

XRD and SEM, ED Analysis Of Solar Dried Vegetables

Comment [DMKSM1]: the title more specifically mentions the type of material being analyzed

Abstract

The practical application of an analytical scanning electron microscope with an EDS system – microanalysis for examining the ash elemental composition of vegetable crop selection was considered. Morphology and surface analysis through XRD and SEM. The content of 14 basic elements (in weight percent) in the mineral component of vegetables was investigated. In the vegetables the accumulation order of the elements is the following: Ca >K >P >Mg >Si >Se >Fe >Mo ≈ S ≈ Cl ≈ Zn >Na >Al. The vegetables are rich in macro – and microelements. In the selecting procedure, vegetables with a high concentration of the components are recommended. The enhanced levels of key macro- and microelements such as Ca, K, P, Mg, Mo, S, and Cl indicate the possibility of developing functional products based on the examined vegetables for dietary enrichment.

Key word: *nutrient content, minerals, xrd, solar-dried, macroelements.*

1.Introduction:

Vegetables are a multi-cultural ingredient found in nearly every Indian dish. They are high in iron, calcium, -carotene, Vitamin C, dietary fibre, and several essential minerals. A substantial amount of leaves from various resources such as aquatic plants, perennial trees, and annuals are consumed in rural communities. These vegetables are a low-cost approach to gain micronutrients. GLV are seasonal and particularly perishable due to their high-water content. A lack of proper storage, transportation, and processing equipment at the time of production has resulted in substantial mortality.(Paul et al., 2012).To preserve nature's reservoir of nutrients, there is a need for simple processing procedures. Dehydration appears to be the simplest way to keep GLV in check, especially if they're numerous. Dehydration of vegetables is commonly done to prevent oxidation of perishable raw materials or to reduce the cost of processing, handling, storing, and shipping them. (Ahmed et al., 2016).Microorganism behaviour is the most important constraint on shelf-life extension. Due to the reduction in water activity, water activity in vegetables is reduced to a very low level after dehydration, resulting in improved microbiological preservation and the postponement of some damaging processes throughout storage.(Ibarz et al., 2000).Dehydration technology is used in this study from a distinct perspective. GLV are significant elements of critical

nutrients, and dehydrating them can provide us with a concentrated source of micronutrients, as previously stated. Using micronutrient-rich GLV in a dehydrated form as a food-based method to treat nutritional deficits that are frequent in our cultures, especially during seasons when food is limited. In recent years, efforts have been made to improve the nutritional availability of dried foods by altering methodological approaches and/or pre-treatment.(Mieszczakowska-Fraç et al., 2021).

The rate of the drying procedure in the Solar Dryer is aided by the flow of hot air, the product's distributing density, the cleanliness of pre-treatment, and the type of the product to be dried. Factors like the starting moisture level and the requisite end % moisture content often affect how long it takes to dry a product. (Phoungchandang et al., 2009).

1.1Solar Drying:

The solar dryer is a passive cabinet dryer that combines direct and indirect rocking thermal storage. The dryer retains heat in a rock bed, which is used to combat temperature fluctuations after the sun sets. The leaves collector's solar dryer had a temperature range of 40 to 73°C, while the drying chamber had a temperature range of 42 to 63°C. The samples were dried in the sun from 10 a.m. to 5 p.m. every day until they reached a constant weight.(Matazu & Haroun, 2004).

The current study looked at the effect of sun and solar drying on the nutritional value of green leafy vegetables.

2.Methodology:

2.1Chemicals:

Analytical grade chemicals were used in entire experiments.

Comment [DMKSM2]: What kind the chemical?

2.2Sample collection:

Spinach (*Spinacia oleracea*), fenugreek (*Trigonella foenumgraecum*), cauliflower (*Brassica oleracea*), tomato (*Solanum lycopersicum*), and chenopodium (*Chenopodium album*) were among the five gathered vegetable from the local market. The study was conducted at USIC lab Department of Environmental studies Babasaheb Bhimrao Ambedkar university Lucknow

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Spinacia oleraceae

2.3 Equipment

Solar dryer, centrifuge, muffle furnace

2.4 Plant preparation:

The dust was removed from the sample by washing it with distilled water, and some of it was dried to a steady weight using two different drying processes. As a control, fresh samples were used. (Kendall et al., 2004).

2.5 Mineral content:

EDS - analysis The chemical composition of the basic ash components (Na, P, S, K, Mn, Fe, Mg, Ca, Al, Si, Cl, Zn, Se, Mo) was determined by the method of energy dispersive spectrometry (EDS) on the analytical raster electron microscope .The microscope resolution is 4 nm at accelerating voltage 20 kV (secondary electrons image), zooming is from x 10 till x 10 000. While performing the elemental analysis the working distance (WD) is 10mm. Energy-dispersive spectrometer allows to carry out the quantitative X-ray microanalysis with the desired analyzing area: in a point or areally, and to receive the maps of elements allocation. X-ray microanalysis data are presented in the form of standard protocols which contain the microstructure picture of the sample under study, the table of the data in weighting and atomic correlation, spectra and histograms. The spectrum example is shown in Figure 1 of EDS(Reuter & Robinson, 1997).

Comment [DMKSM4]: What types of analysis are carried out?

3. Results And Discussion

3.1 X-Ray diffraction (XRD) analysis

Fig. 1(a), (b), (c), (d) and (e) respectively shows XRD pattern of T.F. *T. graecum*, *Solanum lycopersicum*, *spinacia oleracea*, *chenopodium album*, *brassica oleracea* powder. The sharp

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peaks in the given figures indicates the crystalline nature of the particles. The diffraction pattern shows well defined and the observed relative intensity peaks confirms the good crystalline nature and two amorphous nature that is result for good texture analysis. The peaks positioned at 2θ values (in degree) for fig (a,) of 26.68, 30.82, 50.28, 60, 68, (for fig b)26, 29,30 (fig. c) 21, 26.30, 27.83, 36.5, 42.27, 50.51, 55, 57.6, (fig. d) 14.7, 18.20, 24, 26, 28, 29, 31 and (fig.e) 20.71.

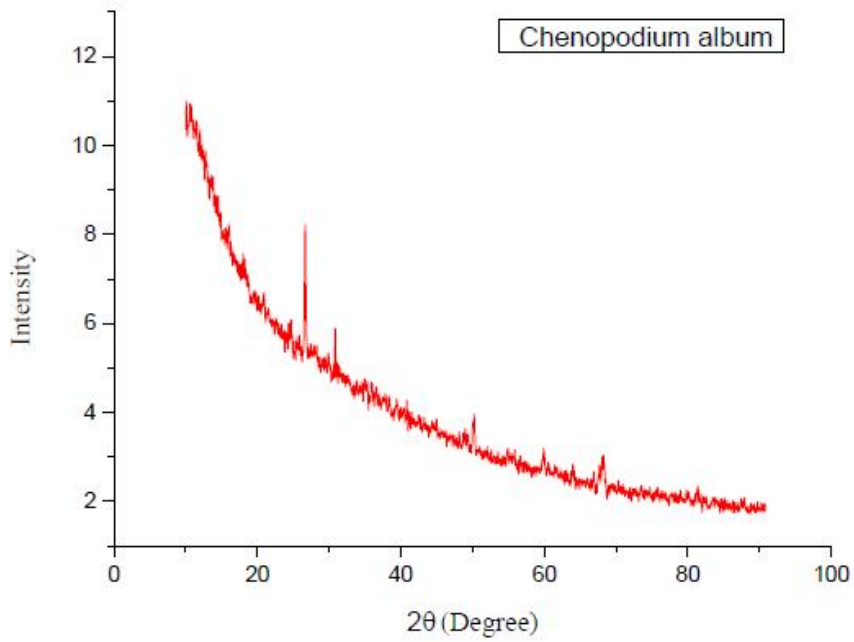


Fig 1(a) XRD image for Chenopodium Album

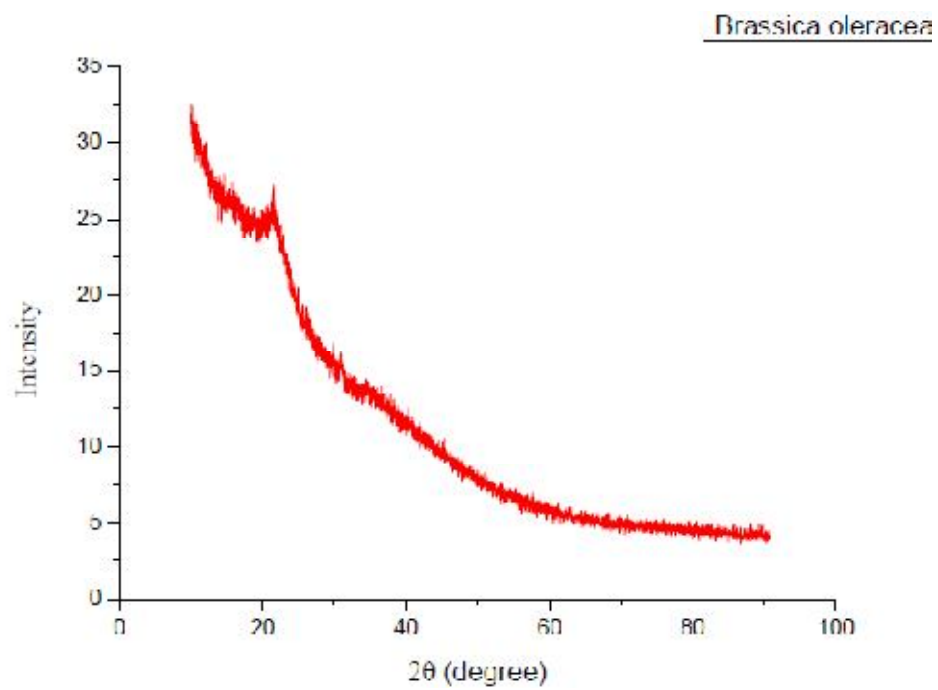


FIG. 1 (b) XRD pattern of Brassica oleracea

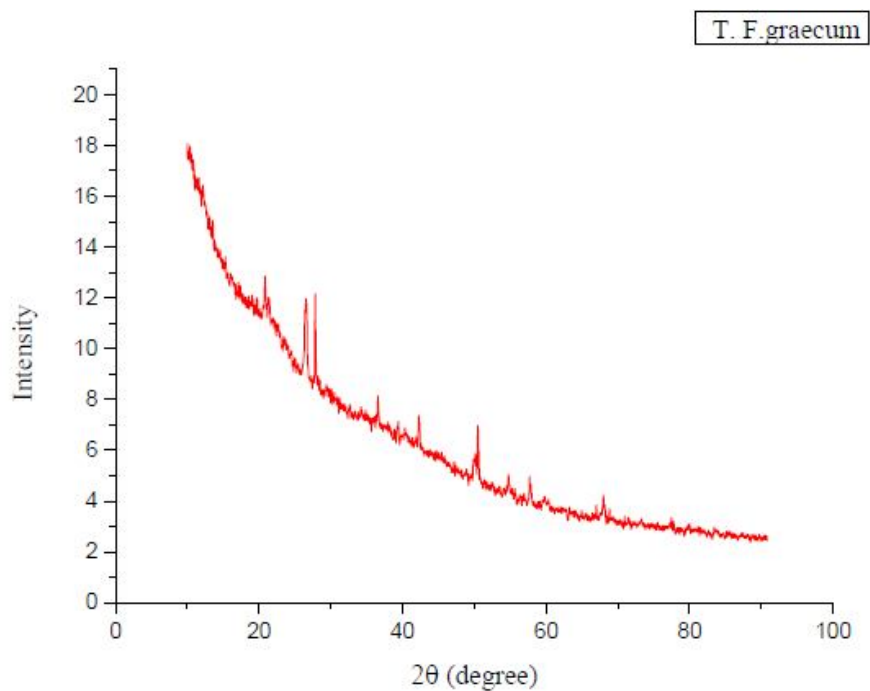


Fig. 1(c) XRD pattern of T. F. Graecum

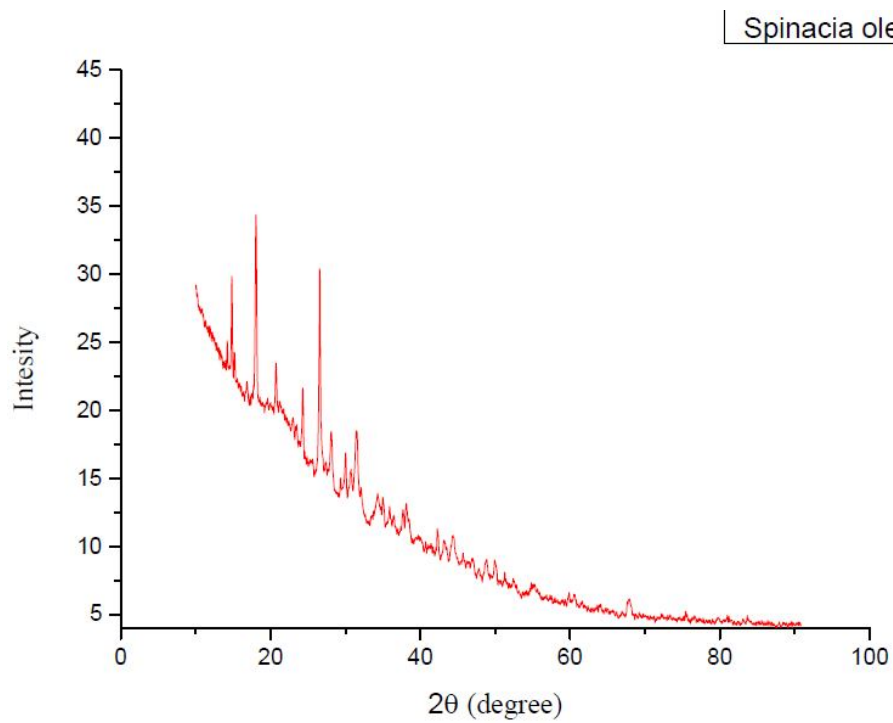


Fig. 1(d) XRD pattern for Spinacia oleracea

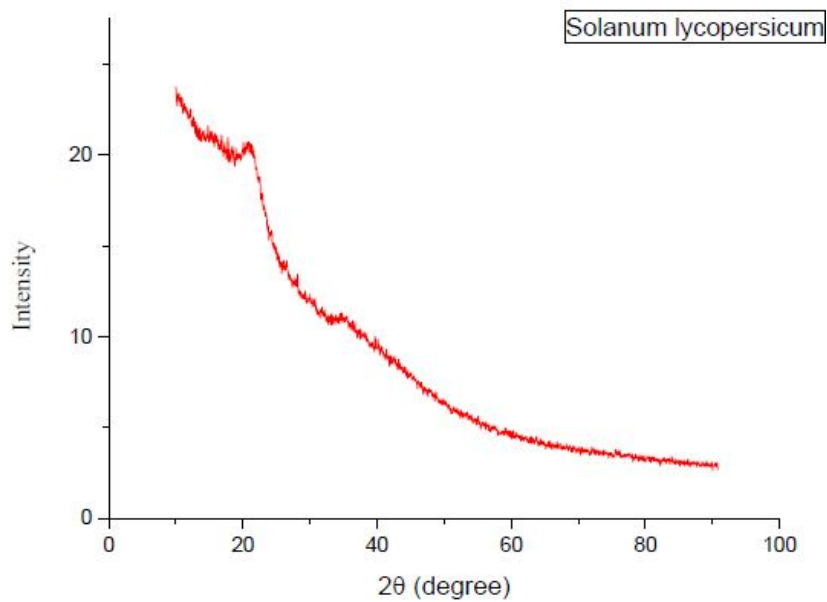


Fig. 1(e) XRD pattern for Solanum lycopersicum

3.2 Scanning electron microscopy (SEM) and EDX analysis:

The surface morphology analysis of the T.F. graecum, *Solanum lycopersicum*, *spinacia oleracea*, *chenopodium album*, *brassica oleracea* powder revealed the different shaped particles with agglomerated cluster-like morphology as shown in fig. 2 (a),(b), (c), (d), and (e). The agglomeration of the particles may be due to the existence of interfacial surface tension phenomenon. The elemental analysis of T.F. graecum, *Solanum lycopersicum*, *spinacia oleracea*, *chenopodium album*, *brassica oleracea* powder are depicted in Fig 3 (a), (b), (c), (d), and (e) respectively. Hence the EDX spectrum shows the peaks which confirms the presence different elements as shown in table. The atomic percentage (Fig. 3) is in good agreement with molar ratio of the different elements as present in the natural above materials.

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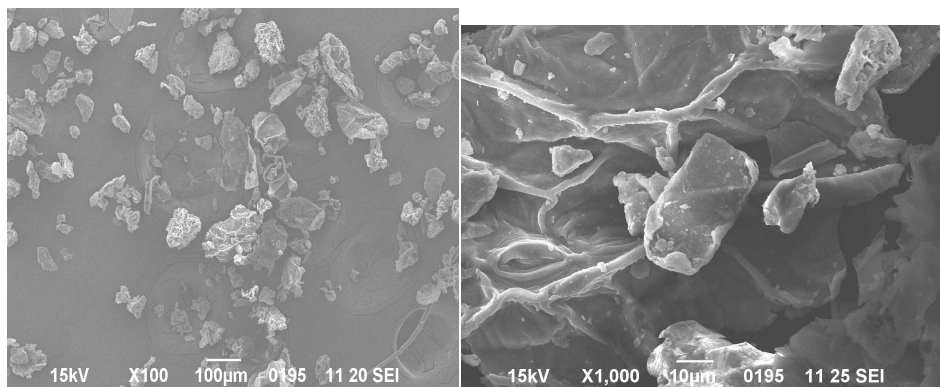


Fig 2(a) SEM image for Chenopodium Album

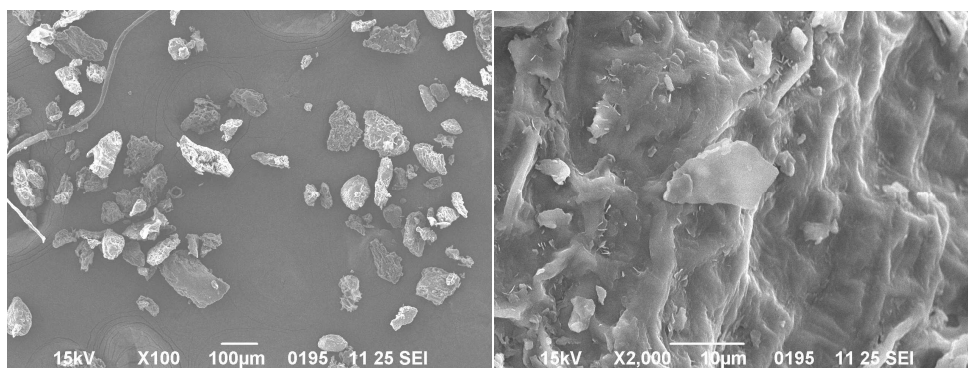


FIG. 2 (b) SEM pattern of Brassica oleracea

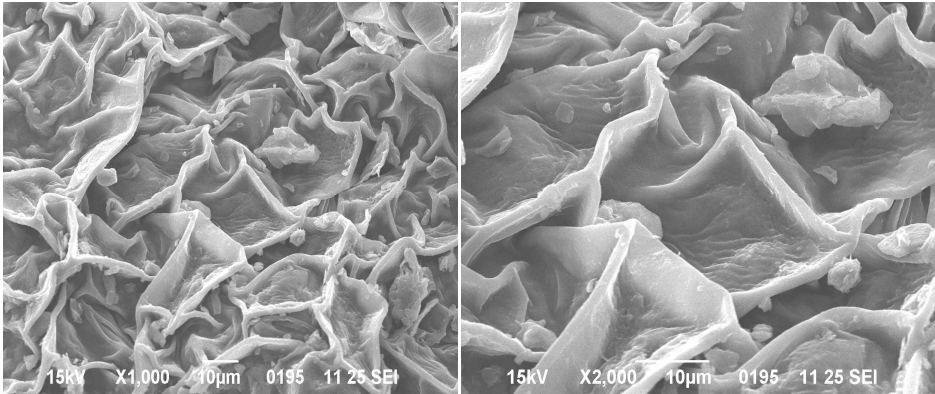


Fig. 2(c) SEM pattern of T. F. Graecum

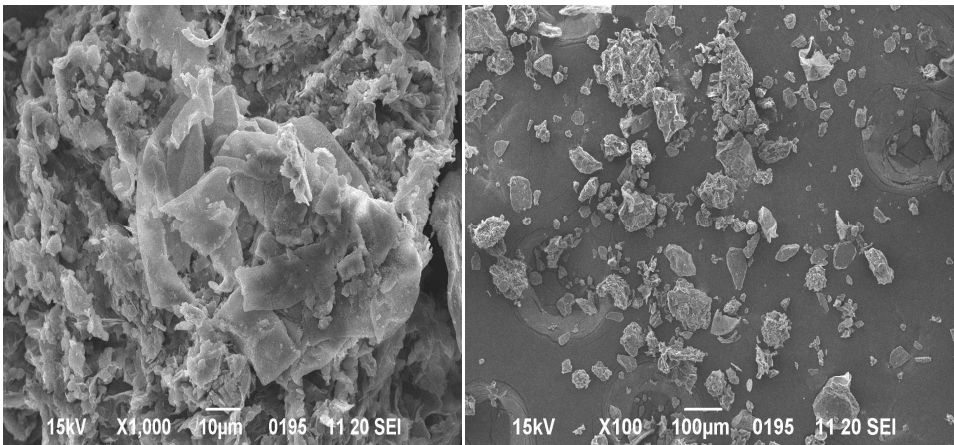


Fig. 2(d) XRD pattern for Spinacia oleracea

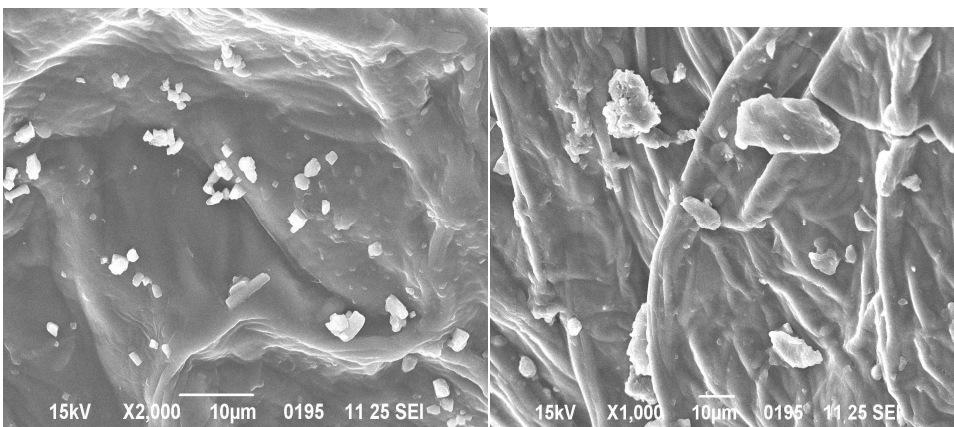


Fig. 2(e) XRD pattern for Solanum lycopersium

3.4 Statistical Analysis:

The data is presented in the form of means and standard deviations. The data was analysed using analysis of variance, complete randomised design, statistical analytical system (1988), SAS/STAT user's guide release (6, 035. A. Cary, N.C., USA). At a probability of 5%, a least significant difference (LSD) was declared significant. (Kuczmariski et al., 1994)

Chart 1 : EDX Analysis

S.No.	Name of sample	Elements Present
1.	Chenopodium Album	K, O, Fe, Na, Mg, Al, Si
2.	Brassica oleracea	K, C, O, Mg, S
3.	T. F. Graecum	K, Ca, Cl, O, Na, Mg, Si
4.	Spinacia oleracea	K, Cl, O, Na, Mg, Al, Si
5.	Solanum lycopersium	K, Cl, O, Na, Mg

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Spectrum of microelements presents in vegetables;

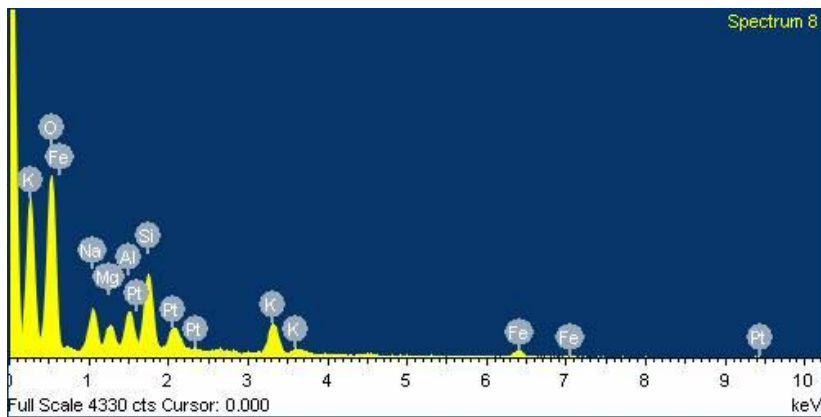


Fig 3 (a) SEM image for Chenopodium Album

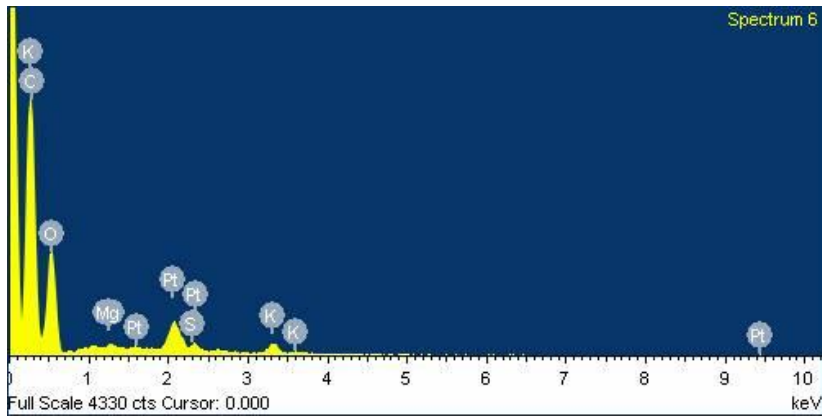


FIG. 3 (b) SEM pattern of Brassica oleracea

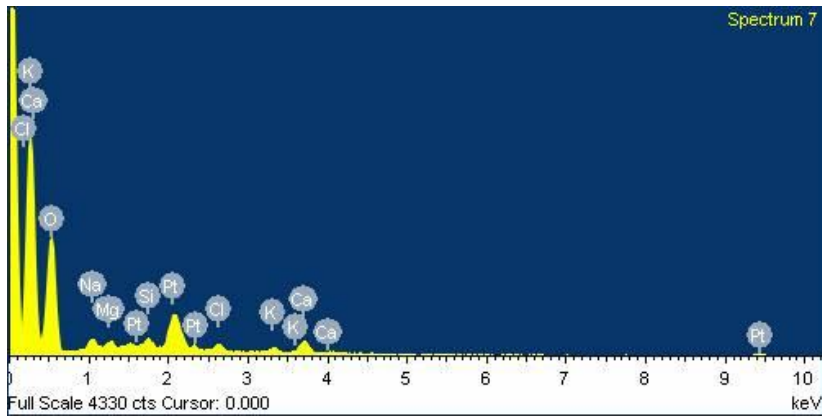


Fig. 3(c) SEM pattern of T. F. Graecum

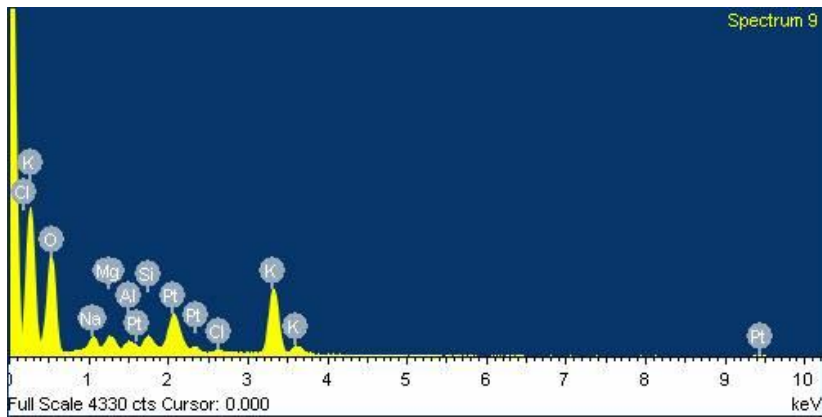


Fig. 3 (d) XRD pattern for Spinacia oleracea

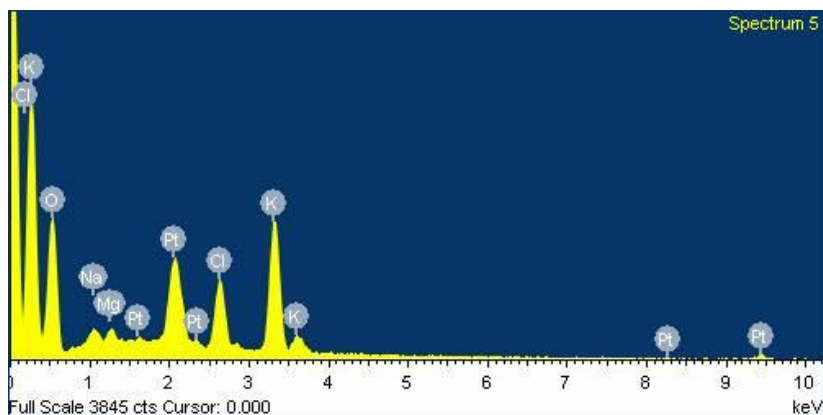


Fig. 3 (e) XRD pattern for Solanum lycopersium

Table 1: symmetrisation of Elements present in vegetables by mass%

Elements	Bathua	Coulliflower	Methi	Spinach	Tomato
Na	0.91 ±0.08	0.91 ±0.08	0.12 ±0.02	0.05 ±0.01	0.09 ±0.02
K	8.99 ±0.60 7	13.77 ±1.67	18.77 ±1.50	7.80 ±1.17	15.47 ±1.38
P	9.13 ±1.18	7.91 ±1.47	14.35 ±1.10	8.85 ±1.49	12.61 ±1.34
Ca	17.23 ±1.23	14.26 ±1.53	10.50 ±1.21	20.36 ±1.77	13.53 ±1.50
Mg	2.56 ±0.15	2.96 ±0.09	3.39 ±0.10	6.87 ±0.1	2.88 ±0.24
Mo	6.20 ±0.08	4.33 ±0.14	6.59 ±0.08 5	2.77 ±0.20	5.23 ±0.06
S	1.77 ±0.04	2.05 ±0.19	2.02 ±0.03	1.90 ±0.20	2.24 ±0.08
Si	0.42 ±0.03 1	1.53 ±0.10	0.22 ±0.01	1.04 ±0.18	3.11 ±0.25
Mn	0.86 ±0.04	3.65 ±0.28	2.02 ±0.23	0.45 ±0.09	0.68 ±0.04
Cl	0.16 ±0,07	0.24 ±0.02	0.06 ±0.02	0.32 ±0.18	0.40 ±0.05
Fe	0.17 ±0.09	0.34 ±0.14	0.33 ±0.07	0.08 ±0.01	0.89 ±0.12
Al	0.19 ±0.04	0.20 ±0.06	0.13 ±0.01	0.24 ±0.02	0.68 ±0.04
Zn	0.20 ±0,03	0.21 ±0.02	0.22 ±0.03	1.73 ±0.03	0.16 ±0.03
Se	0.41 ±0.03	0.32 ±0.03	0.36 ±0.02	0.48 ±0.03	0.20 ±0.03
Σ	48.04	52.70	59.05	59.047	58.16

Figure 4: The comparative concentration of macroelements in the ash of the vegetables.

The concentration of 14 basic elements (in mass %) contained in the vegetables mineral part was studied (Figure4). Herewith the main proportion of the ash elements in the seeds belongs to Ca. Ca takes part in the processes of living organisms growth and development, goes into the composition of coenzymes and cells nucleuses, it also takes part in the most important processes for the organism such as metabolism, immunity, regeneration and others (Gusev, 1998; Gins and Gins, 2011).

The proportion of Ca in the vegetables fluctuates from 13.171 to 20.361; mass %. K is a macroelement that is responsible for the regulation of the majority metabolic reactions that flow in living organisms. The very special role in controlling the homeostasis belongs to K. It controls osmotic pressure transmembrane potential, charges equilibrium, cathodeanion balance, pH – everything that the homeostasis of cells and tissues consists of. In the ionic form K can be found in all the organs, tissues and cell structures in the concentrations that exceed the concentration of other ions (Meathnis et al., 1997) The concentration of K in the vegetables fluctuates from 7.80 ± 1.17 (Spinach) to 18.77 ± 1.50 (methi) mass % relatively.

In the bathua cauliflower, tomato, the fluctuations were not essential – from 8.99 ± 0.60 , 13.77 ± 1.67 , 15.47 ± 1.38 mass % (Figure 2).

More than 50% of P is presented in tissues in the form of inorganic P (Pin). P is a part of DNA and RNA, phospholipids, phosphate esters, nucleoside phosphates – ATP, ADP, NADPH, where it fulfills the structural function (in composition of first two types of compounds), in the rest ones – metabolic. P plays a very important role in the cell energetics. For the plants the analogue of P is phytin – Ca^{2+} – Mg^{2+} - the sol of inositolphosphoric acid, essential quantities of which are accumulated in the seeds.

The concentration of P were detected 9.13 ± 1.18 , 7.91 ± 1.47 , 14.35 ± 1.10 , 8.85 ± 1.49 , 8.85 ± 1.49 , 12.61 ± 1.34 mass % at average in bathua, cauliflower, methi, spinach, tomato wherein in the ash of methi the proportion of P is more than all vegetable (Schachtman et. al., 1998).

Mg is necessary for the processes of regeneration and renewal of cells, tissues and organs. It activates a large number of enzymes that take part in the processes of CO_2 and N assimilation. In cytosol Mg counter-balances organic compounds (sugars groups, nucleotides, organic and amino acids). Mg is necessary for the keeping up of the cation balance and pH regulation. In the cell wall approximately 2.5% of the general concentration of Mg can be found. In the cell wall and in the seeds membrane Mg^{2+} is coordinately connected with carboxylic groups of pectin substances and takes part in the creation of the inner physiological environment of plants. Mg, Ca and N are localized in the seed membrane. ATP, phosphoinositol (phytin) in combination with Mg are accumulated in the seeds in the form which is comfortable for storage (Nechaev et.al., 2007).

In vegetables the concentration of Mg has close values 2.56 ± 0.15 , 2.96 ± 0.09 , 3.39 ± 0.10 , 6.87 ± 0.1 , 2.88 ± 0.245 mass % in bathua, cauliflower, methi spinach and tomato respectively orderly.

Mo fulfills a number of useful functions for the organism: it is a cofactor and an activator of oxidases (xanthine oxidase and serine oxidase), takes part in the amino acids synthesis, in the exchange of the vitamins C, E and B12 inside an organism (Avtsyn et al., 1991). The concentration of Mo in **vegetables** under study fluctuates from 6.20 ± 0.08 , 4.33 ± 0.14 , 6.59 ± 0.08 , 5.27 ± 0.20 , 5.23 ± 0.06 mass % bathua, cauliflower, methi spinach and tomato respectively, (Figure 2).

Firstly S is necessary for the synthesis and regulation of the plant produced protein quantity and quality. S is a biogenous element as a part of proteins and glutathione, it possesses antioxidant activity, provides the process of the energy transfer in the cell by transporting electrons, takes part in the methyl groups transportation and fixation, covalent, hydrogen and mercaptide connections production, enables the genetic information transfer. In vegetables S is contained 1.77 ± 0.04 , 2.05 ± 0.19 , 2.02 ± 0.03 , 1.90 ± 0.20 , 2.24 ± 0.08 bathua, cauliflower, methi spinach and tomato respectively.

(Table 2, Figure 2).

At the present time Cl belongs to the microelements. It is the most important biogenous element of living organisms. Cl ions together with Na and K ones take part in the support of the osmotic equilibrium and salt- water exchange regulation. The transportation of Cl contributes to the realization of the following functions: electrical and Ca^{2+} – signalization, membrane potential and pH gradient control. Cl together with Ca is included in the mechanism of the stomatal movements (Avtsyn et al., 1991). In vegetables Cl is contained 0.16 ± 0.07 , 0.24 ± 0.02 , 0.06 ± 0.02 , 0.32 ± 0.18 , 0.40 ± 0.05 mass % in bathua, cauliflower, methi spinach and tomato respectively more than in the seeds of the vegetable application breeds – 1.818 mass % (Table 1, Figure 2).

Si is an obligatory element for the plants (Kolesnikov and Gins, 2001). It is accumulated in large amount in the leaves cell walls, especially in the ecdemonic tissues of the scape leaves and the roots. Si is not only the base of the tissues framed element, but it also controls a number of biological and chemical processes in the living organism. It influences the seeds emergence and the seedlings growth it quickens the scapes growth and the dry biomass accumulation. Si has a protective effect toward the harmful influence of Al ions, creating aluminosilicates which are included in the composition of phytoliths. Si enlarges the plants resistivity to abioogenous stresses. The concentration of the biogenous eminent – Si in the amaranth seeds of grain breeds is in 2.6 times more than in the seeds of the vegetable forms (Table 1, Figure 3). Se is a powerful antioxidant that increases the living organism resistivity to biogenous and abioogenous stressors influence is the necessary microelement that is a part of active centers in the form of aminoacid selenocysteine (Vikhreva et al., 2001). The concentration of Se in the vegetables is 0.42 ± 0.03 , 1.53 ± 0.10 , 0.22 ± 0.01 , 1.04 ± 0.18 , 3.11 ± 0.25 mass % in bathua, cauliflower, methi spinach and tomato respectively.

Mn is a co-factor and activator of many enzymes (pyruvate kinase, decarboxylase, superoxide dismutase) it takes part in the synthesis of glycoproteins and proteoglycans and possesses antioxidant activity. In the vegetables the concentration of Mn is $0.86 \pm 0.04, 3.65 \pm 0.28, 2.02 \pm 0.23, 0.45 \pm 0.09, 0.68 \pm 0.04$ mass % (Table 2, Figure 3).

Fe as a part of active centers – hemoproteins and iron-sulphur proteins determines the space structure and activity and takes part in oxidation-reduction reactions. The alternative form of Fe is the molecule of protein ferritin that may accumulate up to 4,500 atoms of Fe in soluble nontoxic form. In the amaranth seeds Fe and Cu are localized in the corcule (Shmalko and Roslyakov, 2011). Organic Fe is an essential compound for the human organism. This element is a part of catalytic centres of many oxidation-reduction enzymes. Fe as a part of active centers – hemoproteins and iron-sulphur proteins determines the space structure and activity and takes part in oxidation-reduction reactions. The alternative form of Fe is the molecule of protein ferritin that may accumulate up to 4,500 atoms of Fe in soluble nontoxic form. In vegetables Fe and Cu are localized in the corcule (Shmalko and Roslyakov, 2011). Organic Fe is an essential compound for the human organism this element is a part of catalytic centres of many oxidation-reduction enzymes. Zn stabilizes the molecules structure, plays an important role in DNA and RNA metabolism, in the protein synthesis and cells fission, in the processes of the signal transmission inside the cell (Nechaev et al., 2007; Pedersen et al., 1987). Zn is also an important biogenous element, its concentration in the vegetables does not exceed 0.217 mass %. Na is contained mostly in the intercellular fluid Na in the combination with K takes part in the membrane potential creation, the activation of enzymes and muscular contractions, supports osmosis, acid-alkaline and water balance, provides membrane transport (Avtsyn et al., 1991). In vegetables the concentration of Na is more in bathua and couliflower than in the seeds of vegetable forms (Table 1, Figure 3).

4. CONCLUSION

In this synthesis process, the final synthesized particles were formed by performing many irreversible reactions. A SEM image indicates the good agglomeration of nanoparticles.

Chinopodium, cauliflower, fenugreek, spinach, and tomato are all high in ash compounds, which are minerals like potassium, sodium, calcium, and magnesium (Gins and Gins, 2011). The proportion of elements in the ash was determined, and the variation coefficients were derived using the energy dispersive X-ray spectrometry method to obtain new data on the

diversity of vegetable mineral composition. The predominant accumulation of Ca, K and P is typical for all the vegetables samples under study. In the selecting procedure, vegetables with a high concentration of the components are recommended. The presence of key macro- and microelements such as Ca, K, P, Mg, Mo, S, and Cl in high concentrations in the examined vegetables suggests the possibility of developing functional products based on them for food enrichment. Taking this into account, studying the composition and mineral element concentration in various organs of the plant, as well as their impact on human life activity, is a real (global) problem, because the macro- and microelement deficit in industrial food is extremely large and dangerous to human health, as the majority of food is mineral depleted.

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