



## Effect of Activated Carbon Prepared from Leaflet of Date Palm Tree and Composite Nano silica/Activated Carbon on Growth and Productivity of Fenugreek Plant under Salinity Soil Conditions

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### ABSTRACT

A field experiment was conducted with completely randomized design to assess the impact of the foliar application of using activated carbon from leaflet of date palm tree as by product and composite nano silica/activated carbon to alleviate salinity stress on fenugreek plants. The prepared samples AC and SiO<sub>2</sub>/AC were characterized using zeta potentials, particle size distributions, EDX, FTIR, S<sub>BET</sub> and morphology description were done using SEM and TEM. All techniques confirmed the good distribution of SiO<sub>2</sub> nanoparticles on surface of AC composite sample and particles size of SiO<sub>2</sub>/AC were found to be in the size range of 70–80 nm. Characterization results showed that surface area of AC prepared from leaflet of date palm tree using KOH as activating agent and SiO<sub>2</sub>/AC are 397.47m<sup>2</sup>/g and 656.33m<sup>2</sup>/g respectively. Through five foliar concentrations from sodium silicate, composite nano silica/activated carbon and activated carbon at (100, 300, 500, 700 and 1000 mg l<sup>-1</sup>) compared with untreated plants as control. Using of SiO<sub>2</sub>/AC could be a good way to deal with problem of soil salinity. As for, there were no significant effect among 100, 300, 500 mg l<sup>-1</sup> SiO<sub>2</sub>/AC for EL% and MSI% in plant leaves. Foliar spraying with 500 mg l<sup>-1</sup> SiO<sub>2</sub>/AC improved different aspects of growth parameters, nutrient elements, amino acids and protein content in seeds. Furthermore, 300 mg l<sup>-1</sup> SiO<sub>2</sub>/AC gave the highest weight of 100 seeds, weight of seeds yields ton fed<sup>-1</sup> and improved quality of fixed oil. On other side, proline content through both seasons decreased by 46.02 and 38.78 % compared with control when 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC applied.

**Keywords:** Activated carbon, Nano silica, Date palm tree, Salinity, Fenugreek.

### Introduction

Fenugreek (*Trigonella foenum-graecum*. L) is an annual plant belonging family *Fabaceae*. It is one of the most important medicinal and spice plants in the world (Ahmad *et al.*, 2016). Fenugreek considers a rich source of nutritional benefits as well as biological and pharmacological properties for human as a result of containing some chemical constituents as carbohydrates, proteins, lipids, pyridine type alkaloids such as trigonelline, choline, flavonoids, amino acids, minerals, saponins, β-carotene, sitosterol, vitamins, nicotinic acid, diosgenin and volatile oils (Nathiya *et al.*, 2014 and Ahmad *et al.*, 2016). Moreover, Fenugreek seeds contain fixed oils. The major components of the oil, linolenic acid and oleic acid, pinene, palmitic acid, -Pentyl-1-(4-propylcyclohexyl)-1-cyclohexene, linoleic acid methyl ester (GU *et al.*, 2017). Thus, it improves hyperglycemia and has anticancer, antimicrobial and antioxidant effects. Moreover, fenugreek improves adipocyte differentiation, inhibition of inflammation

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in adipose tissues, decreasing pancreatic and renal damage (Al Mosawi, 2021). The production of fenugreek crop is affected by environmental stress such as: drought, salinity and heat.

Salinity stress is one of the main environmental problems affecting on crops production (Taha *et al.*, 2021). When a plant is exposed to salinity, its production decreases and effects on its physiology and biochemistry properties. The negative effect of salinity on plant growth is linked to the osmotic effect and/or the ionic effect. Both effects are harmful as they may cause changes in cell membrane characteristics, water status, enzymes, protein synthesis and gene expression to growing plants (Prakash *et al.*, 2011).

Silicon (Si) has beneficial roles in growth of plants, development and its metabolism (Arif *et al.*, 2021). Its absence results negative consequences in plant growth and yield (Rajput *et al.*, 2021). Also, Si can alleviate different biotic and abiotic stresses (Afshari *et al.*, 2021 and Bayoumi *et al.*, 2021). Furthermore, Si improves the toughness of cell walls in plants and accumulates in the gaps among cells and outer layers, resulting in larger, thicker, and heavier leaves (Son *et al.*, 2012).

Sodium silicate is the common name for  $\text{Na}_2\text{SiO}_3$  and also called "glass water".  $\text{Na}_2\text{SiO}_3$  had efficacy to relieve the salt stress and showed improvement in growth parameters of wheat plants grown under salt stress (Mushtaq *et al.*, 2021).

Nano materials and their applications are increasing in agricultural biotechnology fields. Nano materials may have special properties due to their small size. When contrasted to bulk materials, they have the ability to modify physico-chemical properties because their surface area is greater than bulk materials, their solubility and surface reactivity tend to be higher. Whereas, Nano silica is considered an important nano fertilizers or nanoparticles with highly reactive surface to volume ratios. The most important agro-application of nano silica is to facilitate plant growth (Al-Saeedi, 2021), stress tolerance (Mathur and Roy, 2020) such as drought and salinity (Bayoumi *et al.*, 2021). The use of nano silica improves pea plants in saline soils (Ismail *et al.*, 2022).

Nowadays many agricultural by-products have received attention in the scientific literature. Date palm tree consider as economic crop especially in the Middle East. These trees generate tons of leaves, rachis, pits and trunk as wastes; a lot of them are converted to compost and using for traditional art and craft or burning causing pollution to environment (Maha *et al.*, 2021). Many products can be extracted from different parts of date palm tree as cellulose fiber from leaves and rachis (Maha *et al.*, 2021) and activated carbon from date palm pits by using different chemical activating agent and used in removal of organic and inorganic pollutant from waste water. Also leaves of date palm tree is consider as by-products which is pruned annually to ensure the healthy growing of the date palm tree and the quality of date fruits, there is about 1562171 tons from leaflet which produced annually as each tree produces 13-20 Kg of leaves (Chandrasekaran and Bahkali, 2013).

Activated carbon produced from agricultural waste materials. Its availability, cheap cost and efficient production might make it a top priority to replace nonrenewable commercial activated carbon in a variety of applications. (Yahya *et al.*, 2018). Active carbon has direct effects on plants as it improves nutrient availability and pH (Kabouw *et al.*, 2010).

Thereupon, the aim of the work dealing with preparation, characterization and application in agriculture effects as:

- 1- Preparation and characterization of activated carbon from leaflet of date palm tree and composite nano silica/ activated carbon using various instrumentation techniques.
- 2- Evaluation of foliar application from different concentration of sodium silicate, active carbon and composite nano silica/activated carbon on alleviation of salinity stress on fenugreek plants.

## 2. Materials and Methods

### 2.1. Raw materials

Date palm tree leaves (*Phoenix dactylifera* L.) were gathered from Egypt (Agricultural research center). The leaves were separated from the stalk, washed with water to get rid of dust and shaded in sunlight for one week, then cut, mild and collected through a 3 mm diameter mesh before chemical analysis.

Potassium hydroxide, Tetra -ethyl ortho silicate (TEOS)  $\text{Si}(\text{C}_2\text{H}_5\text{O})_4$ , formamide  $\text{HCONH}_2$ , ethanol  $\text{C}_2\text{H}_5\text{OH}$ , nitric acid  $\text{HNO}_3$  and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) were obtained from Sigma-Aldrich.

## 2.2. Preparation of Activated carbon (AC) from leaflet of date palm tree and composite nano silica/activated carbon (SiO<sub>2</sub>/AC).

To prepare activated carbon, leaflet will be carbonized at 700 °C for 3h, then the samples was impregnated in solution from KOH by ratio 1:2 (precursor: KOH) using appropriate amount of water for 2 days, then samples dried over night after that the samples fired at 550°C for 3h, washed, dried and stored in bottle.

SiO<sub>2</sub> /AC was prepared as 228 ml from TEOS, 157 ml HCONH<sub>2</sub> which called as dry additive agent, 60ml C<sub>2</sub>H<sub>5</sub>OH and 4ml HNO<sub>3</sub> (1N aq) after that activated carbon sample which prepared from leaflet was added to above solution and stirring the mixture for 2h at 50 °C and aged for three days. The formed product was dried over night at 105°C, after that the samples was fired at 550 °C for 3h to calcined.

## 2.3. Characterization of prepared AC and SiO<sub>2</sub>/AC

Zeta potential and particle size distribution of activated carbon from leaflet of date palm tree and composite nano silica activated carbon samples was done using Zetasizer nano series (NanoZS, Malvern, UK), while, EDX analyses of the samples were done using Oxford Link Isis instrument, UK., IR spectra were done using FT/IR - 4700 type A in the absorbance mode from 4000 to 400 cm<sup>-1</sup>. The morphology and structure of AC were observed by SEM (Quanta 250 FEG) as AC sample were sputter coated with gold for 2 min and then observed under a working voltage of 15 kV. While SiO<sub>2</sub>/AC were studied using TEM Image Tecnai G20, Super twin, double tilt, FEI, Netherland. Surface area were determined using gas sorption analyzer. The adsorption – desorption isotherm of nitrogen was determined at its boiling point -196 °C by Quanta chrom 3000 instruments, the samples are degassed under vacuum at 200 °C for 5 h before measurement.

## 2.4. Field experimental

Two field experiments were carried out at the Experimental Station of the Agricultural Research Center in Tag El- Ezz, Dakahlia Governorate, Egypt (30°57' 25" N latitude and 31°35' 54" E longitude) through winter seasons of 2020/2021 and 2021/2022 to study the impact of different foliar concentrations from sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>), composite nano (SiO<sub>2</sub> /AC) and (AC) from leaflet on plant growth, yield and chemical contents of fenugreek plant grown under saline soil conditions. The Average properties of the experimental soil for the two seasons are presented in Table (1).

**Table 1:** Average physico-chemical properties of the experimental soil during two seasons.

|  |                      |                |   |                        |        |
|--|----------------------|----------------|---|------------------------|--------|
| <b>Particle size distribution</b>            | <b>Coarse sand</b>   | 3.43           | <b>Ions<br/>(meq/100g soil)</b>                     | <b>Ca<sup>++</sup></b> | 1.88   |
|  | <b>Fine sand</b>     | 29.40          |   | <b>Na<sup>+</sup></b>  | 2.88   |
|  | <b>Silt</b>          | 38.05          |   | <b>K<sup>+</sup></b>   | 0.47   |
|  | <b>Clay</b>          | 29.12          |   | <b>Cl<sup>-</sup></b>  | 2.76   |
|  | <b>Texture class</b> | Sand clay loam |   | <b>Mg<sup>++</sup></b> | 1.27   |
| <b>EC dS m<sup>-1</sup></b>                  |                      | 5.69           | <b>Available nutrients<br/>(mg kg<sup>-1</sup>)</b> | <b>N</b>               | 49.93  |
| <b>pH (1:2.5)</b>                            |                      | 8.04           |   | <b>P</b>               | 4.43   |
| <b>Organic matter (g 100 g<sup>-1</sup>)</b> |                      | 1.24           |   | <b>K</b>               | 136.00 |

## 2.5. Layout of Experimental

The experimental design was arranged in a complete randomized blocks with sixteen treatments; each treatment was replicated three times.

Chemical fertilizers (NPK) were added as ammonium sulphate (20.5% N) at 200 kg fed<sup>-1</sup>, superphosphate (15.5% P<sub>2</sub>O<sub>5</sub>) at 200 kg fed<sup>-1</sup> and potassium sulphate (48% K<sub>2</sub>O) at 100 kg fed<sup>-1</sup>. Super phosphate was added during soil preparation, ammonium sulphate and potassium sulphate doses were applied in two equal splits, after 30 and 60 days from sowing. Organic manures (FYM) were added to the soil before sowing at the rate of 10 m<sup>3</sup> fed<sup>-1</sup>. Chemical analyses of the organic manures used are presented in Table (2).

**Table 2:** Average chemical analysis of FYM used for two seasons

| Properties                    | Value |
|-------------------------------|-------|
| pH (1:10)                     | 6.18  |
| EC (1:10)(dSm <sup>-1</sup> ) | 3.84  |
| Organic matter (%)            | 31.89 |
| Total Nitrogen (%)            | 1.14  |
| Total Phosphorous (%)         | 0.39  |
| Total Potassium (%)           | 0.51  |

## 2.6. The experiment treatments.

Foliar application of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC each of them solely were done three times. The first dose was given 40 days after sowing and was repeated twice after 20 days interval, sixteen treatments were arranged as follow:

1) control (untreated plants); 2) Na<sub>2</sub>SiO<sub>3</sub> at 100 mg l<sup>-1</sup>; 3) Na<sub>2</sub>SiO<sub>3</sub> at 300 mg l<sup>-1</sup>; 4) Na<sub>2</sub>SiO<sub>3</sub> at 500 mg l<sup>-1</sup>; 5) Na<sub>2</sub>SiO<sub>3</sub> at 700 mg l<sup>-1</sup>; 6) Na<sub>2</sub>SiO<sub>3</sub> at 1000 mg l<sup>-1</sup>; 7) SiO<sub>2</sub> /AC at 100 mg l<sup>-1</sup>; 8) SiO<sub>2</sub> /AC at 300 mg l<sup>-1</sup>; 9) SiO<sub>2</sub> /AC at 500 mg l<sup>-1</sup>; 10) SiO<sub>2</sub> /AC at 700 mg l<sup>-1</sup>; 11) SiO<sub>2</sub> /AC at 1000 mg l<sup>-1</sup>; 12) AC at 100mg<sup>-1</sup>; 13) AC at 300mg<sup>-1</sup>; 14) AC at 500 mg<sup>-1</sup>; 15) AC at 700 mg<sup>-1</sup>; 16) AC at 1000 mg<sup>-1</sup>.

## 2.7. Cultivation Process

Fenugreek seeds (*Trigonella foenum-graecum* L.) were obtained from Medicinal and Aromatic Plants Research Department, Horticultural Research Institute, Agricultural research center, Egypt. Cultivation process was performed in the second week of October in 1<sup>st</sup> and 2<sup>nd</sup> season respectively. Each treatment was replicated three times, which contained three rows, 5m long and 60 cm width. The seeds were sown in hills on two sides of row and the distances between hills 20 cm. After 20 days from sowing, seedlings were thinned to be two plants / hill. Other field practices for fenugreek growing were followed according to the recommendation of the Ministry of Agriculture and Land Reclamation, Egypt.

## 2.8. Data recorded.

### 2.8.1. Plant growth parameters.

Plant height (cm), leaves number, branch number, fresh and dry weights of the shoot (g) per plant, length of root (cm), fresh and dry weights of the root (g) per plant were estimated and Leaf area plant<sup>-1</sup> (cm<sup>2</sup>) measured according to Alessi and Power (1975) using the following formula:

Leaf area (LA) = leaf length x maximum leaf width x 0.75. Leaf area in cm<sup>2</sup> of three plants was summed and the leaf area/plant was calculated.

### 2.8.2. Seed yield components.

Number of pods plant<sup>-1</sup>, weight of seeds plant<sup>-1</sup> (g) and seed yield fed<sup>-1</sup> (ton) were determined. Also, weight of 100 seeds (g) was estimated.

### 2.8.3. Chemical analysis of fenugreek plant.

**Electrolyte leakage:** Ion leakage was estimated as electrical conductivity (EC%) (Hassanein *et al.*, 2012) by conductivity meter (Jenway 470 portable conductivity meter). The percentage of electrolyte leakage (EL %) was calculated as a formula:

$$EL (\%) = (C1/C2) \times 100.$$

Where C1 and C2 are the electrolyte conductivities determined before and after boiling, respectively.

**Membrane stability index (MSI %):** was determined by Rady (2011). The MSI (%) was calculated by the following equation according to Premchandra *et al.* (1990).

$$MSI (\%) = [1 - (EC1/EC2)] \times 100.$$

Where, EC1 = electrical conductivity value in leaf tissue sample solution after heating at 40 °C for 30 min, and EC2 = electrical conductivity value in another leaf tissue sample solution after heating at 100 °C for 10 min.

**Chlorophyll content** was estimated in the fresh leaves according to Gavrilenko and Zigalova (2003).

Fenugreek seeds samples (0.2 g) were taken to wet digestion using a mixture of sulphuric and perchloric acids (1:1) to determine the following nutrient elements:

**Total Nitrogen (g. 100g<sup>-1</sup>)** was determined by the modified microkjeldahl method as described by Jones *et al.* (1991)

**Total phosphorus (g. 100g<sup>-1</sup>)** was estimated using spectrophotometer by Milten Roy Spectronic 120 at wavelength 725 nm using stannous chloride reduced molybdosulphoric blue colour method in sulphuric system according to Peters *et al.* (2003).

**Total potassium and sodium (mg. 100g<sup>-1</sup>)** was determined using Flame-Photometer by Jenway PFP7 model according to Peters *et al.* (2003).

**Calcium** was determined by atomic absorption spectrophotometer using perkin Elmer Model 370A as described by Chapman and Pratt (1978).

#### 2.8.4. Chemical composition

**Crude protein** in the plant seeds was calculated by multiplying N content by 6.25 (A.O.A.C. 2000).

**Proline** was assayed in fresh leaves according to the method of Marin *et al.* (2010). Two ml of the extract, 2.0 ml of acid ninhydrin and 2.0 ml of glacial acetic acid were added. After that, it incubated for 1 h in a boiling water bath followed by an ice bath. The absorbance was measured at 520 nm using spectrophotometer (Jasco, V-630, B163061148, Japan). Standard curve was determined by using a known concentration of pure proline.

**Total carbohydrate** was estimated in the plant seeds colorimetrically using spectrophotometer (Jasco, V-630, B163061148, Japan) as method given by (Shumaila and Safdar, 2009).

**Total free amino acids** in the plant seeds was determined using spectrophotometer (Jasco, V-630, B163061148, Japan) according to the method described by Jayaraman (1985).

**Fixed oil** of fenugreek seeds was extracted using n-hexane as a solvent in a Soxhlet system HT apparatus according to the methods of (AOAC, 1984). Then, fixed oil yield per plant and per fed. were calculated.

**Fatty Acid Profiles** in fixed oil of fenugreek seeds in the second season were performed in the Regional Center for Food and Feed, ARC, Egypt, using Gas chromatogram with FID detector according to the methods of (AOAC, 2000).

#### 2.9. Statistical analysis

The statistical analysis software; CoStat version 6.4 (CoHort Software) was used for the analysis of variance (ANOVA) of the data, comparison among means was carried out using Duncan's new multiple range test at probability (*P*) level  $\leq 0.05$  (CoStat, 2005).

### 3. Results and Discussion

#### 3.1. Zeta potential

Zeta potential analysis was carried out to spot the surface charges obtained by prepared samples in the colloidal state. Figure (1a and 1b) represented zeta potential of AC from leaflet and composite SiO<sub>2</sub>/AC which showing negative zeta potential -21.8 for activated carbon due to using KOH as activating agent and -9.41 for composite SiO<sub>2</sub>/AC.

#### 3.2. Dynamic light scattering

It is a broadly technique carried out to determine solid particles size in colloidal solution Figure (2a and 2b) represented particle size of AC from leaflet and composite nano SiO<sub>2</sub>/AC. we found that the size in the range of 100-1000 in case of AC which approximately equal 425 nm, while in case of composite nano SiO<sub>2</sub>/AC the particle size in rang of 10–100 nm and PDI = 0.256 which means that the sample is in range of nanomaterials and the particles is monodispersed.

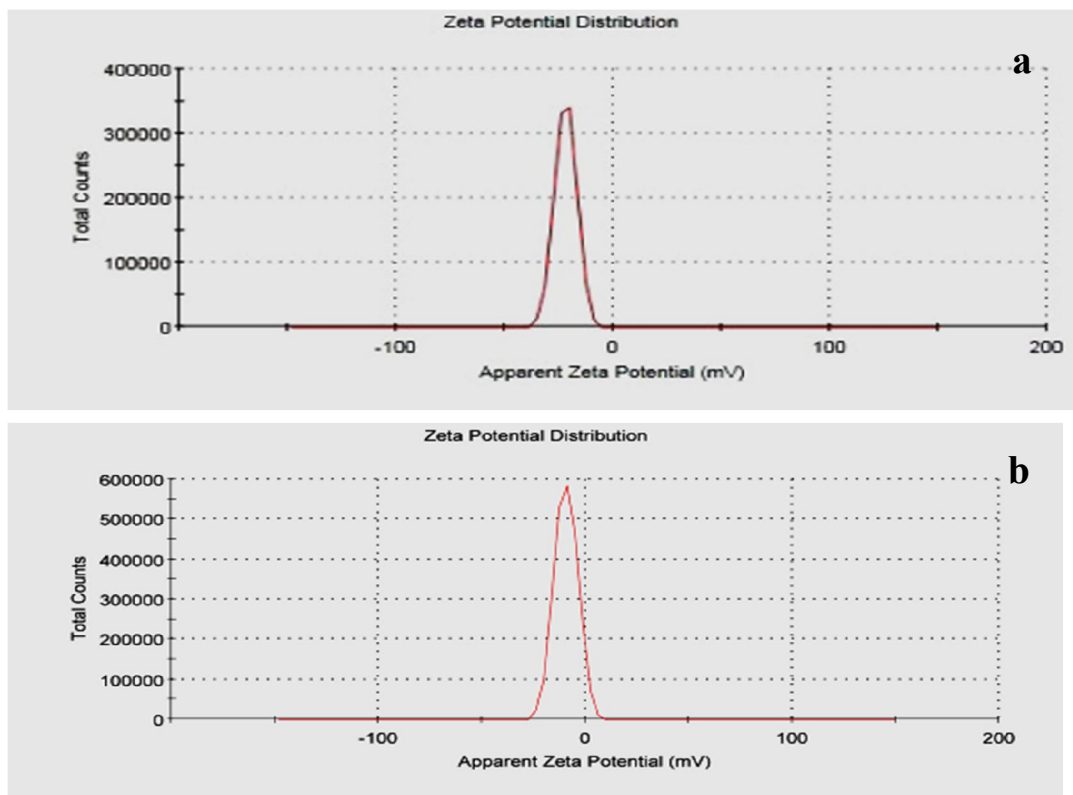


Fig. 1: Zeta potential of (a) activated carbon from leaflet, (b) composite nano silica/ activated carbon.

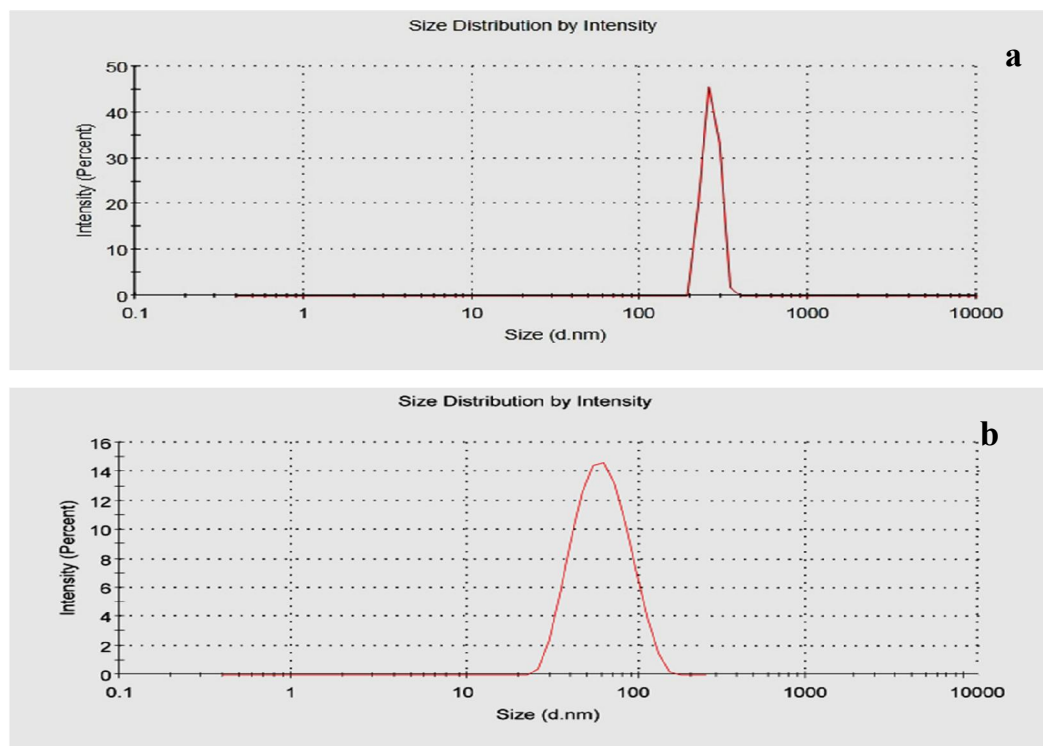


Fig. 2: Size distribution by number using Dynamic Light Scattering for (a) activated carbon from leaflet and (b) composite nano silica/ activated carbon.

### 3.3. Energy dispersive x-ray

It is done to determine elemental analysis of all prepared samples from Figure (3a and 3b) we found that in case of AC sample there is only two peaks occur which corresponding to carbon and oxygen (70.31 wt % and 29.7 wt %) respectively, while in case of composite nano SiO<sub>2</sub>/AC carbon there are three peaks appear which corresponding to carbon (48.05 wt %), silica (8.912 wt %) and oxygen (43.05 wt %). appearance of these peaks confirmed the formation of composite sample.

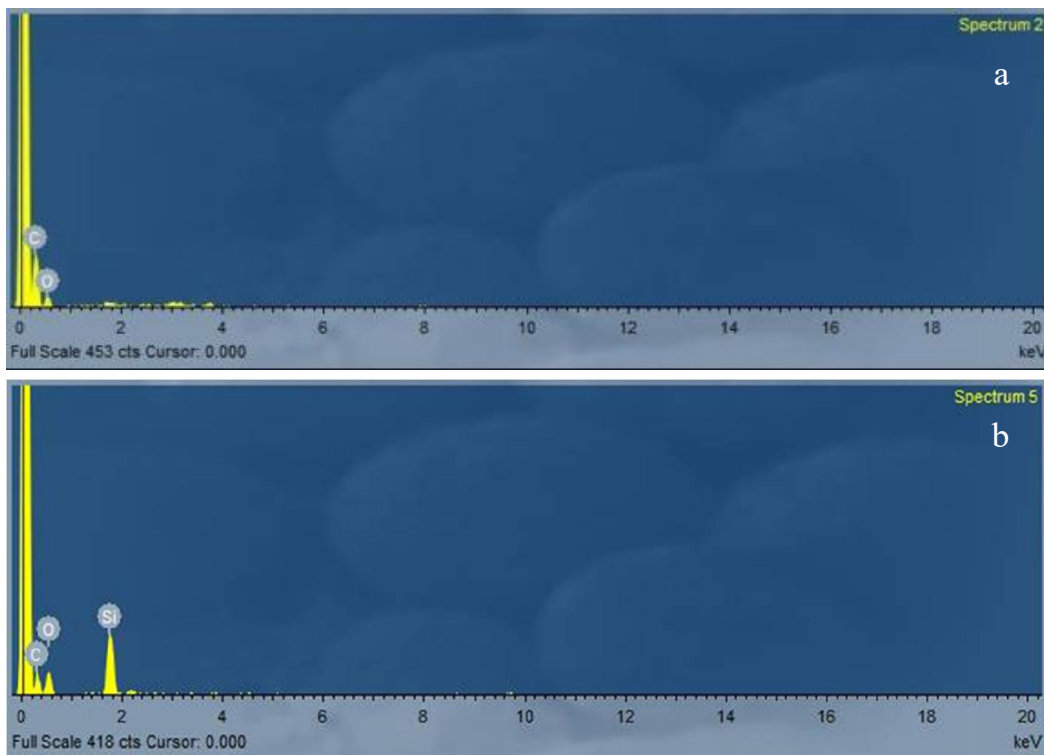


Fig. 3: EDX spectrum of (a) Activated carbon from leaflet, (b) Composite nano silica/activated carbon

### 3.4. FTIR spectroscopy analysis

FTIR spectroscopy is a simple technique that giving information about chemical changes which appears during chemical treatments (Ristolainen *et al.*, 2002). Figure (4) compares the FTIR spectra of AC from leaflet and SiO<sub>2</sub>/ AC. The observation of absorption bands shows that there band around 3500 – 3400 cm<sup>-1</sup> is due to the –OH stretching vibration mode of hydroxyl functional groups and adsorbed water (Aguilar *et al.*, 2003). Small peak around 1520 cm<sup>-1</sup> may due to the C=C stretching vibration in aromatic rings (Foo and Hameed 2009), due to the tars. While in case of nano composite SiO<sub>2</sub>/ AC carbon there is peaks around 1050, 794 and 470 cm<sup>-1</sup> refer to asymmetric, symmetric and the bending modes of SiO<sub>2</sub> (Günzler and Gremlich 2002&Duran *et al.*, 1986).



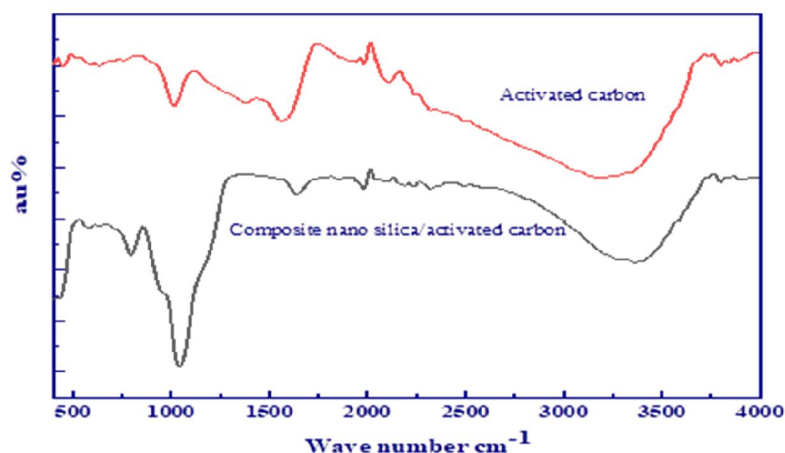


Fig. 4: FT-IR spectra of activated carbon from leaflet and composite nano silica /activated carbon.

### 3.5. Morphology of AC and SiO<sub>2</sub>/AC samples.

SEM is a technique used to detect differences in surface morphologies, while TEM is a major review measurement of the nano-particles size and morphology characterization. Figure (5a) illustrates the SEM image of the AC sample, and we found that activated carbon had randomly distributed pores. Such porosity was generated due to using potassium hydroxide as an activating agent, pyrolytic decomposition of hemicelluloses, and removal of byproducts from washing. Pore formation in activated carbon is important as it is the active site which plays an essential role in the formation of composite nano SiO<sub>2</sub>/AC samples. From Figure (5b) it could be seen that silica particles had a relatively better dispersion on the surface of activated carbon and a tendency to form spherical silica particles with an average diameter of 70 – 80 nm.

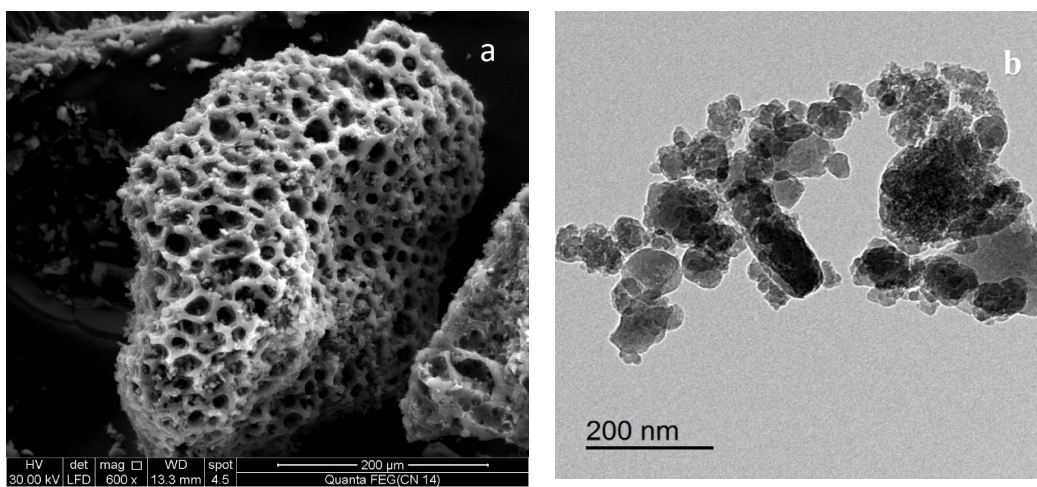


Fig. 5: (a) SEM of activated carbon from leaflet and (b) TEM of composite nano silica / activated carbon

### 3.6. Surface area and pore structure.

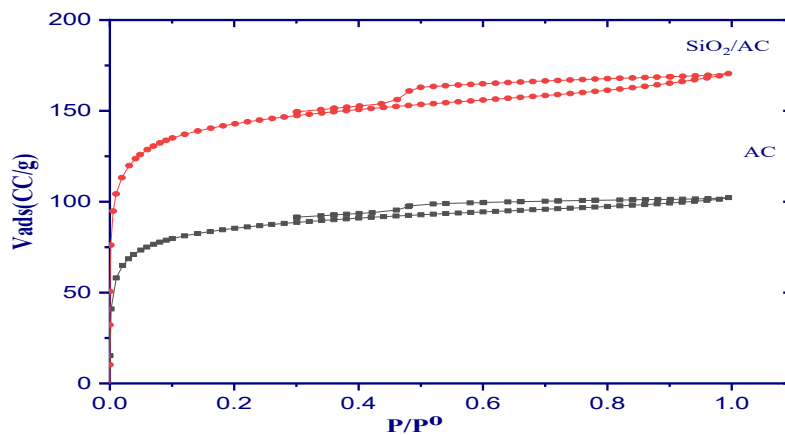
The BET surface area ( $S_{BET}$ ), and the mean pore radius ( $r_p$ ) of the activated carbons were determined by application of Brunauer-Emmett-Teller (BET) analysis software available with the instrument equation and applied in the relative pressure range from 0.06 to 0.30 (Aroua *et al.*, 2008). The data are listed in Table (3).



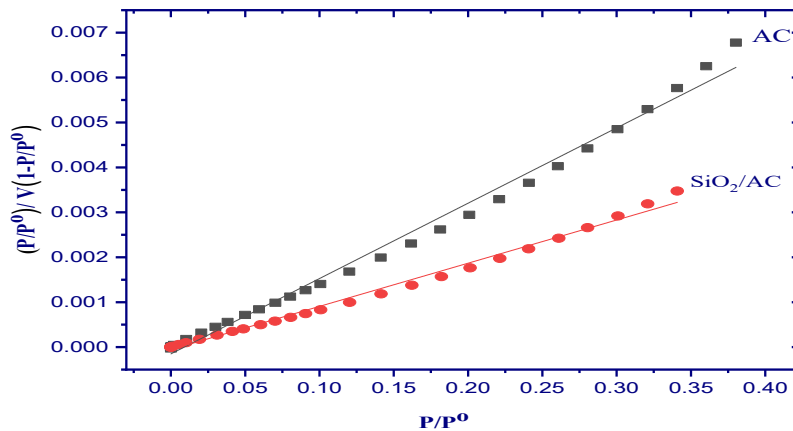
**Table 3:** Textural properties of AC and SiO<sub>2</sub>/AC as determined from nitrogen adsorption at -196°C

| Adsorbents           | S <sub>BET</sub> (m <sup>2</sup> /g) | C <sub>BET</sub> | V <sub>p</sub> (cm <sup>3</sup> /g) | r <sup>*</sup> (nm) |
|----------------------|--------------------------------------|------------------|-------------------------------------|---------------------|
| AC                   | 397.46                               | 3.071            | 0.1608                              | 0.800               |
| SiO <sub>2</sub> /AC | 656.33                               | 2.75             | 0.2643                              | 0.815               |

From Table (3) we can conclude that: the surface area for composite nano silica is higher than surface area of activated carbon from leaflets so this sample of nano silica will give good result with the plant that treated with it. Figure (6) presents the nitrogen adsorption/desorption isotherms at -196 °C for the investigated activated carbon sample. It is clear that all above sample give hysteresis loop, this hysteresis loop is usually attributed to thermodynamic or network effects or combination of two effects (Rouquerol *et al.*, 1999). Also, from Figure (7) we found Good straight line was obtained for points located in the relative pressure range (0.05-0.30). The second step when we applied BET theory is the determination of the surface area S<sub>BET</sub>



**Fig. 6:** Nitrogen adsorption / desorption isotherms at -196 oC for activated carbon from leaflet and composite nano silica / activated carbon



**Fig. 7:** The BET Linear plots of nitrogen adsorption isotherms at 77K for activated carbon from leaflet and composite nano silica / activated carbon

### 3.5. Electrode leakage (EL%) and membrane stability index (MSI%).

Data showed that (EL%) and (MSI%) of salt stressed fenugreek leaves showed various responses when treated with foliar spray of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC at different concentrations. It was noted

that the foliar spray of different concentrations caused a significant decrease in EL% and significantly increase MSI% in plant leaves comparing with control (Table 4). There was no significant effect among 100 mg<sup>l</sup><sup>-1</sup> AC, 100, 300, 500 mg<sup>l</sup><sup>-1</sup> SiO<sub>2</sub>/ AC through 1<sup>st</sup> season. Application of SiO<sub>2</sub>/ AC at 100 mg<sup>l</sup><sup>-1</sup> decreasing EL% by 31.10, 31.05 % and increasing MSI% by 14.13, 16.56% through two seasons, respectively when compared with control. Values of EL% and MSI% can be used to expect cell membrane damage that caused by salt stress (Ali *et al.*, 2008). The stimulation effect of saline on the EL % value might be attributed to injury the plasma membrane, that damage caused by reactive oxygen species (ROS) which could induce lipid peroxidation and consequently electrolyte leakage (Kassab *et al.*, 2012). Decreasing of EL% in response to various foliar application treatments may be due to stimulation of antioxidant responses that protect the plants from oxidative damage, increased membrane stability and tolerance of plants which in sequence enhanced scavenging of harmful free radicals (Rubinowska *et al.*, 2014) that keeps the plant from the oxidative damage by silicon treatments (Salwa *et al.*, 2013). Similar results were obtained by (Abdul Qados, 2015).

**Table 4:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on electrode leakage and membrane stability index of fenugreek plants grown under salinity conditions during 2020/2021 and 2021/2022 seasons.

| Treatments   | Electrode leakage (EL %) |                        | Membrane stability index (MSI %) |                        |       |
|--|--------------------------|------------------------|----------------------------------|------------------------|-------|
|  | 1 <sup>st</sup> season   | 2 <sup>nd</sup> season | 1 <sup>st</sup> season           | 2 <sup>nd</sup> season |       |
| <b>Control</b>   |                          | 31.25                  | 34.78                            | 68.75                  | 65.22 |
| <b>Na<sub>2</sub>SiO<sub>3</sub><br/>(mg<sup>l</sup><sup>-1</sup>)</b> | <b>100</b>               | 28.84                  | 31.37                            | 71.16                  | 68.63 |
|  | <b>300</b>               | 28.58                  | 30.64                            | 71.42                  | 69.36 |
|  | <b>500</b>               | 27.64                  | 28.56                            | 72.36                  | 71.44 |
|  | <b>700</b>               | 26.40                  | 28.00                            | 73.60                  | 72.00 |
|  | <b>1000</b>              | 26.50                  | 26.61                            | 73.50                  | 73.39 |
| <b>SiO<sub>2</sub>/AC<br/>(mg<sup>l</sup><sup>-1</sup>)</b>            | <b>100</b>               | 21.53                  | 23.98                            | 78.47                  | 76.02 |
|  | <b>300</b>               | 21.79                  | 23.40                            | 78.21                  | 76.60 |
|  | <b>500</b>               | 21.88                  | 24.37                            | 78.12                  | 75.63 |
|  | <b>700</b>               | 25.29                  | 25.63                            | 74.71                  | 74.37 |
|  | <b>1000</b>              | 26.43                  | 27.50                            | 73.57                  | 72.5  |
| <b>AC<br/>(mg<sup>l</sup><sup>-1</sup>)</b>                            | <b>100</b>               | 21.97                  | 23.66                            | 78.03                  | 76.34 |
|  | <b>300</b>               | 25.64                  | 26.84                            | 74.36                  | 73.17 |
|  | <b>500</b>               | 27.43                  | 28.68                            | 72.57                  | 71.32 |
|  | <b>700</b>               | 29.50                  | 32.06                            | 70.50                  | 67.94 |
|  | <b>1000</b>              | 30.63                  | 33.89                            | 69.38                  | 66.11 |
| <b>LSD at 5%</b>   |                          | 1.53                   | 1.71                             | 1.73                   | 1.82  |

### 3.6. Plant growth characters.

It was observed that there were no significant differences between 100 mg<sup>l</sup><sup>-1</sup>SiO<sub>2</sub>/ AC and 300 mg<sup>l</sup><sup>-1</sup> AC for shoot length comparison to the control. Foliar spray fenugreek plants with SiO<sub>2</sub>/ AC at 100 mg<sup>l</sup><sup>-1</sup> induced shoot length by (22.68 and 32.54%); root dry weight by (113 and 157%) , respectively through two seasons. While, foliar spray with SiO<sub>2</sub>/ AC at 500 mg<sup>l</sup><sup>-1</sup> revealed significant positive effects for branch number by (127.58 and 95%); leave number by (75.43 and 101%); fresh and dry weight of shoot by (136.18, 123.33 and 133, 130 %), leaf area by (73.62 and 77.01%) during 1<sup>st</sup> and 2<sup>nd</sup> seasons, respectively. concerning with root length, there were no significant difference among plants treated with 100 mg<sup>l</sup><sup>-1</sup> Na<sub>2</sub>SiO<sub>3</sub>, 1000 mg<sup>l</sup><sup>-1</sup> AC and untreated plants (Table 5).

To overcome salinity stress, plants have changed several systemic signals pathways as electricity, calcium, ROS, and hormones (Choudhury *et al.*, 2018), which enables plant tissues to alleviate stress (Fichman and Mittler, 2020). As discussed before the surface area for SiO<sub>2</sub>/AC is 660.33 m<sup>2</sup>g<sup>-1</sup> as mentioned in Table (3). So, composite nano silica activated carbon can improve the plant tolerance to salt stress by decreased ROS production or hormones level changing (Wang *et al.*, 2022), increased antioxidant enzymes activities (Naguib and Abdalla, 2019), increased K<sup>+</sup> uptake and decreased Na<sup>+</sup> uptake by addition of Si were the main mechanisms responsible for improving growth of plants under salinity conditions (Shahzade, 2012). These results are in context with Badawy *et al.* (2021). It was found that foliar applications of SNPs improve saline tolerance, root improvement, growth efficiency of rice. Furthermore, activated carbon had randomly pores on the surface as observed in (Figure 5a).

(Chutipajit and Sutjaritvorakul, 2018) who stated that activated carbon enhanced the callus induction and regeneration of plant in tissue culture medium of rice.

**Table 5:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on plant growth characters of fenugreek plants grown under salinity conditions during 2020/2021 and 2021/2022 seasons.

| Treatments   | Shoot length (cm) |                 | Branch (No.)    |                 | Leaves (No)     |                 | Root length (cm) |                 | Leaf area (cm <sup>2</sup> ) |                 |      |
|--|-------------------|-----------------|-----------------|-----------------|-----------------|-----------------|------------------|-----------------|------------------------------|-----------------|------|
|  | 1 <sup>st</sup>   | 2 <sup>nd</sup> | 1 <sup>st</sup> | 2 <sup>nd</sup> | 1 <sup>st</sup> | 2 <sup>nd</sup> | 1 <sup>st</sup>  | 2 <sup>nd</sup> | 1 <sup>st</sup>              | 2 <sup>nd</sup> |      |
|  | season            | season          | season          | season          | season          | season          | season           | season          | season                       | season          |      |
| Control  | 35.50             | 40.23           | 7.25            | 10.00           | 57.00           | 64.00           | 16.50            | 18.00           | 2.54                         | 2.48            |      |
| Na <sub>2</sub> SiO <sub>3</sub> (mg l <sup>-1</sup> ) | 100               | 36.00           | 44.50           | 8.50            | 10.00           | 73.00           | 77.00            | 16.50           | 17.00                        | 3.89            | 4.01 |
|  | 300               | 37.00           | 47.00           | 10.60           | 11.25           | 77.00           | 83.00            | 15.59           | 16.15                        | 3.60            | 3.80 |
|  | 500               | 38.00           | 47.50           | 12.00           | 14.00           | 86.50           | 80.00            | 13.50           | 15.75                        | 3.49            | 3.56 |
|  | 700               | 40.00           | 49.66           | 13.00           | 15.00           | 92.00           | 100.00           | 13.50           | 14.50                        | 3.49            | 3.50 |
|  | 1000              | 39.50           | 48.00           | 14.00           | 16.50           | 95.50           | 93.00            | 13.75           | 14.59                        | 3.49            | 3.50 |
| SiO <sub>2</sub> /AC (mg l <sup>-1</sup> )             | 100               | 43.76           | 53.32           | 13.50           | 17.75           | 94.00           | 107.50           | 13.00           | 12.00                        | 3.90            | 4.13 |
|  | 300               | 40.00           | 51.50           | 14.50           | 19.00           | 96.50           | 117.50           | 11.50           | 11.00                        | 3.90            | 3.90 |
|  | 500               | 39.15           | 50.45           | 16.50           | 19.50           | 100.0           | 129.5            | 10.50           | 10.00                        | 4.41            | 4.39 |
|  | 700               | 38.55           | 50.00           | 14.00           | 18.50           | 97.50           | 104.00           | 12.15           | 11.50                        | 4.25            | 4.19 |
|  | 1000              | 36.62           | 47.50           | 12.80           | 16.35           | 82.50           | 88.00            | 13.00           | 13.00                        | 3.70            | 3.85 |
| AC (mg l <sup>-1</sup> )                               | 100               | 41.00           | 49.00           | 10.00           | 12.50           | 65.00           | 77.50            | 14.56           | 15.00                        | 4.20            | 4.23 |
|  | 300               | 43.50           | 52.50           | 10.75           | 12.50           | 74.00           | 89.75            | 12.75           | 13.50                        | 3.38            | 3.44 |
|  | 500               | 40.77           | 51.00           | 12.00           | 14.00           | 89.00           | 100.50           | 13.50           | 14.00                        | 3.47            | 3.50 |
|  | 700               | 37.79           | 43.45           | 8.50            | 11.00           | 70.00           | 75.00            | 15.50           | 16.50                        | 3.04            | 2.92 |
|  | 1000              | 33.00           | 41.00           | 7.00            | 9.50            | 63.00           | 65.00            | 16.50           | 18.00                        | 2.70            | 2.61 |
| LSD at 5%  | 1.25              | 1.47            | 1.45            | 1.33            | 5.90            | 7.42            | 1.50             | 1.81            | 0.21                         | 0.18            |      |

**Table 5:** continued.

| Treatments   | Shoot fresh weight (g plant <sup>-1</sup> ) |                 | shoot Dry weight (g plant <sup>-1</sup> ) |                 | Root fresh weight (g plant <sup>-1</sup> ) |                 | Root dry weight (g plant <sup>-1</sup> ) |                 |      |
|--|---|-----------------|---|-----------------|--|-----------------|--|-----------------|------|
|  | 1 <sup>st</sup>                             | 2 <sup>nd</sup> | 1 <sup>st</sup>                           | 2 <sup>nd</sup> | 1 <sup>st</sup>                            | 2 <sup>nd</sup> | 1 <sup>st</sup>                          | 2 <sup>nd</sup> |      |
|  | season                                      | season          | season                                    | season          | season                                     | season          | season                                   | season          |      |
| Control  | 11.22                                       | 12.73           | 1.89                                      | 1.98            | 0.39                                       | 0.45            | 0.15                                     | 0.14            |      |
| Na <sub>2</sub> SiO <sub>3</sub> (mg l <sup>-1</sup> ) | 100   | 13.05           | 14.50                                     | 2.46            | 2.82                                       | 0.55            | 0.53                                     | 0.17            | 0.16 |
|  | 300   | 16.50           | 19.13                                     | 2.83            | 2.43                                       | 0.69            | 0.74                                     | 0.21            | 0.23 |
|  | 500   | 17.37           | 21.12                                     | 3.02            | 3.18                                       | 0.80            | 0.88                                     | 0.28            | 0.32 |
|  | 700   | 20.02           | 22.00                                     | 3.28            | 3.69                                       | 0.85            | 0.92                                     | 0.26            | 0.26 |
|  | 1000  | 23.18           | 25.50                                     | 3.51            | 3.52                                       | 0.77            | 0.81                                     | 0.24            | 0.26 |
| SiO <sub>2</sub> /AC (mg l <sup>-1</sup> )             | 100   | 22.57           | 25.00                                     | 3.24            | 3.73                                       | 0.81            | 0.88                                     | 0.32            | 0.36 |
|  | 300   | 23.52           | 25.34                                     | 3.58            | 4.01                                       | 0.98            | 1.13                                     | 0.31            | 0.33 |
|  | 500   | 26.50           | 28.43                                     | 4.40            | 4.57                                       | 0.86            | 1.01                                     | 0.28            | 0.30 |
|  | 700   | 23.41           | 24.12                                     | 4.16            | 4.18                                       | 0.79            | 0.92                                     | 0.28            | 0.29 |
|  | 1000  | 23.56           | 23.15                                     | 2.41            | 3.12                                       | 0.70            | 0.77                                     | 0.17            | 0.20 |
| AC (mg l <sup>-1</sup> )                               | 100   | 19.24           | 19.00                                     | 2.80            | 2.98                                       | 0.87            | 1.02                                     | 0.20            | 0.23 |
|  | 300   | 20.92           | 23.43                                     | 4.16            | 3.96                                       | 0.75            | 0.98                                     | 0.30            | 0.33 |
|  | 500   | 22.06           | 25.13                                     | 3.56            | 3.78                                       | 0.69            | 0.74                                     | 0.23            | 0.28 |
|  | 700   | 16.57           | 15.97                                     | 2.02            | 2.17                                       | 0.52            | 0.60                                     | 0.18            | 0.20 |
|  | 1000  | 11.00           | 12.45                                     | 1.75            | 1.84                                       | 0.49            | 0.46                                     | 0.15            | 0.15 |
| LSD at 5%  | 1.37  | 1.92            | 0.22                                      | 0.16            | 0.12                                       | 0.12            | 0.021                                    | 0.027           |      |

### 3.7. Yield parameters.

It was obviously clear that the foliar application of SiO<sub>2</sub>/ AC at 500 mg l<sup>-1</sup> induce increasing the number of pods per plant by (89.9 and 102.88 %) comparing with the control through both seasons respectively. While, at concentration 300 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC increased weight of seeds per plant by (34.13 and 37.05%) and weight of 100 seeds by (41.51 and 37.73 %) , through two seasons, respectively when compared with the control. However, 1000 mg l<sup>-1</sup> AC gave poor data for the studied traits (Table 6). Salinity may cause damage to the ovary, and subsequently cause a reduction in the yield (Munns and Tester, 2008) due to harm effects of salinity on some metabolic processes in plant tissues (Al-Ashkar *et al.*, 2019), also plants close stomata to prevent water loss through transpiration. This mechanism limits CO<sub>2</sub> assimilation, which in turn slows down the photosynthesis process and limits the plant yield (El Moukhtari *et al.*, 2020). The present study showed treating with SiO<sub>2</sub>/ AC were the most effective in alleviating the harmful effect of salinity on the plants. Where, Si treatments lead to increase K uptake

and decrease Na uptake, hence increasing plant tolerance to salinity stress, as a result increasing productivity (Meena *et al.*, 2014). Moreover, SNPs improved the mechanical strength of plant tissues because of the amorphous solid Si accumulation on the cell wall layers (Yaghubi *et al.*, 2019), enhancing the activity of antioxidant enzymes (Abdul Qados, 2015). Beside nano silica, activated carbon enhanced the flowering in pineapple plants (van de Poel *et al.*, 2009). Our results agree with Ayman *et al.* (2020) who found that nano silica improves wheat yield under salinity stress and Taha *et al.* (2021) on lupine.

**Table 6:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on yield characters of fenugreek plants grown under salinity conditions during 2020/2021 and 2021/2022 seasons.

| Treatments   | No of pods plant <sup>-1</sup> |                 | No of seeds pod <sup>-1</sup> |                 | Weight of seeds plant <sup>-1</sup> (g) |                 | Weight of 100 seeds (g) |                 | Weight of seeds yield fed <sup>-1</sup> (ton) |                 |       |
|--|--------------------------------|-----------------|-------------------------------|-----------------|---|-----------------|-------------------------|-----------------|---|-----------------|-------|
|  | 1 <sup>st</sup>                | 2 <sup>nd</sup> | 1 <sup>st</sup>               | 2 <sup>nd</sup> | 1 <sup>st</sup>                         | 2 <sup>nd</sup> | 1 <sup>st</sup>         | 2 <sup>nd</sup> | 1 <sup>st</sup>                               | 2 <sup>nd</sup> |       |
|  | season                         | season          | season                        | season          | season                                  | season          | season                  | season          | season  | season          |       |
| <b>Control</b>   | 20.80                          | 20.43           | 15.00                         | 14.50           | 2.90                                    | 2.78            | 1.06                    | 1.06            | 0.406   | 0.389           |       |
| <b>Na<sub>2</sub>SiO<sub>3</sub> (mg l<sup>-1</sup>)</b> | 100                            | 25.17           | 24.00                         | 15.75           | 17.15                                   | 3.09            | 2.99                    | 1.09            | 1.11  | 0.433           | 0.419 |
|  | 300                            | 27.75           | 24.00                         | 16.00           | 17.75                                   | 3.31            | 3.41                    | 1.17            | 1.19  | 0.463           | 0.477 |
|  | 500                            | 29.50           | 30.75                         | 17.00           | 19.00                                   | 3.09            | 3.15                    | 1.13            | 1.12  | 0.433           | 0.441 |
|  | 700                            | 31.25           | 30.66                         | 17.00           | 17.50                                   | 3.01            | 3.02                    | 1.13            | 1.14  | 0.421           | 0.423 |
|  | 1000                           | 33.50           | 33.50                         | 15.50           | 16.00                                   | 3.01            | 2.94                    | 1.07            | 1.09  | 0.421           | 0.412 |
| <b>SiO<sub>2</sub>/ AC (mg l<sup>-1</sup>)</b>           | 100                            | 34.50           | 32.50                         | 18.50           | 19.85                                   | 3.57            | 3.52                    | 1.17            | 1.13  | 0.500           | 0.493 |
|  | 300                            | 36.25           | 37.00                         | 16.50           | 20.45                                   | 3.89            | 3.81                    | 1.50            | 1.46  | 0.545           | 0.533 |
|  | 500                            | 39.50           | 41.45                         | 16.50           | 23.00                                   | 3.78            | 3.78                    | 1.23            | 1.30  | 0.529           | 0.529 |
|  | 700                            | 38.00           | 38.86                         | 20.00           | 18.60                                   | 3.60            | 3.65                    | 1.13            | 1.27  | 0.504           | 0.511 |
|  | 1000                           | 37.05           | 38.50                         | 15.50           | 17.00                                   | 3.58            | 3.57                    | 1.20            | 1.23  | 0.501           | 0.500 |
| <b>AC (mg l<sup>-1</sup>)</b>                            | 100                            | 37.50           | 39.75                         | 22.00           | 18.50                                   | 3.76            | 3.70                    | 1.17            | 1.17  | 0.526           | 0.518 |
|  | 300                            | 31.55           | 33.00                         | 16.25           | 17.00                                   | 3.57            | 3.57                    | 1.11            | 1.14  | 0.500           | 0.500 |
|  | 500                            | 28.50           | 29.18                         | 17.00           | 20.00                                   | 3.49            | 3.57                    | 1.07            | 1.12  | 0.489           | 0.500 |
|  | 700                            | 20.00           | 22.00                         | 23.50           | 19.50                                   | 3.06            | 3.20                    | 1.09            | 1.12  | 0.428           | 0.448 |
|  | 1000                           | 18.00           | 20.00                         | 18.25           | 17.88                                   | 2.80            | 2.96                    | 1.00            | 1.07  | 0.392           | 0.414 |
| <b>LSD at 5%</b>   | 2.38                           | 1.80            | 2.20                          | 2.44            | 0.24                                    | 0.19            | 0.059                   | 0.098           | 0.034   | 0.026           |       |

### 3.8. Photosynthetic pigments.

In our present study, the data registered in (Table7) cleared that there were non-significant effect between application of 300 and 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC for Chl a and total chlorophyll through both seasons. While, at 300 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC, Chl b increased by 44.4 and 37.56% when compared with the control, respectively through two seasons. Treated plants with 700 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC increased carotenoid content by (24.29 and 27.85%) over control during 1<sup>st</sup> and 2<sup>nd</sup> seasons, respectively. Salinity induces the activity of some enzymes that degrade chlorophylls (Banakar *et al.*, 2022). Different foliar application from Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC reduced the side effects of salinity stress on photosynthetic pigments through reduction EL% and rising MSI % as displayed before. Furthermore, the decreasing the ROS and elevating the antioxidant systems stabilize the chloroplast membrane and protect the chloroplasts from salt stress and stopped degradation of leaf chlorophyll (Sevengor *et al.*, 2011 & Siddiqui and Al-Wahaibi, 2014). Our results were matched with El- Emary and Amer (2018) on faba bean and maize plants.

### 3.9. Nutrient elements.

Data in Table (8) cleared that foliar application with 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC significantly increased the N by (21.26, 19.35%), P by (37.27, 42.39%), K by (9.71, 9.22%) and Ca content by (28.49, 20.57%) in fenugreek seeds respectively, comparing with control plants. On other hand, Na content decreased with the same treatment by (38.75, 34.15%) through two seasons respectively. It was noticed that there was no significant effect for P content among 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC and 100 mg l<sup>-1</sup> AC through 1<sup>st</sup> season. N, P, K and Ca are very important elements for membrane structure, cell division, stomatal function, and plant growth cell wall synthesis, involved in plant defence and repair the damage from biotic and abiotic stress (Hu and Schmidhalter, 2005). Also, high potassium content improves water potential and sustainability of plant finally ameliorates the Na<sup>+</sup> ion toxicity. Hence, it is concluded that foliar application of 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC increases potassium uptake and alleviates Na<sup>+</sup> toxicity. It may be due to the effect of nano silica on the uptake of the minerals and the reduction in the absorption of Na. These

results were on line with reported by (Abdul Qados, 2015) on faba bean, Sadak and Ahmed (2016) on wheat plant and Banakar *et al.* (2022) on fenugreek.

**Table 7:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on photosynthetic pigments of fenugreek plants grown under salinity conditions during 2020/2021 and 2021/2022 seasons.

| Treatments  | Chl a<br>(mg g <sup>-1</sup> f. wt) |                 | Chl b<br>(mg g <sup>-1</sup> f. wt) |                 | Total chlorophyll<br>(mg g <sup>-1</sup> f. wt) |                 | Carotenoid<br>(mg g <sup>-1</sup> f. wt) |                 |       |
|---|-------------------------------------|-----------------|-------------------------------------|-----------------|---|-----------------|--|-----------------|-------|
|   | 1 <sup>st</sup>                     | 2 <sup>nd</sup> | 1 <sup>st</sup>                     | 2 <sup>nd</sup> | 1 <sup>st</sup>                                 | 2 <sup>nd</sup> | 1 <sup>st</sup>                          | 2 <sup>nd</sup> |       |
|   | season                              | season          | season                              | season          | season  | season          | season                                   | season          |       |
| Control   | 0.958                               | 0.923           | 0.389                               | 0.426           | 1.347   | 1.349           | 1.066                                    | 1.034           |       |
| Na <sub>2</sub> SiO <sub>3</sub><br>(mg l <sup>-1</sup> ) | 100                                 | 1.107           | 1.321                               | 0.540           | 0.532   | 1.647           | 1.853                                    | 1.217           | 1.210 |
|   | 300                                 | 1.102           | 1.333                               | 0.536           | 0.551   | 1.638           | 1.884                                    | 1.268           | 1.243 |
|   | 500                                 | 1.180           | 1.352                               | 0.530           | 0.543   | 1.71            | 1.895                                    | 1.231           | 1.228 |
|   | 700                                 | 1.195           | 1.386                               | 0.520           | 0.519   | 1.715           | 1.905                                    | 1.235           | 1.237 |
|   | 1000                                | 1.212           | 1.402                               | 0.499           | 0.505   | 1.711           | 1.907                                    | 1.241           | 1.249 |
| SiO <sub>2</sub> / AC<br>(mg l <sup>-1</sup> )            | 100                                 | 1.265           | 1.385                               | 0.541           | 0.558   | 1.806           | 1.943                                    | 1.283           | 1.274 |
|   | 300                                 | 1.300           | 1.432                               | 0.562           | 0.586   | 1.862           | 2.018                                    | 1.291           | 1.288 |
|   | 500                                 | 1.308           | 1.428                               | 0.551           | 0.574   | 1.859           | 2.002                                    | 1.296           | 1.303 |
|   | 700                                 | 1.264           | 1.390                               | 0.503           | 0.532   | 1.767           | 1.922                                    | 1.325           | 1.322 |
|   | 1000                                | 1.229           | 1.336                               | 0.471           | 0.499   | 1.700           | 1.835                                    | 1.252           | 1.273 |
| AC<br>(mg l <sup>-1</sup> )                               | 100                                 | 1.121           | 1.227                               | 0.481           | 0.512   | 1.602           | 1.739                                    | 1.271           | 1.278 |
|   | 300                                 | 1.133           | 1.235                               | 0.494           | 0.507   | 1.627           | 1.742                                    | 1.274           | 1.282 |
|   | 500                                 | 1.157           | 1.210                               | 0.514           | 0.536   | 1.671           | 1.746                                    | 1.294           | 1.299 |
|   | 700                                 | 1.190           | 1.249                               | 0.551           | 0.549   | 1.741           | 1.798                                    | 1.311           | 1.311 |
|   | 1000                                | 0.973           | 0.953                               | 0.431           | 0.455   | 1.404           | 1.408                                    | 1.225           | 1.242 |
| LSD at 5%   | 0.0259                              | 0.0286          | 0.0306                              | 0.029           | 0.048   | 0.0365          | 0.0215                                   | 0.0262          |       |

**Table 8:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on nutrient contents of fenugreek plants grown under salinity conditions during 2020/2021 and 2021/2022 seasons.

| Treatments  | N<br>(g. 100g <sup>-1</sup> ) |                 | P<br>(g. 100g <sup>-1</sup> ) |                 | K<br>(mg. 100g <sup>-1</sup> ) |                 | Na<br>(mg. 100g <sup>-1</sup> ) |                 | Ca<br>(mg. 100g <sup>-1</sup> ) |                 |     |
|---|-------------------------------|-----------------|-------------------------------|-----------------|--------------------------------|-----------------|---------------------------------|-----------------|---------------------------------|-----------------|-----|
|   | 1 <sup>st</sup>               | 2 <sup>nd</sup> | 1 <sup>st</sup>               | 2 <sup>nd</sup> | 1 <sup>st</sup>                | 2 <sup>nd</sup> | 1 <sup>st</sup>                 | 2 <sup>nd</sup> | 1 <sup>st</sup>                 | 2 <sup>nd</sup> |     |
|   | season                        | season          | season                        | season          | season                         | season          | season                          | season          | season                          | season          |     |
| Control   | 3.34                          | 3.41            | 0.220                         | 0.217           | 752                            | 748             | 111                             | 110             | 172                             | 175             |     |
| Na <sub>2</sub> SiO <sub>3</sub><br>(mg l <sup>-1</sup> ) | 100                           | 3.38            | 3.53                          | 0.214           | 0.219                          | 744             | 753                             | 104             | 108                             | 174             | 172 |
|   | 300                           | 3.50            | 3.58                          | 0.237           | 0.242                          | 755             | 765                             | 100             | 102                             | 186             | 181 |
|   | 500                           | 3.72            | 3.80                          | 0.270           | 0.264                          | 785             | 773                             | 95              | 94                              | 201             | 196 |
|   | 700                           | 3.74            | 3.81                          | 0.277           | 0.270                          | 787             | 775                             | 95              | 93                              | 204             | 198 |
|   | 1000                          | 3.90            | 3.90                          | 0.282           | 0.288                          | 793             | 779                             | 93              | 93                              | 210             | 200 |
| SiO <sub>2</sub> /AC<br>(mg l <sup>-1</sup> )             | 100                           | 3.66            | 3.74                          | 0.276           | 0.285                          | 786             | 776                             | 94              | 90                              | 199             | 197 |
|   | 300                           | 3.97            | 3.96                          | 0.293           | 0.300                          | 813             | 803                             | 83              | 85                              | 216             | 206 |
|   | 500                           | 4.05            | 4.07                          | 0.302           | 0.309                          | 825             | 817                             | 80              | 82                              | 221             | 211 |
|   | 700                           | 3.88            | 3.90                          | 0.289           | 0.289                          | 791             | 786                             | 91              | 89                              | 212             | 199 |
|   | 1000                          | 3.76            | 3.83                          | 0.280           | 0.278                          | 780             | 780                             | 96              | 92                              | 191             | 196 |
| AC<br>(mg l <sup>-1</sup> )                               | 100                           | 3.93            | 3.98                          | 0.300           | 0.293                          | 810             | 800                             | 82              | 83                              | 209             | 209 |
|   | 300                           | 3.86            | 3.75                          | 0.287           | 0.282                          | 790             | 780                             | 88              | 87                              | 198             | 193 |
|   | 500                           | 3.75            | 3.72                          | 0.270           | 0.267                          | 781             | 768                             | 93              | 90                              | 189             | 189 |
|   | 700                           | 3.63            | 3.57                          | 0.254           | 0.257                          | 769             | 761                             | 93              | 93                              | 187             | 178 |
|   | 1000                          | 3.57            | 3.52                          | 0.231           | 0.242                          | 760             | 757                             | 91              | 94                              | 179             | 177 |
| LSD at 5%   | 0.084                         | 0.078           | 0.010                         | 0.009           | 6.48                           | 4.90            | 2.70                            | 2.00            | 2.18                            | 1.86            |     |

### 3.10. Chemical composition.

Different foliar treatments decreased proline accumulation particularly when plants sprayed with SiO<sub>2</sub>/ AC. There were non-significant difference between 100, 300 and 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC for proline through two seasons. Proline content through both seasons decreased by (46.02 and 38.78%) compared with control when 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC treated with. Furthermore, treated with 500 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC gave significant value of amino acids and protein through two seasons where the increasing by (73.03, 79.13% and 21.21, 19.38%) over the control, respectively. Concerning with total carbohydrate there were no significant differences between 1000 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC and 100 mg l<sup>-1</sup> AC through both seasons (Table 9).

Proline plays an important role in plant osmotic regulation, where it accumulates under stress conditions (Nayyar and Walia, 2003). The accumulation of proline and amino acids permits plants to keep cell turgor pressure for cell expansion under salinity stress conditions; act as osmo-protectants (Ruiz-Carrasco *et al.*, 2011). Proline has the ability to scavenge ROS and other free radical compounds, preventing protein denaturation during stress (Szabados and Saviouré, 2010). Proline content increasing consistent with the suggestion that nitrogen forwarded to the synthesis of proline instead of chlorophyll. Improvement of protein and carbohydrate content might be due to the role of Si-NPs in the activation of various enzymes associated with protein and amino acids biosynthesis beside carbohydrate metabolism (Ma and Yamaji, 2008; Wang *et al.*, 2013 and Mahmoud *et al.*, 2020). Beside the role of activated carbon that randomly distributed pores on the surface as observed from (Figure 5a) The results of our study are in line with those obtained by Rahimi *et al.* (2012) on fennel; Kalteh *et al.* (2014) on basil and Ayman *et al.* (2020) on wheat.

**Table 9:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on chemical compositions of fenugreek plants grown under salinity conditions during 2020/2021 and 2021/2022 seasons.

| Treatments       | Proline content<br>( $\mu\text{mol g}^{-1}$ ). |                 | Amino acid<br>(%) |                 | Protein content<br>( $\text{g. } 100\text{g}^{-1}$ ) |                 | Total carbohydrates<br>(%) |                 |
|------------------|--|-----------------|-------------------|-----------------|--|-----------------|----------------------------|-----------------|
|                  | 1 <sup>st</sup>                                | 2 <sup>nd</sup> | 1 <sup>st</sup>   | 2 <sup>nd</sup> | 1 <sup>st</sup>                                      | 2 <sup>nd</sup> | 1 <sup>st</sup>            | 2 <sup>nd</sup> |
|                  | season   | season          | season            | season          | season   | season          | season                     | season          |
| <b>Control</b>   | 2.89   | 2.63            | 0.152             | 0.139           | 20.88  | 21.31           | 54.72                      | 55.32           |
| <b>100</b>       | 2.33   | 2.35            | 0.154             | 0.144           | 21.13  | 22.06           | 55.85                      | 57.50           |
| <b>300</b>       | 2.26   | 2.29            | 0.157             | 0.148           | 21.88  | 22.38           | 57.35                      | 58.00           |
| <b>500</b>       | 2.15   | 2.24            | 0.159             | 0.153           | 23.25  | 23.75           | 59.22                      | 60.07           |
| <b>700</b>       | 2.13   | 2.18            | 0.159             | 0.157           | 23.38  | 23.81           | 59.47                      | 60.39           |
| <b>1000</b>      | 2.07   | 2.11            | 0.161             | 0.166           | 24.38  | 24.38           | 62.50                      | 63.50           |
| <b>100</b>       | 1.64   | 1.77            | 0.224             | 0.203           | 22.88  | 23.38           | 62.37                      | 64.00           |
| <b>300</b>       | 1.58   | 1.68            | 0.237             | 0.218           | 24.79  | 24.75           | 62.37                      | 64.18           |
| <b>500</b>       | 1.56   | 1.61            | 0.263             | 0.249           | 25.31  | 25.44           | 61.04                      | 65.43           |
| <b>700</b>       | 1.94   | 1.95            | 0.191             | 0.200           | 24.25  | 24.38           | 61.65                      | 66.32           |
| <b>1000</b>      | 2.11   | 2.14            | 0.152             | 0.194           | 23.50  | 23.94           | 67.80                      | 67.00           |
| <b>100</b>       | 2.23   | 2.18            | 0.147             | 0.140           | 24.56  | 24.88           | 67.77                      | 66.67           |
| <b>300</b>       | 2.17   | 2.03            | 0.153             | 0.151           | 24.13  | 23.44           | 63.70                      | 61.50           |
| <b>500</b>       | 2.54   | 2.21            | 0.151             | 0.149           | 23.44  | 23.25           | 56.79                      | 57.37           |
| <b>700</b>       | 2.78   | 2.29            | 0.149             | 0.140           | 22.69  | 22.31           | 54.22                      | 53.20           |
| <b>1000</b>      | 2.88   | 2.50            | 0.138             | 0.130           | 22.31  | 22.00           | 52.55                      | 51.08           |
| <b>LSD at 5%</b> | 0.18   | 0.17            | 0.015             | 0.011           | 0.53   | 0.48            | 2.10                       | 2.02            |

### 3.11. Fixed oil content.

Foliar spraying with Na<sub>2</sub>SiO<sub>3</sub> and SiO<sub>2</sub>/AC improve the fixed oil percentage. It was noted that, no significant effect among 1000 mg l<sup>-1</sup> Na<sub>2</sub>SiO<sub>3</sub> and 300 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC. Fixed oil percentage was increased At 300 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC by (100.4 and 97.87%) over the control, respectively during the two seasons (Table 10). Oil yield influenced by salinity (Farouk and Omar, 2020). Si might be act as an important factor in elicitor accelerated secondary metabolite production that might improve various transcriptional modifications. Si may be enhance oil yield by enhancing cell development and ion uptake (Esmaeilzadeh and Rezaei, 2018). This result is matched with previous studies that showed using of Si enhanced oil accumulation and possibly served as an effective stimulant to increase the production of secondary metabolites (Farouk and Omar, 2020). Our results agree with El-Leithy *et al.* (2019) on black cumin and Farouk *et al.* (2020) on sweet basil.

### 3.12-Fixed oil constituents.

The analysis of fenugreek seeds oil illustrates that, the oil composed of 12 well known fatty acids (Table 11). The results declared that foliar spray of 300 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC resulted in decrement in palmitic acid, heptadecanoic acid, stearic acid, vaccinic acid and gondoic acid by 5.34, 24.4, 2.1, 20 and 16.67%, respectively compared to the control. Myristic acid disappeared in SiO<sub>2</sub>/ AC treated plants. On the other hand, SiO<sub>2</sub>/ AC treatment increased arachidic and behenic saturated fatty acids. Moreover, foliar spray with 300 mg l<sup>-1</sup> SiO<sub>2</sub>/ AC increased the palmitoleic and linoleic omega-6 unsaturated fatty acids by 88.8 and 5.91%, receptively compared to untreated plants but declined oleic and linolenic unsaturated fatty acids by 0.45 and 2.84%, respectively compared with untreated plants. So, according to the present

results, 300 mg l<sup>-1</sup> SiO<sub>2</sub>/AC improve the oil quality of the studied fenugreek plants, that it reduces the concentration of saturated fatty acids by 4.64% and increasing the unsaturated fatty acids by 1.12% compared to control. The oil was rich with unsaturated fatty acids, chiefly linoleic, linolenic and oleic fatty acids acids, which have therapeutic importance. The major compounds fenugreek oil is linoleic acid omega-6 (Akbari *et al.*, 2019). The occurrence of saturated acids (18.1 and 17.26%) in fenugreek seeds oil in control and SiO<sub>2</sub>/ AC at (300 mg l<sup>-1</sup>), respectively represent a chief quantitative variation against marketable oils. Our results agree with Sulieman *et al.* (2008) on fenugreek seed oil and Farouk *et al.* (2022) on borage seeds oil. Such positive role may be achieved by silicate that lead to increase unsaturation fatty acids (Wang and Galletta, 1998).

**Table 10:** Effects of Na<sub>2</sub>SiO<sub>3</sub>, SiO<sub>2</sub>/AC and AC on fixed oil content of fenugreek plants grown under salinity conditions during seasons of 2020/2021 and 2021/2022.

| Treatments   | Fixed oil percentage (%) |                        | Fixed oil content plant <sup>-1</sup> (g) |                        | Fixed oil content fed <sup>-1</sup> (Kg) |                        |       |
|--|--------------------------|------------------------|---|------------------------|--|------------------------|-------|
|  | 1 <sup>st</sup> season   | 2 <sup>nd</sup> season | 1 <sup>st</sup> season                    | 2 <sup>nd</sup> season | 1 <sup>st</sup> season                   | 2 <sup>nd</sup> season |       |
|  | <b>Control</b>           | 6.95                   | 7.03                                      | 0.20                   | 0.19                                     | 28.32                  | 27.20 |
| <b>Na<sub>2</sub>SiO<sub>3</sub> (mg l<sup>-1</sup>)</b> | 100                      | 10.50                  | 10.37                                     | 0.32                   | 0.31                                     | 45.39                  | 43.42 |
|  | 300                      | 11.07                  | 11.13                                     | 0.37                   | 0.38                                     | 51.23                  | 53.09 |
|  | 500                      | 11.51                  | 11.49                                     | 0.36                   | 0.36                                     | 49.72                  | 50.69 |
|  | 700                      | 12.32                  | 12.51                                     | 0.37                   | 0.38                                     | 51.81                  | 52.86 |
|  | 1000                     | 13.71                  | 13.80                                     | 0.41                   | 0.41                                     | 57.74                  | 56.88 |
| <b>SiO<sub>2</sub>/ AC (mg l<sup>-1</sup>)</b>           | 100                      | 13.26                  | 13.32                                     | 0.47                   | 0.47                                     | 66.31                  | 65.59 |
|  | 300                      | 13.93                  | 13.91                                     | 0.54                   | 0.53                                     | 75.92                  | 74.11 |
|  | 500                      | 10.98                  | 11.07                                     | 0.42                   | 0.42                                     | 57.99                  | 58.52 |
|  | 700                      | 9.5                    | 9.62                                      | 0.34                   | 0.35                                     | 47.88                  | 49.16 |
|  | 1000                     | 9.37                   | 9.41                                      | 0.34                   | 0.34                                     | 46.99                  | 46.98 |
| <b>AC (mg l<sup>-1</sup>)</b>                            | 100                      | 9.87                   | 9.93                                      | 0.37                   | 0.37                                     | 51.95                  | 51.36 |
|  | 300                      | 8.41                   | 8.62                                      | 0.30                   | 0.31                                     | 42.03                  | 42.99 |
|  | 500                      | 8.01                   | 8.00                                      | 0.28                   | 0.29                                     | 39.07                  | 39.96 |
|  | 700                      | 7.39                   | 7.47                                      | 0.23                   | 0.24                                     | 31.62                  | 33.55 |
|  | 1000                     | 6.35                   | 6.89                                      | 0.18                   | 0.20                                     | 24.83                  | 28.62 |
| <b>LSD at 5%</b>   | 1.09                     | 1.05                   | n.s                                       | n.s                    | 5.02                                     | 4.62                   |       |

**Table 11:** Fixed oil constituents of the selected treatments in the second season.

| Fatty acids                    | Relative distribution (%) |  |
|--------------------------------|---------------------------|--|
|                                | Control                   | SiO <sub>2</sub> / AC (300mg l <sup>-1</sup> ) |
| Myristic acid (C14:0)          | 0.19                      | --   |
| Palmitic acid (C16:0)          | 11.43                     | 10.82  |
| Heptadecanoic acid (C17:0)     | 0.41                      | 0.31   |
| Stearic acid (C18:0)           | 4.29                      | 4.20   |
| Arachidic acid (C20:0)         | 1.33                      | 1.41   |
| Behenic acid (C22:0)           | 0.45                      | 0.52   |
| Palmitoleic acid (C16:1)       | 0.27                      | 0.51   |
| Vaccinic acid (C18:1) omega-7  | 0.40                      | 0.32   |
| Oleic acid (C 18:1) omega-9    | 15.67                     | 15.60  |
| Linoleic acid (C18:2) omega-6  | 31.16                     | 33.0   |
| Linolenic acid (C18:3) omega-3 | 34.17                     | 33.20  |
| Gondoic acid (C20:1)           | 0.24                      | 0.20   |
| No. of fatty acids             | 12                        | 11   |
| Total saturated%               | 18.1                      | 17.26  |
| Total unsaturated%             | 81.91                     | 82.83  |



## Conclusion

Activated carbon were prepared from date palm tree leaflet using KOH as activating agent and composite SiO<sub>2</sub>/ AC. S<sub>BET</sub> ranged between 397.46 and 656.33 m<sup>2</sup>/g. It is obvious that the use of SiO<sub>2</sub>/ AC enhanced fenugreek crop productivity under salinity soil conditions. Thus, in this context, the usage of 500mg l<sup>-1</sup> SiO<sub>2</sub>/ AC could be more effective for growth parameters and chemical composition while 300mg l<sup>-1</sup> SiO<sub>2</sub>/ AC improved yield characters and fixed oil percentage. On other hand, the application of higher concentration from activated carbon had poor effects for different aspects compared with other treatments. Also, the preparation of activated carbon from leaflet of date palm tree and its application in agriculture field consider a new way to get rid of leaflet of palm tree as waste causing problem in environment.

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