Carpathian Journal of Earth and Environmental Sciences, April 2010, Vol. 5, No. 1, p. 25 - 32

THE EFFECT OF ORGANIC FERTILIZER, COMPOSITE NPK AND CLINOPTILOLITE ON CHANGES IN THE CHEMICAL COMPOSITION OF DEGRADED VERTISOL IN WESTERN SERBIA

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Abstract: Over 2003-2008, a study was conducted on the combined effect of clinoptilolite commercially termed “Agrozel”, organic fertilizer (cattle manure) and composite NPK fertilizer (15:15:15) on changes in the chemical composition of shallow eroded i.e. degraded vertisol in Cacak area (Western Serbia). The mineral clinoptilolite derived from natural zeolitic tuff obtained at the Katalenac deposit near Vranjska Banja (South-eastern Serbia) was used in the study. Agrozel has a particle size of 0-1.0 mm, cavity volume of about 34%, high thermal stability (600-650ºC), and the cation exchange capacity (CEC) of 153 mmol M^+/100 g. The following fertilization treatments were employed: i) control (without fertilization), ii) A_1 = NPK (0.1 kg/m^2) + cattle manure (5 kg/m^2), iii) A_2 = NPK (0.1 kg/m^2), iv) A_3 = Agrozel (1 kg/m^2) + cattle manure (5 kg/m^2) + NPK (0.1 kg/m^2), and v) A_4 = Agrozel (1 kg/m^2) + NPK (0.1 kg/m^2). The obtained results showed that the combined use of fertilizers induced changes in soil chemical composition as compared to the control. All treatments (A_1, A_2, A_3 and A_4) led to increases in humus, total nitrogen (N_{TOT}) and, in particular, phosphorus (P_2O_5) and potassium (K_2O) levels within all depths. The humus and N_{TOT} increases were not significant, as opposed to P_2O_5 and K_2O increases that were significant relative to the control. Soil pH at all depths was significantly increased by the A_1, A_3 and A_4 treatments and decreased by the A_2 treatment. The highest increase was recorded in the 0-20 cm soil depth and the lowest in the 40-60 cm depth.

Key words: cattle manure, clinoptilolite, composite NPK fertilizer, degraded vertisol, soil.

1. INTRODUCTION

According to Cakmac (2002), 60% of world soils exert limiting effects on food production due to their degradation, micronutrient deficiencies and toxicity traits. Serbia is characterized by a substantial proportion of soils having unfavourable properties (pronounced acidity, disturbed structure, compaction, exhaustion, tiredness, etc.). Specific crops, fruit ones in particular, are being grown on these soils, yielding, however, considerably lower yields and deteriorated fruit quality. The non-planned and routine fertilization of these soils only induces deterioration of its properties [chemical (pH), physical (structure) and, particularly, biological traits (the presence of beneficial macro- and microorganisms)]. Regeneration of these soils is a long-term process which can, assisted solely by nature, last for more than 20 years (Ruecker et al., 1998). Their remediation, i.e. conversion into cropland is costly and long-term.

Organic fertilizers, such as cattle manure, have long since been known to improve physical, chemical and biological properties of soils, particularly increasing the humus content and decreasing acidity (Zhukov et al., 1993; Ganzhara, 1998; Lal, 2006).

One of the measures considered highly effective, biologically justified and environmentally safe, especially on degraded and other soils having unfavourable productive traits for crop cultivation, is the use of natural zeolite mineral (Polat et al., 2004; Beqiraj et al., 2008). There are several zeolite types, one of them being clinoptilolite, a hydrated alumosilicate of alkali and alkaline earth metals (Na^+, K^+, Ca^{2+}, Mg^{2+}, Ba^{2+}) having an infinite threedimensional crystal structure, a polyedric shape and a great open cavity (Wise & Kleck, 1988). Anonymous (2004) reported that zeolites can be successfully used in cultivating different crops such as cereals, forage crops, vegetables, vine and fruit crops due to their exceptionally high ion-exchange capacity (Mumpton, 1999; Baerlocher et al., 2001; Butorac et al., 2002; Lee et al., 2006). Their porous structure assures a permanent water reservoir in the root zone, improving the horizontal spread of water after irrigation (Treacy & Higgins, 2001; Polat et al., 2004). Zeolites prevent unnecessary losses of nutrients, making them available exactly when needed (Flanigen & Mumpton, 1981). They are excellent carriers, stabilizers and regulators of mineral fertilizers, themselves being a source of certain nutrients (Podlešáková et al., 1967; Bagdasarov et al., 2004). As carriers of nitrogen and potassium fertilizers, they increase their efficacy by decreasing application rates for equal yields to be achieved (Polat et al., 2004). The stated author also suggested that zeolites, being of weakly alkaline reaction, can be combined with mineral fertilizers to maintain soil buffering and indirectly regulate soil pH. According to Anonymous (2004), zeolites improve the growth and development of plants.

The basic objective of this study was to examine changes in the soil chemical properties as induced by the effect of combined fertilization with the clinoptilolite type Agrozel, cattle manure and composite NPK fertilizer on changes in soil chemical composition on shallow eroded, i.e. degraded vertisol during 2003-2008.

2. MATERIALS AND METHODS

2.1. The study area

The western Serbia region is formed from the rocks of different petrographic composition and age. Great areas are covered by paleozoic schists, mesozoic limestone and sandstone. Of the eruptive rocks, the most widespread are serpentine rocks, followed by granite, andesite and diabase. Some areas abound in neogenic sediments as well as in quaternary sediments as loess and aluvium, flysch, deluvial sediments, diabase-chert formations and other rocks (Skoric et al., 1985). The above rocks served for soil formation, which, coupled with other factors, contributed to the great diversity of the pedological cover in western Serbia, including Cacak area (Fig. 1).

The investigation was conducted in 2003-2008 in the mature plum orchard established in 1990, at the village of Prislonica, 15 km north-east of Cacak (43°53′N; 20°21′E, altitude 390 m), western Serbia. The spacing employed was 5 m × 3 m (667 trees/ha).

Soil maintenance in the orchard involved grass-sown inter-row area and row grass and weed control by the Glifosat total herbicide.

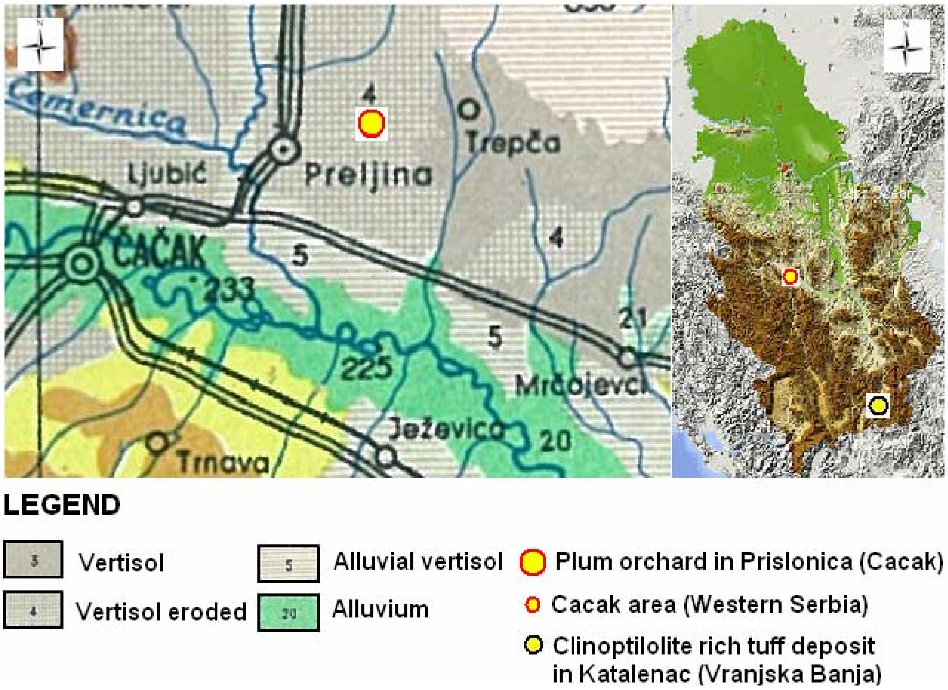

Figure 1. Pedological map of the Cacak area and clinoptilolite rich tuff deposit in Katalenac near Vranjska Banja (South-eastern Serbia)

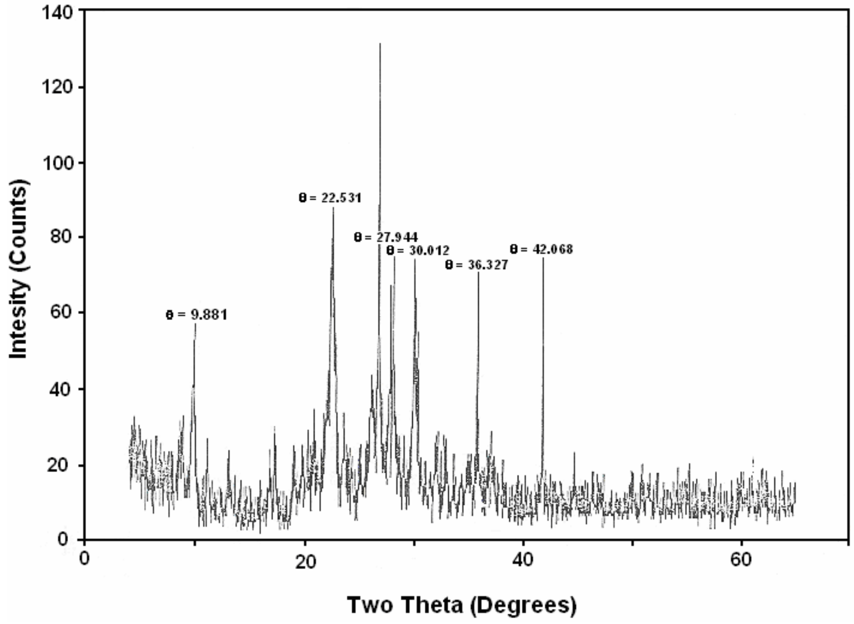

Figure 2. X-ray diffraction analysis of the clinoptilolite rich tuff originating from the Katalenac deposit (Vranjska Banja)

Soil samples for chemical analysis were collected from a 1-m wide herbicide-treated band of soil. The orchard was not irrigated.

The orchard soil was shallow eroded i.e. degraded vertisol (USDA Soil Taxonomy, 1999). The distribution of this type of vertisol in western and north-western Serbia is larger than that of typical vertisol, as it covers covering an area of 51,000 ha or 2.81% of the total acreage (Protic et al., 2003). It occurs on sloping relief along the rim of the Cacak area i.e. on higher-altitude steep terrains providing less favourable conditions for the formation of deeper soil or it was developed by erosion of typical vertisol. In morphological terms, this type of vertisol has the properties of normal vertisol (A-AC-C profile), with only the horizons, the A horizon in particular, being more poorly developed (Tanasijevic et al., 1966). In our study, A horizon is 31 cm deep. It is black, turning lighter with depth. In terms of composition, it is a heavy textured loam, having a prismatic polyhedral structure. There appears a gradual transition into the AC horizon. The AC horizon is located at a depth of 31-67 cm. It is dark brown, heavy-textured loam, with a short prismatic polyhedral structure. There is an abrupt transition into the C horizon. The C horizon is 67-180 cm deep, composed of calcerous loam, pale-yellow in colour. The number of pale-grey rusty spots and soft lime concretions increase with depth. The total content of clay in the surface layer is 63.65%, and that of sand 36.35%. The predominating fractions include coloidal clay particles, silt and fine sand, whereas the proportion of coarse sand is negligible. Hygroscopic moisture in the upper layers is 5.10%.

2.2. Origin and characteristics of the clinoptilolite and cattle manure

The natural zeolite sample used in this study was a clinoptilolite rich tuff obtained from the Katalenac deposit (Vranjska Banja, south-eastern Serbia). In terms of mineralogical composition, the natural zeolitic tuff primarily contained clinoptilolite (minimum 60%) with trace amounts of feldspar, quartz and pyrite as determined by X-ray powder diffraction (XRPD) analysis (Philips PW-1051, Cu anti-cathode) (Fig. 2). The predominant cation associated with the clinoptilolite rich tuff is calcium (Radosavljevic-Mihajlovic et al., 2003). The exchangeable cations range within the following values (mmol M^+/100 g): Ca^{2+}-62.12 to 100.94; Mg^{2+}-11.63 to 36.89; Na^+-25.31 to 44.68 and K^+-22.37 to 28.96. Specific mass is 2.24 g cm^{-3}, bulk density 1.425 η m^{-3}, and hardness (by Moss) is 3.0-3.5. The chemical composition of the mineral (wt%) was: SiO_2-62.65, Al_2O_3-14.10, Fe_2O_3-3.07, TiO_2-0.49, CaO-3.04, MgO-1.64, Na_2O-0.92, K_2O-3.30, Cu-0.007, Mn-0.018, Zn-0.007 and H_2O-10.45 (Tomasevic-Canovic et al., 2001). The selectivity towards cations was as follows: Cs>Rb>K>NH_4>Ba>Sr>Na>Ca>Fe>Al>Mg>Li.

Agrozel originated from Institute for Technology of Nuclear and Other Raw Materials in Belgrade. The final processing (grinding and packaging) of the natural zeolite type clinoptilolite, commercially termed Agrozel is performed at the A.D. "MineraliCO - Nemetali", Vranjska Banja (Fig. 1). It is being marketed as Agrozel for use in plant production. Agrozel has a particle size of 0-1.0 mm, cavity volume of about 34%, high thermal stability (600-650ºC), and the cation exchange capacity (CEC) of 153 mmol M^+/100 g (Tomasevic-Canovic et al., 2001).

The cattle manure used in our study was provided by a local dairy farm. The chemical composition of the manure was as follows: N-0.5%, P_2O_5-0.3%, K_2O-0.6, electrical conductivity - 6.32, organic matter - 25%, and the C:N ratio - 18:1 (DeLuca & DeLuca, 1997; Larney et al., 2006).

2.3. Methods

The experiment was set up as a randomized block design in four replications, each comprising ten plum trees. In mid-November of 2003-2008, the fertilizers used in the treatments were applied in 1.0 m wide bands on both row sides. Upon broadcasting, the fertilizers were rotary tilled into the soil. The treatments involving the application of fertilizers were as follows:

i) Prior to trial - no fertilizer (control);

ii) Treatment 1 (A_1) = NPK (0.1 kg/m^2) + cattle manure (5 kg/m^2);

iii) Treatment 2 (A_2) = NPK (0.1 kg/m^2);

iv) Treatment 3 (A_3) = Agrozel (1 kg/m^2) + cattle manure (5 kg/m^2) + NPK (0.1 kg/m^2); and

v) Treatment 4 (A_4) = Agrozel (1 kg/m^2) + NPK (0.1 kg/m^2).

In mid November 2003, immediately prior to treatments, a chemical soil analysis was conducted to determine pH and the contents of humus, N_{TOT} and available $P_2O_5$and K_2O. The samples were collected from bands on the left- and right-hand sides of the plum rows in the root system zone at a distance of 1.0 m from the trees. Each replication included three soil samplings conducted on both row sides from three soil depths of 0-20, 20-40 and 40-60 cm. The analysis was repeated at the end of the experiment, i. e. in mid-November 2008 for determination of any changes in the soil chemical composition.

A Pye glass electrode pH-meter-potentiometer (W. G. Pye, Cambridge) was used to measure pH in 0.01 M KCl. The humus content was determined by oxidation with $KMnO_4$ solution (according to Kotzman). Total nitrogen was measured using the Kjeldahl method with sulphuric acid and a metal catalyst. Available P_2O_5 and K_2O were determined by extraction with Al solution, and P_2O_5 and K_2O by colorimetry with molybdate and flame photometry, respectively (Egner et al., 1960).

The experimental data were subjected to analysis of variance. An LSD test at $P \leq 0.01$ was used for mean separation. The data were analyzed by the ANOVA statistical programme (SPS Statistica 6.0 Software).

3. RESULTS AND DISCUSSION

The table 1 data show that, prior to trial (control) establishment (mid-November 2003), the plum orchard soil had an acid pH (of 4.89±0.10 to 5.19±0.13), a poor to moderate humus supply (1.03±0.05 to 3.01±0.09%) and a very low N_{TOT} content (0.06±0.01 to 0.15±0.01%), the values thereof gradually decreasing with depth. The contents of P_2O_5 and K_2O in the 0-60 cm soil depth ranged from 31.5±2.08 to 73.0±2.11 mg/kg and from 174.5±2.43 to 332.0±4.71 mg/kg, respectively.

The soil chemical analysis conducted at the end of the trial (November 2008, i.e. after a six-year period) suggest that the soil chemical composition underwent certain changes as compared to the composition prior to treatment (control) (Figs. 3-7). All treatments (A_1, A_2, A_3 and A_4) led to increases in humus, N_{TOT} and particularly P_2O_5 and K_2O within all depths (Figs. 4-7). The humus and N_{TOT} increases were not significant, as opposed to P_2O_5 and K_2O increases which were significant relative to the control. Soil pH at all depths was significantly increased by the A_1, A_3 and A_4 treatments and decreased by the A_2 treatment (Fig. 3).

The A_3 treatment induced the highest increase in soil pH (from 5.19±0.13 units before the trial to 6.74±0.62 after six years at 0-20 cm depths).

Table 1. Soil chemical properties prior to trial establishment*

Soil depth (cm)	pH	Content (%)		mg/kg (AL-method)	
		Humus	Total N	P_2O_5	K_2O
00-20	5.19±0.13	3.01±0.09	0.15±0.01	73.0±2.11	281.0±5.66
20-40	4.91±0.11	1.89±0.08	0.08±0.01	26.5±1.94	332.0±4.71
40-60	4.89±0.10	1.03±0.05	0.06±0.01	31.5±2.08	174.5±2.43

* Soil sampling for chemical analysis was conducted in mid-November 2003

The humus content slightly increased, especially at the 0-20 cm soil depth (from 3.01±0.09% before the trial to 3.44±0.14% after six years in the A_3 treatment) (Fig. 4).The N_{TOT} content slightly increased, especially in the A_3 treatment (from 0.15±0.01% before the trial to 0.18±0.04% after six years at the 0-20 cm soil depth) (Fig. 5). The content of P_2O_5 at the 0-20 cm depth increased from 73.0±2.11 mg/kg before the trial to 333.0±4.99 mg/kg in A_3. At a depth of 20-40 cm, it increased from 26.5±1.94 mg/kg before the trial to 217.0±3.89 mg/kg in A_3. At a depth of 40-60 cm, it increased from 31.5±2.08 mg/kg to 149.0±3.33 mg/kg in A_1 and 149.0±3.19 mg/kg in A_3 (Fig. 6).

The content of K_2O increased from 281.0±5.66 mg/kg to 355.0±5.97 mg/kg in A_3 at a depth of 0-20 cm. It increased from 332.0±4.71 mg/kg to 380.0±5.32 mg/kg in A_3 at a depth of 20-40 cm and from 174.5±2.43 mg/kg to 224.0±4.88 mg/kg in A_3 at a depth of 40-60 cm (Fig. 7). With the exception of the A_2 treatment which caused an increase in acidity, all treatments led to a significant reduction in acidity at all depths (Fig. 3). The incorporation of composite NPK fertilizers into the soil having an optimized content of K_2O and a low to medium content of P_2O_5 caused additional increases in these contents, especially within the 0-20 cm soil depth (Figs. 6 & 7). Jelic et al. (2006) reported that mineral fertilization, primarily with large potassium and nitrogen rates applied to the vertisol undergoing degradation, caused a substantial increase in acidity (0.29 to 0.37 pH units in active acidity and 0.18 to 0.24 units in substitutionary acidity), humus, N_{TOT} and available P_2O_5 and K_2O in the soil.

Stevanovic et al. (1999) and Glisic et al. (2009) determined that high rates of composite NPK fertilizers broadcast annually across the rows in pear, blackberry and strawberry orchards, induced disturbances in mineral nutrition, primarily due to the accumulation of higher amounts of K_2O and acidity increases in the soil. The results of the above authors were confirmed by the results of the our study.

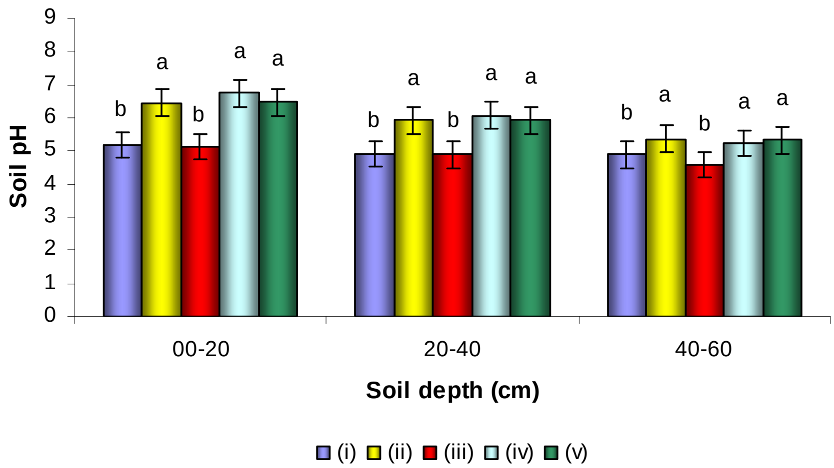

Figure 3. Soil pH at all depths was significantly increased by the A_1, A_3 and A_4 treatments and decreased by the A_2 treatment (mean±SE). The same letters at the top of the columns indicates non significant differences at $P \leq 0.01$ by LSD test

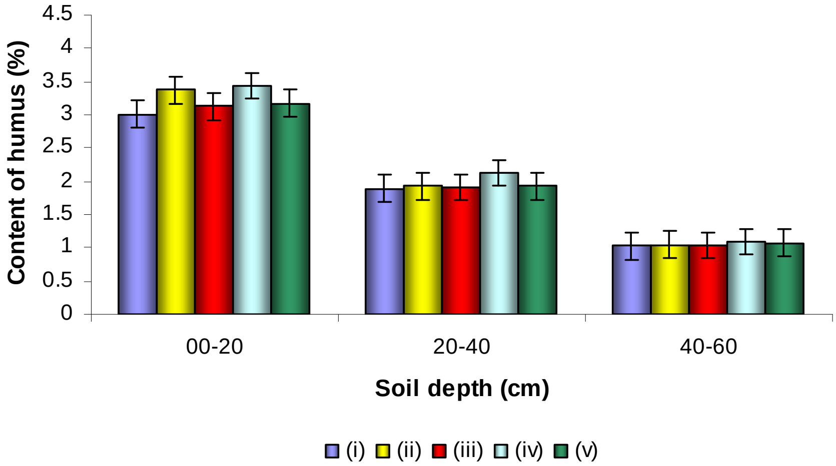

Figure 4. Increases in humus content at all soil depths were not significant in any of the treatments (mean±SE)

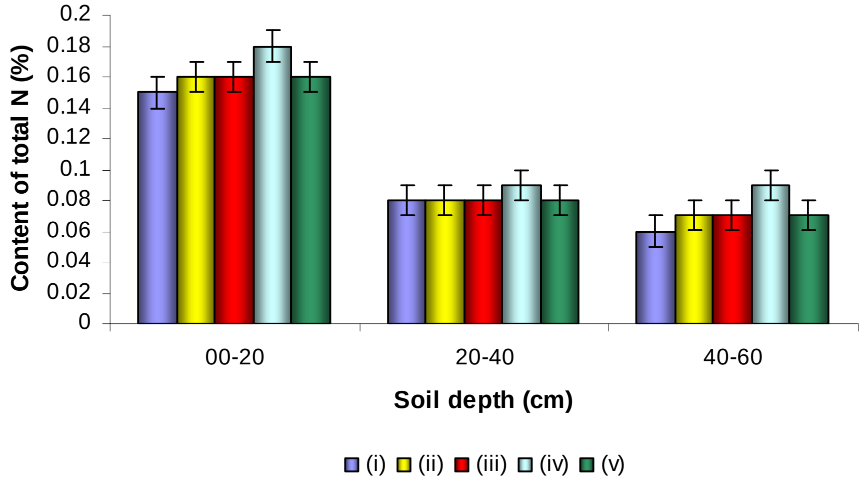

Figure 5. Increases in total nitrogen content at all soil depths were not significant in any of the treatments (mean±SE)

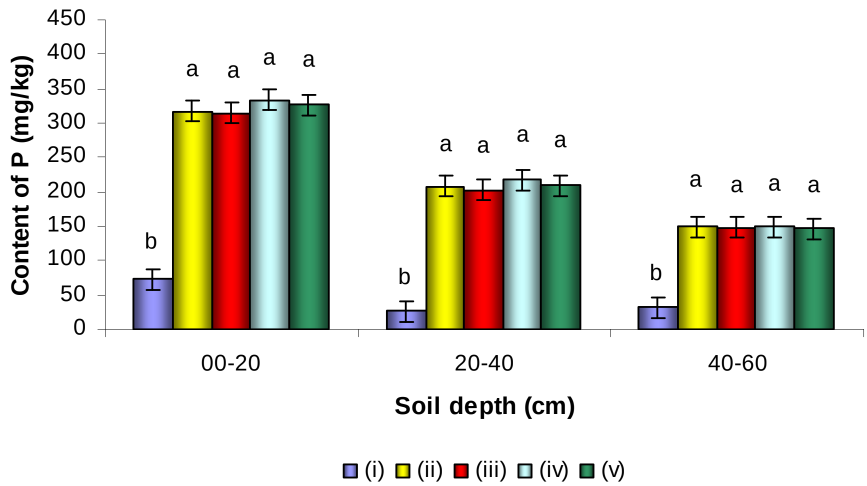

Figure 6. Phosphorus content at all soil depths was significantly increased by all treatments (mean±SE). The same letters at the top of the columns indicates non significant differences at $P \leq 0.01$ by LSD test

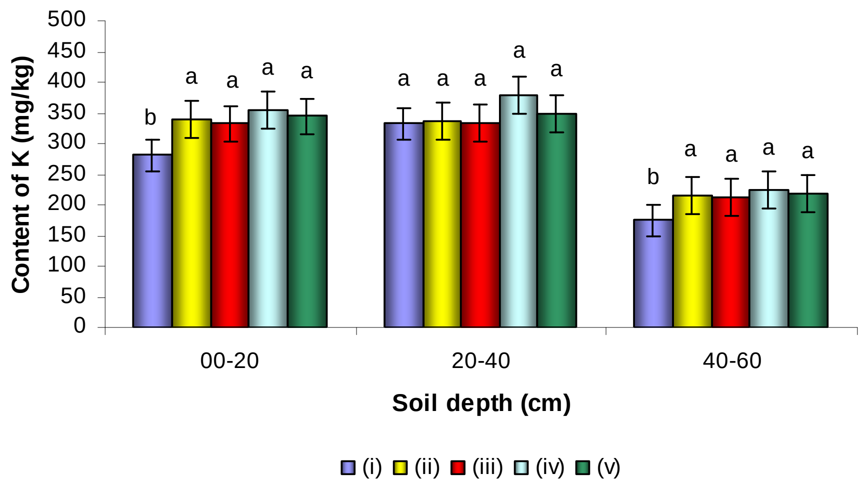

Figure 7. Potassium content at all soil depths, except at a depth of 20-40 cm, was significantly increased by all treatments (mean±SE). The same letters at the top of the columns indicates non significant differences at $P \leq 0.01$ by LSD test

The combined fertilization with manure, NPK and Agrozel (A_3) resulted in an increase in humus content and a decrease in acidity in our study (Iakimenko et al., 1996), particularly in the 0-20 cm soil depth (Figs. 3 and 4). Given the average organic matter content of 25.0% in well-mature beef manure (DeLuca & DeLuca, 1997; Larney et al., 2006), the reaction was as expected and in line with the results of the stated authors. Apart from increasing the humus content of soil, cattle manure has a favourable effect through decreasing acidity due to the significant proportion of H_2CO_3 reacting with the soil adsorption complex (Jakovljevic & Pantovic, 1999). This results in the formation of $NaHCO_3$ which in an aqueous solution dissolves into weak non-dissociating H_2CO_3 and strong dissociating NaOH, with several OH^- ions occurring in the soil, thus resulting in an acidity decrease (Augstburger, 1983; Chander & Joergensen, 2002). The exchangeable cation sites of Agrozel are occupied by exchangeable divalent metal Ca^{2+} ions (Baerlocher et al., 2001; Lee et al., 2006) being substituted in the soil solution with other ions. Once in the soil solution, they indirectly decrease acidity. Chander & Joergensen (2002) report that "A_4"-type zeolite increases soil pH as $CaCO_3$ does, which was confirmed in our study.

The N_{TOT} content of the soil in our study was low, the value obtained being the most common one for vertisols in Serbia (Tanasijevic et al., 1966). Its increase induced by different treatments was not significant. Vertisols in Serbia are generally poor in available P_2O_5 (Skoric et al., 1985). However, the incorporation of the composite NPK fertilizer with a 15% P_2O_5 content was determined to be unnecessary in our study due to its superfluous content in the soil (Fig. 6). The natural zeolite i.e. Agrozel structure is mostly made up of the K^+-ion-containing clinoptilolite mineral. One of major natural zeolite traits is that of ion-exchange, zeolite having the cation exchange capacity (CEC) of 153 mmol M^+100g^{-1} (Wise & Kleck, 1988; Baerlocher et al., 2001), potassium being among elements having the highest ion-exchange capacity (Treacy & Higgins, 2001) and therefore being very easily released from the crystal zeolite structure and into the soil solution, eventually experiencing an increase in its total soil content (Podlešáková et al., 1967; Beqiraj et al., 2008). The incorporated NPK mineral fertilizer with a 15% K_2O content also gave rise to an increase in soil potassium content in our study.

4. CONCLUSION

The combined fertilization of soil in a mature plum orchard with clinoptilolite, commercially termed Agrozel, cattle manure and composite NPK fertilizer over a six-year period induced increases in soil pH, humus and N_{TOT} as well as a substantial increase in K_2O and P_2O_5 contents of the soil in the mature plum orchard.

The highest increase was recorded in the 0-20 cm soil depth and the lowest in the 40-60 cm depth. The soil pH was made to reach the optimum level for plum trees in six years. The humus content reached the moderate supply level for plum trees, and the N_{TOT} content, although increased, showed that the soil was poorly supplied with this biogenic element. The highest increase was achieved in available K_2O and, particularly, P_2O_5.

5. ACKNOWLEDGEMENTS

This study is part of the 02E40 project financially supported by the Ministry of Science of the Republic of Serbia. We hereby express our sincerest gratitude to them for their financial support. Special thanks to Ms. Jelena Krstic, Faculty of Agronomy, Cacak, for translating the paper into English.

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Received at: 06. 08. 2009
Revised at: 20. 10. 2009
Accepted for publication: 28. 12. 2009
Published online at: 13. 01. 2010