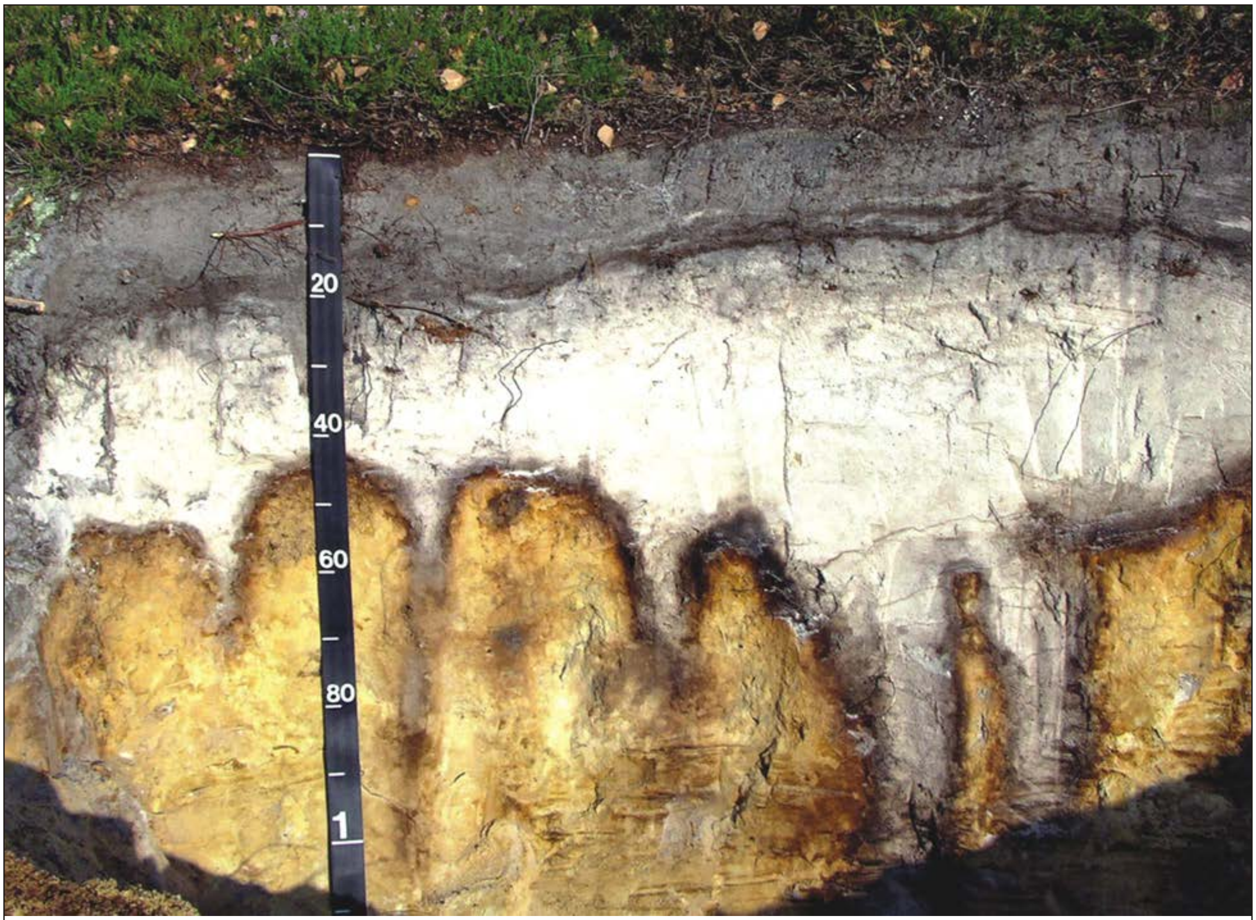

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Use of Clinoptilolite Zeolite to Reduce Christmas Island Rock Phosphate use in *Zea mays* Cultivation on an Acid Soil

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ABSTRACT

This study was conducted to (i) Determine dry matter production, nutrients concentration, nutrients uptake and use efficiency of *Zea mays* by including clinoptilolite zeolite in the fertilization program of *Zea mays* planted on an acid soil and (ii) Determine the effect of including Clinoptilolite zeolite in the fertilization program of *Zea mays* on selected chemical properties of an acid soil. The effect of Clinoptilolite zeolite application with 75% of fertilizers (C2) and 100% fertilizers (C1) on soil chemical properties were statistically similar. Similar observation was made for dry matter production, nutrients concentration, nutrients uptake and nutrients use efficiency. These suggest that substituting 25% of N, P and K fertilizers with Clinoptilolite zeolite is more beneficial compared to 100% application of these fertilizers. Clinoptilolite zeolite strong affinity for monovalent cations ensured timely availability of P by exerting more soil exchange sites to occupy Al^{3+} and Fe^{2+}.

Key words: Zeolites, acid soils, nutrient use efficiency, fertilizers management

INTRODUCTION

The global food demand by 2050 is estimated to increase by at least 50% and majority of the increase in demand will be in the tropical region. Dominance of metal hydroxides such as Al and Fe in acid soils of the tropics is a major factor limiting profitable and sustained agricultural production. As soil pH declines, supply of most plant nutrients decreases. Sufficient supply of P from the beginning is essential for optimum crop production. Phosphorus deficiency does not only limit N availability but it also leads to poor yield of crops. Thus, efficient use of P is crucial especially for developing countries where the need for achieving sustainable food security to sustain their growing human population.

The concerns for sustaining quality and soil resources as well as improving agriculture productivity under intensive production system had led to the use of soil amendments such as zeolites. Clinoptilolite zeolite is the most abundant natural zeolite with an infinite three-dimensional crystal structure, a polyhedric shape, with a great open cavity (Ajirloo *et al*., 2013; Dakovic *et al*., 2007). Unlike other amendments, zeolites do not break down over time but remains in the soil to improve nutrients retention. Therefore, its addition to soil will significantly reduce water and fertilizer costs by retaining water and beneficial nutrients in the root zone

(Polat *et al*., 2004). A number of studies on zeolites reported improved nutrients uptake and nutrients use efficiency of *Zea mays* cultivated on acid soils (Omar *et al*., 2011; Ahmed *et al*., 2010), increasing P availability from RP (Pickering *et al*., 2002), improving use of NH-N and NO-N by reducing leaching losses of exchangeable cations. Zeolites also act as slow-release fertilizer (Rabai *et al*., 2012; Lija *et al*., 2012), improves rice grain yield, N recovery and use efficiency (Kavoosi, 2007).

Although zeolites have been used to improve crops production, plant nutrients uptake and use efficiency, there is dearth of information on the use of zeolites to reduce P fixation not to mention reduction of N, P and K fertilizers use in agriculture. Thus, this study was carried out to (i) Determine dry matter production, nutrients concentration, uptake and use efficiency of *Zea mays* by including Clinoptilolite zeolite in the fertilization program of *Zea mays* planted on an acid soil and (ii) Determine the effect of including clinoptilolite zeolite in the fertilization program of *Zea mays* on selected chemical properties of an acid soil.

MATERIALS AND METHODS

Soil and clinoptilolite zeolite preparation and characterization: The Clinoptilolite zeolite used in this study was imported from Indonesia. The mineral soil (Typic Paledults, Bekenu series) used in this study was sampled in an undisturbed area of Universiti Putra Malaysia, Bintulu Sarawak Campus, Malaysia using an auger. The soil sample was taken at 0-15 cm depth, air dried and ground to pass 5.0 mm sieve for pot study and 2.0 mm for laboratory analysis. Soil texture was determined using hydrometer method (Bouyoucos, 1962), field capacity and bulk density were determined by the method described by Tan (2005). The pH of soil and Clinoptilolite zeolite was determined in a 1:2 (soil: distilled water) using a digital pH meter (Peech, 1965). The soil organic matter was determined using the loss-on-ignition method (Piccolo, 1996), available NO_3 and exchangeable NH_4 using Keeney and Nelson (1982). Soil available P was extracted using the double acid method (Tan, 2005) followed by blue color development (Murphy and Riley, 1962) and determination using UV-spectrometer. Exchangeable cations were extracted using the leaching method (Cottenie, 1980) and their concentrations determined using Atomic Absorption Spectrometry (AAS). Soil CEC was determined by the leaching method followed by steam distillation (Bremner, 1965). However, the CEC of the Clinoptilolite zeolite was determined using the CsCl method (Ming and Dixon, 1986). The CsCl method used avoids underestimation CEC of zeolites as the method does not lead to entrapment of ammonium ions in its channels. Soil exchangeable aluminium was extracted using 1 M KCl (1:10 soil/solution) and determined using colorimetric method (Rowell, 1994).

Pot study: The pot experiment was conducted at Universiti Putra Malaysia, Bintulu Sarawak Campus. The size of each pot was 22×28 cm and 8 kg soil (based on soil bulk density) was used. Maize (*Zea mays* L.) Hibrimas variety was used as test crop. The recommended rates of N, P and K fertilizers used were 60 kg N ha^{-1}, 60 kg P ha^{-1} and 40 kg K ha^{-1} (MARDI, 1990). The rates used in this study were a scaled down of the standard fertilizer recommendation per plant basis. The soil moisture was maintained at 60% of field capacity.

Treatments and their preparations: Evaluated treatments were as follows: (i) No fertilizer (T0), (ii) 4.85 g urea +2.47 g MOP +7.42 g CIRP (C1), (iii) 3.64 g urea +1.85 g MOP +5.57 g CIRP +12.29 g zeolite (C2). The treatments consisted of without fertilizer (T0), application of 100%

recommended fertilizers (C1) and 75% recommended fertilizers plus replacing 25% of the fertilizers with Clinoptilolite zeolite (C2). C2 was arrived at based on previous laboratory incubation study (unpublished).

The Clinoptilolite zeolite was mixed thoroughly with soil a day before planting. Five seeds were sown and thinned to one at the seven Days After Sowing (DAS). The MOP (60% K_2O) and CIRP (28% P_2O_5) were surface applied while urea (46% N) was buried in the soil to avoid loss of urea through ammonia volatilization. Split applications of fertilizers were carried out on 10 and 28th DAS and plants were harvested at 50 DAS (tasseling stage). Tasselling stage is the maximum growth stage for the plants before they go to reproductive stage (Kasim *et al.*, 2009). A day before harvesting, soil samples were taken and analyzed using standard procedures as described previously.

Plant analysis: The plants harvested were partitioned into roots, stems, tassels and leaves. Afterwards, they were washed using tap water and distilled water before being oven dried at 60°C until constant weight was attained. The dry weight were recorded and separately, plant parts were ground and analyzed for total N, P and K uptake and their use efficiency. Total N in plant tissues was determined using the Kjedahl method followed by steam distillation (Bremner, 1965). Total P and K in plant tissues were extracted using single dry ashing method (Tan, 2005). Total P was determined using UV-Vis spectrometer after blue color development (Murphy and Riley, 1962). Potassium values were determined using AAS. Nitrogen, P and K uptake in leaves, stems and roots were determined by multiplying their concentrations with the dry weight of the plant parts while N, P and K use efficiency (agronomic effectiveness) were determined by the equation shown below (Dobermann, 2005):

$$\text{Nutrient use efficiency (\%)} = \frac{\text{(Uptake with fertilizer-Uptake without fertilizer)}}{\text{Total amount of fertilizer that had been applied}} \times 100$$

Experimental design and statistical analysis: The experimental design was completely randomized design with four replicates. Analysis of variance (ANOVA) was used to test significant effect of treatments while means of treatments were compared using Tukey test. Statistical Analysis System (SAS, 2008) was used for the statistical analysis.

RESULTS AND DISCUSSION

The selected physico-chemical properties of Bekenu series, zeolite and CIRP are shown in Table 1. The soil pH in water, CEC and total N were 4.32, 5.33 cmol kg^{-1} and 0.06%, respectively. These results were consisted with those reported by Paramananthan (2000). Total carbon, exchangeable K, Ca and Mg were slightly higher than the standard range and this could possibly be due to litter decomposition at the soil surface of undisturbed area with time.

Table 2 shows selected soil chemical properties at 50 DAS. Irrespective of treatment, the selected soil chemical properties showed no significant differences. However, such observation was the contrary for K. Potassium in C1 was the highest and significant reduction in C2 was due to nutrients concentration and the availability is primarily regulated by soil properties and plant requirements. Stronger affinity for divalent cations towards Clinoptilolite zeolite exchange site may have suppressed monovalent ions thus, the low K availability in C2.

Table 1: Selected chemical properties of soil, zeolite and Christmas Island Rock phosphate (CIRP)

	Soil			
Property	Value obtained	Standard data range*	Zeolite	CIRP
pH (water)	4.32	4.6-4.9	8.54	Nd
Bulk density (g cm^{-3})	1.01	Nd	Nd	Nd
CEC (cmol kg^{-1})	5.33	3.86-8.46	75.4	Nd
Total Nitrogen (%)	0.06	0.04-0.17	Nd	Nd
Total Phosphorus (%)	0.005	nd	Nd	8.27
Available Phosphorus (mg kg^{-1})	2.48	nd	Nd	Nd
Organic Matter (%)	5.60	nd	Nd	Nd
Organic Carbon (%)	3.25	0.57-2.51	Nd	Nd
Exchangeable Aluminium (cmol kg^{-1})	0.9	nd	Nd	Nd
Exchangeable Hydrogen (cmol kg^{-1})	0.48	nd	Nd	Nd
Total acidity (cmol kg^{-1})	1.38	nd	Nd	Nd
Exchangeable Potassium (cmol kg^{-1})	0.24	0.05-0.19	6.16	8.06
Exchangeable Calcium (cmol kg^{-1})	0.76	0.05-0.19	22.30	3.28
Exchangeable Magnesium (cmol kg^{-1})	0.45	0.07-0.21	2.36	20.31
Texture	Sandy loam	Sandy loam	Nd	Nd

*Subjected to the soil development, standard data range by Paramananthan (2000), Nd: Not determine

Table 2: Selected chemical properties of soil without fertilization, recommended fertilization and 75% fertilization with 25% clinoptilolite zeolite at 50 days after seedling

			Exchangeable cation (cmol kg^{-1})				
Treatments	pH	OM (%)	K	Ca	Mg	Exchangeable ammonium (%)	Available nitrate (%)
T0	5.31^{ab}	5.75^{a}	0.10^{c}	0.53^{b}	0.41^{b}	0.019^{b}	0.001^{a}
C1	5.46^{a}	5.75^{a}	0.55^{a}	0.59^{a}	0.53^{a}	0.023^{a}	0.002^{a}
C2	5.14^{b}	5.80^{a}	0.31^{b}	0.43^{a}	0.49^{ab}	0.016^{ab}	0.001^{a}

Means within column with different letters indicate significant difference using Tukey test at $p \leq 0.05$, OM: organic matter, K: Potassium, Ca: Calcium, Mg: Magnesium, T0: Soil without fertilization, C1: Recommended fertilization, C2: 75% fertilization with 25% Clinoptilolite zeolite

Although more fertilizers were used in C1 compared to C2, exchangeable Ca and Mg contents were statistically similar suggesting better retention of Ca and Mg in C2. A Similar observation was reported in tomato cultivated with zeolite (Stylianou *et al*., 2004). These cations also partly attribute to RP dissolution (Wright *et al*., 1991). No statistical difference in soil pH, OM, NH_4^+ and NO_3^- is attributed to presence of Clinoptilolite zeolite as it preserves soil buffering capacity by reducing leaching losses of bases. In a related study, where zeolite was used as amendment, soil pH significantly improved with higher amount of zeolite used (Ahmed *et al*., 2010). Possibly the lower amount of Clinoptilolite zeolite used in this study was unable to preserve sufficient amount of bases to improve soil pH. Other authors reported that soil buffering capacity, microbial activities and plant genotypes also influence significant soil pH changes (Hinsinger *et al*., 2005; Vance *et al*., 2003). No significant differences in NH_4^+ and NO_3^- contents regardless of fertilization was possible because Clinoptilolite zeolite in C2 had higher CEC and greater affinity for NH_4^+ and hence minimized nitrification and NO_3^- ion from leaching (Rabai *et al*., 2013; Lija *et al*., 2012; Ahmed *et al*., 2010).

Soil available P as affected by treatments is shown in Fig. 1. Available P of C1 and C2 were similar although more fertilizers were used in C1 compared to C2. This observation was possible

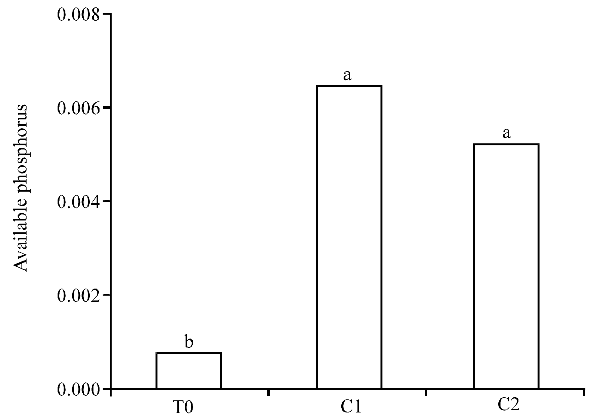

Fig. 1: Available P of solis without fertilization (T0), recommnded fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letter indicates significant differences using Tukey test at p≤0.05

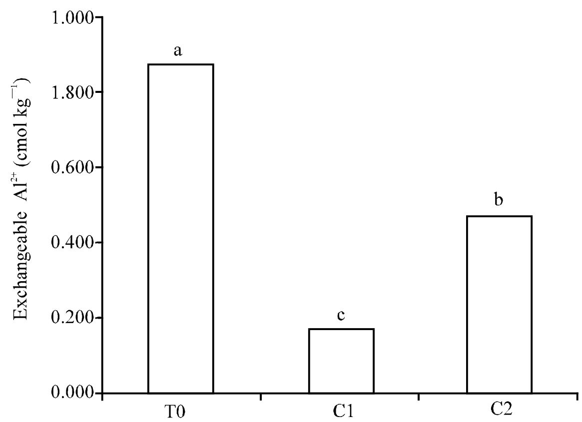

Fig. 2: Exchangeable Al^{3+} of soils without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letter indicates significant differences using Tukey test at p≤0.05

because of active induce-exchange mechanism of zeolite-RP which may have enhanced RP dissolution. Plant uptake and zeolite inclusion also induced soil capacitance by providing vacant sites onto which cations are held. Cations removal from soil solution induces RP dissolution in order to maintain soil-plant equilibrium (Mahabadi *et al.*, 2007). Besides, significant difference in exchangeable Al known to fix available P in C1 and C2 partly explains those observations (Fig. 2). Soil exchangeable Fe regardless of treatment however showed no statistical difference (Fig. 3). Better Al saturation as compared to Fe was due to larger Al sorption capacity and limited P availability to satisfy the sorption sites (Maguire *et al.*, 2000; He *et al.*, 2002). It was reported in other studies where higher rates of P fertilizers were needed to satisfy the sorption sites so as to increase P available for better crop production (Anetor and Akinrinde, 2007).

H^+ recorded for C1 and C2 were similar (Fig. 4). The acidification was due to nitrification, root exudation and high RP dissolution. This explains the significant increase of H^+ ion in C1 and C2 as compared to soil alone. Total soil acidity is the sum of H^+ and Al^{3+}. The significant reduction of total soil acidity in C1 and C2 was because of Al^{3+} reduction (Fig. 5).

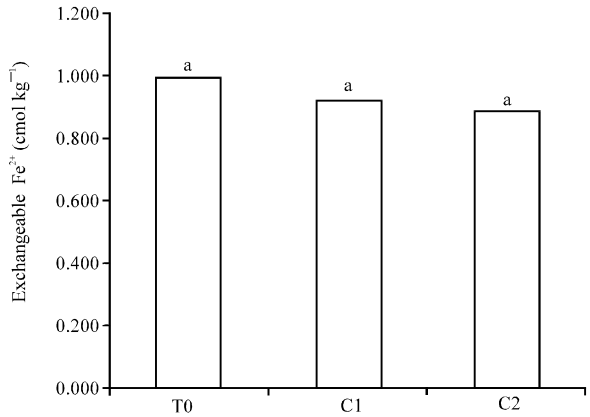

Fig. 3: Exchangeable Fe^{2+} of soils without fertilization (T0), 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letter indicates significant differences using Tukey test at $p \leq 0.05$

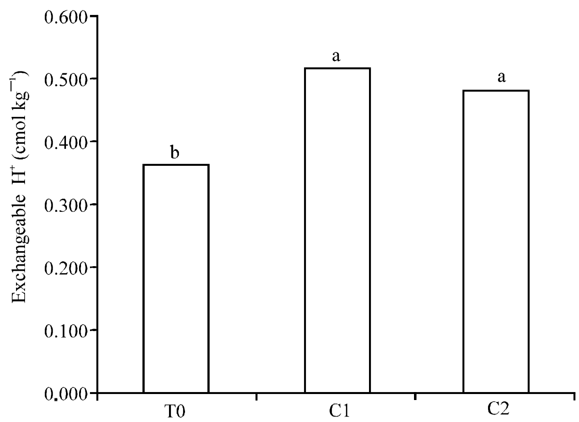

Fig. 4: Exchangeable H^+ of soils without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letter indicates significant differences using Tukey test at $p \leq 0.05$

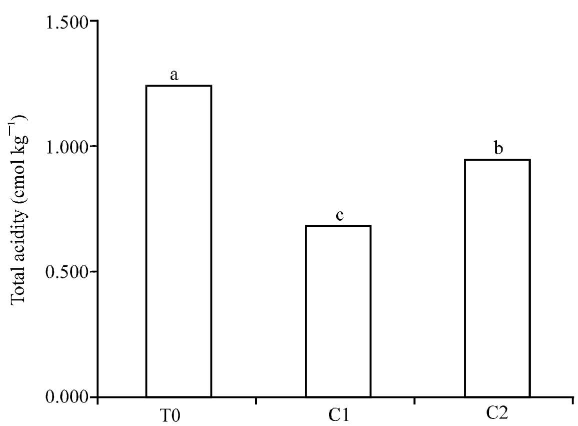

Fig. 5: Total acidity of soils without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letter indicates significant differences using Tukey test at $p \leq 0.05$

Table 3: Dry matter production and nutrients concentration of plant without fertilization, recommended fertilization and 75% fertilization with 25% clinoptilolite zeolite at 50 days after seeding

Parameters	Dry matter (g)	N (%)	P (%)	K (%)
Root				
T0	0.95[b]	0.35[b]	0.14[a]	0.90[a]
C1	14.81[a]	1.04[a]	0.08[a]	1.01[a]
C2	16.41[a]	0.80[a]	0.07[a]	0.83[a]
Stem				
T0	0.37[b]	0.41[b]	0.19[a]	3.87[a]
C1	8.39[a]	1.70[a]	0.18[a]	5.14[a]
C2	8.92[a]	1.08[a]	0.17[a]	4.34[a]
Leaf				
T0	0.97[b]	0.50[b]	0.27[a]	3.25[a]
C1	9.73[a]	2.03[a]	0.18[b]	3.25[a]
C2	12.16[a]	0.91[a]	0.16[c]	3.00[a]
Total				
T0	2.29[b]	1.26[b]	0.61[a]	8.03[a]
C1	32.93[a]	4.78[a]	0.44[b]	9.24[a]
C2	37.48[a]	2.78[b]	0.40[b]	8.17[a]

Means within column with different letters indicate significant difference using tukey test at $p \leq 0.05$, N: Nitrogen, P: Phosphorus, K: Potassium, T0: Soil without fertilization, C1: Recommended fertilization, C2: 75% fertilization with 25% clinoptilolite zeolite

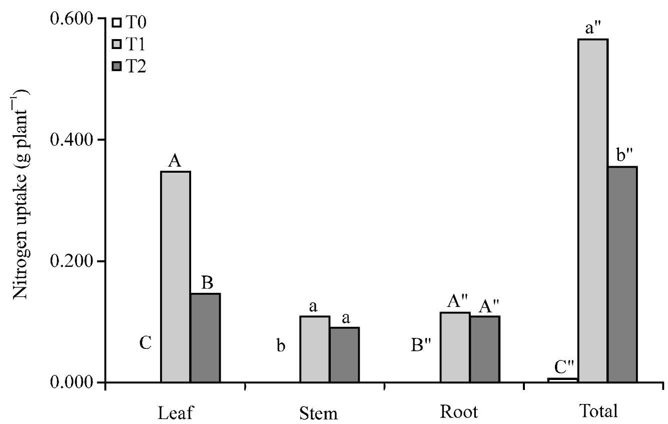

Fig. 6: Nitrogen uptake of plant without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letters indicate significant difference using Tukey test at $p \leq 0.05$

Plant dry matter production and nutrients concentration results are presented in Table 3. Dry matter production irrespective of plant part showed significant increase in C1 and C2 compared to soil alone (T0). Low dry matter for T0 was due to no fertilization to sustain plant growth and development. No significant difference in the effects of C1 and C2 on dry matter production suggests that inclusion of Clinoptilolite zeolite contributed to better nutrients retention and their timely release for plant growth and development. Dilution effects perhaps caused the insignificant differences in nutrients concentration regardless of plant part (Marschner, 1995). Similar observation has been reported by other authors (Ahmed *et al*., 2010; Mengel and Kirby, 1996). Significant dry matter production, yield and nutrients uptake have been associated with root development, translocation and shoot demand per unit of nutrients absorbed (Baligar *et al*., 1993). As shown in Fig. 6, the use of Clinoptilolite zeolite with reduced amount of fertilizers (C2) showed significant reduction in N uptake irrespective of plant part compared those of C1 (standard fertilization). However, this observation was not true for K and P. Similar uptake of P and K in C1 and C2 (Fig. 7 and 8) suggests a need to reduce excessive use of fertilizers to avoid loss and environmental pollution. Nitrogen, P and K use efficiency in C2 was comparable to those of C1 (Fig. 9, 10 and 11). Perhaps efficient use of these nutrients in C2 as compared to C1 was due to successful retention of cations and their timely releases by Clinoptilolite zeolite for crop use. Improvement in N, P and K uptake and use efficiency in *Zea mays* were noted in studies using inorganic fertilizers mixed with zeolite (Ahmed *et al.*, 2010) and mixing zeolite with compound fertilizer (Rabai *et al.*, 2013). However, such observation was not true in this study due to the lower amount of Clinoptilolite zeolite and fertilizers applied for economic purposes. Inclusion of zeolite in fertilization is reported to cause a phenomenal growth of the microorganisms in the soil, increased nutrient intake of plants, reduced effects of soil acidity, prevented soil erosion and increased storage

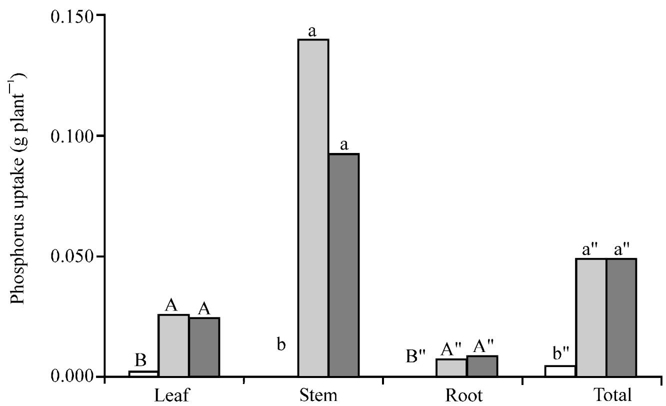

Fig. 7: Phosphorus uptake of plant without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letters indicate significant difference using Tukey test at $p \leq 0.05$

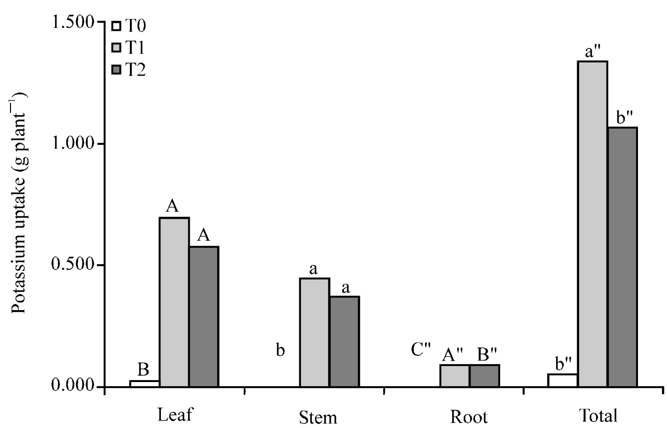

Fig. 8: Potassium uptake of plant without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letters indicate significant difference using Tukey test at $p \leq 0.05$

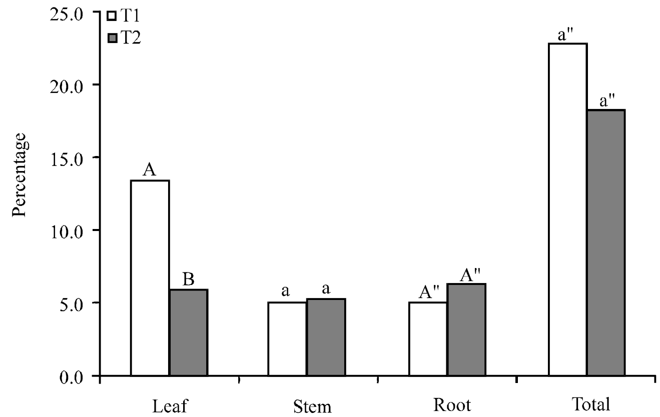

Fig. 9: Nitrogen use effeicency of plant without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letters indicate significant difference using Tukey test at p≤0.05

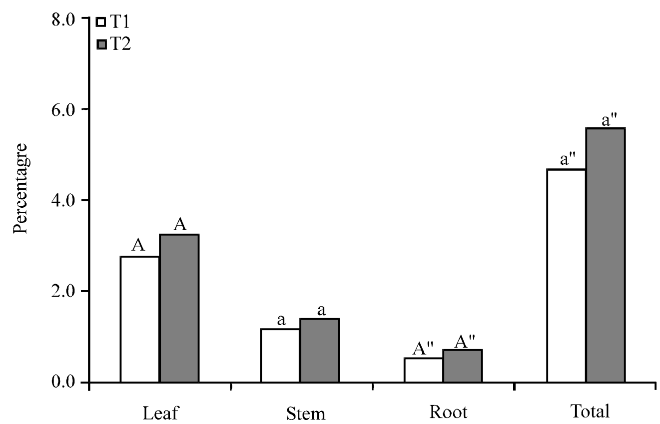

Fig. 10: Phosphorus use effeicency of plant without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letters indicate significant difference using Tukey test at p≤0.05

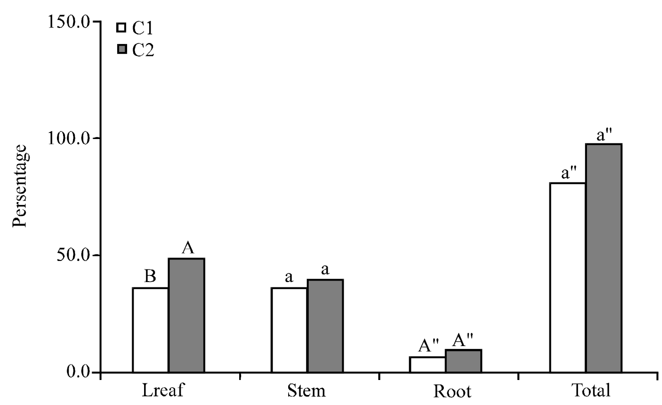

Fig. 11: Potassium use effeicency of plant without fertilization (T0), recommended fertilization (T1) and 75% fertilization with 25% clinoptilolite zeolite (T2) at 50 DAS. Means with different letters indicate significant difference using Tukey test at p≤0.05

capacity of soil as well as contributing to building of precious humus complexes (Grant *et al.*, 2005; Beqiraj *et al.*, 2008). Although in this study the effect of Clinoptilolite zeolite was not glaring on the selected soil chemical properties, perhaps long term application could cause significant effect.

CONCLUSION

The effect of Clinoptilolite zeolite application with 75% of fertilizers (C2) and 100% fertilizers (C1) on soil chemical properties were statistically similar. Similar observation was made for dry matter production, nutrients concentration, nutrients uptake and nutrients use efficiency. These suggest that substituting 25% of N, P and K fertilizers with Clinoptilolite zeolite is more beneficial compared to 100% application of these fertilizers. Clinoptilolite zeolite strong affinity for monovalent cations ensured timely availability of P by exerting more soil exchange sites to occupy Al^{3+} and Fe^{2+}. At least three cropping cycles are recommended to confirm the findings of this study. It is also essential to estimate the economic benefits of including Clinoptilolite zeolite in *Zea mays* cultivation. These aspects are being investigated in on-going field experiments.

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