STUDYING CONCENTRATION OF MINERALS IN SOME LOCAL AND IMPORTED RICE BY USING ENERGY-DISPERSIVE X-RAY FLUORESCENCE (EDXRF)

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ABSTRACT

Rice is an important food in the world, particularly in Asian countries, it is consumed as a staple food. This study analyzes the 5 different samples (imported basmati rice such as India-1, India-2, India-3, Iraqi- PDS, Russian and local Kurdish rice) by using most covenant, economical technique Energy-dispersive X-ray Fluorescence (EDXRF) spectroscopy). Techniques were used after grounding, and pressing the samples in the sample containers to analyze 15 minerals (Al, Ca, Cl, Cr, Fe, K, Mg, Mn, P, S, Si, Ti, Zn, Na, and Sr). The results of variance showed that there is a significant difference ($p<0.01$) in the minerals content of the rice samples and the main trends show that all rice samples were not contained sodium (Na) and strontium (Sr). On the other hand, the dominant minerals in all samples were silicon (Si) > phosphate (P) > Sulfur (S) than other minerals, while rice as a plant is considered to be one of the most accumulator of Si, P and S minerals. In conclusion, EDXRF offers a reliable and fast approach to screen of minerals in different sample of foods and the differences in minerals content in the study may refer to the influence of different factors such as irrigation, rice verities, type of soil, and fertilization.

Keywords: EDXF; Rice; Minerals content; Spectroscopy; chemical analysis; micronutrient.

دراسة تركيز المعادن في بعض أنواع الأرز المحلية والمستوردة عن طريق استخدام الأشعة السينية المشتتة للطاقة

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المستخلص:
يعتبر الارز احد الاغذية المهمة عالمياً، وخاصتا في الدول الآسيوية، حيث يعتبر كغذاء أساسي. الدراسة بحثية تمت بتحليل 5 عينات مختلفة من الأرز (رز بسمتي مستورد مثل الهندي -١، الهندي-٢، الهندي -٣، عراقي (لبطاقة تموينية) ، رز المستورد الروسي مع عينة رز المحلي مثل الأرز الكردي) باستخدام احد التقنيات القابلة للتطبيق و الاكثر اقتصادية, المعروفة بتشتت طاقة الاشعة السينية الطيفي. بعد جمع العينات تم التحليل 15 معدنًا مثل (الالمنيوم، الكالسيوم، الكروم، الحديد، البوتاسيوم، المغنيسيوم، المنغنيس، الفوسفور، الكبيريت، السليكون، التيتانيوم، الزنك، الصوديوم، السترونتيوم). أظهرت نتائج في عينات الارز أن هناك فروقات معنويا بمستوى ($p<0.01$) في محتوى المعادن و نتيجة كانت أن جميع عينات الارز لا تحتوي على الصوديوم والسترونتيوم. ومن جانب أخر ، كانت السيليكون ثم الفوسفات ثم الكبريت من المعادن السائدة في كل العينات مقارنتا بالمعادن الأخرى ، بينما يعتبر الارز من نباتات أكثر تراكما للسيليكون و الفوسفات و الكبريت. في الختام ، يقدم تقنية تشتت الطاقة بالأشعة السينية الطيفي طريقا موثوقًا وسريعًا لفحص المعادن في عينات الأطعمة المختلفة ، وقد تشير الاختلافات في محتوى المعادن في الارز إلى تأثير عوامل مختلفة مثل ماء الري ، صنف الارز ، ونوع التربة و سماد.

Introduction

Rice (*Oryza sativa L.*) is a most important dietary component in most of the countries. It is more consumed in Asia and Africa and fewer in the European countries [1] . Asia as archaeological sources has shown that it has been planted before the 4000 AD. Every continent on the planet produces rice except Antarctica [2]. Rice also highly consumed in Iraq, it is essential food that must have in the meals [3]. White rice is the name given to milled rice or polished rice that has had its husk, bran, and germ removed [4]. Rice is a rich source of starch 75%–80%, also contain 12% water, and only 7% protein [5, 6]. Minerals that found in rice such as calcium (Ca), magnesium (Mg) and phosphorus (P) are present along with some traces of iron (Fe), copper (Cu), zinc (Zn) and manganese (Mn) [7]. Although the nutritional values of rice especially minerals, varies with different varieties, soil fertility, fertilizer application, the processing of rice such as polishing and other environmental conditions. Due to its nutritional quality and higher digestibility, rice is considered as the best cereals [8]. The amount of macronutrients such as P, S, K, Ca, and Mg in rice are higher usually than micronutrients like Mn, Fe, Cu, Zn, and Se, while Aluminum (Al) and heavy metals are undesirable because of their harmful effects [9]. Consumers have become more quality and healthy aware about the rice cultivars they consume. Hence, it needs to be focused on the quality of rice along with production. When farmers become conscious of their rice quality, they were driven to produce better quality rice [10, 11]. The rice quality, including minerals and toxic elements, also their percentages, is closely related to the health of people who take rice as the main food [12]. Meanwhile, elements toxicity has proven to be related with several

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health risks such as cancer and neurological disorder. Various public health measures have been undertaken to control, prevent and treat metal toxicity occurring at various levels, such as occupational exposure, accidents and environmental factors [13].

While, the quality of food become important, the needs for high performance analysis also is required in terms of quality analysis of minerals in food samples for that reasons a fast, reliable and simple technique is required, the analytical technique of Energy Dispersive X-ray fluorescence (EDXRF) spectroscopy work with simple analytical preparation compared to other analytical techniques for analyzing food samples. EDXRF also offers numerous benefits including non-destructive analysis and multi-element qualitative and quantitative analysis. Analysis can be performed on a wide range of types of sample including solids, liquids, powders, pastes and films [14]. The major advantage of EDXRF is that equipment is simpler and cheaper with lower output X-ray tubes, albeit with lower resolving power and higher background. If compared to other technique [15]. This technique could use in agriculture, petrochemical, general analysis, cement and so many other areas. In the area of plant and food science, applications have been found for EDXRF in the determination of iodine and calcium in bread improver [16], minerals in milk powder [17, 18], Fe, Cu and Zn in food premixes [19, 20], and phosphorus in potato starch [21].

This study has been designed to measure the concentration of minerals in polished imported rice with local Kurdish rice at local Iraqi market by using new technology Energy Dispersive X-ray fluorescence (EDXRF) for analyze.

Materials and Methods

Materials

In total, six commonly consumed and popular white rice varieties were purchased from a local Supermarket in Kurdistan region, Iraq. Imported rice varieties with one sample from local area, which were used for comparison included: (India-1, PDS-Ration card rice (the rice that come from Iraqi public distribution system (PDS)), India-2, India-3 rice, Russian round rice, and local Kurdish round rice. All sample milled rice samples were individually ground to a fine powder (30 mesh size) using a mill (Severin, made in Germany), packed in air tight plastic bags and were stored at 4°C for future analysis. In addition, the Oxford Instruments X-Supreme 8000 model (made in USA) uses the analytical technique of Energy Dispersive X-ray Fluorescence (EDXRF) spectroscopy [22]. with automatic hydraulic press (made in Germany) to press the sample in the poly ethylene cups of instrument. The analyzing conducted at the mechanical and Mechatronic department, College of Engineering, Salahaddin University-Erbil.

Sample Preparation and Presentation

In order to ensure the high accuracy required for cost optimized process control, firstly, the samples were individually ground to a fine powder (30 mesh size) using a mill (Severin), packed in air tight plastic bags and were stored at 4°C for future analysis. After that, the resulting powder pressed into a strong pellet (40mm diameter) using an automatic hydraulic press set to 20 tons. The pellet was then placed into a standard Oxford Instruments' sample holder and the holder placed onto the X-Supreme sample carousel in the sake of minerals analyzing such as Aluminum (Al), Calcium (Ca), Chlorine (Cl), Chromium (Cr), Iron (Fe), Potassium (K), Magnesium (Mg), Manganese (Mn), Phosphate (P), Sulfur (S), Silicone (Si), Titanium (Ti), Zinc (Zn), Sodium (Na), and Strontium (Sr).

Qualitative Analysis

The Oxford Instruments X-Supreme 8000 uses the analytical technique of Energy Dispersive X-ray Fluorescence (EDXRF) spectroscopy and this offers the ability to simply and rapidly observe a sample's X-ray elemental spectrum.

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EDXRF was fitted with a 10 place auto sampler. Total analysis time for each sample was 186 s. Scans were conducted in sample cups assembled from 21 mm diameter Al cups combined with polypropylene inner cups sealed at one end with 4 µm Poly-4 XRF sample film. Cups containing samples were gently shaken to evenly distribute grains. sample mass was fixed at 4 g. According to the manufacturer, the X-Supreme 8000 scans a circle of 21 mm diameter with the sample spinner on. All scans in this study were performed in this mode, so the scanned area was 346 mm2. For the determination of Al, Si, P, Mn and Fe in Iron ore, two optimized "instrument conditions" are used. For the first determination of these low atomic number elements, i.e. Al, Si and P, the X-Supreme uses the combined power of the Tungsten Target X-ray tube, and high resolution of the Silicon Drift Detector. The Tungsten Target X-ray tube provides excellent elemental excitation, for example measuring the lowest atomic number of Al2O3 (leading to high sensitivity), while the high elemental resolution of the high performance Silicon Drift SDD allows the determination of the element P at a low concentration of approximately 250 parts per million (0.025%) even in the presence of high concentration of Si (50%). Whereas, for the second determination, the detect high atomic number elements such as Fe and Zn the X-Supreme uses power of the Tungsten Target X-ray tube [14, 22].

Statistical data analysis

The rice samples data were analyzed by one-way analysis of variance (ANOVA) for each minerals and the means were compared by (Duncan) test as described by Statistics Analysis Program (XLSTAT, 2016) with significant differences were defined at ($P < 0.01$) level. Results of minerals concentration were expressed as mean ± Standard error of the mean (SEM).

Results:

The EDXRF spectroscopy technique was used for detecting minerals in the different imported rice and local Kurdish rice samples and results of 15 minerals (Al, Ca, Cl, Cr, Fe, K, Mg, Mn, P, S, Si, Ti, Zn, Na, and Sr) concentration were shown in the (Table1). In addition, the total milled rice samples with three replicate for each samples were from different origins such as Kurdish, Russia, India, and Iraqi Rational card-PDS rice). The samples are represented as India-1 Sella basmati rice, Iraqi PDS rice, India-2 Sella basmati rice, India-3 Sella basmati rice, Russian round rice, and local Kurdish round rice. Analytical results presented as mean ± standard error of mean in the (Table1) and showed that the all rice samples were zero mg/kg contain of Na and Sr. On one hand, Rice samples of Iraqi PDS and India-3 not contain Cr, whereas, the India-1, India-2 and India-3 rice were zero mg/kg content of Zn. On the other hand, the maximum amount of mineral was Si for all the samples that ranged between 41.564 mg/kg to 52.347 mg/kg for India-1and Iraqi PDS, respectively.

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Table 1. The mean and standard error of minerals content that were analyzed by EDXRF that were conducted by ANOVA test and Duncan comparison at level of P < 0.01.

Samples	Minerals (mg/Kg) (Mean ± Standard error of mean (SEM))														
	Al	**Ca**	**Cl**	**Cr**	**Fe**	**K**	**Mg**	**Mn**	**P**	**S**	**Si**	**Ti**	**Zn**	**Na**	**Sr**
India-1	3.54 ± 0.02^{f}	1.02 ± 0.01^{d}	1.43 ± 0.01^{c}	0.07± 0.09 ^{a}	0.13± 0.01^{a}	9.15± 0.01^{a}	1.90± 0.02^{d}	0.09± 0.01^{ab}	25.07± 0.04^{c}	15.89± 0.04^{a}	41.56± 0.01^{f}	0.07± 0.01^{a}	0.0± 0.01^{d}	0	0
PDS-Iraqi	5.65± 0.02^{a}	0.86 ± 0.01^{e}	1.01± 0.01^{e}	0.00± 0.09 ^{b}	0.07± 0.01^{d}	4.28± 0.01^{f}	1.30± 0.02^{f}	0.12± 0.01^{ab}	18.41± 0.04^{f}	15.68± 0.04^{b}	52.34± 0.01^{a}	0.06± 0.01^{cd}	0.14± 0.01^{a}	0	0
India-2	3.81± 0.02^{e}	1.03 ± 0.01^{c}	1.08 ± 0.01^{d}	0.02± 0.09 ^{b}	0.09± 0.01^{c}	7.99± 0.01^{b}	1.71± 0.02^{e}	0.07± 0.01^{ab}	20.37± 0.04^{e}	15.26± 0.04^{d}	48.45± 0.01^{b}	0.06± 0.01 ^{c}	0.0± 0.01^{d}	0	0
India-3	4.53± 0.02^{d}	0.82 ± 0.01^{f}	0.92 ± 0.01^{f}	0.00± 0.09 ^{b}	0.068± 0.01^{e}	7.46± 0.01^{c}	2.33± 0.02^{c}	0.05± 0.01^{b}	22.19± 0.04^{d}	15.41± 0.04^{c}	46.03± 0.01^{c}	0.06± 0.01^{b}	0.0± 0.01^{d}	0	0
Russian	4.70± 0.02^{b}	1.06± 0.01^{a}	1.60 ± 0.01^{a}	0.01± 0.09 ^{b}	0.06± 0.01^{e}	6.26± 0.01^{e}	3.27± 0.02^{a}	0.15± 0.01^{a}	27.20± 0.04^{a}	11.25± 0.04^{e}	44.21± 0.01^{d}	0.06± 0.01 ^{cd}	0.07± 0.01 ^{b}	0	0
Local Kurdish	4.58± 0.02^{c}	1.04 ± 0.01^{b}	1.54 ± 0.01^{b}	0.06± 0.09 ^{a}	0.13 ± 0.01^{b}	7.12± 0.01^{d}	2.68± 0.02^{b}	0.06± 0.01^{b}	26.34± 0.04^{b}	11.10± 0.04^{f}	43.35± 0.01^{e}	0.06± 0.01^{d}	0.05± 0.01 ^{c}	0	0

a, b, c, d, e, f = Different alphabets in the same column means significantly different at P < 0.01.

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Discussion:

Nowadays, there are many new high performance and low cost technologies use to detect amount of minerals in foods such as EDXRF spectroscopy was a reliable and quick alternative to traditional analytical methods, as well as it is allowing for much larger sampling regimes in relatively shorter times and could be applied in other different areas of studies [23]. Minerals are vital nutrient elements that required in the normal diet as it can support health to an ideal level and some of them may toxic for health if consumed in large amount or expose to it. Minerals are naturally found in different kinds of food, and are at higher amount in those, which are grown up directly from the soil such as rice. With concern to metals, human interference such as use of fertilizers in agriculture and mining can be the route cause for their accumulation. Since rice is a staple food, growing them in contaminated soils with toxic compound can cause serious health risks to humans [24]. Generally, there is a significant difference ($P < 0.01$) between minerals concentration in all the samples. The minerals content in Russian round rice and local Kurdish round rice were close in quantity and the other rice basmati samples were nearly same in amount of minerals such as showed in (Table 1). Minerals such as P and S were high in all six samples if compared of the rest of other minerals in the study especially P that was 27.231 mg/kg for Russian rice, whereas, the highest amounts of S were seen in India-1 rice (15.893 mg/kg). On the other hand, the S content in the round rice such as Kurdish (11.10 mg/kg) and Russian rice (11.25 mg/kg) was lower from the other rice samples, however the Russian was 27.20 mg/kg and Kurdish was 26.34 mg/kg in the P content and higher than other rice samples. In addition, S and P play essential role in cellular energy, transfer of energy and protein metabolism [25]. Macronutrients such as Na and Sr, were not found in all samples. The level-off of Na in rice can be advantageous for those people who require to restrict the intake owing to high hypertension associated problems [26]. Also the Sr have a side effect on health especially renal function [27] so that is mean this rice sample will be safe in terms of Sr. also the concentration of Sr In Uruguayan rice was close to our results that was contain a very small amounts[28] . According to [29], the samples of Iraqi PDS rice and India-3 rice not contain Cr and other samples have a small amount that were not exceed more than 0.070 mg/kg and lower than safe limits that is conclude that in terms of Cr concentration the all rice samples is acceptable for health. On the other hand, the all samples contain small quantity of Ti that is between 0.065 mg/kg and 0.078 mg/kg and it source may came from soil or fertilizer or during whitening process for rice to look more bright and white [30, 31]. Among all the six rice samples analyzed, the macro-mineral K, which was between 4.287 to 9.154 mg/kg, and for Mg, was between of 3.273-1.334 mg/kg, respectively, that result of study was comparable to the observations of Malaysian rice [24]. In addition to these, the essential micronutrients such as Mn, Zn, Fe and macronutrient Ca were found in low amounts in all samples but Ca was higher than Mn, Zn and Fe. Generally, low level of Zn in polished rice is reported compared to unpolished rice [32]. In this study, Zn level was higher in Russian rice (0.053 mg/kg) and Kurdish rice (0.073 mg/kg), in addition, the rest of samples not contain Zn. For Mn, Fe and Ca, all samples contain in it, but in a small amount that for Mn ranged between 0.057 and 0.151 mg/kg between Indian-3 rice and Russian rice, respectively. Staple food such as rice have deficiency in Zn and Fe as the results is showed in this study. Furthermore, Zn, Fe, Ca and Mn being an essential micronutrient is required to be a part of the normal human diet, and their deficiency can lead to health problems [33]. The Al and Cl contents in all the rice samples is characterized by lower values than in samples from Sri Lanka rice that

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reported in study by using titanium dioxide [34]. For Al and Cl content were ranged between 3.548 to 5.651 mg/kg and 1.017 to 1.608 mg/kg, respectively. Rice is an identical silicon accumulating plant, moreover it is essential for nutrition and growth of rice. The source of Si in rice was mostly from Si soil that absorbed by rice [35]. The all samples of rice dominant in Si content and higher than other minerals that analyzed in this study. The Iraqi-PDS rice contain higher amount (52.347 mg/kg) of Si than other samples and the lowest was for Indian-1 rice (41.564 mg/kg) and these results were comparable with results of Si concentration in rice that conducted by another study [36].

Conclusion

The mineral content and concentration for various kinds of rice that have been tested is varied and these variations may because different factors such as rice cultivars, water of irrigation, type of fertilization, and type of soil. The results showed all rice samples not contain Na and Sr. In addition, the highest Si, P and S contents were found in all rice samples, and the lowest was Cr and Al that is at safe level for health. In addition, the rice was also had deficiency in Fe, Mg, Mn, Zn, K and Ca contents. In this work, Energy-dispersive X-ray Fluorescence (EDXRF) was used for the evaluation of rice minerals. This technique was offered an economical, convenient and reliable tool for finding minerals concentration in rice samples. Because of these advantages, EDXRF is anticipated to largely replace Inductivity coupled plasma ICP and spectrometric methods in the great method for detecting mineral in food samples. In future work, I recommend the consideration of EDXRF to analyze the heavy metals in cooked rice samples that might be a high risk of toxicity.

Name	Abbreviation
Energy-dispersive X-ray Fluorescence	EDXRF
Inductivity coupled plasma	ICP
Standard error of mean	SEM
Iraqi public distribution system	Iraqi- PDS
Milligram	mg
Kilogram	Kg
Aluminum	Al
Calcium	Ca
Chlorine	Cl
Chromium	Cr
Iron	Fe
Potassium	K
Magnesium	Mg
Manganese	Mn
Phosphorus	P
Sulfur	S
Silicone	Si
Titanium	Ti
Zinc	Zn
Sodium	Na
Strontium	Sr

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