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Effects of adding synthetic Zeolite at the expense of Mineral Fertilizer on the Sandy Soil parameters and Nutrition conditions: A case study on Papaya Plant

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ABSTRACT

The present study is testing the feasibility of using environmental friendly-blended mixture containing a combined fertilizer (Z/F) made up of synthetic zeolite (Z), derived from local natural resource, and NPK (19 N: 19 P_2O_5: 19 K_2O) mineral fertilizer (F), aiming to help in fertilizer reduction, and improving the plant nutrition and sandy soil quality. Different Z/F rates of 1.0 to 8.0 (g/ pot/plant) were respectively added to 1.0 - 5.0 (g/ pot/plant) amounts of NPK and used to grow papaya seedlings in the Pomology greenhouse, under Egyptian conditions during summer season. The XRD, SEM-Eds, and XRF were used to identify the mineral and chemical composition of the rock and zeolite, whereas, the starting (SS) and ending soils (ES) chemical and physical parameters and nutrient concentrations were measured by spectrophotometer, atomic absorption among other tools. Obtained results indicated that, applying (Z/ F) fertilizer mixture resulted in soil improvement with enhanced plant nutrition, where the optimum results obtained on increasing the zeolite contents at the expense of NPK amounts to certain limits. The highest performance was recorded for samples containing 2-3 Z: 1 F, meanwhile the extremes of having all mixture made completely of either zeolite or fertilizer implied the lowest improvement values of all treatments. Additionally, applying (Z/ F) seemed to directly influence the soil quality by upgrading its fine fraction, elevating water retention and organic matter content. Whereas, improved nutrient concentration were the main benefits of papaya on using lower rates of fertilizer (1.0-2.0 g/ pot/ plant). The indirect impact of using (Z/ F) fertilizer on the sandy soils is the substitution of the expensive mineral fertilizer with more appropriate, cost-effective, and environmentally safe alternative with retail of adding new reclaimed areas.

Keywords: Soil amendment, Combined fertilizer, Zeolite + NPK, Papaya, Nutrition

1. Introduction

The preservation of local resources as rocks, minerals and soils, needs much more attention to prevent their depletion or diminish due to inappropriate consumption. Clays and aluminosilicate zeolites are two groups of minerals that have relatively similar composition (alumina, silica and cations) but different building construction; where clays have layered structure, zeolites have naturally designed micro porous one. Both materials have important industrial applications, besides their role played as media for agriculture. Zeolite ores are not mined in Egypt, thus its preparation from available local source as the clays is of economic value. Zeolites have unique nature and performance, as being the world's only negatively charged minerals in existence. They implied breakthrough possibilities especially in agriculture among many other applications containing water softening and purification, catalysis, detergency, oil refining, absorbency etc… (Breck, 1974; Barrer, 1982; Szostak, 1998 and Cie´sla *et al*., 2019).

Natural zeolite family has Alumino-silicate minerals with valuable agriculture effects containing; soil fertility enhancement, temperature restore and water retention (Kazemian, 2005). Different types

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(more than 40) are present in nature in very complex three-dimensional porous frameworks, "open structures", with a high specific surface area and high cation exchange capacity in their internal cavities (Anderson, 2001and Szerementa, *et al*., 2021). Therefore, it can accommodate different cations as calcium, magnesium and sodium to be exchanged with other cations or anions like ammonia and/or potassium (Barros *et al*., 2003). Aluminosilicate zeolites are good absorbents, owing to their surface possession of elevated electrical charges, along with internal charged cavities that make them behave as "molecular sieves" (Sersale, 1985 and Ramôa, 1993). For the previous reasons, they have been used in agricultural applications (Boettinger and Ming, 2002). Satisfactory results were reported on applying different zeolites as soil amendment (Lai *et al*., 2000 and Al-Busaidi *et al*., 2008) and as component of growing media in containerized seed propagation and seedlings growth (Ayan and Tillki, 2007). In addition, it promoted plant growth by improving the value of fertilizers; retaining valuable nitrogen, enhancing quality of resulting manures, enriching plant yield and quality (Ayan *et al*., 2006 and Karami *et al*., 2020).

Papaya (*Carica papaya* L.) is a tropical fruit with commercial importance due to the high nutritive and medicinal values obtained from all parts of the plant (Karuna S. Verma and Kaushal, 2014). It has fast growing capability especially at the first six months of its plantation, thus requiring plentiful nutrition with extensive nitrogen, phosphors, and potassium fertilization.In addition, their roots are superficial and not extended to great depth in soil so must be sufficiently and continuously irrigated (Mohamed, 1995). Papaya is very responsive to the application of inorganic fertilizers along with organic manures and the balanced nutrition plays an important role in its growth, yield and fruit quality (Krishi, 2015).

Responding to the great needs for cultivating much more areas, for offering sustainable food source and supporting the use of natural and non-toxic materials in agriculture, the current study was aimed. In the present work, four-fold benefit were targeted; the first is to elevate the value of a natural resource (Kaolin rock) by converting it into zeolite-A (Z) which is the worldwide product of valuable applications. The second is to introduce "zeolite-based fertilizer" model, with different rates of zeolite to NPK commercial fertilizer, as a new candidate in the agriculture field. The third is to evaluate the performance of such Z/ F fertilizer admixture and measure the actual effects on the physico-chemical properties of the poor sandy soil, as a model for huge areas covering most of the desert areas. Finally, is to determine economic impact of the best-performed rates of Z/ F on both the fertilizer reduction and cost.

2. Experimental

2.1. Materials

2.1.1. Zeolite Synthesis

In the present work, "Kalabsha kaolin" from Aswan governorate, Egypt utilized as the starting material in zeolite preparation for economic reason. Kaolinite, the main mineral constituting kaolin rock, is a clay mineral with the chemical formula; Al_2O_3 $2SiO_2$ $2H_2O$. Kaolin is commonly used as a silica and alumina source for zeolite-A synthesis since both of them has nearly the same Si/Al ratio that near to unity (Rosha *et al.*, 1991; Dongeard *et al.*, 2000 and Szerementa, *et al*., 2021). The chemical analysis of the parent rock as given by X-Ray Fluorescence analysis included (in Wt%); 53.23 SiO_2, 41.58 Al_2O_3, 2.25 TiO_2, 1.83 Fe_2O_3 and other minor oxides of MgO, Na_2O, K_2O, MnO and CaO.

In this work, zeolite is prepared following the method of (Youssef *et al*., 2008). In a typical synthesis, natural clay rock was calcined at 750 ^oC for 4 hours. The thermally treated material was reacted with caustic soda with solid / liquid ratio of 10:1 to produce a dense gel containing monomer species of both silicate and aluminate. The final step was the hydrothermal treatment of the fresh gel at a temperature of 80^{o}C for 2 hours. The produced powder was collected and washed with distilled water several times to eliminate the excess alkali and dried overnight in an electric oven at 80^{o}C.

2.1.2. Soil Preparation

The composition of the starting soil (SS) contained mixture of sand and peat moss at 2:1 ratio, respectively; one seedling per pot is applied. Six treatments with five replicates (seedling/pot/replicate) were prepared, using NPK fertilizer compound (F) 19 N; 19 P_2O_5: 19 K_2O and zeolite-A powder (Z) in the following admixture rates given in Table 1. Each treatment is signed in the form of (Z/F/Pot/plant) as follows:

Table 1: The applied Z/ F admixture rates

Treatments	Zeolite/ Fertilizer Mixture (Z/ F) in grams	
	Zeolite-A	Fertilizer (NPK)
T_1 (Z_0F_5)	0.0	5.0
T_2 (Z_1F_4)	1.0	4.0
T_3 (Z_2F_3)	2.0	3.0
T_4 (Z_4F_2)	4.0	2.0
T_5 (Z_6F_1)	6.0	1.0
T_6 (Z_8F_0)	8.0	0.0

During the whole duration of the experiment, the monthly fertilizer's addition is stabilized, whereas foliar application with micronutrients compound (3.0% Fe, 3.0% Zn and 3.0% Mn) at 5 g L^{-1} was twice added (April and June) after the soil fertilization.

2.1.3. Plant Material

Papaya (*Carica papaya* L.) seedlings of Solo cultivar (2 months old) were used in this work with uniform seedlings of similar vigor, age and size to be transplanted (late of February). This research was conducted during 2014 season in pots (45 cm) and started from March $1^{st}$to the end of August.

2.2. Methods

2.2.1. Zeolite Characterization

In our study, the elemental chemical analysis of the starting kaolin is obtained by X-ray fluorescence technique using XRF instrument model AXIOS, WD-XRF sequential spectrometer (Analytical, 2005). Meanwhile, the determination of the mineralogical constituents of the Kalabsha kaolin and zeolite is investigated by X-ray diffraction method; using BRUKUR D8 ADVANCE with secondary monochromatic beam Cu Ka radiation at Kv = 40 and mA = 40. Microstructures of the synthesized materials are scanned using TEM, SEM model Philips XL30 attached with EDX unit, using an accelerating voltage of 30 kV, magnification 109 up to 400,0009 and resolution for W (3.5 nm), respectively.

2.2.2. Soil Analysis

Soil physico-chemical properties were determined at the beginning and end of all treatments. Soil samples were analyzed for their texture; pH and electric conductivity (E.C) using water extract (1: 2.5) method. Total calcium carbonate ($CaCO_3$ %) was detected by Calcimeter, organic matter (O.M %) by potassium dichromate method (Chapman and Pratt, 1978). Phosphorus was extracted using sodium bicarbonate (Olsen *et al.,* 1954); meanwhile potassium (K), calcium (Ca), magnesium (Mg) and sodium (Na) were measured using ammonium acetate (Jackson, 1973).

2.2.3. Nutrient concentrations

Nutrient elemental compositions in the leaves (the 4^{th} top leave of plant), stems and roots were analyzed for the macro- and micronutrient contents. Nitrogen (N) was detected by using the Kjeldahl method, Phosphorous (P) by spectrophotometer (Chapman and Pratt, 1978). Potassium (K) and micronutrients (Fe, Mn and Zn) were measured in the suspension using (Perkin-Elmer 100 B) Atomic Absorption Spectrophotometer (Lindsay and Norvell, 1978).

2.3. Statistical Analysis

The whole results were submitted to analysis of variance according to (Snedecor and Cochran, 1967). Differences among treatments means were determined by using the LSD test at a significance level of 0.05 (Waller and Duncan, 1969).

3. Results and Discussions

3.1. Zeolite Results

Figure1 shows the X-ray diffraction pattern of the mineral composition for the prepared zeolite. The strong, thin, and sharp peaks with high intensity of the XRD profile were matching well with zeolite-A (PDF #39-0222) with $Na_{96}Al_{96}Si_{96}O_{384}.216\ H_2O$ chemical formula. It can also be noted that, Anatase (TiO_2) and Quartz (SiO_2) co-crystallized with zeolite-A, the presence of which is common in zeolites developed from kaolinitic rocks, and not affecting either crystallization process or the product performance.

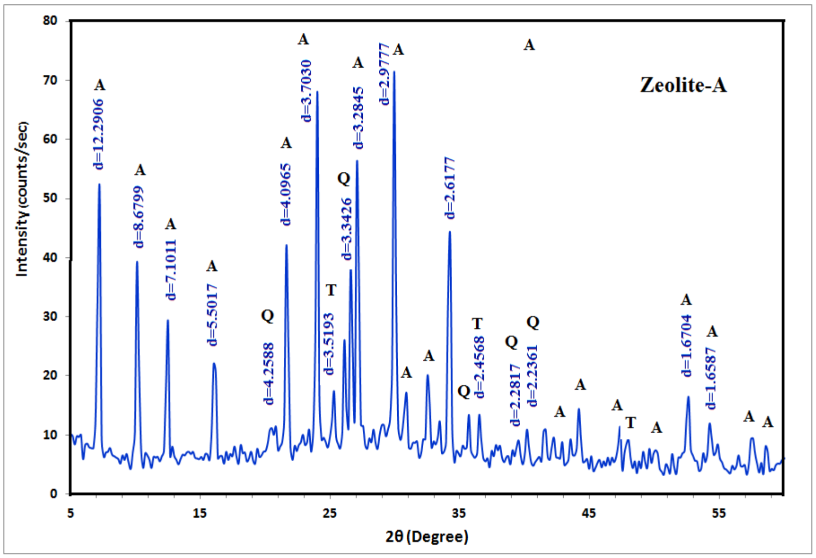
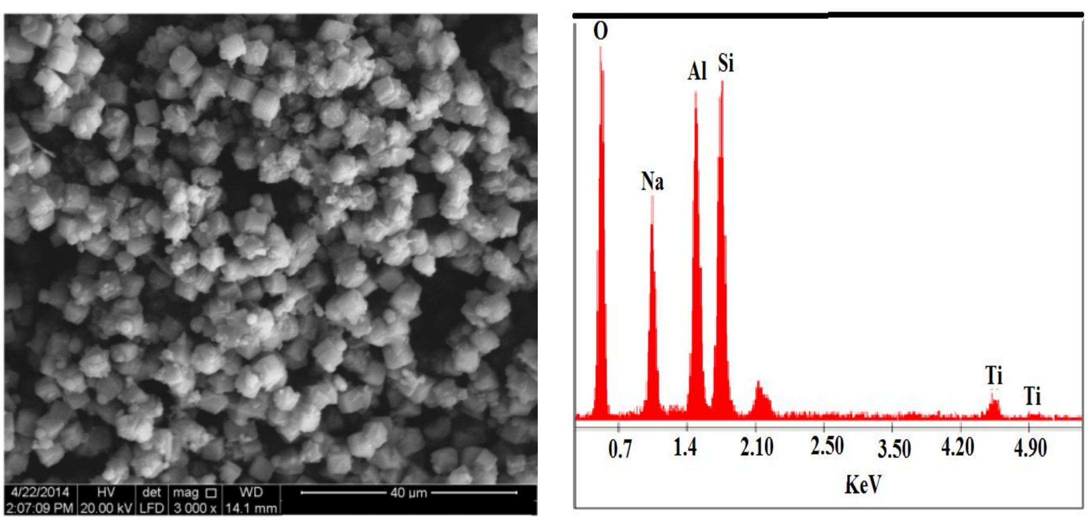


Fig. 1: XRD pattern of zeolite-A prepared under hydrothermal conditions of 80°C for 2h.

Figure 2 illustrated the microstructure of the synthetic powder (Fig.2a) and its chemical microanalysis (Fig. 2b). In Figure 2a, the cubic structure of zeolite-A with an average crystallite size of 3-4 µm covered all over the scanned area. The crystals seemed uniform in size and densely packed with very few debris from the parent un-reacted metakaolinite source. This intensive structure indicated the effectiveness and completion of the crystallization process of zeolite-A from Egyptian kaolin. Figure 2b implied the elemental microanalysis of the product (Z) which confirmed the crystallization of zeolite-A with its characteristic identity of nearly equal amounts of Si and Al.



(a) (b)

Fig. 2: SEM (a) and EDX (b) microanalysis for zeolite product.

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3.2. Soil Properties

Table 2 represented the physical and chemical parameters of the starting and ending soil (SS & ES) used in this work. The SS structure showed a sandy soil with very low percentage of the fine-grained clay and silt fractions (silt + clay=1%). The physical parameters figured out a slightly acidic soil with $CaCO_3$ and OM deficiencies. Meanwhile, the chemical properties emphasized nutrients-poor soil with high deficiency in all elements, particularly for K, Ca, Mg, and Na (Ankerman and Large, 1974).

After one rune of Z/ F fertilizer, the physical parameters measured some improvement, which appeared in the positive increase in organic matter percent (Min. of 0.90% in SS to Max. of 1.25% in Z_8F_0) and in the pH (from 6.8 to 8.04) with increasing Z/ F amounts. Also, the EC (1.07 to 0.26 dS/m) and $CaCO_3$ (0.34 to 0.24 Z_6 F_1) are relatively decreased. Chemically, some elements were increased in there measured amounts than the original ones namely; P (from 1.50 to 2.45 mg/100g in Z_0F_5) and (K) (from 13.0 in SS to 17.0 mg/100g in Z_0F_5), whereas others presented depletion in their amounts as Ca (from 259 in SS to 155 mg/100g in Z_8F_0), Mg (from 4.9 in SS to 3.1 mg/ 100g in Z_8F_0) and Na (24 in SS to 18 in Z_8F_0 and Z_6 F_1) noticed.

Table 2: Physico-chemical properties of the starting (SS) and ending soil (ES).

Soil properties		Starting Soil (SS)	At the end (ES) Treatments						Range
			Z_0 F_5	**Z_1 F_4**	**Z_2 F_3**	**Z_4 F_2**	**Z_6 F_1**	**Z_8 F_0**	
Sand	%	**99**	98.5	98.0	98.0	98.0	98.0	98.0	
Silt		**0.8**	1.2	1.5	1.5	1.5	1.4	1.4	
Clay		**0.2**	0.3	0.5	0.5	0.5	0.6	0.6	
Texture	**Sandy Soil**								
Physical									
pH		**6.8**	7.00	7.20	7.37	7.40	7.67	8.04	**6.7-7.2**
Electric Conductivity (EC) (dS/m)		**1.07**	0.28	0.26	0.27	0.29	0.34	0.39	**0.3-0.4**
Calcium Carbonate (%)		**0.34**	0.26	0.26	0.26	0.28	0.24	0.28	**2.1-8.0**
Organic Matter (OM)(%)		**0.90**	0.95	1.08	1.10	1.18	1.22	1.25	**2.0-3.0**
Chemical									
P	**(mg/ 100g)**	**1.51**	2.45	2.20	2.01	1.82	1.56	1.50	**1.2-2.7**
K		**13.0**	17.0	16.2	15.1	14.3	14.0	13.0	**21-30**
Ca		**259**	179	178	176	172	171	155	**91-140**
Mg		**4.9**	3.9	3.5	3.3	3.8	4.8	3.1	**30-180**
Na		**24.0**	20	19	18	20	18	20	**26-30**

In the current research, the application of the combined fertilizer (Z/ F) affected positively on the soil structure by enhancing its fine fraction content and raising the (silt + clay) from 1 in SS to 2 % at the end of treatment of only one run. The fine material part of soil (silt + clay) is an effective property concerning the soil fertility due to their capability of holding nutrients, microorganisms, and water. Irrespective to the rise of such fine earthy fraction, the soil is still poor and its fertility up-grade might need repetitive cycles of plantation with this combined fertilizer.

The up rise of the soil pH from slightly acidic (pH=6.8) to slightly alkaline (max. pH= 8.04) was the direct effect of zeolite addition, the result which agreed well with (Chander and Joergensen, 2002; Oste LA *et al*., 2002 and Hermassi *et al*., 2020), who reported similar batch soil-pH rise from 6 to 8 due to zeolite-A addition. Therefore, it was relied on the alkaline ability of zeolite to replace lime in soil applications for reducing acidity, improving the nutrients availability and cation exchange capacity (CEC) (Nibou *et al*., 2009 and Ramesh and Reddy, 2011).

It is a fact that, the CEC is directly proportional to the zeolite negative charges within the pores and is resulting from the isomorphous substitution of Si by Al, as ascribed to that of the 2:1 layered clay of montmorillonite structure (McBride, 1989).The CEC is constant for each zeolite species at different pH values and is dependent on its Si/Al ratio. Very recently, (Munthali, *et al*., 2014; Hermassi *et al*., 2020 and Szerementa, *et al*., 2021) reported the decrease in the CEC (*i.e.* the amount of Na^+ retention) of the low Si/Al zeolites (as A and X zeolite types) in the pH region of 7 to 9 and even more. The previous finding was explained by the high affinity of the negative charge sites of zeolite to H^+, originated from the dissociation of water.This H^+ high affinity recorded for the high negative charge density zeolite types with low Si/Al ratios as zeolite-A, is due to the more effective small ionic radius of H^+ (1.0 Å) (Marcus, 2012), compared to the larger one of Na^+ (1.9 Å) (Bonenfant *et al*., 2008). In addition, H^+ is bound to zeolite through covalent bonding, which is stronger than the electrostatic one originated between Na^+ and the negative charge sites. Accordingly, the said high (H^+) affinity of zeolite-A, as a low Si/Al zeolite with high charge density, could be the reason of sodium depletion in the treated soil here in. In the same trend, the high selectivity and saturation of H^+ in zeolite pores might also cause the decrease in the Ca and Mg retention of our soil by preventing their exchange with the negative zeolite sites. Thus, the replaced sodium by H^+ and the non-exchanged calcium and magnesium probably found their way out through leached from the sandy soil due to the elevation of pH value (Oste LA *et al*., 2002 and Hermassi *et al*., 2020). In addition, part of the Ca and Mg amounts were normally taken by plant daily nutrition. According to the previous authors, the increase of the soil pH caused partial dissolution in the organic matter, followed by high metal leaching through the metal-OM complexes. In the sandy soil, like one in this work, the leaching of dissolved components is not excluded, especially at the beginning of zeolite/soil amendment application. The leaching of Ca and Mg could also explain the slight decrease in the $CaCO_3$ content and the electric conductivity EC.

The results of physico-chemical properties of the biomass clearly indicated thankful effect on the soil quality when applying Z/F admixture, particularly for the organic matter (OM) content. The amount of (OM) has a direct relation to the water holding capacity of the soil (Fontaine *et al*., 2003 and Sayer *et al*., 2011). Recently, (Lai *et al*., 2013) record that carbon holding and water with high nutrients storing capacity as the benefits of high OM content of the soil**.** The OM content enrichment caused the improvement of micro and macro-pores number in the soil through "gluing" soil particles together and/or by creating favorable living conditions for soil organisms (June, 2008**)**. In this work, the addition of NPK in the combined fertilizer raised the K and P levels in the soil whereas the presence of zeolite and OM helped in storing those important elements for plant.

3.3. Nutrient concentrations

The convenient supply of macronutrient is crucial and positively affecting on plant growth and photosynthesis; while nitrogen is an integral part of all protein and enzymes, phosphorus is vital for energy transport (Amaliotis, *et al.* 2004). Also, potassium has an important role in soil fertility and plant health by improving each of the following: activating ATP-production enzymes, Enhancing photosynthesis and Phenolic biosynthesis (Krauss, 1999)**,** Water absorption and retention, along with root strengthening and sturdy stems (Hafez-Omaima, *et al*. 2012), and transporting nutrients to the xylem (Turner and Barkus, 1983).

Because the fourth leaf is the best indicator for the whole plant nutrition status of the plants, its analysis is separated in the following table.

Table (3) showed the analysis of the fourth paper nutrients after adding different Z/F amounts. It is clear from the table that, all macro and microelements were sufficient as nutrients for papaya according to (Jones, 1991), except for the K and Mg. Since potassium (K) was not an integral part of the initial soil, so its availability to the plant was dependent on the amount of the added NPK. In Table (3), all samples showed descending amounts of K content with decreasing the NPK rates, which is a quite normal effect of increasing zeolite parts at the expenses of the mineral fertilizer in the combined batches. In the case of Mg, the notable low levels of its concentration were the normal effect of unavailability from the poor mother sandy soil.

Table 3: Effect of applying Z/ F rates on the fourth papaya leaf analysis

Treatments	Macronutrients (%)				Micronutrients (ppm)		
	N	P	K	Mg	Fe	Mn	Zn
T_1 (Z_0F_5)	3.66	0.46	2.05	0.12	131	80	40
T_2 (Z_1F_4)	3.84	0.40	1.90	0.11	128	71	50
T_3 (Z_2F_3)	3.80	0.40	1.81	0.11	128	64	52
T_4 (Z_4F_2)	3.67	0.33	1.60	0.10	124	62	79
T_5 (Z_6F_1)	3.42	0.33	1.41	0.10	124	59	88
T_6 (Z_8F_0)	2.26	0.26	1.10	0.10	108	58	90
$LSD_{0.05}$	**0.06**	**0.02**	**0.04**	**0.01**	**4.6**	**2.5**	**2.5**
Ranges *	**1.2-1.35**	**0.17-0.21**	**2.7-3.4**	**0.4-1.2**	**25-100**	**20-150**	**15-40**

* Jones (1991)

3.4. Economic Effect

In the normal conditions of cultivation papaya under the Egyptian conditions, the actual needed amounts of the NPK (19 Nitrogen: 19 Phosphorus: 19 Potassium) fertilizer equals about 2.38 ton/ha. In the current work, the best treatments were recorded with corresponding fertilizer reduction percentages of 60 - 80%, which is economically feasible.

4. Conclusion and Recommendation

In conclusion, the addition of zeolite at the expense of fertilizer experimentally influenced the sandy soil parameters and plant quality. As fare from the obtained results, authors concluded that the addition of micro porous zeolite to the usual NPK fertilizer (19 N: 19 P_2O_5: 19 K_2O), for growing papaya was proved effective. It improved the soil properties, by increasing its fine fraction and reduced the fertilizer amounts.

The advantages of using the prepared zeolite-based fertilizer are of low-cost due to simple processing from local resource, reducing fertilizer consumption, and retail of high possibility for amending the lean sandy soil. Supplementary studies are needed to decide whether comparable effects will be found under field conditions and/or for other types of plants and crops.

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