

Quantitative Analysis of Nutrient Minerals in Malagasy Rice Species by EDXRF and Dietary Intake Assessment

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Abstract: The elemental analysis of rice samples sold in local market in Antananarivo are carried out at Institut National des Sciences et Techniques Nucléaires-Madagascar (INSTN-Madagascar) using Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer. The study aims to assess the nutritional and mineral elements contents of rice which is the staple dietary food for Malagasy people. The Malagasy rice is divided into three species according to its color: red, pink and white rice. Essential major elements namely sodium (Na), magnesium (Mg), phosphorus (P), chloride (Cl), potassium (K), calcium (Ca) and trace elements such as chromium (Cr), manganese (Mn), iron (Fe), copper (Cu), zinc (Zn), selenium (Se) and iodine (I) are quantitatively determined. The accuracy of the instrumental method and analytical procedures are checked by using the Reference Material IPE 135 (Rice sample) from the Wageningen Evaluating Programmes for Analytical Laboratory (WEPAL) in Netherland. The experimental values are in good agreement with the certified values because of a coefficient of determination R^2 of 0.999 and a recovery rate %R between 87% and 114%. The comparison of the elemental content of the studied rice samples with the Recommended Dietary Allowance (RDA) reveals that Red rice foods have sufficient capacity to provide adult male consumers with P, Mn, Mg, Fe, Cu and Zn. Certainly insufficient daily intake of Mg (55%), Fe (58%), Cu (89%) and Zn (79%) could be met by ingestion of vegetables and meats that are served as a side dish.

Keywords: Rice, EDPXRF, Madagascar, Recommended Dietary Allowance

1. Introduction

Rice (*Oryza sativa* L.), “vary” in Malagasy language is the most prevalent cereal crop in Madagascar with an annual global production of approximately 4 million tons [1]. It is the main caloric intake of Malagasy people, most households consume rice three times a day and the per-capita annual consumption of rice is almost double that of Japan, reaching 120 kilograms [2].

Available in most parts of the country, rice appears to be the staple food in Madagascar. As for Malagasy people, a

meal without rice is not a meal. The importance of rice is also evident in the language. The Malagasy word for “rice” indicates at the same time the “meal”.

Food is basically composed of macronutrients and micronutrients. Among them, mineral nutrients are the inorganic chemical elements contained in foods and which are essential to participate in the functioning of enzymes, tissue composition, muscle contraction and blood clotting [3]. These mineral elements are essentially needed in important quantities (major elements) and smaller quantities (trace elements) and work with other food micronutrients such as

vitamins, fats and protein. Thus, a deficiency or an excess of a mineral can produce an imbalance of organism even a disease, this may be important causes of malnutrition, especially in the populations with a monotonous diet [4].

This is why analyses of nutritional and trace elements related to three (03) types of Malagasy rice are carried out. In the present work, Energy Dispersive X-ray Fluorescence (EDXRF) method has been used; it is a simplest, most accurate and most economical analytical technique for the determination of the chemical composition of different types of materials including the food sample [5, 6]. The elemental range goes from sodium (Na) to uranium (U), and provides detection limits at the low level ($\sim 10^{-1}$ mg.kg⁻¹) [7].

The objective of this study is to quantify essential major elements (Na, Mg, P, Cl, K, and Ca) and trace elements (Cr, Mn, Fe, Cu, Zn, Se and I) in rice samples which are rounded up into 3 groups according to the color and to evaluate how much mineral elements the rice foods can supply regarding the dietary intake.

2. Materials and Methods

2.1. Sample Collection and Preparation

Eleven raw rice samples, 4 of which are red (R), 4 of pink (P) and 3 of white (W) are randomly purchased from different local markets in Antananarivo Madagascar and are

analyzed in the laboratory. These three species show a fair representation of the rice consumed in Madagascar. Samples are put in plastic bags, coded and transported to the laboratory for analysis.



Figure 1. Rice offered on a market.

All the samples have been washed by double distilled deionized water to remove all kind of dirt. After oven-drying in a Sanyo oven at 65°C for 48 hours, about 100 mg of the dried rice samples are powdered; homogenized using an agate mortar and pestle and pelletized (3mm thick and 25mm diameter) using a SPECAC tabletop pelletize machine (10-15 tons pressure). No binder material was applied. The pellets, ready for measurements are stored in PTFE circular boxes well codified.

Table 1. The collected raw rice samples and their codification.

Sample species	Red rice				Pink rice				White rice		
N°	1	2	3	4	5	6	7	8	9	10	11
Sample Code	R01	R02	R03	R04	P01	P02	P03	P04	W01	W02	W03

2.2. Experimental Studies and Method of Validation

2.2.1. Elemental Analysis

Quantitative analysis of rice samples is performed using Energy Dispersive benchtop X-Ray Fluorescence (EDXRF), mark SPECTRO-XEPOS with 50Watt Palladium end-window X-Ray tube that produces the primary beam, and a target changer including Bragg and Barkla polarizers and secondary targets. The tube current and high voltage

settings depend on the targets. The measurement parameters are given in Table 2.

A silicon drift detector (SDD) is used to collect the fluorescence radiation from the sample. The spectral resolution of the SDD is ≤ 130 eV for Mn K α line [8]. The measurements are carried in vacuum mode and the test for one sample lasts approximately 1200s with the four targets. The analysis is controlled by a PC data acquisition system using a specialized software program SPECTRO X-Lab Pro [9].

Table 2. Measurement conditions.

Excitation	Tube voltage/current	Elements of interest	Measurement time
Molybdenum (Mo)	40kV/0.88mA	Fe, Cu, Zn, Se	300 s
BARKLA scatter (Al ₂ O ₃)	49.5kV/0.7 mA	I	300 s
Bragg crystal (HOPG)	17.5kV/2 mA	Na, Mg, P, Cl	300 s
Cobalt (Co)	35kV/1 mA	K, Ca, Cr, Mn	300 s

2.2.2. Method of Validation

The accuracy of the measurements and the efficiency of the method to be used are checked using WEPAL Certified Reference Materials IPE 135 (rice sample). Table 3 and Table 4 report the mean concentration and standard deviation values of 4 replicates.

Quality assessment statistics are applied to present both bias and precision data. This is about Linear Regression and

Recovery Analysis.

In Linear Regression Analysis, coefficients of determination R² of 1 or 0 would indicate the regression line represents all or none of the data, respectively. A higher coefficient is an indicator of a better goodness of fit for the observations [10].

In Recovery Analysis, the range of the acceptable criteria for recovery rate is between 80% and 120% [11]. The analytical recovery rate %R is expressed as the following

equation [12]:

$$\%R = 100 \times V_{\text{exp}}/V_{\text{assigned}} \quad (1)$$

where: V_{exp} is the value reported by the laboratory and V_{assigned} is the assigned value.

According to the above mentioned statistical criteria, it is

shown that the experimental values are in good agreement with the certified values because of a coefficient of determination R^2 of 0.999 (Figure 2) and a recovery rate %R between 87% and 114% (Tables 3 and 4).

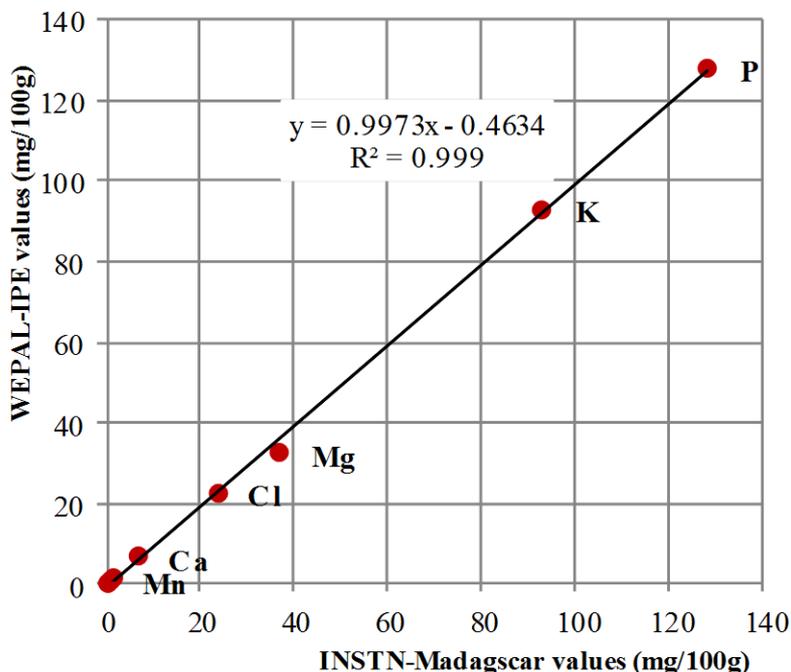


Figure 2. Plotted of INSTN-Madagascar values vs WEPAL-IPE values.

Table 3. Comparison between WEPAL IPE-35 certified values and experimental values (Nutritional elements).

Elements	IPE-135 Concentration*		Recovery Rate (%)
	Assigned values	Experimental values	
Nutritional elements			
Na	< 1	< 3	-
K	92.9	92.9 ± 4.6	100.0
Ca	6.6	6.6 ± 0.6	99.5
Mg	32.4	37.0 ± 2.6	114.2
P	127.8	127.9 ± 3.8	100.1
Cl	22.6	23.6 ± 1.7	104.6

*Data are expressed as mg/100g on a dry weight basis.

Table 4. Comparison between WEPAL IPE-35 certified values and experimental values (Trace elements).

Elements	IPE-135 Concentration*		Recovery Rate (%)
	Assigned values	Experimental values	
Trace elements			
Fe	1.0	0.9 ± 0.1	87.0
Cu	0.3	0.3 ± 0.04	96.8
Zn	1.6	1.4 ± 0.1	89.4
I	< 0.1	< 0.02	-
Mn	0.9	0.9 ± 0.1	104.6
Rb	0.2	0.2 ± 0.03	90.0

*Data are expressed as mg/100g on a dry weight basis.

3. Results and Discussion

Overall, eleven raw rice samples used in various Malagasy dishes were analyzed. Table 5 presents analysis results of

these samples for nutritional elements (Na, Mg, P, Cl, K, Ca) and Table 6 the trace elements (Mn, Fe, Cu, Zn, Cr, Se, I) compositions. Once the daily intake data are processed (Tables 7 and 8), the calculated mineral nutrient amounts are then compared to the Recommended Dietary Allowance values

(Table 9).

3.1. Element Contents of the Raw Rice Samples

Table 5 and Table 6 below present the analytical results of different varieties of rice available on the market insofar as

mineral elements are concerned. In this table are added the mean, minimum and maximum values for each variety of rice which is subject to the study.

Table 5. Nutritional elements contents in the studied rice samples.

Sample species	Sample code	Na	Mg	P	Cl	K	Ca
Red (R)	R01	<3	44.9	224.1	20.9	206.1	31.5
	R02	<3	68.9	309	41.3	235.5	13.5
	R03	<3	66	355.2	31.3	313.2	6.9
	R04	<3	50.3	280.2	26.8	244.6	38.3
	Mean		57.5	292.1	30.1	249.9	22.6
	Min		44.9	224.1	20.9	206.1	6.9
	Max		68.9	355.2	41.3	313.2	38.3
Pink (P)	P01	<3	52	222.3	42.8	171.2	26.8
	P02	<3	39.6	180.1	31	165.6	25.1
	P03	<3	36.6	202.7	25	154.8	26.4
	P04	<3	37.1	209	34	177.7	69.8
	Mean		41.3	203.5	33.2	167.3	37.0
	Min		36.6	180.1	25	154.8	25.1
	Max		52	222.3	42.8	177.7	69.8
White (W)	W01	<3	25.1	116.6	35.3	78.8	61.7
	W02	<3	31.6	158.2	27.3	110.4	79.7
	W03	<3	11	86.7	30.9	68	41.9
	Mean		22.6	120.5	31.2	85.7	61.1
	Min		11	86.7	27.3	68	41.9
	Max		31.6	158.2	35.3	110.4	79.7

Data are expressed as mg/100g on a dry weight basis.

Table 6. Trace elements contents in the studied rice samples

Sample species	Sample code	Mn	Fe	Cu	Zn	Cr	Se	I
Red (R)	R01	1.4	0.8	0.2	1.7	<0.02	<0.01	<0.02
	R02	1.6	1.6	0.2	2	<0.02	<0.01	<0.02
	R03	3.7	1.5	0.2	2.5	<0.02	<0.01	<0.02
	R04	1.7	0.7	0.2	2.5	<0.02	<0.01	<0.02
	Mean	2.1	1.2	0.2	2.2			
	Min	1.4	0.7	0.2	1.7			
	Max	3.7	1.6	0.2	2.5			
Pink (P)	P01	1.7	1.6	0.2	2.1	<0.02	<0.01	<0.02
	P02	0.9	0.7	0.2	1.9	<0.02	<0.01	<0.02
	P03	1.8	1.8	0.3	2	<0.02	<0.01	<0.02
	P04	1.1	1.1	0.4	2	<0.02	<0.01	<0.02
	Mean	1.4	1.3	0.3	2.0			
	Min	0.9	0.7	0.2	1.9			
	Max	1.8	1.8	0.4	2.1			
White (W)	W01	1.2	1.9	0.3	1.7	<0.02	<0.01	<0.02
	W02	1.4	1.2	0.2	1.6	<0.02	<0.01	<0.02
	W03	1	1.8	0.2	1.5	<0.02	<0.01	<0.02
	Mean	1.2	1.6	0.2	1.6			
	Min	1	1.2	0.2	1.5			
	Max	1.4	1.9	0.3	1.7			

Data are expressed as mg/100g on a dry weight basis.

In comparative assessment of the various species, the results reported in Tables 5 and 6 allow to show that:

Red rice and *Pink rice* are most significant species having higher concentrations of magnesium, phosphorus, potassium and manganese as compared to the *White rice*. The mean concentrations of magnesium are 57.5, 41.3 and 22.6 mg/100g in *Red rice*, *Pink rice* and *White rice* respectively. Phosphorus contents of the studied samples are found as 292.1, 203.5 and 120.5 mg/100g for *Red rice*, *Pink rice* and

White rice respectively. In case of potassium, *Red rice* has the highest concentration of 249.9 mg/100g followed by *Pink rice* then *White rice*, which have respectively a concentration of 167.3 and 85.7 mg/100g. *Red rice* is found to be in greater amount in manganese (2.1 mg/100g) among all the species.

The reverse case appears to calcium; its content turned out to be higher in *White rice* (61.1 mg/100g), and lower in *Red rice* (22.6 mg/100g).

The micronutrients including Fe, Cu, Zn and Cl are found

to be in the range of 1.2-1.6, 0.2-0.3, 1.6-2.2, and 30.1-33.2 mg/100g respectively. In other words, the variation is not significant for a given species.

All the studied species are low in sodium, chromium, selenium and iodine. Their contents are below the detection limit values which are 3 mg/100g, 0.02 mg/100g, 0.01 mg/100g and 0.02 mg/100g respectively.

3.2. Daily Nutrient Intakes for an Adult Male Through Rice Consumption

The traditional Malagasy formula is characterized by a daily diet based on three main meals of rice to which can be added one or two snacks. The daily intake of nutritional and trace elements from rice food have been estimated based on

the ingested quantities in the day. In this study, it is assumed that a male Malagasy citizen, aged 19 to 50 consumes approximately 400 g of rice on a daily basis. In fact, the most active period of human life is in the age range 19 - 50 years old. Tables 7 and 8 summarize the estimate of daily nutrients intake for an adult male.

The results indicate that all the analyzed rice samples have the potential to provide essential nutrients to the human body. Some species are found significantly useful in terms of minerals sources, particularly the Red rice with its high contents of Mg, P, K, Ca, Mn, Zn comparing to the two others species. However, it is now questionable whether once individuals have access to various foods in sufficient quantities, are their main nutritional needs met?

Table 7. Estimation of Daily nutrient intakes for a male, aged 19-50 (Nutritional elements).

Sample species	Sample code	Mg [mg/d]	P [mg/d]	Cl [mg/d]	K [mg/d]	Ca [mg/d]
Red (R)	R01	179.6	896.4	83.6	824.4	126
	R02	275.6	1236	165.2	942	54
	R03	264	1420.8	125.2	1252.8	27.6
	R04	201.2	1120.8	107.2	978.4	153.2
	Mean	230.1	1168.5	120.3	999.4	90.2
	Min	179.6	896.4	83.6	824.4	27.6
	Max	275.6	1420.8	165.2	1252.8	153.2
Pink (P)	P01	208	889.2	171.2	684.8	107.2
	P02	158.4	720.4	124	662.4	100.4
	P03	146.4	810.8	100	619.2	105.6
	P04	148.4	836	136	710.8	279.2
	Mean	165.3	814.1	132.8	669.3	148.1
	Min	146.4	720.4	100	619.2	100.4
	Max	208	889.2	171.2	710.8	279.2
White (W)	W01	100.4	466.4	141.2	315.2	246.8
	W02	126.4	632.8	109.2	441.6	318.8
	W03	44	346.8	123.6	272	167.6
	Mean	90.3	482.0	124.7	342.9	244.4
	Min	44	346.8	109.2	272	167.6
	Max	126.4	632.8	141.2	441.6	318.8

Table 8. Estimation of Daily nutrient intakes for a male, aged 19-50 (Trace elements).

z	Sample code	Mn [mg/d]	Fe [mg/d]	Cu [mg/d]	Zn [mg/d]
Red (R)	R01	5.6	3.2	0.8	6.8
	R02	6.4	6.4	0.8	8
	R03	14.8	6	0.8	10
	R04	6.8	2.8	0.8	10
	Mean	8.4	4.6	0.8	8.7
	Min	5.6	2.8	0.8	6.8
	Max	14.8	6.4	0.8	10
Pink (P)	P01	6.8	6.4	0.8	8.4
	P02	3.6	2.8	0.8	7.6
	P03	7.2	7.2	1.2	8
	P04	4.4	4.4	1.6	8
	Mean	5.5	5.2	1.1	8
	Min	3.6	2.8	0.8	7.6
	Max	7.2	7.2	1.6	8.4
White (W)	W01	4.8	7.6	1.2	6.8
	W02	5.6	4.8	0.8	6.4
	W03	4	7.2	0.8	6
	Mean	4.8	6.5	0.9	6.4
	Min	4	4.8	0.8	6
	Max	5.6	7.6	1.2	6.8

3.3. The Daily Nutrient Intakes for an Adult Male Compared with the Recommended Dietary Allowance

The following is the definition of the RDA: "Recommended Dietary Allowance is the average daily dietary intake level that is sufficient to meet the nutrient requirements of nearly all (97–98 percent) individuals in a specific life stage and gender group." [13]

RDA applies to mineral nutrients from daily food and supplements. The purpose of this guideline is to inform on

how much of a specific nutrient the human body needs on a daily basis. It is important to meet the daily recommended dietary allowance so that the body gets everything it needs to function normally and to maintain good health. It is reported in Table 9 the comparison of the average daily intake for an adult male to the recommended values. Figure 3 illustrates the contribution of the various species of rice among the adult males.

Table 9. Contribution of the various species of rice to mineral intakes in adult males.

Elements	RDA* [mg/d]	Red Rice		Pink Rice
		Mean ± SD [mg/d]	Mean/RDA %	Mean ± SD [mg/d]
Mg	420	230.1 ± 40.6	55	165.3 ± 25.1
P	700	1168.5 ± 190.1	167	814.1 ± 61.1
Cl	2300	120.3 ± 29.8	5	132.8 ± 25.7
K	4700	999.4 ± 157	21	669.3 ± 33.6
Ca	1000	90.2 ± 51.2	9	148.1 ± 75.7
Mn	2.3	8.4 ± 3.7	365	5.5 ± 1.5
Fe	8	4.6 ± 1.6	58	5.2 ± 1.7
Cu	0.9	0.8 ± 0	89	1.1 ± 0.3
Zn	11	8.7 ± 1.4	79	8 ± 0.3
Na	1500	< 12	-	< 12
Cr	0.035	< 0.08	-	< 0.08
Se	0.055	< 0.04	-	< 0.04
I	0.15	< 0.08	-	< 0.08

Table 9. Continued.

Elements	RDA* [mg/d]	Pink Rice	White Rice	
		Mean/RDA %	Mean ± SD [mg/d]	Mean/RDA %
Mg	420	39	90.3 ± 34.4	22
P	700	116	482 ± 117.3	69
Cl	2300	6	124.7 ± 13.1	5
K	4700	14	342.9 ± 72	7
Ca	1000	15	244.4 ± 61.8	24
Mn	2.3	239	4.8 ± 0.7	209
Fe	8	65	6.5 ± 1.2	81
Cu	0.9	122	0.9 ± 0.2	100
Zn	11	73	6.4 ± 0.3	58
Na	1500	-	< 12	-
Cr	0.035	-	< 0.08	-
Se	0.055	-	< 0.04	-
I	0.15	-	< 0.08	-

* RDA for adult male.

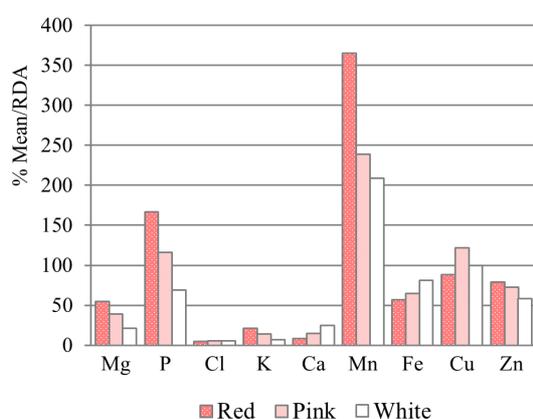


Figure 3. Diagram of contribution of red, pink and white rice to mineral intakes in adult males.

The concentration values of the nutrients elements in the various species of rice when compared with dietary allowance show that there are some whose mineral contents can meet with the daily requirements. This is because the elements in those rice foods are more than 50% close to the allowed value. The value of about 50% implies that the containing foods need to be completed to meet up the requirements. Fortunately, Malagasy rice dishes are usually taken with meats (beef, pork, poultry), fishes, green vegetables,... as side dishes generally salted. Rare are the Malagasy people who eat the rice without a side dish.

Based on the computed mean values and the comparison with dietary allowance, red rice foods have sufficient capacity to provide consumers with P, Mn, Mg, Fe, Cu and Zn. Certainly, as mentioned above, insufficient daily intake of Mg

(55%), Fe (58%), Cu (89%) and Zn (79%) could be met by ingestion of vegetables and meats that are served as a side dish. Moreover, it is found that in rice-based foods, the daily requirements of calcium, potassium, sodium, chloride and iodine are far from being met. Therefore, additional intakes through other foods are required; the main sources of calcium and potassium are dairy products (milk, cheese, yogurt), fruits and green vegetables such as spinach and broccoli.

As for the insufficient intake of sodium, chloride and iodine in the daily meal of rice, the iodized salt, widely used in food processing and manufacturing, could partly solve the shortfall. Indeed, inadequate intake of iodine leads to brain damage and irreversible mental retardation [14]. In parallel, sodium is an essential nutrient necessary for maintenance of plasma volume, acid–base balance, transmission of nerve impulses and normal cell function [15].

The presence of these essential elements in the tested rice samples is quite revealing.

4. Conclusion

The adopted procedure using XRF spectrometry to quantify mineral element contents produced accurate results. The various species of Malagasy rice differ in their essential elements variable amounts, offering the consumers a wide range of choices. Red rice appears to be generally good source of essential major and trace elements. Indeed, the comparison of the Red rice contents with the RDA reveals that P, Mn, Mg, Fe, Cu and Zn requirements are met.

The data in this report can therefore be used by nutrition stakeholders and decision-makers for food safety and security, as well as researchers to support their work related to the nutrition of the Malagasy population. This study could contribute to solving the problem of malnutrition that remains severe in southern Madagascar, where it is necessary to improve the population nutrition issues through government policies and programs. Food and Agriculture Organization of the United Nations (FAO) reports: “in 2017/2018, over 407,000 people are estimated to be severely food insecure in the south and southeast of Madagascar and, without adequate external assistance, they are unable to meet their food needs” [16].

One of the ways to reduce malnutrition includes adequate communication-training and sensitization of the population to comply with correct dietary plans, as well as the optimal use of nutrient-rich local foods such as fruits, vegetables, cereals and fish products. This dietary diversification is the most suitable solution, but is often unaffordable for the majority of the population in low-income countries such as Madagascar. Generally, the resource-poor people are at greater risk of deficiencies. Given the existing situation, raising public awareness on these crucial issues is of prime importance.

This study on rice food is limited to how much mineral elements the rice foods can supply regarding the dietary intakes. To complete this investigation, further tests need to be conducted, in particular for the evaluation of

macronutrients such as proteins, fat, carbohydrates, dietary fiber, vitamins which play vital roles in the normal functioning of the body.

Abbreviation

INSTN:	Institut National des Sciences et Techniques Nucléaires
EDXRF:	Energy Dispersive X-Ray Fluorescence
WEPAL:	Wageningen Evaluating Programs for Analytical Laboratory
RDA:	Recommended Dietary Allowance
SDD:	Silicon Drift Detector
IPE:	International Plant-Analytical Exchange
FAO:	Food and Agriculture Organization of the United Nations
XRFE:	X-Ray Fluorescence and Environment

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