Effect of waste Al-phosphate on soil and plant

N.R. Amaizah[1], D. Cakmak[2], E. Saljnikov[2], G. Roglic[1], N. Kokovic[2], D. Manojlovic[1]

[1]*Faculty of Chemistry, University of Belgrade, Belgrade, Serbia*
[2]*Institute of Soil Science, Belgrade, Serbia*

ABSTRACT

Irreplaceability of phosphorus as a necessary macroelement in crop production is due to limited resources and costly processing of ores and immobilization in soil, which force for seeking an alternative sources or the use of waste materials. In this paper, the waste aluminum phosphate from pharmaceutical factory used as phosphate fertilizer and its effects were compared with other phosphorus fertilizers (superphosphate and rock phosphate). Except the analysis of available phosphorus (AL-method) the sequential extraction of phosphorus (modified Chang and Jackson) and sequential extraction of aluminum (modified Tessier) were performed. The experimental plant was mustard (*Sinapis alba*). The pot experiment was carried out on two soil types: Stagnosol and Vertisol. Application of phosphorus with aluminum phosphate had the same effect as the application of other phosphatic fertilizers in both soil types. In Stagnosol Al-phosphate directly influenced the increase in plant fresh weight by 39% and dry weight by 43% compared to the control, and also decreased the content of mobile Al for 40% and Pb for 47% in plant biomass. Based on these results, the use of waste aluminum phosphate has a potential to be used as a phosphorus fertilizer under given conditions.

Keywords: macroelement; fertilizer; phosphorus; aluminum; plant biomass

Phosphorus (P) has an indispensable role in the growth and development of plants. The amount of total P in soil varies from 50 to 5000 mg/kg in top 0–30 cm soil (Mengel and Kirkby 2001, Saljnikov and Cakmak 2012). However, compared with the other major nutrients, the P is the least mobile and less available to plants in most soil conditions.

Due to limited resources of phosphorus, its expensive ore processing, and immobilization in soil there are many attempts to use waste material (sewage sludge) (O'Riordan et al. 1987) and rock phosphate (Bolland et al. 1988) as source of P-fertilizer. The use of waste materials may result in aggravation of soil properties and can threat crop production with harmful consequences for end users. Therefore, before using such materials it is necessary to implement the experiments, which may reveal the beneficial and harmful effects of fertilization with waste materials, especially, considering the complexity of the behavior of P in soil (Matula 2010).

The use of aluminum phosphate is not usual because of possible harmful effect of Al on soil and plant especially in acid environment (Kabata-Pendias and Pendias 2001). The aim of this research was to find out the possibility of using the waste Al-phosphate as a fertilizer. For this purpose the detailed sequential analyses of different fractions of P and Al in soil was performed to reveal the effect of application of waste aluminum phosphate on two soil types, Stagnosol and Vertisols, and the quality and quantity of plant biomass.

MATERIAL AND METHODS

Two soil types, Stagnosol and Vertisol (WRB 2006) were chosen for the experiment from Varna at 44°41'38''N and 19°39'10''E and Bozurna at 44°13'42''N and 20°42'45''E experimental fields of the Institute of Soil Science, near Belgrade,

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Table 1. The initial soil characteristics of the studied soil types: Stagnosol and Vertisol (WRB 2006)

Soil type	pH (KCl)	pH (H_2O)	C_{org}	N_{tot}	$CaCO_3$	P_{AL}	K_{AL}
				(%)		(mg/kg)	
Stagnosol	3.85	5.10	1.51	0.13	0	58.9	103.3
Vertisol	6.38	7.04	2.28	0.20	0	107.4	229.0

Serbia. The Stagnosol soil from Varna is strongly acidic, with medium humus content, high content of available potassium and phosphorus. The Vertisol soil from Bozurna is weakly acidic, with medium humus content, with high amount of available potassium and phosphorus (Table 1).

The experiments were performed under semi-controlled conditions, where the soil moisture was controlled at WHC (water holding capacity), while the temperature and light were non-controlled and the same as in the surrounding environment, in three repetitions with mustard (*Sinapis alba* L.). The experiment was designed in 4 kg pots, with watering with distilled water. Except common phosphorus fertilizers (superphosphate and rock phosphate) the application of waste aluminum phosphate (WAP) in a form of gel from a pharmaceutical factory in Belgrade was studied (Table 2). The concentration of aluminum in WAP was 5.6%, concentrations of elements (in dry matter): Ca 322, Cd 0.16, Co 0.3, Cr 1.07, Cu 6.41, Fe 123, K 47, Mg 100.7, Mn 3.99, Ni 5, Pb 1.3, Sr 1.3, Zn 20.1 mg/kg.

Soil pH was determined with a glass electrode pH meter in a 1:2.5 distilled water and KCl (mol/L). Soil total C and N were measured with an elemental CNS analyzer, Vario model EL III (Elemental Analysis systems GmbH, Hanau, Germany). Available P and K were determined by the AL-method by Egner and Riehm (Djamic et al. 1996). Exchangeable Al was determined by the titration method after Sokolov (Jakovljevic et al. 1985). Speciation of phosphorus was done using sequential extraction procedure: a modified Chang and Jackson method (Manojlovic et al. 2007): step I – water soluble P (NH_4Cl mol/L); step II – Al bound P (NH_4F 0.5 mol/L); step III – Fe bound P (NaOH 0.1 mol/L); step IV – reducible P (Na dithionite, Na citrate 0.3 mol/L); step V – occluded P (NaOH 0.1 mol/L) and step VI – Ca bound P (H_2SO_4 0.25 mol/L).

Furthermore, the soil was subjected to a 5-phase sequential extraction by modified Tessier method in order to study the substrates of Al (Petrovic et al. 2009): step I – exchangeable (CH_3COONH_4 mol/L); step II – bound to carbonates, and easily reducible ($NH_2OH{\cdot}HCl$ 0.1 mol/L); step III – moderately reducible ($(NH_4)_2C_2O_4$ 0.2 mol/L and $H_2C_2O_4$ 0.2 mol/L); step IV – organic-sulphide (30% H_2O_2 + CH_3COONH_4 3.2 mol/L); step V – residual (HCl 6 mol/L).

For the analyses of plant material, one gram of sample was digested with mix of HNO_3 and H_2O_2 (8 mL of mixture at ratio HNO_3: H_2O_2= 7:1 was added per 1 g of plant material). The content of phosphorus and metals in all the extracts were determined by inductively coupled plasma atomic emission spectrometry, iCAP 6500 Duo (Thermoscientific, Cambridge, UK). For the verification of the results, the reference soil sample was determined for all metals ERM-CC135a Contaminated Brickworks Soils.

Statistical analyses were performed with SPSS version 16 software (Chicago, USA). The effect of

Table 2. The design of the experiment with application of different phosphate fertilizer at the two soil types in Serbia

Treatment		N (LAN) (kg N/ha)	Superphosphate	Rock phosphate	Waste Al-phosphate (WAP)
				(kg P/ha)	
1	0 (control)	0	0	0	0
2	nitrogen	120	0	0	0
3	superphosphate S	120	53	0	0
4	rock phosphate S	120	0	53	0
5	waste Al-phosphate S	120	0	0	53
6	waste Al-phosphate D	120	0	0	106

LAN – limestone ammonium nitrate (27%), 0.06 g per pot; superphosphate (7.86%), 0.9 g per pot; rock phosphate (11.35%), 0.616 g per pot; WAP (5.59%), 1.25 and 2.5 g, for 5 and 6 treatments, correspondingly; S – single dose of phosphorus; D – double dose of phosphorus

Table 3. The content of available P and exchangeable Al in soil after completion the experiment

Treatment	Stagnosol		Vertisol
	P_{AL}	$Al_{Sokolov}$	P_{AL}
	(mg/kg)		
0 (control)	$57.8^a \pm 1.3$	$10.26^a \pm 1.39$	$118.3^a \pm 18.2$
Nitrogen	$58.9^a \pm 1.6$	$9.74^a \pm 1.42$	$108.4^a \pm 0.8$
Superphosphate S	$82.9^b \pm 11.8$	$7.60^b \pm 0.20$	$125.9^a \pm 5.8$
Rock phosphate S	$89.2^b \pm 12.6$	$5.79^b \pm 1.31$	$151.1^b \pm 6.0$
Waste Al-phosphate S	$67.1^a \pm 3.0$	$6.86^b \pm 2.47$	$122.3^a \pm 25.0$
Waste Al-phosphate D	$65.7^a \pm 6.2$	$5.80^b \pm 1.67$	$127.0^a \pm 2.3$

The different letters denote the statistical difference at $P = 0.95$. S – single dose of phosphorus; D – double dose of phosphorus

treatments on all the variables was tested by the ANOVA software. Statistical differences between the treatments were determined using the *t*-test (95%) by Pearson for Fisher *LSD*. The significance of their correlations was analyzed by the Pearson correlation matrix (SPSS 2007).

RESULTS AND DISCUSSION

The content of available phosphorus (AL-method) increased only after the use of rock phosphate, which is quite available in acid soils (Mengel 1997) and superphosphate (Table 3). However, the sequential analysis (modified Chang and Jackson method) showed significant changes in P content. The first water-soluble phase of P is increased under P fertilization, while the lowest values were observed in the treatment fertilized with N only. The highest values were under application 106 kg P/ha of WAP. There was no difference between treatments with the same amount of P-fertilizer (Table 4).

In the Vertisol the influence of P-fertilizer on an increase of available P_{AL} was less pronounced since only rock phosphate increased its content in the soil (Table 3). It is probably due to the method procedure and the acidity of extraction solution with pH 3.7. The sequential analysis showed that major changes were observed in water soluble phase of P, with the highest values recorded in the treatment fertilized with WAP. The phase of P bound to aluminum in the treatments with WAP differs from other treatments and from each other depending on the amount of fertilizer used (Table 4).

The content of P in the plant material in both soil types and all treatments ranged within the usual limits 4 to 5 g/kg dry matter (Mengel and Kirkby 2001). The absence of correlation of P content in plant material with applied fertilizer is determined by sufficient amount of available P_{AL} in the initial state of soil (Table 1), which is confirmed by earlier studies (O'Connell and Grove 1985). Plant material grown on Stagnosol has significantly lower P content (4.24 g/kg) in the treatment where only nitrogen fertilizer was applied, which is probably due to the inability of plants to balance the concentrations of P with N that was available (Table 5).

Control plants in Vertisol have the highest content of P (5.22 g/kg) in dry material (Table 5), because the lack of fresh most available water-soluble P can lead to rapid and non-controlled uptake of phosphate (Clarkson and Scattergood 1982). However, increase in fresh biomass in Stagnosol clearly demarcates treatments that were fertilized with phosphorus from the others, in accordance with applied P, where the biggest increase in fresh plant weight was under a double dose of WAP. Also, in Stagnosol WAP application had the same affect on biomass similar to raw phosphate and superphosphate (Table 5). In Vertisol a high initial content of available P_{AL} in the soil disabled such pronounced effect. Only treatments with superphosphate and double amount of WAP were different from the treatment with only N fertilizer, which was responsible for the increase in dry yield (Poulton et al 1997).

The influence of P fertilization on phosphorus in the soil and the plant is clearly evident through the first and second phases of sequence analysis of phosphorus and it correlate with other indicators, such as P first phase with fresh plant mass ($P = 0.750^{**}$) and dry plant mass ($P = 0.864^{**}$), or P second phase with the fresh plant mass ($P = 0.773^{**}$) and with a dry plant mass ($P = 0.867^{**}$). Intertwining of the two phases is also confirmed on Stagnosol in the previous results (Amaizah et al. 2012).

Table 4. Sequential analyses of soil P (mg/kg) by modified Chang and Jackson method on Stagnosol and Vertisol at the P-fertilization experiment

Treatment	P phase I	P phase II	P phase III	P phase IV	P phase V	P phase VI
Stagnosol						
0 (control)	2.97^{a} ± 0.14	69.30^{ab} ± 4.6	431.11^{ab} ± 12.5	0.89^{ab} ± 0.2	22.03^{a} ± 2.9	17.66^{ac} ± 0.5
Nitrogen	2.29^{b} ± 0.2	66.59^{a} ± 2.6	415.46^{ab} ± 15.5	0.92^{ab} ± 0.2	21.51^{a} ± 1.2	16.92^{acd} ± 0.5
Superphosphate S	3.97^{c} ± 0.6	87.85^{cd} ± 5.8	439.93^{a} ± 19.2	1.06^{a} ± 0.2	21.24^{a} ± 2.3	17.77^{a} ± 1.1
Rock phosphate S	4.10^{c} ± 0.3	75.13^{b} ± 4.6	407.91^{b} ± 16.7	1.03^{a} ± 0.3	14.87^{b} ± 5.3	19.19^{b} ± 7.7
WAP S	3.51^{c} ± 0.3	82.96^{c} ± 2.5	402.54^{b} ± 23.6	0.66^{b} ± 0.12	13.51^{b} ± 0.4	16.52^{cd} ± 5.5
WAP D	4.78^{d} ± 0.4	93.26^{d} ± 1.3	417.69^{ab} ± 15.6	0.84^{ab} ± 0.2	17.92^{ab} ± 4.1	16.27^{d} ± 0.6
Vertisol						
0 (control)	6.40^{a} ± 0.5	59.84^{a} ± 2.5	172.09^{ab} ± 6.8	3.68^{a} ± 0.8	19.56^{a} ± 10.5	14.17^{ab} ± 0.3
Nitrogen	6.58^{a} ± 0.2	62.87^{a} ± 5.1	173.3^{ab} ± 1.9	3.93^{a} ± 0.2	30.19^{ab} ± 11.3	13.62^{a} ± 0.3
Superphosphate S	12.02^{b} ± 3.2	70.44^{a} ± 5.0	176.8^{ab} ± 5.3	2.70^{a} ± 0.3	30.30^{ab} ± 13.3	16.46^{b} ± 1.5
Rock phosphate S	7.94^{c} ± 0.9	61.83^{a} ± 2.6	167.0^{b} ± 11.7	3.67^{a} ± 0.8	28.92^{ab} ± 6.6	26.11^{c} ± 3.5
WAP S	11.12^{bc} ± 3.4	93.24^{b} ± 9.0	182.6^{a} ± 0.5	2.84^{a} ± 0.5	23.71^{ab} ± 8.6	15.22^{ab} ± 0.5
WAP D	11.64^{bc} ± 2.1	126.4^{c} ± 25.5	181.7^{a} ± 8.2	3.36^{a} ± 0.7	39.62^{b} ± 2.7	13.57^{a} ± 0.2

The different letters denote the statistical difference at $P = 0.95$. S – single dose of phosphorus; D – double dose of phosphorus; WAP – waste Al-phosphate

In the Vertisol positive correlations were found between water-soluble phase (phase I) and the fresh ($P = 0.535$*) and dry biomass ($P = 0.627$**). The higher biomass yield is due to the higher amount of freshly added P, which is represented by the phase I (Chang and Jackson method, Tables 4 and 5). In Stagnosol the content of exchangeable Al by Sokolov method was significantly reduced under P-fertilizer, where the lowest values were observed at the highest dose of WAP and rock phosphate (Table 3) due to the presence of CaO in WAP and $CaCO_3$ in the rock phosphate, because liming at low rates results in neutralization of exchangeable Al in soil solution (Mengel and Kirkby 2001). Sequential analysis of Al by the modified Tessier method showed that the amount of phase I of the most soluble Al were the lowest under WAP and statistically differed from the treatment with N fertilizer (Table 6). The reason of the increased content of Al phase I in the treatment with LAN is due to substitution of Al by NH_4 ions from fertilizer, where the greater availability of P did not intensify the root activity and thus the uptake of Al was reduced similar to the P-fertilization treatments. Such processes resulted in higher content of Al phase I in soil with LAN after finishing the experiment. This also affected the amount of Al in plant material, where control plants have significantly higher content.

In the Vertisol content of exchangeable Al was not recorded due to soil pH, because the Al release from the reserves starts mainly at the values of active acidity below 5.5, i.e. exchangeable below 4.5_{KCl} (Mrvić et al. 2007). For Vertisol the increased content of Al phase IV, which is the phase of complexation of Al ions, occurs under fertilization with the largest quantities of WAP (Table 6). In this soil plant biomass has the highest content of Al under double dose of WAP that significantly differed from control (Table 6).

Except chemical influences an immobilization of Al is subject to physiological influences from plant activity. The exchangeable Al determined by Sokolov was in a positive correlation with Fe ($P = 0.624$*) and Al ($P = 0.523$*) in plant material, which might be partly due to the applied phosphorus. However, further correlation between Al and Fe ($P = 0.985$**), and Al and P ($P = 0.489$*) in plant material, and statistically significant difference in the content of Al phase I (Tessier, Table 6) between the treatments with superphosphate and WAP, same as absence of difference in Al content in plant material under the mentioned treatments (Table 5) indicate that not only chemical immobilization of Al is taking a place in a soil. Absence of fresh phosphorus in Stagnosol (control) might result in an increased activity of plant roots e.g., increased exudates (malates, citrates, oxalates, piscidic acid, phytosiderophores), which is confirmed by statistically significant difference between the control and

Table 5. Fresh and dry biomass and the content of P, Al, Fe and Pb in dry biomass

Treatment	Fresh biomass	Dry biomass	P	Al	Fe	Pb
	(g)		(g/kg)		(mg/kg)	
Stagnosol						
0 (control)	$36.19^{a} \pm 0.18$	$3.28^{a} \pm 0.07$	$5.26^{a} \pm 0.39$	$0.78^{a} \pm 0.34$	$0.87^{a} \pm 0.28$	$2.02^{a} \pm 0.46$
Nitrogen	$44.05^{b} \pm 1.0$	$3.44^{a} \pm 0.34$	$4.24^{b} \pm 0.36$	$0.39^{b} \pm 0.06$	$0.50^{b} \pm 0.07$	$1.47^{b} \pm 0.28$
Superphosphate S	$49.58^{c} \pm 0.7$	$4.59^{b} \pm 0.19$	$4.99^{a} \pm 0.25$	$0.45^{b} \pm 0.06$	$0.57^{b} \pm 0.06$	$1.43^{bc} \pm 0.16$
Rock phosphate S	$50.36^{c} \pm 0.6$	$4.52^{b} \pm 0.03$	$4.94^{a} \pm 0.61$	$0.36^{b} \pm 0.02$	$0.48^{b} \pm 0.06$	$1.23^{bc} \pm 0.16$
WAP S	$50.36^{c} \pm 0.6$	$4.68^{b} \pm 0.37$	$5.12^{a} \pm 0.36$	$0.42^{b} \pm 0.06$	$0.51^{b} \pm 0.08$	$1.08^{c} \pm 0.16$
WAP D	$57.13^{d} \pm 0.7$	$5.79^{c} \pm 0.26$	$5.23^{a} \pm 0.10$	$0.46^{b} \pm 0.10$	$0.56^{b} \pm 0.07$	$1.10^{c} \pm 0.10$
Vertisol						
0 (control)	$26.83^{a} \pm 0.2$	$4.15^{a} \pm 0.13$	$5.22^{a} \pm 0.28$	$0.22^{ab} \pm 0.05$	$0.27^{a} \pm 0.05$	$0.69^{a} \pm 0.21$
Nitrogen	$51.64^{b} \pm 2.0$	$6.69^{b} \pm 0.72$	$4.69^{bc} \pm 0.38$	$0.28^{ac} \pm 0.04$	$0.36^{b} \pm 0.03$	$1.01^{ab} \pm 0.00$
Superphosphate S	$56.10^{c} \pm 1.9$	$7.70^{c} \pm 0.38$	$4.75^{bc} \pm 0.04$	$0.29^{bc} \pm 0.05$	$0.37^{b} \pm 0.06$	$0.91^{ab} \pm 0.24$
Rock phosphate S	$52.39^{b} \pm 2.4$	$6.83^{b} \pm 0.20$	$4.43^{b} \pm 012$	$0.29^{abc} \pm 0.02$	$0.35^{b} \pm 0.03$	$0.97^{ab} \pm 0.08$
WAP S	$53.87^{bc} \pm 1.8$	$6.98^{b} \pm 0.21$	$4.96^{ab} \pm 0.05$	$0.25^{abc} \pm 0.02$	$0.34^{ab} \pm 0.03$	$1.06^{ab} \pm 0.24$
WAP D	$53.22^{bc} \pm 0.9$	$7.63^{c} \pm 0.06$	$4.97^{ab} \pm 0.37$	$0.31^{c} \pm 0.06$	$0.39^{b} \pm 0.05$	$1.21^{b} \pm 0.24$

The different letters denote the statistical difference at $P = 0.95$. S – single dose of phosphorus; D – double dose of phosphorus; WAP – waste Al-phosphate

P-treatments and the content of Al in plants. The root exudates increase solubility of inorganic P, and also divalent and trivalent ions of metals, which resulted in the highest concentration of Al and Fe in the control treatment (Hisinger 2001). On the other hand, this effort requires certain energy from plants, which reflected in reduction of the plant biomass. Application of WAP led to an increase of

Table 6. Sequential analyses of soil Al (mg/kg) by modified Tessier method on Stagnosol and Vertisol at the P-fertilizer experiment

Treatment	Al phase I	Al phase II	Al phase III	Al phase IV	Al phase V
Stagnosol					
0 (control)	$3.46^{abc} \pm 1.14$	$378.66^{a} \pm 59.5$	$1588^{a} \pm 48.1$	$130.8^{a} \pm 41.5$	$11324^{a} \pm 128$
Nitrogen	$6.27^{a} \pm 3.2$	$411.23^{ab} \pm 58.5$	$1561^{a} \pm 75.1$	$121.5^{a} \pm 36.6$	$10492^{a} \pm 2107$
Superphosphate S	$4.23^{ab} \pm 1.97$	$468.12^{ab} \pm 130.4$	$1575^{a} \pm 89.5$	$100.6^{a} \pm 19.2$	$10776^{a} \pm 1268$
Rock phosphate S	$1.02^{bc} \pm 0.6$	$498.64^{ab} \pm 13.2$	$1556^{a} \pm 145$	$122.7^{a} \pm 12.7$	$10900^{a} \pm 86.5$
WAP S	$0.84^{c} \pm 0.7$	$889.09^{b} \pm 646.7$	$1571^{a} \pm 36$	112,.9$^{a} \pm 5.2$	$10352^{a} \pm 671$
WAP D	$1.23^{bc} \pm 1.8$	$445.76^{ab} \pm 92.6$	$1649^{a} \pm 73.7$	$138.6^{a} \pm 17.0$	$9799^{a} \pm 1535$
Vertisol					
0 (control)	$1.07^{a} \pm 1.00$	$306.89^{ab} \pm 11.3$	$1350^{ab} \pm 153$	$134.9^{a} \pm 16.3$	$11556^{a} \pm 295$
Nitrogen	$3.18^{a} \pm 0.9$	$297.46^{a} \pm 15.3$	$1362^{ab} \pm 112$	$104.2^{a} \pm 11.1$	$11010^{a} \pm 532$
Superphosphate S	$0.40^{a} \pm 0.3$	$290.73^{a} \pm 18.3$	$1352^{ab} \pm 70.7$	$132.6^{a} \pm 6.6$	$10587^{a} \pm 403$
Rock phosphate S	$1.00^{a} \pm 1.2$	$302.49^{ab} \pm 16.9$	$1291^{a} \pm 105$	$121.7^{a} \pm 19.7$	$10652^{a} \pm 468$
WAP S	$4.18^{a} \pm 7.0$	$309.51^{ab} \pm 5.2$	$1396^{ab} \pm 214$	$125.1^{a} \pm 31.0$	$10295^{a} \pm 2223$
WAP D	$0.87^{a} \pm 0.74$	$328.84^{b} \pm 1.3$	$1569^{b} \pm 12$	$183.2^{b} \pm 39.6$	$9990^{a} \pm 2591$

The different letters denote the statistical difference at $P = 0.95$. S – single dose of phosphorus; D – double dose of phosphorus; WAP – waste Al-phosphate the phase II of aluminum bound to carbonate that can be available under certain conditions.

In the Vertisol the initial amount of available and water-soluble P is double than in Stagnosol (Table 1), which results in decreased exudation by roots and, therefore, the absence of P fertilization did not lead to increased adsorption of aluminum in control (Table 5). Increasing Al content in the fertilizer evidently led to an increase of Al in plant material in Vertisol (Table 6).

In Stagnosol the content of Pb in plant was reduced because the phosphate decreased availability of Pb in the presence of Al in soil (Ma et al. 1994). The explanation for this phenomenon is partly due to the root exudation that helps to dissolve phosphorus, which is confirmed by positive correlation coefficient between Pb and Al (P = 0.668**) and between Pb and Fe (P = 0.729**) in plant material. In Vertisol the effect of Al on phosphorus solubility (Ma et al. 1994) is less obvious, same as reduced root exudation due to the availability of phosphate.

The application of waste Al-phosphate in both soil types showed similar concentrations of P and Al in soil and plant as under the use of other P-fertilizers used in the study, in Stagnosol, the WAP reduced the concentration of mobile Al in soil and concentrations of Al and Pb in plants same as other used P-fertilizers.

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Corresponding author:

Dr. Dragan Cakmak, Senior Scientist, Institute of Soil Science, Teodora Drajzera 7, Belgrade 110 00, Serbia
phone: + 381 698 223 524, fax: + 381 112 667 199, e-mail: cakmakd@yahoo.com