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DETERMINATION OF CALCIUM CONTENT IN RICE VARIETIES USING FLAME PHOTOMETRY AND TITRIMETRIC METHODS

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ABSTRACT

The study aims to determine the calcium content in the Indian rice varieties and to compare the result analysis between flame photometric and titrimetric methods. A total of eight rice varieties have been procured from the Indian market. The samples were extracted by liquid-liquid extraction and the supernatant was analysed by two methods *viz*. flame photometry and titrimetric methods. Comparative study between flame photometry and titrimetric methods indicates that there is a variation with a low correlation coefficient (r^2 = 0.79). Besides, the paired t-test for all rice samples showed that there was a statistically significant difference ($p < 0.05$) in calcium concentration determined by both the methods. Furthermore, the mean recoveries of both analytical methods (flame photometric & titrimetric methods) were found to be 99.26 ± 0.40 and 95.66 ± 0.57, respectively. It is quite clear that the flame photometric method offers more accurate results than the titrimetric method, thus making it more reliable in determining the calcium content in the rice samples. In addition, rice varieties (Boiled sella and Chhapi rice) with higher calcium content can be recommended for daily consumption among the population with lower plasma calcium.

Key words : Complexometric titration, Flame photometry, Food nutrition, Rice variety, Quality control.

Introduction

Calcium is an essential mineral for human health, playing a crucial role in bone and teeth formation, blood clotting and muscle function. Understanding the calcium content in rice is important for assessing its contribution to overall dietary calcium intake, especially in regions where rice is a staple food (Munshi *et al*., 2020). The determination of calcium content is important for regulatory compliance with food safety standards (Cormick *et al*., 2020). Reliable analytical methods can be crucial for the elemental assessment of food quality and safety requirements.

Flame photometry is based on atomic emission spectroscopy wherein atoms get excited by the excitation energy of the flame and the intensity of emitted radiations by the atoms as they return to the ground state. It is crucial to routinely analyze certain group 1 and group 2 metals and widely used in the beverage, agriculture and food processing industry (Chu and Taylor, 2016; Banerjee and Prasad, 2020; Ren *et al*., 2019; Chen *et al*., 2005; Pragathi, 2023). Flame photometry is often considered a cost-effective method for routine analyses and is generally less expensive compared to some other analytical techniques such as atomic absorption spectroscopy and ICP-MS (ion-coupled plasma mass spectrometry) (Siong *et al*., 1989 and Cubadda, 2004). Similarly, traditional methods such as titrimetric analysis can be used to determine the calcium element by complexometric titration (Simoes *et al*., 2020). Titrimetric methods are often more economical than some other analytical techniques. The equipment required is relatively simple, and titrants are generally less expensive compared to reagents used in other methods. Nevertheless, titrimetric methods may lack the sensitivity required for the analysis of trace or low-concentration analytes.

The rationale of this study was to explore flame photometry and titrimetric methods to evaluate the accuracy and practicality of these techniques in determining calcium content in rice. This can contribute to developing simple methods in the field of food analysis. Further, estimating calcium content in Indian rice allows for regional comparisons, helping identify variations that may exist due to geographical and agricultural differences (Kure *et al*., 2022). This information can be valuable for tailoring nutritional recommendations to specific populations with osteoporosis (Sumardiono *et al*., 2020 and Qi *et al*., 2019).

Therefore, the study aims to determine calcium content by flame photometric and titrimetric methods in different rice varieties readily available in the Indian market and compare the accuracy of sample results using both analytical methods.

Materials and Methods

Reagents : Calcium carbonate, acetic acid (3 %), disodium Ethylenediaminetetraacetic acid, mordant black T was purchased from Merck Life Sciences Pvt Ltd., Mumbai, India. All the other solvents and chemicals used were of analytical grade.

Stock solution of calcium carbonate : To prepare 1000 ppm stock solution of calcium ion, about 2.5 g of calcium carbonate was weighed, dissolved in 300 mL of distilled water and acidified with 10 ml of conc. HCl. The resultant solution was further diluted to 1 litre using distilled water.

Working standard solution : The calibration standards were prepared by appropriately diluting stock solution to produce the working standards of concentration ranging from 20 to 300 ppm. For instance, to prepare a dilution of 20 ppm, transfer 1 mL of stock solution into 50 mL of volumetric flask and dilute with distilled water up to the mark.

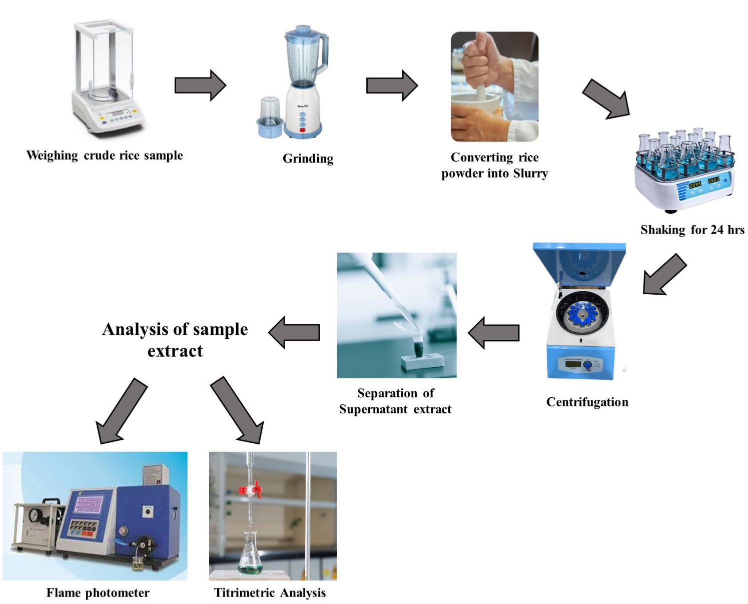

Fig. 1 : Schematic representation of sample extraction procedure.

Sampling preparation : Different varieties of rice samples were procured from the local market of Delhi. Accurately weighed 100 g of each rice sample in a weighing balance and then crushed with a household grinding machine. The powdered sample was further converted into a uniform mixture in a mortar pestle by adding 100 mL of acetic acid (3%) and triturating it for 25 to 30 minutes. The target analyte was extracted from the slurry by horizontal shaking for 24 hours at 35°C. The samples were then transferred in conical tubes and centrifuged at 5000 rpm for 10 min. The supernatant was separated into fresh tubes and stored in a refrigerator for further analysis. The sample procedure is summarized in Fig. 1.

Analytical methods

Flame Photometer : Flame photometer (Systronics Auto Ignition Model 1027) aspirates distilled water from sample holders until uniform flame is achieved and the desired element is selected from the list for automated selection of the wavelength filter. Initially, a calibration curve was generated by employing standard calibration solutions with known concentrations (20 to 300 ppm) of the calcium under investigation. The calibration data were stored in the developed method in a flame photometer. Subsequently, the prepared samples were aspirated into the instrument and automatically determined the concentration of a sample by the saved calibration data points and comparing its scale reading to the curve.

Complexometric titration : The titration was performed using 0.05M disodium EDTA which was standardized with zinc sulphate to confirm its concentration. 10 ml of rice sample extract was transferred into a 50 mL conical flask and dissolved in 50 mL of distilled water. Further, 10 mL of ammonia buffer solution was added to the mixture with two drops of mordant black T indicator. Then the resultant solution was titrated with 0.05 M disodium EDTA filled in a burette until a blue colour was obtained. The titration was repeated twice for each sample and the average titre value was calculated.

Equivalent factor

1 mL 0.05 M disodium edetate ≡ 0.005 g of Calcium carbonate

Formula used to determine calcium content in eight samples of rice :

$$\text{Calcium content} = \frac{(\text{Titre volume} \times 0.005)}{0.05} \times 100$$

Results and Discussion

The purpose of the study was to determine the calcium content in the varieties of rice available in the local market of northern India. A total of eight different rice samples were procured and labelled according to the vernacular names (Table 1). The rice samples were extracted into a neat sample according to the procedure mentioned in section 2.4. The calcium content was determined by using two methods *viz*. flame photometric and titrimetric (complexometry) methods. Out of 8 rice samples, Chhapi and Boiled sella revealed the highest calcium content and were found to be 32 and 29 mg/100g (flame photometric method) and 28.4 and 26 mg/100g (titrimetric method), respectively.

However, the result in Table 1 clearly shows that there is variation in the result from both analytical

Table 1 : Calcium content determined in the rice samples by flame photometry and titrimetric methods.

Sample ID	Rice variety	Ca content(mg/100g)	
		[a]Titrimetric Method	[b]Flame Photometric Method
Sample 1	Boiled sella	26	29
Sample 2	Chhapi	28.4	32
Sample 3	Parmal	21.4	31
Sample 4	Basmati	18.6	20.4
Sample 5	Jeeri	19.1	24
Sample 6	Golden sella	12.2	18
Sample 7	Brown rice	18.2	22.4
Sample 8	Golden Basmati	15.4	22

Each value is the mean of duplicate analysis
Standard deviation is 5.14[a] and 5.17[b]
Coefficient of variation is 25.8[a] and 20.8[b]
t-test is 2.37 ($p < 0.05$)

Table 2 : Percent recovery study of calcium determined by Flame photometric method.

Concentration taken (mg/100mL)	Found concentration (mg/100mL)	% Recovery
5	4.96	99.2
10	9.89	98.9
20	19.94	99.7

Mean: 99.26, Standard Deviation:0.40, % RSD: 0.41

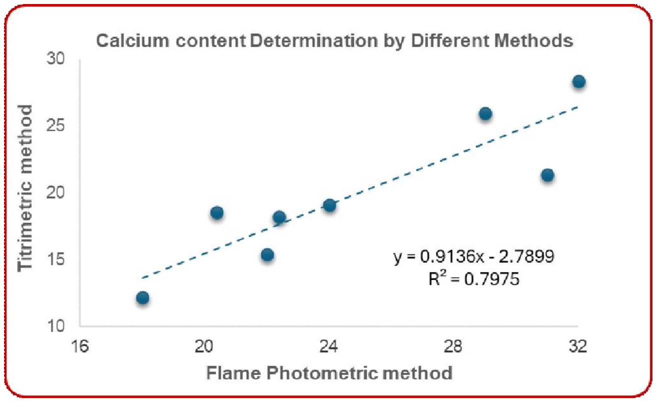

Fig. 2 : Calcium content determination in rice samples by flame photometric and titrimetric methods.

methods. The correlation coefficient ($r^2 = 0.79$) obtained for all 8 pairs of results by different analytical methods was found to be lesser than 0.99 and the data is scattered but not linear (Fig. 2). This highlights that the calcium detection sensitivity of two analytical methods differ rather than homogenous. On the other hand, the results of the paired t-test for all rice samples showed that there was a statistically significant difference ($p <0.05$) in calcium concentration determined by the flame photometry and titrimetric methods. The coefficient of variation for each series revealed more variance (25.8) in the case of the titrimetric method, while relatively lesser variation (20.8) was observed in the flame photometric method. The pooled standard deviation obtained for all 8 rice samples studied was 5.17 and 5.14, respectively, for the flame photometric and titrimetric methods. The significant difference can be attributed to the higher sensitivity of flame photometric over titrimetric methods.

Besides, to validate the performance of the analytical methods, recovery studies have been carried out for three known concentrations from the range of 2-30 mg/100 mL. The mean recoveries of both analytical methods (flame photometric & titrimetric methods) were found to be 99.26 ± 0.40 and 95.66 ± 0.57, respectively (Tables 2 and 3). It is quite clear that the flame photometric method offers more accurate results than the titrimetric method, thus making it more reliable in determining the calcium content in the rice samples. The similar observation was also reported in the determination of salinity in processed foods as well as in soya sauce wherein flame photometer showed rapid and more accurate results (Chen *et al.*, 2005; Chu and Taylor, 2016). In addition, this method can serve the purpose of elemental estimation in a laboratory which are not equipped with costly instruments such as atomic absorption spectrometry.

It is further proposed that the rice variety (Boiled sella and Chhapi rice) can be recommended for daily

Table 3 : Percent recovery study of calcium determined by Titrimetric method.

Concentration taken (mg/100mL)	Found concentration (mg/100mL)	% Recovery
5	4.75	95
10	9.6	96
20	19.2	96

Mean: 95.66, Standard Deviation:0.57, % RSD: 0.60

consumption among the population with lower plasma calcium as a source of food supplement. This piece of work can be used as a piece of general information among consumers to get aware of calcium content in different rice varieties available in the local market.

Conclusion

The calcium content in the eight different varieties of rice that are consumed widely in the Indian market was determined by two analytical methods *viz*. flame photometry and titrimetry. Boiled sella and Chhapi rice revealed the highest calcium content estimated by both selected methods. The result from the analytical methods showed statistical differences because of the higher sensitivity of the flame photometer over the titrimetric method. In addition, the flame photometric method was found to be more accurate and reliable. The method can also be used for routine analysis for estimation of calcium content in different foodstuffs.

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Declaration

The authors do not have any conflict of interest.

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