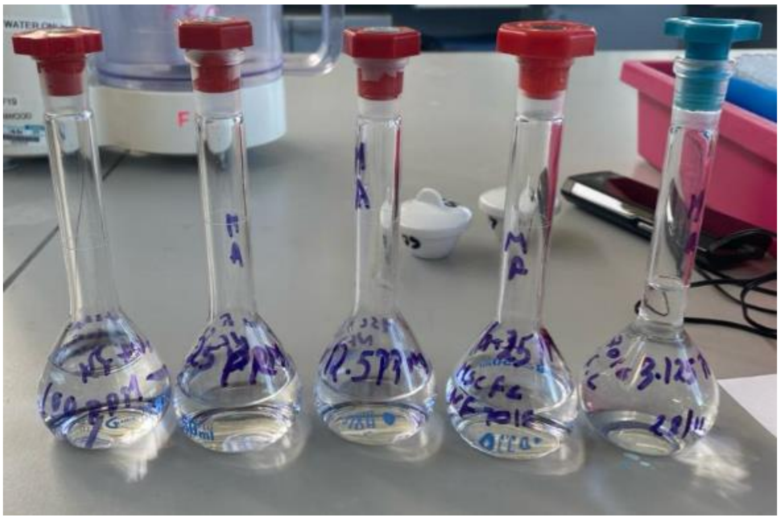

Figure 2.3 Sodium standard solutions

2.4 MP-AES Analysis

Agilent Technologies MP-AES 4100 equipment was used for sample analysis to determine salt content in the juice samples. The wavelength for Sodium was set at 588.995nm (Nelson et al., 2015). This equipment has a magnetron which produces electromagnetic energy at 2.5 GHz focused on the torch to form plasma as a light source. The continuous supply of nitrogen is provided by nitrogen generator which goes into the torch where the plasma forms. Sample is absorbed through capillary tubes into the sample introduction system through a 3-channel peristaltic pump and is transferred to cyclonic spray chamber nebulizer at 340kPa pressure where aerosol is created and sprayed to the atomizer. The light wavelength which is passed through sodium passes through monochromator which directs the wave to detector. The detector then gives the result for the intensity of wavelength on the screen which is calculated by a software MP Expert. (Drvodelic and Cauduro, 2013).

3. Results

Initially standard solutions were introduced one by one to get the intensity value for each standard solution of known concentration. Now concentration and value for the standards is given in Table 1. A graph for calibration curve was plotted using these values in which X-axis represented the concentration in ppm and Y-axis represented the intensity (Figure 3.1).

Sno.	**Standards**	**Concentration**	**Intensity**
1.	Blank	0	0
2.	Standard	0.78	496187.53
3.	Standard 2	1.56	935649.34
4.	Standard 3	3.13	1703121.35
5.	Standard 4	6.25	3165694.47

Table 1. Sodium Standard concentrations and Intensities

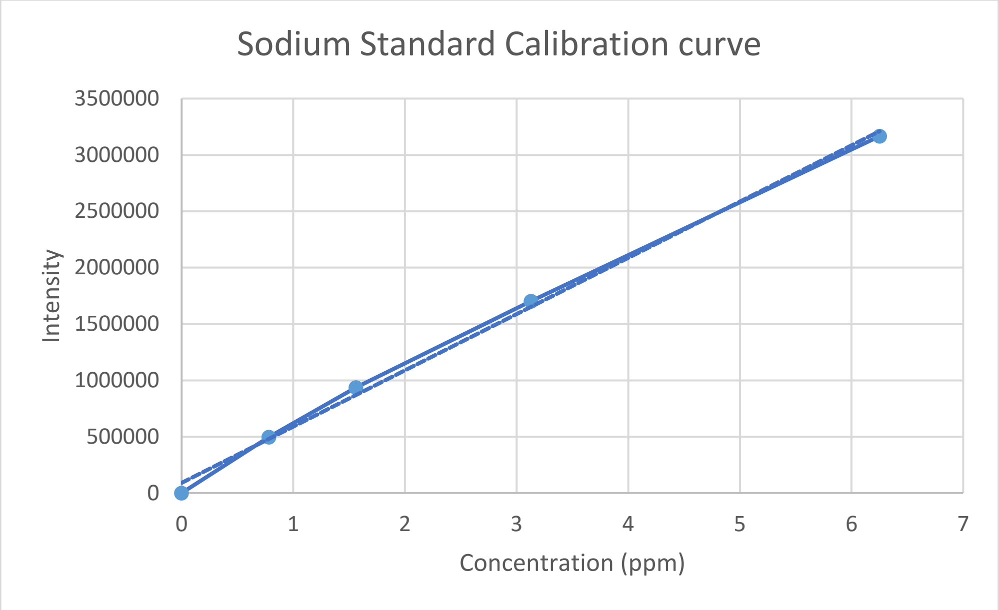

Figure 3.1 Calibration Curve

The value for correlation coefficient shows perfect linearity because $R^2 = 0.997$. The analysis time for each sample was approximately 30-45 seconds. After standards juice samples were introduced. The equation for graph is y = mx + c.

The equation deduced from standard curve was y = 499157x + 90106. The 'm' and 'c' values for this equation were obtained from Microsoft 365 Excel insert equation feature in the graph.

By putting the intensity values for juice samples in 'y' variable and then X will be our sodium concentration in ppm.

The intensity values for juice samples are mentioned in Table 2.

Sample 1 (Pressed Juice)	1109719.73
Sample 2 (Concentrate)	752001.85

Table 2. Intensity for Juice samples

So,

For 100% Pressed Juice sample the sodium concentration will be-

1109719.73 = 499157x + 90106

x = (1109719.73 - 90106)/ 499157

x = 2.0426 ppm

1ppm = 1mg/ litre So, 2.0426 ppm = 2.0426 mg/ litre

Converting it to per 100ml so divide by 10.

0.20426mg/100ml. This is the concentration for diluted 0.1% Pressed Juice sample. To get the sodium content for original juice sample multiply by the dilution factor which is 1000.

The amount of Sodium present in the sample is 204.26mg/100ml.

Concentration of salt is determined by Salt equivalent which is

Salt concentration = Concentration of Na x $\frac{\text{Molecular mass of Nacl}}{Molecular\ mass\ of\ Na}$

Salt content in the juice = 204.26 x (58.5/ 23) = 518.82 mg/100ml

To convert it to g/ 100ml which is mentioned in the label of packaging 1g has 1000mg So, The Salt concentration for Pressed juice will be 0.51g /100ml.

For 100% Pressed Juice sample the sodium concentration will be-

752001.85 = 499157x + 90106

x = 1.3260 ppm

1ppm = 1mg/ litre So, 1.3260 ppm = 1.3260 mg/ litre

Converting it to per 100ml so divide by 10.

0.13260 mg/100ml. This is the concentration for diluted 0.1% Pressed Juice sample. To get the sodium content for original juice sample multiply by the dilution factor which is 1000.

The amount of Sodium present in the sample is 132.60 mg/100ml. Concentration of salt is determined by Salt equivalent which is

Salt concentration = Concentration of Na x $\frac{\text{Molecular mass of Nacl}}{Molecular\ mass\ of\ Na}$

Salt content in the juice = 132.60 x (58.5/ 23) = 336.8 mg/100ml

To convert it to g/ 100ml which is mentioned in the label of packaging 1g has 1000mg So, The Salt concentration for Pressed juice will be 0.33g /100ml.

The values which were mentioned on the label of juices and the results calculated are compared in Table 3.

Samples	**Value on Label**	**Value from result**	**Difference**
Pressed Juice	0.50	0.51	0.01
From Concentrate juice	0.43	0.33	0.10

Table 3. Result comparison

The difference in From concentrate juice value is significantly higher than the Pressed Juice. There are multiple possibilities for this error. Lubinska-Szczygeł et al., (2023) also used the MP-AES for determination of Sodium and other macroelements in their Citrus peel based by-products. Authors have taken ± 0.02g/kg error range in the results. MP-AES provided fast results and was efficient with accuracy and hence can be recommended routine analysis in small to medium sized enterprises or for lab based analysis. To check the accuracy of this method more samples can be made or analysis of samples with known concentration can be analysed after calibration According to the values presented in Table 3 pressed juice had more salt content.

4. Discussion

Fruit juice concentrates go through a lot of processing procedures which cause loss of heat labile Vitamins and active compounds. Fruit juice concentrates are easier to preserve as they have low water activity and are easy to handle ask compared to raw tomatoes. Compared to 100% pressed juice, the juice which comes from concentrate need less barriers to prevent microbial spoilage. The reason for high salt content in pure juices is because salt has anti-microbial properties hence it is added in larger amounts due to high water activity and raw materials. Juice from concentrates are necessary to increase availability because fresh fruits or juices are not an option in many locations due to geographical constraints as specific species grow in different regions with favourable temperature zones. A large percentage of fruits are seasonal hence maintaining availability of the stock can be only achieved through storage and processing for preservation (Adnan, Mushtaq and ul Islam, 2018). From an economic point of view it is better to transport the concentrates to the targeted region and then process the juices by rehydrating although multiple dehydration an rehydration steps may affect the nutritional and sensory properties of juices.

In case of result value in From concentrate juice, The difference in values can be due to human errors like error in sample dilution, malfunctioning of auto pipette, manufacturing defect in volumetric flasks, cross contamination of samples. These errors can be rectified by conducting the experiment again with different approach and equipments. The bigger concern is if the values are a consequence of food fraud. Salt is a macroelement needed by the body for regulating blood pressure. So if the company is claiming the amount of salt on label and adding decreased amount of salt in the actual product during processing then it is a direct violation of food authenticity (Reid, O'donnell and Downey, 2006). This is considered as a fraudulent and deceptive practice because the decreased salt content will also affect the shelf life of product. The Date of Expiry is not valid if there is an unprecedented change in recipe or processing technique which were not considered during the shelf life study of the product.

Reid, O'donnell and Downey, (2006) have discussed different spectroscopy techniques to test for adulterants and determination of food authenticity in food products. Though MP- AES has some range limitations as compared to Inductively coupled mass spectroscopy but it is an effective rapid method.

5. Conclusion

Salt determination for two variants of Tomato juice were done, both from same brand using MP- AES method. The method for sample preparation was straightforward and the equipment was easy to use, reliable, accurate and timesaving. Concentration of salt in pressed juice sample was more than from concentrate juice and was similar to the amount mentioned on the label. The result for From concentrate juice can be due to error or after further analysis can be detected as a food authenticity issue. Pressed juice is healthier because it retains most of the nutritional properties of tomato but if the consumer is a patient of hypertension then From concentrate juice can fit their preferences. The product can be consumed by all age groups as it has moderate levels of salt and only contains 10-15% of the Reference intakes mentioned in the standards for adults.

References

Adnan, A., Mushtaq, M. and ul Islam, T., 2018. Fruit juice concentrates. In Fruit Juices (pp. 217-240). Academic Press.

Brzezińska-Rojek, J., Rutkowska, M., Ośko, J., Konieczka, P., Prokopowicz, M. and Grembecka, M., 2023. Evaluation of the Safety and Potential Benefits of Beetroot-Based Dietary Supplements According to Their Elemental Composition. Biological Trace Element Research, pp.1-15.

Drvodelic, N. and Cauduro, J., 2013. Determination of metals in wine using the Agilent 4100 microwave plasma-atomic emission spectrometer. Application Note. Agilent Technologies, Victoria, Australia.

Dwivedi, S., Prajapati, P., Vyas, N., Malviya, S. and Kharia, A., 2017. A review on food preservation: Methods, harmful effects and better alternatives. Asian J. Pharm. Pharmacol, 3(6), pp.193-199.

Khandpur, P., & Gogate, P. R. (2016). Evaluation of ultrasound-based sterilization approaches in terms of shelf life and quality parameters of fruit and vegetable juices. Ultrasonics Sonochemistry, 29, 337–353. doi:10.1016/j.ultsonch.2015.10.008.

Lubinska-Szczygeł, M., Kuczyńska-Łażewska, A., Rutkowska, M., Polkowska, Ż., Katrich, E. and Gorinstein, S., 2023. Determination of the Major By-Products of Citrus hystrix Peel and Their Characteristics in the Context of Utilization in the Industry. Molecules, 28(6), p.2596.

Nelson, J., Hopfer, H., Gilleland, G., Cuthbertson, D., Boulton, R. and Ebeler, S.E., 2015. Elemental profiling of Malbec wines under controlled conditions using microwave plasma-atomic emission spectroscopy. American Journal of Enology and Viticulture, 66(3), pp.373-378.

Nielsen, S.S., 2010. Sodium and Potassium Determinations by Atomic Absorption Spectroscopy and Inductively Coupled Plasma-Atomic Emission Spectroscopy. Food Analysis Laboratory Manual, pp.87-93.

O'Donnell, M., Mente, A. and Yusuf, S., 2015. Sodium intake and cardiovascular health. Circulation research, 116(6), pp.1046-1057.

Reid, L.M., O'donnell, C.P. and Downey, G., 2006. Recent technological advances for the determination of food authenticity. Trends in Food Science & Technology, 17(7), pp.344-353.

Sánchez-Moreno, C., Plaza, L., de Ancos, B. and Cano, M.P., 2006. Nutritional characterisation of commercial traditional pasteurised tomato juices: carotenoids, vitamin C and radical-scavenging capacity. Food Chemistry, 98(4), pp.749-756.

Thangavelu, S., Chiranjivi Rao, K., & Rakkiyappan, P. (2003). Sodium content in juice of sugarcane clones and its relationship with other traits at different stages of maturity. Sugar Tech, 5(1-2), 85–88. doi:10.1007/bf02943772.

Tian, Y., Gou, X., Niu, P., Sun, L., & Guo, Y. (2018). Multivariate Data Analysis of the Physicochemical and Phenolic Properties of Not from Concentrate Apple Juices to Explore the Alternative Cultivars in Juice Production. Food Analytical Methods, 11(6), 1735–1747. doi:10.1007/s12161-018-1169-2

Wu, X., Yu, L. and Pehrsson, P.R., 2022. Are processed tomato products as nutritious as fresh tomatoes? Scoping review on the effects of industrial processing on nutrients and bioactive compounds in tomatoes. Advances in Nutrition, 13(1), pp.138-151.

Xu, L., Xu, Z., Kelly, S. and Liao, X., 2020. Integrating untargeted metabolomics and targeted analysis for not from concentrate and from concentrate orange juices discrimination and authentication. Food chemistry, 329, p.127130.

Yasunaga, N., 2020. Value and Consumption Conditions of Locally Processed Food: Market Strategy of Tomato Juice. Farm and Rural Community Management in Less Favored Areas, pp.127-146.

Yeung, V., Miller, D.D. and Rutzke, M.A., 2017. Atomic absorption spectroscopy, atomic emission spectroscopy, and inductively coupled plasma-mass spectrometry. Food Analysis, pp.129-150.