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PHYSICOCHEMICAL CHARACTERIZATION OF ZEOLITE AND NUTRACEUTICAL QUALITY OF *Cucurbita pepo* AMENDED WITH THIS ALUMINOSILICATE AND ROWS COVERED WITH PLASTIC MULCH

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Abstract: The physical and chemical characteristics of the substrates employed in the present study with zuchinni plants were improved by the addition of zeolite to the soil at 20 and 40 T ha^{-1}. Zeolite increased total porosity and bulk density of the substrate, and also promoted a greater water retention capacity compared to control treatment. Concerning to fruit quality, zeolite significantly increased fruit diameter and in a similar way, plastic mulching increased fruit diameter and total yield. The interaction zeolite-PM showed highly significant differences in fruit length, diameter, total soluble solids and firmness of zucchini fruits, also they were positively affected by the interaction of zeolite and plastic mulch. Therfore, we conclude that growth, yield and quality of *C. pepo* plants could be improved by amending the soil with natural zeolite and by row covering with plastic mulching.
Keywords: Cucurbits, zeolite clinoptilolite, water efficiency, natural fertilizers.

INTRODUCTION

Since the publication of "La roca magica: Uses of natural zeolites in agriculture and industry", Mumpton (1999) pointed out that natural zeolites like those found in volcanogenic sedimentary rocks have been and are being used as building stone, as lightweight aggregate in cements and concretes, as filler in paper, in the take-up of Cs and Sr from nuclear waste and fallout, as soil amendments in agronomy and horticulture, in the removal of ammonia from municipal, industrial, and agricultural waste and drinking waters, as dietary supplements in animal diets, as consumer deodorizers, and many more usages, Figure 1.

In fact, Hu et al. (2023) reported that zeolite recycled from the drainage ditch was able to reduce N concentration caused by straw decomposition in the surface water. Zeolite adsorption reduced the peak values of NH^{4+}-N, Total N, and Total P by 30, 19, and 5%, respectively. Based on these findings and conventional field designs, the use of 20 t ha^{-1} zeolite in the field was effective for recycling N and P. Therefore, this research provides a sustainable development method to mitigate the water quality deterioration caused by straw returning to the field.

According to Jarosz et al. (2022) the geometrical parameters of zeolites are one of the most important characteristics responsible for their adsorption capacity. As a result, zeolites can not only serve as a sorbent for pollutants in the environment, but also as a reservoir of water and nutrients for plants (anions and cations). Due to their unique properties, zeolites have become more and more popular in recent years and find practical application in many branches of the agronomy sector.

Use of nanocarriers in agro-practices for sustainable farming contributes to achieving up to 75% nutrient delivery for a prolonged period to maintain nutrient availability in soil for plants in adverse soil conditions. In this context Sharma et al. (2022) stated that sieve-like zeolites and the diversity in their structural morphologies, have attracted increasing interest over recent years. Engineered nano-porous zeolites, also called aluminosilicates, are defined based on the presence of micro- (<2 nm), meso- (2–50 nm), and macropores (>50 nm), which can be employed as carriers of fertilizers due to their enhanced ion-exchange properties and adsorption capabilities.

Méndez-Argüello et al. (2018) studied the effect of zeolite-clinoptilolite on *Solanum lycopersicum* seedlings growth. An assay was set to test and compare the physical properties of three substrates: peat moss (pm), perlite (per) and zeolite (zeo), and their mixtures (pm:per:zeo) at different proportions. Compared to control plants,

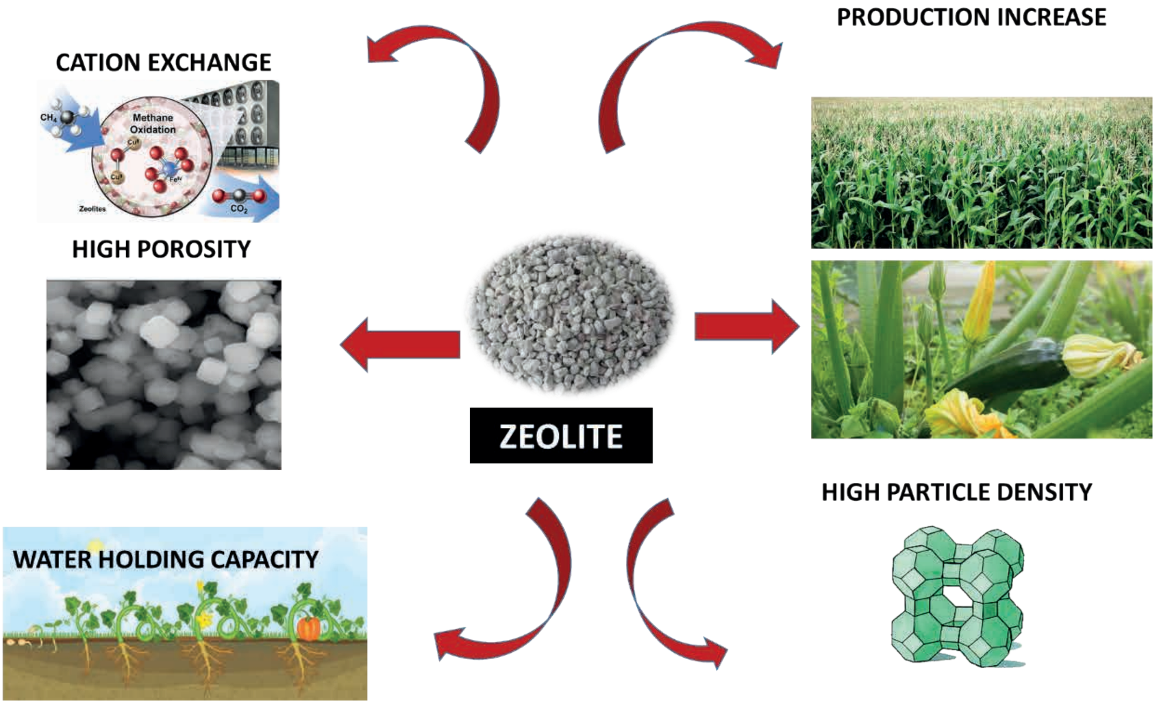

Figure 1. important properties of zeolite and benefits in agriculture

Physical-chemical characterization of zeolite									
Chemical composition (ppm kg^{-1})									
N	P	K	Ca	Mg	Fe	Cu	Zn	Mn	B
17.2	1.43	17.36	2.641	385	3.8	0.31	0.64	6.77	0.75
Texture %									
Sand	Silt	Clay							
52.2	34	13.8							
M.O %	pH								
0.4	8.75								

ppm: parts per million; Kg^{-1}: kilogram; M.O %: organic material %

Table 1. Chemical properties of zeolite clinoptilolite used in this experiment.

substrates containing 30% zeolite increased their water holding capacity (260%), total porosity (8.47%), bulk density (212%) and particle density (230%). The overall outcomes indicated that substrates amendment with zeolite could effectively improve tomato plants growth.

In a similar way Lira-Saldivar et al. (2017) incorporated 0, 10, 20 and 40 t ha^{-1} of zeolite clinoptilolite (ZC) to the soil established with *Cucurbita pepo* plants, with and without plastic mulch (PM). Zucchini plants showed higher yields when soil was amended with 40 t ha^{-1} of ZC than those growing on soil without ZC. Also there was a significant interaction between both factors (ZC and PM), with the highest yield when plants were cultured in bare soil plus 40 t ha^{-1} ZC.

Vitamin C concentration in fruits was reduced by 30.0, 53.6 and 38.8%, and chlorophyll index by 5.45, 7.20 and 5.84% with the incorporation of 10, 20 and 40 t ha^{-1} ZC, respectively. It was concluded that PM significantly improved biomass production (29.9%), leaf area (35.3%) and yield (22.3%). Based on the above described the objective of the present work was to perform the physicochemical characterization of the zeolite and its effect in conjunction with the PM in the nutraceuticals quality of zucchini fruits.

MATERIALS AND METHODS

The physicochemical characterization of the zeolite was carried out in the Soil Physics Laboratory of the Universidad Autónoma Chapingo, Estado of Mexico. Zeolite was used from a natural deposit of the state of San Luis Potosí México. The nutraceuticals quality of fruits was carried out in the Centro de Investigación en Química Aplicada (CIQA), in Saltillo, Coahuila México.

ZEOLITE PHYSICAL-CHEMISTRY CHARACTERIZATION

Physicochemical analysis: pH was measured with a potentiometer in a distilled water/ZC ratio 2:1; organic matter; N was extracted with potassium chloride 2 N and determined by Kjeldahl; phosphorous (P) was calculated by the Bray P1 method; potassium (K) was extracted by means of ammonium acetate 1.0 N and pH 7.0 set by spectrometry of flame emission; calcium (Ca) and magnesium (Mg) were extracted with ammonium acetate 1.0 N, pH 7.0 and determined by atomic absorption.

Physical characterization of substrate blends: An assay was set to test and compare the physical properties of three substrates: peat moss (pm), perlite (per) and zeolite (zeo), and mixtures (pm:per:zeo) at different proportions: T1 control = 100:0:0; T2 = 70:30:0; T3 = 70:20:10; T4 = 70:10:20 and T5 = 70:0:30 (v/v), to produce five treatments with three replicates, being each container a replicate. Total porosity, water holding capacity and bulk and particle densities were determined for all substrates mixtures by using the following equations (1-4) and using the methodology of Pire and Pereira 2003:

$$Total\ porosity\ (\%) = \frac{Dv + \frac{Sfw - Sdw}{Wsw}}{Cv}\ x\ 100 \quad (1)$$

$$Water\ holding\ capacity\ (\%) = \frac{Sfw - Sdw}{Cv}\ x\ 100 \quad (2)$$

$$Bulk\ density\ (Mg\ m^{-3}) = \frac{\mathrm{Sdw}}{\mathrm{Cv}} \quad (3)$$

$$\text{Particle density}\ (Mg\ m^{-3}) = \frac{Bd}{1 - \frac{Tp}{100}} \quad (4)$$

Where:
Dv = Drainage volume (cm^{-3})
Sfw = Sample fresh weight (g)
Sdw = Sample dry weight (g)
Wsw = Water specific weight (g cm^{-3})
Cv = Container volume (cm^{-3})
Tp = Total porosity (%)
Bd = Bulk density (Mg m^{-3})

Data collected were processed by analysis of variance (ANOVA), to statistically analyze the differences among group variable means.

Fruit quality: The evaluations of diameter, length and firmness of fruit were performed at 62, 69 and 74 days after sowing (DAS), for this purpose, 8 fruits per treatment with a uniform size were selected, the measurements were made with a rule of 30 cm (size) and with a Vernier (diameter). The firmness was measured with a texturometer model TA-XT Express enhanced of the Stable brand, at a speed of insertion and output of 10 mm/s and a force of 0.5 Newton with a penetration of 5 mm.

Nutraceuticals quality: Ascorbic acid concentration in fruit (vitamin C), was estimated by the method used by the AOAC 967.21. 20 grams of fruit were macerated with 10 ml of HCl (2%), maceration was gauge to 100 ml with distilled water and filtered. The filtrate was titrated an aliquot of 10 ml of reagent of Thielman until obtaining a pink coloration for 30 seconds, at this point the reading was taken in spent milliliters of the reagent.

The concentration of ascorbic acid was calculated by the following equation:

$$AC = \frac{Vg * 0.088 * VT * 100}{P * VA}$$

Where:

AC = Ascorbic acid in mg (100 gr sample)

Vg = Total volume of Thielman reagent (ml)

VT = Total volume of vitamin C filtering in HCl (ml)

VA = Volume of the assessed aliquot (ml)

P = Sample weight (gr)

0088 = milligrams of ascorbic acid equivalent to 1 ml of the Thielman reagent

100 = Total volume of distilled water gaug (ml)

Extraction of total chlorophylls (A and B) in leaves: Was determined by the technique reported by Abraham et al., (2010). 100 mg zucchini leaf is macerate with 2 ml of cold ethanol, 200 microliters of maceration were placed in a vial with 1.5 ml of cold ethanol, this solution is incubating one hour at 4 °C, then centrifuged at 12,000 rpm for 5 minutes. After centrifugation, 1 ml was extracted and placed in the spectrophotometer, where the diffraction of the light was measured by two wavelengths (663 and 645 nm).

Chlorophyll concentration was calculated using the following equations:

C_A mg/L = 12.7 (A663)-2.63 (A645)

C_B mg/L = 22.9 (A645)-4.68 (A663)

Where:

C_A = Chlorophyll concentration A

C_B= Chlorophyll concentration B

A663 = absorbance at 663 nm

A645 = absorbance at 645 nm

Determination of soluble solids (SST) in fruits: Was carried out by means of a refractometer model HI 96801 of Hanna Instruments Inc., (Woonsocket, Rhode Island, 02895, USA). Where approximately 100 µl of zucchini fruit extract solution was placed.

Statistical design: A completely randomized bivariate design with 4 levels for factor A (zeolite) and 2 levels for factor B (plastic mulch) is employed, having 8 treatments in interaction, (NZC + NPM (Control), ZC 10 + NPM, ZC 20 + NPM, ZN 40 + NPM, ZC 0 + PM, Zc 10 + PM, Zc 20 + PM, ZC 40 + PM), with 3 repetitions each. With the data obtained an analysis of variance (ANOVA) and multiple range tests were performed according to Tukey with one ($P < 0.05$) with the statistical program InfoStat, version 2015. (InfoStat group, FCA, National University of Córdoba, Argentina).

RESULTS AND DISCUSSION

The chemical tests of the natural ZC employed in this study (Table 1) point out that this type of aluminosilicate is rich in potassium and calcium and that it also harbors other macro-and micronutrients for plants. Regarding the physical characterization of substrates (Figure 2), treatments containing 20 and 30% ZC, presented an increase ($p \leq 0.05$) of total porosity (Figure 2a), particularly T4 (16.6%) and T5 (8.47%) compared to the control treatment (T1).

All substrates with ZC added promoted greater water retention capacity (WRC), since T4 and T5 exhibited a superior volume of water retained (35.33% and 33.71%, respectively), followed by T3 (25.85%) (Figure 2b). The lesser WRC was attained by T1 (9.34%), without ZC added. Regarding to bulk density (Figure 2c), this variable was increased significantly ($p<0.05$) by T4 (0.21 Mg m^{-3}) and T5 (0.25 Mg m^{-3}), compared to T1 (0.08 Mg m^{-3}). The addition of ZC to the substrate also improved particle density (Figure 2d), since T4 and T5 reported 0.37 Mg m^{-3} and 0.43 Mg m^{-3} respectively, compared to T1 (0.13 Mg m^{-3}).

Fruit quality: Zeolite with a dosage of 20 and 40 T ha^{-1} significantly increases fruits diameter by 4.7 and 6.8% with respect to the control (Table 2). The plastic mulch significantly increases the fruit diameter at 69 DAS by 10.3% compared to soil without plastic mulch. The zeolite-PM interaction showed highly significant differences on length and fruit diameter with the treatment ZC 20+NPM.

Length increased by 6.8 and 10.3% and the fruit diameter increased 8.6 and 10.8% at 69 and 76 DAS, compared to control. The fruit firmness did not show significant differences in the evaluations carried out (Table 2). However, an increase in firmness was observed as the dose of zeolite increased. The PM affects the firmness of fruit compared to fruits obtained in bare soil.

Amer (2011) remarks that optimal irrigation (100% evapotranspiration) increases fruit length in zucchini plants. The increase of fruit size in the present study may have a similar origin because the application of zeolite provides a greater water retention (Gholamhoseini et al., 2018) and nutrients, that promotes a greater development of diameter and fruits length.

On the other hand, in crops such as Strawberry (Abdi et al., 2006) and Ghazvini et al. (2007), zeolite does not affect fruits diameter. The effect of plastic mulch in fruit quality is widely reported. Shiukhy et al. (2014) remarks that this is due to an increase in temperature in the plant rhizosphere as well as soil moisture retention which generates a thermal and water optimal environment that leads to higher quality fruits.

Nutraceuticals quality: During the first harvest (69 DAS) zeolite affected the production of total soluble solids in fruits up to 20.5% with the highest doses of zeolite (40 T ha^{-1}), however, at 76 DAS, this variable is increased by 19.5% (10 t ha^{-1}) in the treatments with low doses of zeolite. Plastic mulch increases total soluble solids. The interaction zeolite and plastic mulch (ZC 10 + NPM) increased total soluble solids up to 42.54% at 79 DAS.

The increase in fruit-soluble solids according to Heeb et al. (2005), may be due to the presence of N in the soil and plant, which is transformed into carbohydrates and organic acids. On the other hand, Sheta et al. (2003) and Ghazvini et al. (2007) attribute the increases in sugars due to the characteristic that zeolites present by absorbing and releasing nutrients in the soil, mainly nitrogen in the form of ammonium.

An increase in total soluble solids by the PM has already been studied by Wang et al. (1998) and Sharma et al. (2013), these authors mention that it is mainly due to the light reflected by the film of plastic mulch which increases the photosynthetic rate and the

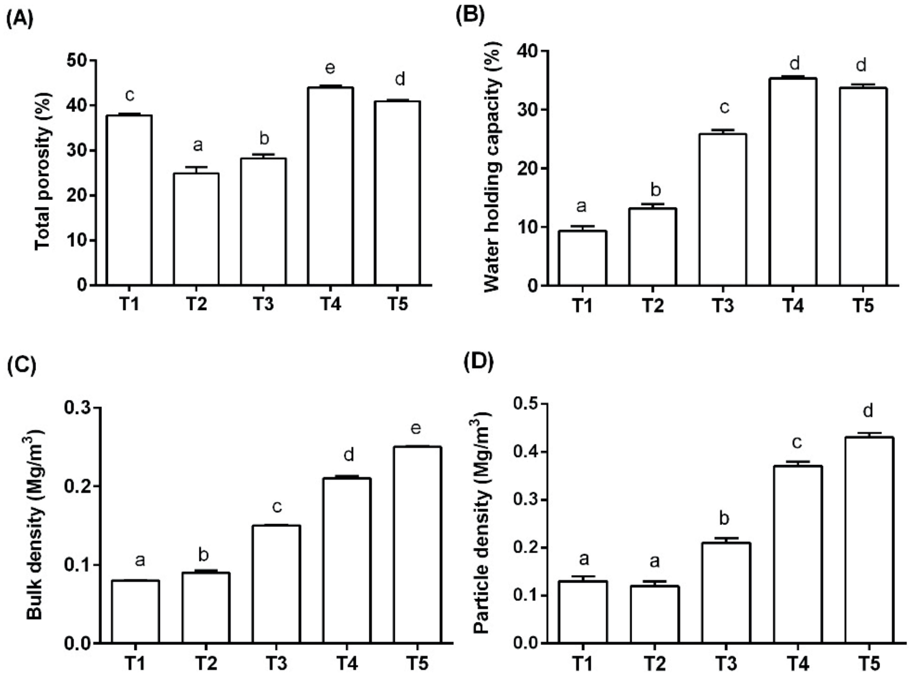

Figure 2. Physical properties of the substrate mixtures tested with *Cucurbita pepo* seedlings. Error bars represent the standard error of the mean (n = 3); columns within the same graph with different letter are statistically different according to Tukey test (α = 0.05).

Treatments	Length (cm)			Diameter (mm)			Firmness ($N\ cm^{-2}$)		
	62 DAS	69 DAS	76 DAS	62 DAS	69 DAS	76 DAS	62 DAS	69 DAS	76 DAS
Zeolite ($t\ ha^{-1}$)									
0	12.02 a	12.68 a	13.63 a	43.21 a	43.07 ab	45.71 bc	3.09 a*	2.89 a	2.92 a
10	12.20 a	12.35 a	13.43 a	41.55 a	41.27 b	45.17 c	3.18 a	3.04 a	3.01 a
20	11.84 a	13.19 a	14.27 a	40.47 a	45.10 a	48.59 ab	2.99 a	3.06 a	3.04 a
40	12.56 a	12.81 a	14.00 a	43.46 a	34.87 c	48.84 a	3.17 a	3.10 a	2.90 a
S.E	± 0.23	± 0.24	± 0.24	± 0.87	± 0.83	± 0.81	± 0.08	± 0.08	± 0.07
Plastic mulch									
PM	12.13 a	12.79 a	13.68 a	42.39 a	43.11 a	45.79 b	3.08 a	2.99 a	2.93 a
NPM	12.18 a	12.72 a	13.98 a	41.96 a	39.05 b	48.37 a	3.14 a	3.06 a	3.01 a
S.E	± 0.16	± 0.17	± 0.17	± 0.61	± 0.58	± 0.57	± 0.06	± 0.06	± 0.05
Interaction									
ZC 0+NPM	12.91 a	12.59 ab	13.54 ab	44.29 a	43.11 ab	46.80 bc	3.06 a	2.92 a	3.02 a
ZC 10+NPM	12.29 ab	11.51 b	13.81 ab	43.24 ab	38.91 b	47.02 abc	3.25 a	3.15 a	3.04 a
ZC 20+NPM	12.51 ab	13.64 a	14.94 a	37.94 b	46.85 a	51.89 ab	3.07 a	3.02 a	3.00 a
ZN 40+NPM	12.36 ab	13.14 a	13.63 ab	42.38 ab	27.33 c	47.76 bc	3.17 a	3.14 a	2.96 a
ZC 0+PM	11.13 b	12.76 ab	13.71 ab	42.14 ab	43.04 ab	44.62 c	3.12 a	2.86 a	2.82 a
ZC 10+PM	12.11 ab	13.19 a	13.05 b	39.85 ab	43.62 ab	43.31 c	3.11 a	2.94 a	2.98 a
ZC 20+PM	11.18 a	12.75 ab	13.60 ab	43.01 ab	43.35 ab	45.29 bc	2.91 a	3.09 a	3.08 a
ZC 40+PM	12.75 a	12.48 ab	14.38 ab	44.55 a	42.42 ab	49.93 ab	3.17 a	3.06 a	2.84 a
S.E	± 0.32	± 0.34	± 0.34	± 1.22	± 1.17	± 1.14	± 0.12	± 0.11	± 0.10

DAS: days after sowing; ZC: zeolite; PM: plastic mulch; NPM: no plastic mulch; S.E: standard error. Means (n = 3) ± standard errors of the means with common letters in columns, within each section, are statistically similar according to the Tukey test (P > 0.05).

Table 2: Length, diameter and firmness of zucchini fruits cultivated in a soil with different levels of zeolite applied and with and without plastic mulch.

carbohydrates content of fruits.

In the present study zeolite does not have a significant effect on total chlorophylls (Table 3). The PM treatment increased total chlorophylls at 69 DAS by 12.2%. The zeolite-PM interaction, significantly increased total chlorophylls by 26% in the treatment with ZC 10+PM and 15.6% in treatment with ZC 20+NPM at 76 DAS. Various authors such as Issa et al. (2001), mention that zeolite did not have a significant effect on total chlorophylls in gerbera plants, which is consistent with our results with zucchini plants.

Abdi et al. (2006) studying the effect of zeolite added to strawberry plants, founded that there is a direct relationship in the increment of total chlorophylls with the doses of zeolite applied. Likewise, Krutilina et al. (2000) mention that chlorophylls are increased in treatments with zeolite compared to control plants due to the high cation exchange capacity of zeolite and its ability of slow release cations.

The production of ascorbic acid was significantly reduced in those treatments where zeolite was added up to 20 t ha^{-1}. The PM reported a 24% reduction in the production of ascorbic acid compared to treatments without plastic mulch. The PM and zeolite reduces the production of vitamin C proportionally with high doses of zeolite, up to 40 t ha^{-1}. In the present work we got low concentrations of ascorbic acid, which could be owing to such factors as salinity, radiation, temperature, fruit maturity, mechanical damage, because these factors according to Lee and Kader (2000) and Rouphael et al. (2006), affect the synthesis of vitamin C.

No statistical differences were found regarding to yield of zucchini fruits, however, we found a higher yield related to the dose of zeolite applied. The plastic mulch increased the yield by 23.7% compared to treatments with bare soil. The interaction zeolite plus PM increased yield proportionately with the higher doses of zeolite applied to the soil.

Zeolite added to the soil increases the absorption of nitrogen, high levels of this mineral in the soil increases yield (Zheng et al., 2018), which is consistent with what was observed in this work. Greater performance by effect of the PM, could be attributed to modifications of soil microclimate, to the energy balance at soil level, to the control of weeds, less loss of humidity, good structure of the soil by effect of mulching, improvement of soil fertility and to the reflection of solar radiation by the PM.

CONCLUSIONS

Treatments containing 20 and 30% zeolite increased total porosity and bulk density of the substrate, also promoted a greater water retention capacity compared to the control treatment. Regarding to fruit quality, zeolite applied at 20 and 40 T ha^{-1} significantly increased fruit diameter. In a similar way, plastic mulch increased fruit diameter and total yield. The interaction zeolite-PM showed highly significant differences in length, fruit diameter, total soluble solids and firmness of zucchini fruits, also were positively affected by the interaction of zeolite and plastic mulch. Therfore, we conclude that growth, yield and quality of *C. pepo* plants can be improved by amending the soil with natural zeolite and by using plastic mulch.

Treatments	Total chlorophylls (mg/L)		TSS		VC (mg 20 g^{-1} muestra)			Fruit yield (kg planta^{-1})
	69 DAS	76 DAS	69 DAS	76 DAS	62 DAS	69 DAS	76 DAS	---------
Zeolite (t ha^{-1})								
0	17.42 a	18.22 a	6.75 a*	5.63 b	12.73 a*	5.69 ab	3.06 a	3.48a
10	16.63 a	20.51 a	5.65 bc	6.73 a	7.21 b	6.92 a	2.13 ab	3.92a
20	16.28 a	20.27 a	6.25 ab	5.65 b	7.44 b	4.54 b	1.42 b	3.59a
40	18.38 a	17.63 a	5.37 c	5.72 b	8.59 b	5.04 ab	1.87 b	4.08a
S.E	± 0.6	± 0.79	± 0.18	± 0. 16	± 0.79	± 0.49	± 0.23	± 0.39
Plastic mulch								
PM	18.17 a	19.03 a	5.83 a	5.79 a	8.48 a	5.30 a	1.83 b	4.17a
NPM	16.19 b	19.28 a	6.18 a	6.08 a	9.50 a	5.79 a	2.41 a	3.37a
S.E	± 0.43	± 0.56	± 0.13	± 0.11	± 0.56	± 0.35	± 0.16	± 0.27
Interaction								
ZC 0+NPM	16.93 a	18.65 ab	6.97 a	5.03 d	11.54 ab	7.84 a	4.00 a	3.05a
ZC 10+NPM	15.66 a	17.53 bc	5.80 a	7.17 a	6.05 b	7.24 ab	1.91 b	3.61a
ZC 20+NPM	15.53 a	21.56 abc	6.47 a	5.80 bcd	8.93 ab	3.29 c	1.32 b	3.00a
ZN 40+NPM	16.65 a	19.39 abc	5.47 a	6.30 ab	11.49 ab	4.79 abc	2.41 ab	3.83a
ZC 0+PM	17.92 a	17.78 b	6.53 a	6.23 abc	13.92 a	3.53 bc	2.12 b	3.91a
ZC 10+PM	17.61 a	23.5 a	5.50 a	6.30 ab	8.38 ab	6.59 abc	2.35 ab	4.24a
ZC 20+PM	17.02 a	18.98 abc	6.03 a	5.50 bcd	5.94 b	5.80 abc	1.52 b	4.18a
ZC 40+PM	20.11 a	15.87 c	5.27 a	5.13 cd	5.69 b	5.29 abc	1.33 b	4.34a
S.E	± 0.86	± 1.12	± 0.26	± 0.22	± 1.12	± 0.70	± 0.33	± 0.55

DAS: days after the transplant; ZC: zeolite; PM: plastic mulch; NPM: no plastic mulch; S.E: standard error. Means (n = 3) ± standard errors of the means with common letters in columns, within each section, are statistically similar according to the Tukey test (P > 0.05).

Table 3: Total chlorophylls, soluble solids (TSS), vitamin C (VC) and yield fruit of zucchini cultivated in soil with different levels of zeolite and plastic mulch.

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