Detection of Macro and Micro Nutrients in Potatoes using Elemental Analysis Techniques

R. Parameswaran, P. T. V. Bhuvaneswari

Abstract: ***The elemental technique used for the detection of macro and micro nutrients in dry potato and fresh potato has been carried out in this research. Two techniques, namely Laser Induced Breakdown Spectroscopy (LIBS) and Scanning Electron Microscope (SEM) with Energy Dispersive Analysis of X-rays (EDAX) have been employed. LIBS provides the spatial distribution of elements in the considered sample, while SEM with EDAX provide a quantitative percentage of elements in the sample. In the first technique, plasma is created in the sample (dry and fresh potato)when photons emitted from the laser source are impinged on the sample. The elemental distribution in plasma is captured using a spectrometer. Then, with the aid of NIST database, the relationship between wavelength and the intensity of the element is determined and the following observations are made by using origin pro tool. The major macro nutrients detected in the sample are nitrogen, calcium, potassium, phosphorus, and magnesium. The micro nutrients, namely sodium and iron are also detected. In the second technique, the samples are pulverized and placed in the sputtering instrument and scanned by SEM. From the scanned image,the macro and micro nutrient present in the sample are determined. The quantity of the element along with atomic percentage is determined. The investigation was extended to potato based food products, namely lays and potato chips. From the experiment, in addition to the macro and micro nutrient, toxic elements, namely lead and indium present in the sample have also determined. The fundamental point of this examination is to serve the cultivation plants to know the elements of the specific plants.***

Keywords: LIBS, SEM, EDAX, Potatoes and nutrients.

I. INTRODUCTION

Healthy living of human being is dependent on the nutritive level of the food consumed. As per the statistics [1] available, fruits and vegetables contain most of the minerals that are required for human beings. But to enhance their yield, cultivation in recent years uses more inorganic compounds in the form of pesticides, fungicides, insecticides and fertilizers abundantly in an unregulated manner. The macro nutrient present in potato comprises of nitrogen, phosphate, potassium, calcium and magnesium [2]. The micronutrient present in potato includes iron, sodium, calcium and zinc [2]. The functions of each nutrient are [3]: Nitrogen is important in fuelling the growth by ensuring optimal photosynthetic production in leaves. Phosphate, particularly foliar phosphate is applied during inhabitation to increase the tuber and tuber size, thereby increasing the tuber yield.

Potassium is absorbed in larger quantities throughout the growing seasons; it is required for high yields, it also influences the number of tubers.

Revised Manuscript Received on January 15, 2020

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Calcium and Magnesium are applied during the bulking process to increase the size of the tuber. Micronutrients such as boron, copper, iron, sodium and zinc present in potato are essential for plant growth. So the deficiency of anyone may affect their growth. Boron is important for growth of plants in the structural and functional virtue of the plant cell. Copper aids in protein synthesis. Iron and zinc are essentially useful for crop growth and high yielding of plants.

Several processed foods such as potato chips, ketchups, fruit juice are made from fruits and vegetables. In order to extend their lifetime, preservatives are added [4]. The inorganic chemicals added in the name of pesticides or preservative will certainly reduce the nutritive value of the raw food.

The unorganized usage of inorganic compounds to enhance the yield of the horticulture crops and adulteration in their by products lead to several health hazards such as Obesity, Heart disease, Cancer and Diabetes [5].

In this research, potato is considered as the case for the study. The main objective of this research is to detect and analyze the spatial distribution of macro and micro nutrients in potato through elemental analysis techniques so that the presence of toxic element in the considered sample can be identified.

The rest of the paper is organized as follows: In section II, the state-of-the-art related to the existing work is discussed. In section III, the proposed work is detailed. In section IV, the results obtained are discussed. Section V concludes by highlighting the contribution of the proposed work.

II.MATERIALS AND METHODS

In [6] the authors have investigated the time resolved characteristics of laser induced plasma on a fresh potato sample. Through experimental analysis, the temporal evolution of plasma formulated in LIBS has been investigated to determine the concentration of trace elements in fresh potato. The presented work has optimized the procedures involved in the experimental set- up and protocols utilized in the spectroscopic data analysis technique, that are used in determining the quantity of trace element present in the fresh potato.

In [7] the authors have evaluated the minor elements in potato using LIBS. Flesh and skin part of fresh potatoes have been investigated using Nd:YAG laser. A plasma has been created on the potato and the spectra has been recorded on the Echelle spectrometer. Eleven minor elements have been identified and their relative concentrations have been estimated. From the investigation, it has been concluded that the LIBS is a promising tool that can be used to measure the elemental composition of fresh vegetables without using sample preparation.

In [8] the authors have developed a new approach to detect the presence of organic phosphorus pesticide in fruits and vegetables. Different concentrations of pesticides have been analysed and compared with the nitro cellulose strip method. From the analysis, it has been inferred that grape juice contains more concentration of pesticides.

In [9] the authors have used XRD, TEM, and EDAX elemental analysis techniques to investigate the bacterial activity of the neem leaf.

In [10] the role of EDAX in biomedical research and diagnosis process are discussed. It is clearly mentioned that EDAX micro analysis technique can be used to study the composition of drugs, presence of nano particles, environmental pollution and minerals accumulated in the tissue. It concludes that EDAX is a useful tool that can determine elements, endogenous or exogenous in tissue on any other sample.

In [11] the elemental composition of the vulcanized silicon rubber sample under room temperature and high temperature is analyzed. Further, six elements, namely carbon, oxygen, aluminium, silicon, iron and zinc have been identified using EDAX, The micro morphology and depth of ablation have been analyzed through SEM. From all the three techniques, it has been concluded that LIBS is the best elemental analysis technique as it clearly detects the composition of it.

In [12] the authors have evaluated the presence of adulterant in various food materials such as milk, butter, edible oil, honey, pulses, wheat flour, black pepper, chili powder, coffee powder through traditional qualitative analysis. The presence of adulteration is indicated through color change.

From the above literature review, it is found that out of several elemental techniques LIBS, SEM, and EDAX are predominantly used in the detection of elements. Hence, in the proposed research an attempt has been made to detect the presence of macro and micro nutrients present in potato. It is also extended to processed potato food products.

The Botanical name of potato is Solanum tuberosum [13]. This root vegetable is rich in starch and carbohydrates. The duration of cultivation of this crop is 3 to 4 months. It is one of the main food crops which grows in cold climates. The yield of this crop is predominantly affected by "late blight disease". Hence, the farmers use inorganic pesticides to control them. The macro and micro nutrients present in potato are nitrogen, phosphate, potassium, calcium, magnesium, iron, sodium, boron, copper and zinc. The proposed methodology adopts two techniques to find the presence of macro and micro nutrients in the sample as shown in Fig. 1.

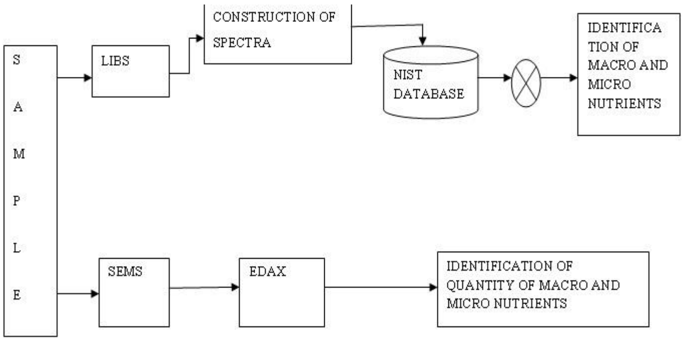

Fig. 1Proposed techniques for detection of macro and micro nutrients in potato

Nutrient plays a major role in the growth of every crop and increases the yields. Each nutrient contributes significantly for plant growth and deficiency of any of them may lead to diseases such as [14] bacterial wilt, early blight, late blight, sepatoria leaf spot, common scab, and pink rot.

Food adulteration is becoming the major cause for health issues as it contains the presence of unwanted toxic elements [15]. The usage of adulterants is increasing day by day. Some authorities are intentionally adding substance to the packed food products and if the usage extends beyond the allowable level, it may be harmful to consumers, leading to food poisoning, vomiting and diahorrea. Further food adulteration increases the impurity in the food. Consumption of adulterated food for long will have both short term and long term impact on the health.

In this research, elemental analysis techniques, namely LIBS and SEMS with EDAX are adopted to detect the presence of macro and micro nutrients in the potatoes.

A.*LASER INDUCED BREAKDOWN SPECTROSCOPY (LIBS):*

The operating principle of LIBS is quite simple. An intense, highly focused laser pulse is injected on the sample to create plasma. The elements present in the sample are excited from their ground states and emit characteristic wavelengths of light. The emitted light is carried out by a lens and or a fiber optic system on the instrument and finally as a graph will be displayed on the screen. The block diagram of the proposed LIBS based macro and micro nutrient detection is shown in Fig. 2. The spectrum of the emitted light is then analyzed for the presence of atomic emission lines to detect the presence of macro and micro nutrients.

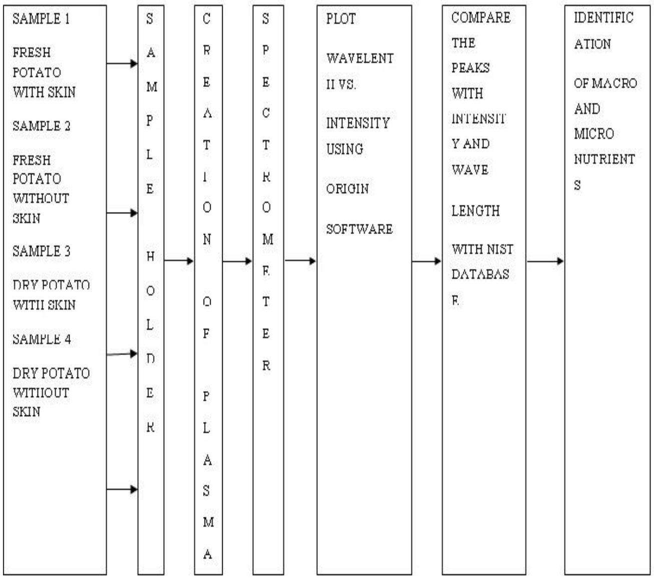

Fig. 2 Working mechanism of LIBS

In the proposed research, potato is taken as a sample to analyze the presence of macro and micro nutrients. Initially the samples are cleaned, ablated and sliced and the sample is placed in the sample holder and shot pulse from the laser source is injected onto the sample, plasma is created. Through collecting lens present in the spectrometer, traces of elements contained in the sample are obtained.

The statistical analysis on the spectra is made using origin pro software [16] and a graphical representation between the wavelength and the intensity is plotted. Then using NIST database [17], the wavelength peaks are mapped and macro and micro nutrients are found out.

The above experiment is made for two types of potato samples, namely fresh potatoes and dry potatoes. To obtain the dry potato sample the fresh potato is dried in the presence of sunlight at 25 degree celsius for five days. Both the samples are tested with skin and without skin (flesh). A short pulse with energy ranging from 5 to 20mJ and pulse width of 5 to 6ns is injected from the laser source of 532nm on the sample.

Based on the energy emitted from the laser source, the fluence level applied on the sample is determined using the expression given in equation (1)

$$E = F / \pi * r^2 \quad (1)$$

where

E is the energy (mJ)

F is the fluence

r is radius of the sample

The experiments were repeated for different energy levels for four samples, namely dry potato with skin, dry potato with flesh, fresh potato with skin and fresh potato with flesh.

The above experiment is repeated for the remaining three different samples. In all cases, interactions between the charged particles and the neutral particles are important in determining the behavior and the usefulness of plasma. The type of atoms in a plasma, the ratio of ionized to neutral particles and the particle energies that result in a broad spectrum of plasma types, are the factors that influence the characteristics and behaviors.

Origin is a licensed tool [16] and mainly used for data analytics and graph plotting. Data analyses in origin include statistics, signal processing and peak analysis.

B.SEM WITH EDAX

A Scanning Electron Microscope (SEM) is a type of electron microscope that produces images of the sample by scanning the surface with a focused beam of electrons. The electrons interact with the atoms in the sample and produce various signals that contain information about the surface topography and composition of the sample [17].

Energy Dispersive X-ray Spectroscopy (EDAX) is a technique used to identify the elemental composition of materials. In a multi technique approach, EDAX becomes very powerful, particularly in contamination analysis and industrial forensic investigations [18]. The technique can be qualitative, semi quantitative, quantitative and also provides spatial distribution of elements through mapping.

In SEM, there are several electromagnetic lenses such as condenser lens and objective lens as shown in Fig. 3. The condenser lenses reduce the crossover diameter of the electron beam while the objective lens reduces the cross section of the electron beam and focuses the beam on the specimen surface to magnify the region of interest.

Electron beam scans the specimen. The signal electrons emitted from the specimen are collected by the detector and amplified. Reconstruction of the image is done by performing correlation between scanning points on the specimen. The picture on the screen of Cathode Ray Tube (CRT) converts the electronic signals to a visual display.

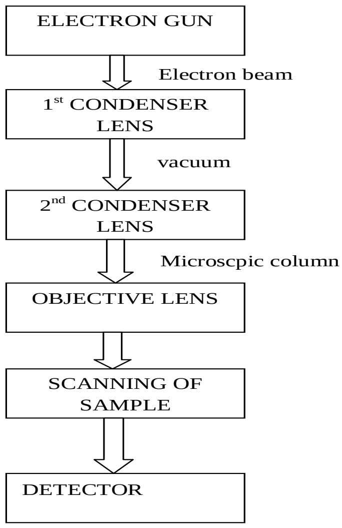

Fig. 3 Block Diagram and Working Process of SEM

The main types of signals that are detected are the backscattered (BSE) and secondary electrons (SE) which generate a grayscale image of the sample at very high magnifications. The smart phase mapping provides a higher level of analysis by automatically collecting spectra and phase maps with elemental distribution on it.

The steps involved in SEM and EDAX method are detailed in this section:

Preparation of the sample is minimal. In this experiment potato is kept in sunlight and grained to fine particles as shown in Fig. 4. The prepared sample is placed in the SEM specimen holder.

Fig. 4 Grained potato sample

The stub is shown in Fig. 5.

Fig. 5 sample placed in the stub

The stub is placed on the sputtering tool kit and preset energy is applied to create plasma over the sample as shown in Fig. 6.

Fig. 6 sputtering instrument

In EDAX method, the X-ray is generated by the interaction of the electron beam with the sample using which the chemical analysis of the sample can be performed. As high energy electrons strike the sample surface, along with secondary and backscattered electrons, characteristic X-rays are emitted whose wavelength depends on the nature of atoms in the sample. X-ray is generated when an inner shell electron is knocked out of the atom and the vacant site is filled with the electron from the outer shell. The excess energy is released and the amount of releasing energy, corresponding to the X-ray range, is the characterization of a particular atom.

III. RESULTS AND DISCUSSION

The LIBS experimental set-up is having laser source, collecting lens, cuvette, laser control unit, spectrometer with fiber cable and system for viewing the spectrum as shown in Fig. 7.

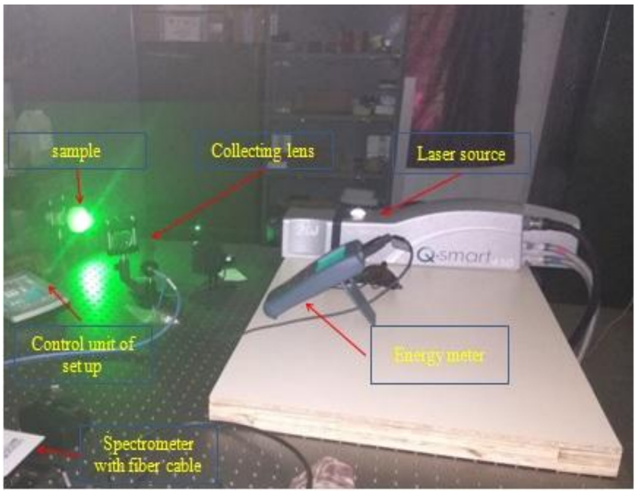

Fig. 7 Real Time Set-up

A.Experimental Parameters

The LIBS based experimental parameters are presented in Table 1.

Table 1 Parameter values for the experiment

s.no	Features	value
1.	Samples	Dry potato (with skin and flesh) Fresh potato (with skin and flesh)
2.	Number of samples	4
3.	Wavelength of LASER	532 nm
4.	Energy applied	5 to 20 mJ
5.	Repetition rate	10 to 20 Hz
6.	Pulse width	5 to 6 ns

B.Performance of LIBS

In LIBS two samples, namely dry potato and fresh potato are taken for investigation as shown in Fig. 8

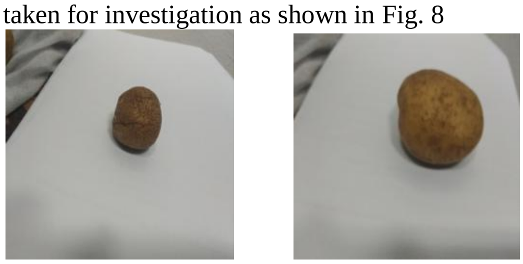

Fig. 8 dry potato and fresh potato

Case study 1 : Dry potato

The laser emits photons of wavelength 532 nm on the dry potato. The following observations are made when a fluence of 1.6 J/cm^2 is applied to the flesh part of the dry potato. From Fig. 9 (a), it is observed that by the application of low energy only one macronutrient (calcium) and one micronutrient (iron) can be identified. From the flesh part of potato nitrogen and from the skin part calcium can be identified as seen from Fig. 9 (b).

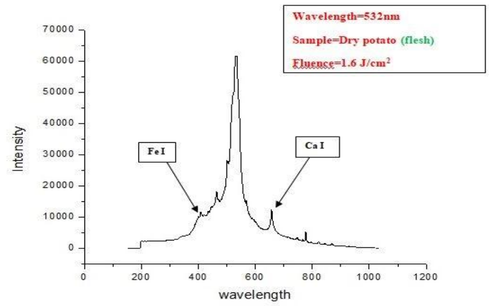

Fig. 9 (a) wavelength vs. intensity of dry potato flesh with energy 5 mJ

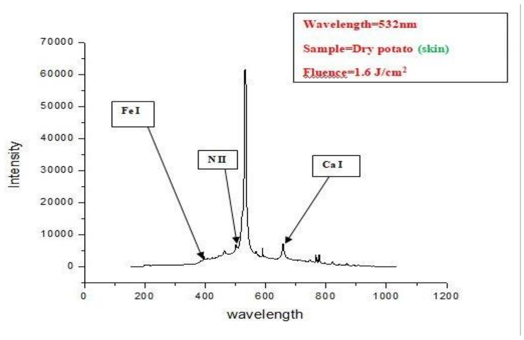

Fig. 9(b) wavelength vs. intensity of dry potato skin with energy 5 mJ

On doubling the fluence level to 3.2 J/cm^2 with same 532 nm and applying on the fleshy part of the dry potato, the following observations are made. In the flesh part of the potato, the macro nutrients present are calcium, phosphorus, and potassium. No micronutrient is detected at this energy level. In the skin part of the potato, the macro nutrients present are calcium, phosphorus, nitrogen, and potassium. The micronutrients are iron and sodium. Thus by increasing the energy, the ability of identifying macro nutrients and macro nutrients can be increased.

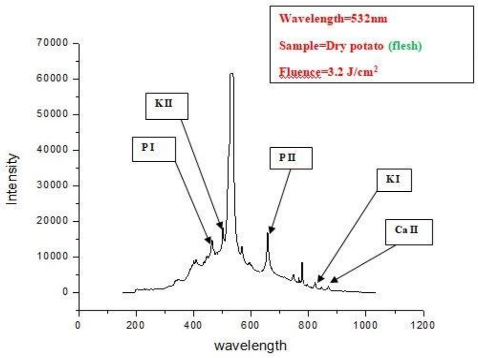

Fig. 10(a) wavelength vs. intensity of dry potato flesh with energy 10 mJ

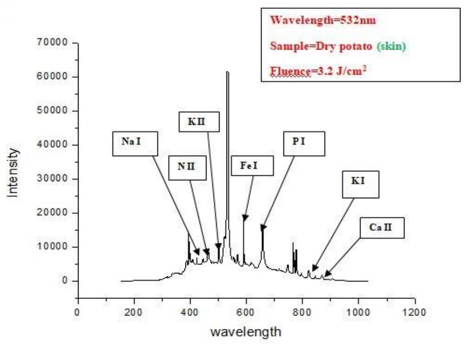

Fig. 10(b) wavelength vs. intensity of dry potato skin with energy 10 mJ

Similarly on increasing the fluence of 4.8 J/cm^2, in flesh part of the potato the macro nutrients nitrogen, calcium and phosphorus are identified. The micronutrients present are iron and sodium as shown in Fig. 11 (a). In the skin part of the potato are found nitrogen, phosphorus and potassium as macronutrients and iron and sodium as micronutrients as shown in Fig. 11(b).

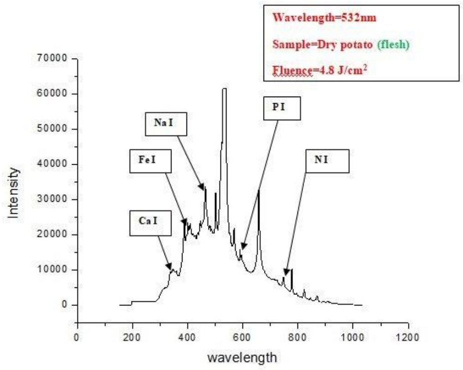

Fig. 11(a) wavelength vs. intensity of dry potato flesh with energy 15 mJ

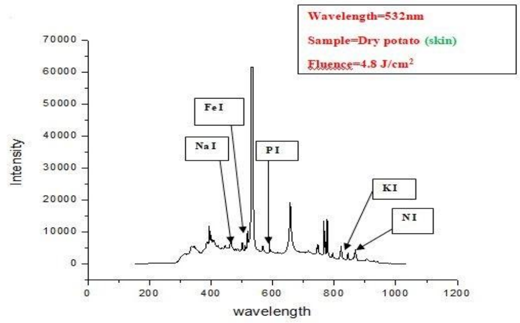

Fig. 11(b) wavelength vs. intensity of dry potato skin with energy 15 mJ

Finally for fluence more than 6.4 J/cm^2 , in the flesh part of the potato the macro nutrients found are nitrogen, calcium, phosphorus, and potassium and the micronutrient is sodium as shown in Fig. 12(a).

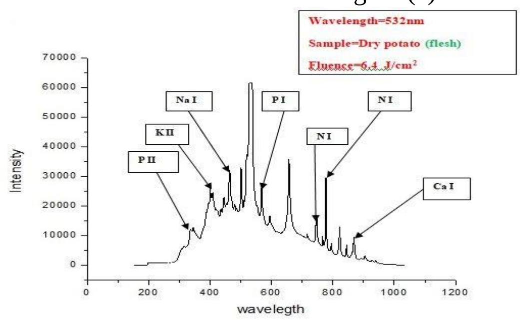

Fig. 12(a) wavelength vs. intensity of dry potato flesh with energy 20 mJ

In the skin part of the potato, the macro nutrients present are nitrogen, calcium, magnesium, and phosphorus and no micronutrient is observed on the skin part of dry potato which is seen in Fig. 12(b).

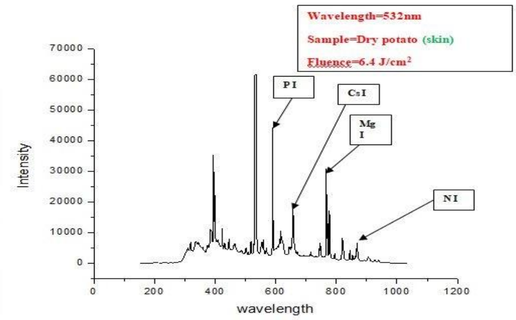

Fig. 12(b) wavelength vs. intensity of dry potato skin with energy 20 mJ

The presence of nitrogen on the skin part is detected at different energy levels. The presence of magnesium is at 20 mJ while the presence of calcium is even at the lowest energy levels, namely 5 -10 mJ. The presence of phosphorus and potassium are identified only at the higher energy levels.

However, the micronutrients, namely boron, copper, and zinc are not identified with this wavelength and energy levels. To conclude macro nutrients identified are nitrogen, calcium, potassium, phosphorus, and magnesium while the identified micronutrients are sodium and iron.

Table2 presents the macro and micro nutrients identified in the dry potato sample while Table 3 presents those of fresh part which are varied from the energy levels 5-20mJ.

Table 2 Presence of macro and micro nutrients in Dry potato with energies 5-20 mJ

		Wavelength=532 nm							
NUTRIENTS	ELEMENTS	Energy =5 mJ Fluence =1.6 J/cm² Sample=Dry potato		Energy =10 mJ Fluence =3.2 J/cm² Sample=Dry potato		Energy =15 mJ Fluence =4.8 J/cm² Sample=Dry potato		Energy =20 mJ Fluence =6.4 J/cm² Sample =Dry potato	
		Skin	Flesh	Skin	Flesh	Skin	Flesh	Skin	Flesh
MACRO	Nitrogen	✓		✓		✓	✓	✓	✓
	Calcium	✓	✓	✓	✓				✓
	Phosphorus			✓	✓	✓	✓	✓	✓
	Magnesium							✓	
	Potassium			✓	✓	✓	✓		✓
MICRO	Iron	✓	✓	✓		✓	✓		
	Sodium			✓		✓	✓		✓
	Boron								
	Copper								
	Zinc								

The above experiment is repeated for fresh potato. The macro and micro nutrients detected/identified are summarized in Table 2. The macro nutrients are identified in the above experimental techniques with the wavelength being 532 nm. The micronutrients, namely boron, copper, and zinc are not identified with this wavelength and energy levels. The macro nutrients identified are nitrogen, calcium, potassium, phosphorus, and magnesium while the micronutrients are sodium and iron.

Table 3 Presence of macro and micro nutrients in Fresh potato with energies 5-20 mj

		Wavelength=532 nm							
NUTRIENTS	ELEMENTS	Energy =5 mJ Fluence =1.6 J/cm² Sample=Fresh potato		Energy =10 mJ Fluence =3.2 J/cm² Sample=Fresh potato		Energy =15 mJ Fluence =4.8 J/cm² Sample=Fresh potato		Energy =20 mJ Fluence =6.4 J/cm² Sample =Fresh potato	
		Skin	Flesh	Skin	Flesh	Skin	Flesh	Skin	Flesh
MACRO	Nitrogen	✓		✓	✓	✓	✓	✓	✓
	Calcium			✓	✓	✓		✓	✓
	Phosphorus	✓		✓			✓	✓	✓
	Magnesium							✓	
	Potassium	✓			✓	✓	✓	✓	✓
MICRO	Iron			✓		✓	✓	✓	
	Sodium	✓		✓		✓	✓		✓
	Boron								
	Copper								
	Zinc								

C.Performance of SEMS With EDAX

The sample is grained and sputtered in the instrument which creates a plasma over the top of the sample. After the sputtered sample is kept inside the SEM instrument with the aid of EDAX, the chemical elements present in the sample are determined.

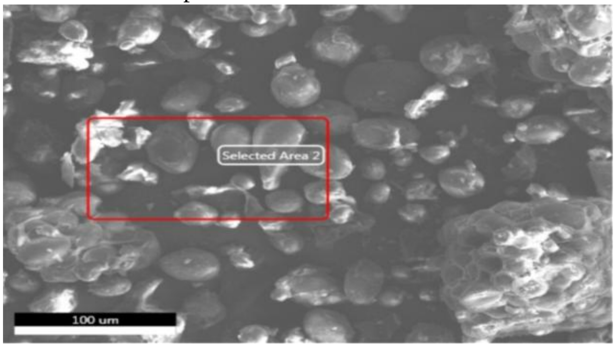

Fig. 13 potato sample SEM image

In SEM the sample is scanned with a depth level of 100 micrometers. An area is selected for the analysis and the presence of macro and micro nutrients. The distribution of elements in this area is shown in Fig. 13.

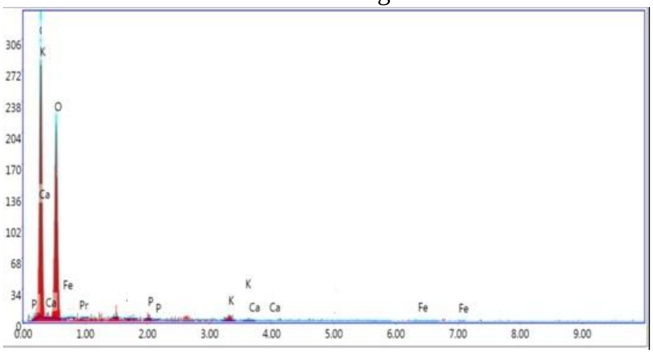

Fig. 14 EDAX output of potato sample

The macro and the micro nutrients present in the sample and determined by EDAX are calcium, phosphorus, iron and potassium as shown in Fig. 14 and the composition of elements and percentages of weight and atomic percentages are given in Table 4

Table 4 Element weight and atomic percentage in dry potato

ELEMENT	WEIGHT %	ATOMIC %
Potassium	46.22	54.63
Phosphorus	0.44	0.20

Calcium	0.22	0.08
Iron	0.54	0.14

Further analyses are made to detect the toxic elements present in the potato chips, lays and small potatoes scenario.

Case 1: Sample potato

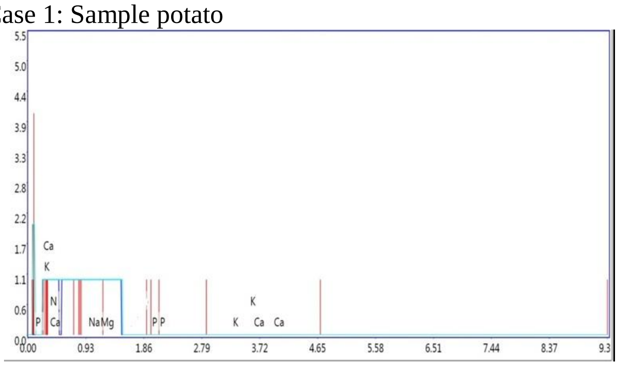

Fig. 15 EDAX output of small potato

The macro nutrients present in small potato are potassium, calcium, phosphorus, nitrogen and magnesium. Sodium is the only micronutrient present. These are shown in Fig. 15. Table 5 provides the information about the weight and atomic percentage present in this sample.

Table 5 Element weight and atomic percentage in small potato

ELEMENT	WEIGHT %	ATOMIC %
Phosphorus	0.00	0.00
Potassium	0.00	0.00
Calcium	8.47	5.18
Nitrogen	0.00	0.00
Magnesium	49.17	49.61
Sodium	42.36	45.20

Case 2: Potato chips

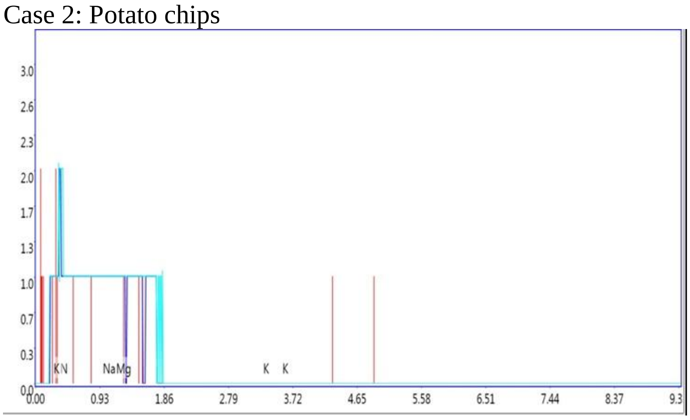

Fig.16 EDAX output of potato chips

Similarly, the macro and the micro nutrients present in potato chips and are identified are nitrogen, potassium, sodium, and magnesium and it shown in Fig. 16. Presence of

toxic elements is noted and identified.Further, the weight and atomic percentage are shown in Table 6.

Table 6 Element weight and atomic percentage in potato chips

ELEMENT	WEIGHT %	ATOMIC %
Nitrogen	100.00	100.00
Magnesium	0.00	0.00
Sodium	0.00	0.00
Potassium	0.00	0.00

Case 3: Lays

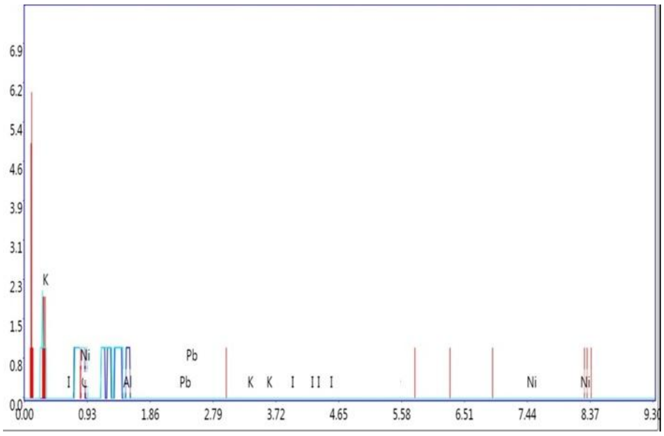

Fig. 17 EDAX output of Lays

The elements present in EDAX are potassium, cerium, lead, nickel, and indium as shown in Fig. 17. If these elements are taken by any human, it can lead to several problems such as heart disease, cancer and other disorders. The composition of elements and percentages of weight and atomic percentages are given in Table 7.

Table 7 Element weight and atomic percentage in Lays

ELEMENT	WEIGHT	ATOMIC
Potassium	16.67	25.70
Indium	16.67	7.92
Lead	16.67	4.85
Nickel	16.67	17.12

IV. CONCLUSION AND FUTURE WORK

In this paper, the essential examination procedures are used to establish out the supplements of potato. It manages the ends drawn in the wake of examining the consequence of the framework. The exploration is for breaking down the nutritive components present in the potato and if any dangerous component is available or not. By doing tries different things with various levels like changing the gathering focal point and spectrometer varieties will prompts get distinctive spectra and may build the nearness of macro and micro nutrients on the sample.

The unregulated usage of pesticides in the potato may lead to health issues and the macro and micro nutrients may get lost in percentage level, so the elemental detection methods are applied here in this research. The two main approaches, namely LIBS and SEM with EDAX area were applied. LIBS detected the presence of macro and micro nutrients in potatoes; SEM was used for obtaining the morphological structure of the sample while EDAX was used to identify the presence of macro and micro nutrients in the sample. The conclusion drawn are; macronutrients, viz., nitrogen, calcium, phosphorus, magnesium, potassium and the micronutrients, namely iron, sodium, boron, copper, zinc were detected by varying fluence level from 5mJ to 20mJ and the toxic elements, namely indium, lead, nickel were also detected. The future scope of this research is to make a handheld device to execute the above study. Also the investigation can be extended to other fruits and vegetables.

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