

# Aluminum Exchangeable and Phosphorous Availability on Ultisol Using Humic Substance and Synthetic Organic Acid

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## ABSTRACT

Humic substance (HS) extracted from composted agricultural waste contains organic acids that potential for an acid soil amendment. Functional group of COOH and OH in synthetically organic acid is higher than in HS originated from composted agricultural waste. Addition of synthetically organic acid to the HS may increase its functional group and therefore it will increase effectiveness in detoxifying aluminum (Al) and desorbing phosphorus (P) from an acid soil. Objective of research was to determine effect of synthetic organic acid addition to HS extracted from composted organic matter in desorbing P and detoxify Al. Research was conducted at Soil Laboratory of Agricultural Faculty of Jember University from February to May 2010. Treatment consisted of combination of four levels of HS concentration extracted from composted rice straw (0; 1,000; 2,000; and 5,000 mg kg<sup>-1</sup>) with two kind of synthetic organic acid: ethylenediaminetetraacetic acid (EDTA) and acetic acid (CH<sub>3</sub>COOH) at concentration of 5 mM. The treatment laid out in a randomized complete by design with three replications. Ultisol collected from Kentrong Banten was used in this research. Observation consisted of pH, Al and P concentration in the suspension at 0, 1, 2, 3, and 4 weeks after incubation. The result showed that EDTA or acetic acid treatment at concentration of 5 mM effectively increased soil pH and decreased exchangeable Al (exch-Al). However, EDTA was more reactive and having higher capability in increasing pH and detoxifying exch-Al than acetic acid. Humic substance extracted from composted rice straw at concentration of 1,000 to 5,000 mg kg<sup>-1</sup> also effectively increased soil pH, decreased exch-Al, and increased P availability during a 4 weeks incubation period. The HS effect in Al detoxification and P desorption in acid soil could be boosted by addition of EDTA or acetic acid synthetic

**Keywords:** Aluminum, humic substance, organic acid, phosphorus

## INTRODUCTION

Acid upland in Indonesia based on exploration soil map of Indonesia year 2000 is about 102.8 million hectare distributed mainly in Sumatera, Kalimantan, and Papua, but only 54.3% suitable for agriculture development (Balai Penelitian Tanah 2006). Major problems of acid upland are low pH and fertility, high exchangeable acidity (Ward *et al.* 2010), high aluminum (Al) saturation and monomeric Al (Heim *et al.* 2003). Low pH and high Al content significantly affect phosphorous (P) retention (Ige *et al.* 2007), and hence lower its availability.

Soil amelioration using agricultural lime decreased exchangeable Al (Rahman 2002). Organic matter (OM) addition is also effective for

acid soil amelioration. Humic acid as product of OM decomposition has an important role in controlling many reactions in the soil, and it depends on the amount and activity of functional groups, especially carboxylate (COOH) and hydroxide (OH) (Stevenson 1982; Ait Baddi *et al.* 2003). Application of composted rice straw increased soil pH and reduced exchangeable Al (Anwar *et al.* 2006). Aluminum toxicity in acidic soils could be reduced by application of feedlot manure and poultry litter (Tang *et al.* 2007). Peat and wheat straw application to Oxisol and Ultisol at the rate of 80 Mg ha<sup>-1</sup> decreased P adsorption (Yusran 2010). Addition of humic extracted from composted rice and soybean straw to solution containing 0.2 mM AlCl<sub>3</sub> reduced Al concentration in the solution (Winarso *et al.* 2010). Haynes and Mokolobate (2001) reviewed from a number of reports and concluded that additions of organic residues to acid soils can reduce Al toxicity and improve P availability, and two important groups in relation to Al toxicity and P

availability are soluble humic molecules and low molecular weight aliphatic organic acids.

Organic agriculture waste is abundance source of humic substance (HS). The HS extracted from composted rice and soybean straw contain high organic acid, especially acetic acid, oxalic acid, succinic acid, and citric acid (Winarso *et al.* 2010), hence it prospective as an acid soil ameliorant to detoxify Al. It also contains base cations (Na, Ca, K and Mg) with their proportion better than commercial one. The base cations containing in commercial humic acid are dominated by Sodium (Cerdan *et al.* 2007). Sodium is non essential nutrient for plant and on high concentration can disperse soil colloids and hence the soil becomes susceptible to erosion and compaction.

Concentration of COOH and OH functional groups per unit of molecular weight of HS is lower than in the synthetic organic acid especially ethylenediaminetetraacetic acid (EDTA). The EDTA have short carbon chain and therefore chelated-Al or metals is mobile in the soil and water bodies (Bedswort and Sedlak 2000; Cho *et al.* 2002). Contrary, humic compound have long carbon chain and therefore chelated-Al is relatively immobile. Molecular weight of humic acid extracted from peat vary from 4 to 50 kDa (Li *et al.* 2003). Addition of synthetic organic acid to humic compound might increase its functional group and hence will increase the activity that make more effective in detoxify Al and desorb P in acid soil. Reducing Al concentration will reduce lime rate.

Objective of research was to determine effect of synthetic organic acid addition to HS extracted from composted OM in desorbing P and detoxify Al.

## MATERIALS AND METHODS

### Site and Soil Description

Research was conducted in The Soil Laboratory of Agricultural Faculty, Jember University from February to May 2010. Humic substances used in the research were extracted from composted rice straw. Acidic soil used was Ultisol collected from Kentrong Banten with its characteristics were pH 4.8, exchangeable Al 6.02 Cmol kg<sup>-1</sup>, available P (Bray-1) 7.13 mg kg<sup>-1</sup> P, and organic-C 2.41%. Chemical characteristics of the HS are presented in Table 1.

### Compost Extraction

Composting and extracting rice straw was conducted without addition of any chemical

materials that could affect its characteristic. Extraction was done mechanically by pressing the composted material and dropped liquid was collected.

### Experimental Design

Treatments consisted of a combination of four levels of HS concentrations (0; 1,000; 2,000; and 5,000 mg kg<sup>-1</sup>) with ethylenediaminetetraacetic acid (EDTA) and acetic acid (CH<sub>3</sub>COOH) at concentration of 5 mM. The purpose of mixing is to increase or enrichment functional group of COOH concentration. The treatment laid out in a randomized completely design with three replications.

### Incubation and Observation

A twenty gram of soil was poured into 200 mL of bottle and added with the solution of HS and synthetic organic acid until the volume in the bottle reach 100 mL (proportion between soil and solute was 1 : 5). The suspension was incubated for four weeks. The suspension was shaken at speed of 250

Table 1. Characteristics of humic substance extracted from composted rice straw.

Variable	Value
Acetic acid (mg kg <sup>-1</sup> )	94
Citric acid (mg kg <sup>-1</sup> )	12
Oxalic acid (mg kg <sup>-1</sup> )	17
Propionic acid (mg kg <sup>-1</sup> )	6
Butyric acid (mg kg <sup>-1</sup> )	1
Succinic acid (mg kg <sup>-1</sup> )	16
Fumaric acid (mg kg <sup>-1</sup> )	11
Cetoglutamic acid (mg kg <sup>-1</sup> )	tt
pH	7.60
C-organic (%)	0.17
C/N ratio	15.0
N (g kg <sup>-1</sup> )	0.1
P <sub>2</sub> O <sub>5</sub> (g kg <sup>-1</sup> )	0.1
K <sub>2</sub> O (g kg <sup>-1</sup> )	0.6
NaO (g kg <sup>-1</sup> )	0.1
CaO (g kg <sup>-1</sup> )	0.5
MgO (g kg <sup>-1</sup> )	1.3
Fe (mg kg <sup>-1</sup> )	1.3
Cu (mg kg <sup>-1</sup> )	tt
Zn (mg kg <sup>-1</sup> )	2.38
Mn (mg kg <sup>-1</sup> )	679
Mo (mg kg <sup>-1</sup> )	71
B (mg kg <sup>-1</sup> )	250

tt = undetectable

rpm for two hours a day during a week in ambient temperature. Observation consisted of pH, Al and P concentration in the suspension at 0, 1, 2, 3, and 4 weeks after incubation. Available P (Bray-1 extractable) was determined using colorimetric and exch-Al *N* KCl extractable was measured by titration.

**Statistical Analysis**

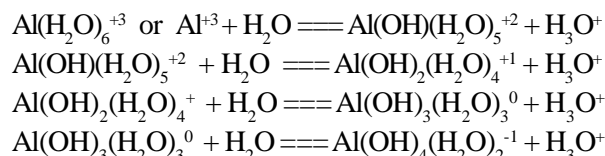
After normalization of the data, results of all experiments were analyzed by one-way analysis of variance (ANOVA) and then by Least Significance Different (LSD) at the significance level of 5%. Relationships among variables were analyzed using a regression and a correlation analysis.

**RESULTS AND DISCUSSION**

**Soil pH**

Addition of 5 mM EDTA or acetic acid increased soil pH started at 0 week after incubation (WAI) up to 4 WAI (Figure 1). The pH increased drastically in treatment EDTA after one week and gradually increased after one up to three WAI and then relatively constant after three WAI, however using acetic acid the pH still increased from three to four WAI even though the pH relatively similar at four WAI. The pH reaches 6.0 at one WAI with EDTA and at two WAI with acetic acid. It means that reaction between soil and acetic acid was slower than with EDTA and this was because EDTA contain functional group four times higher than that of acetic acid.

Every mole of EDTA (C<sub>10</sub>H<sub>16</sub>O<sub>8</sub>N<sub>2</sub>) has four carboxylate functional group (Reaves 2004), while acetic acid (CH<sub>3</sub>COOH) has one carboxylate functional group. Carboxylate functional group has an important role in reducing exchangeable Al concentration through chelation and precipitation, and as a result soil pH increase. Based on Lindsay (1979), precipitation reaction of Al<sup>+3</sup> due to increasing pH is as follow:



Ethilendiamintetraacetic acid addition increased pH by 0.8 unit at 0 WAI, and less than 0.2 unit at 1 and 2 WAI, and zero in the end of incubation. pH increased by using acetic acid was less than 0.4 unit at 0 WAI then increased to 0.6 unit at 1 and 2 WAI, and about 0.1 unit at the end of incubation (Figure 2). During four weeks incubation, total soil pH (initial soil pH was 4.8) increased by addition of 5 mM EDTA and acetic acid was 1.62 unit and 1.54 unit respectively. Its mean that EDTA was more reactive and having higher neutralizing pH capability than acetic acid.

Increasing HS concentration from 0 to 5,000 mg kg<sup>-1</sup> significantly increased soil pH in all incubation period, and higher pH gained at concentration of 5,000 mg kg<sup>-1</sup> (Table 2). The trend of HS effect on pH was still continued until four week period of incubation, and this effect was more or less similar to acetic acid. It may because of HS from composted rice straw contain 94% acetic acid (Table 1).

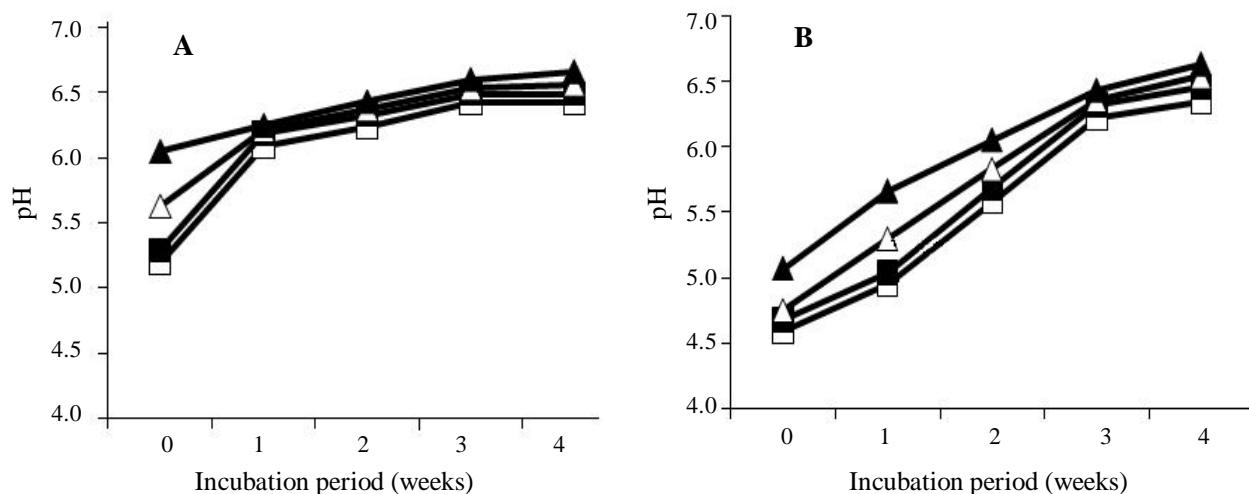


Figure 1. Effects of HS and 5 mM EDTA (A) or 5 mM acetic acid (B) addition on soil pH during four weeks incubation.  $\square$  = without HS,  $\blacksquare$  = + 1,000 mg kg<sup>-1</sup> HS,  $\triangle$  = + 2,000 mg kg<sup>-1</sup> HS, and  $\blacktriangle$  = 5,000 mg kg<sup>-1</sup> HS.

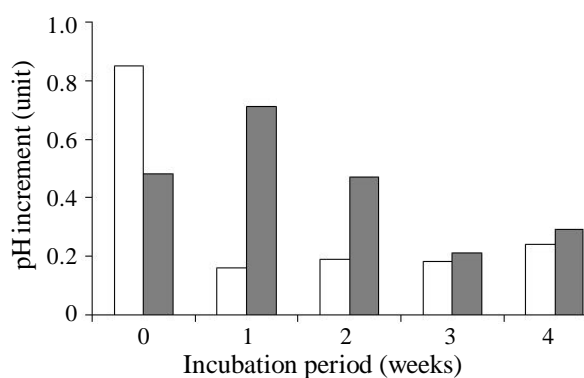


Figure 2. Effects of EDTA and acetic acid addition on soil pH increment during four weeks incubation. □ = 5 mM EDTA and ■ = 5 mM acetic acid.

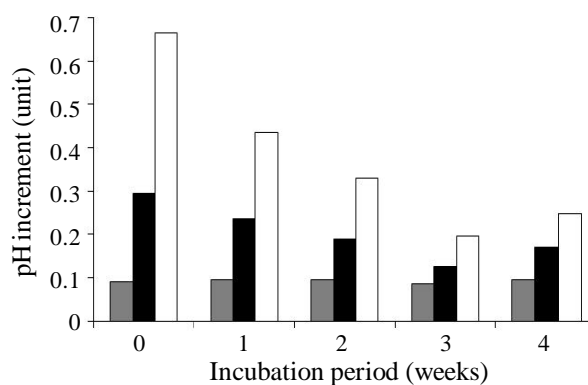


Figure 3. Effects of HS concentration on soil pH increment at variable incubation period. ■ = HS 1,000 mg kg<sup>-1</sup>, ■ = HS 2,000 mg kg<sup>-1</sup>, and □ = HS 2,000 mg kg<sup>-1</sup>.

Table 2. Effects of HS concentration on soil pH at various incubation periods.

Humic Substance Concentration (mg kg <sup>-1</sup> )	Soil pH <sup>1</sup>				
	0 WAI	1 WAI	2 WAI	3 WAI	4 WAI
HS 0	4.89 d	5.52 d	5.91 d	6.32 d	6.38 d
HS 1,000	4.99 c	5.61 c	6.00 c	6.40 c	6.48 c
HS 2,000	5.19 b	5.75 b	6.10 b	6.45 b	6.55 b
HS 5,000	5.56 a	5.96 a	6.24 a	6.51 a	6.64 a

Note: numbers in the same column with same letter is not significantly different at LSD test 5%

<sup>1</sup>WAI= week after incubation

The HS capability to change pH increased with increasing concentration, and averaged by 0.1, 0.2 and 0.4 unit at 1,000; 2,000 and 5,000 mg kg<sup>-1</sup>, respectively, in each incubation period compared to control (without HS addition). The HS addition at concentration of 1,000 ppm increased pH constantly in all incubation periods, while pH changed at concentration of 2,000 and 5,000 ppm was relatively high in 0 WAI and was likely to decrease linearly until 4 WAI (Figure 3). Total pH increased due to HS treatment at concentrations of 1,000, 2,000 and 5,000 ppm during 4 week incubations were 0.5, 1.0, and 1.9 units, respectively.

Combination of HS at various concentration with 5 mM EDTA and acetic acid gave more effect to pH, except with acetic acid at 0 WAI (Table 3). Comparing to initial soil pH (4.8), addition of 5 mM acetic acid decreased pH at 0 WAI and then increased after 1 WAI, except if it was combined with 5,000 mg kg<sup>-1</sup> HS. It means that acetic acid could acidify the soil just after addition and this effect could be neutralized by 5,000 mg kg<sup>-1</sup> HS. It was likely that it had correlated with COOH (source

of H<sup>+</sup> ion) per unit molecular weight which was higher in acetic acid than EDTA. Acetic acid have a molecular weight of 60.05 and pKA of 4.75 (Lindsay 1979), while EDTA have molecular weight of 372.24 and pKA of 1.99, 2.67, 6.16, and 10.26 (Dawson *et al.* 1969).

This result showed that humic substance, EDTA, and acetic acid individually can be used to increase soil pH, and combination of HS with EDTA or acetic acid make it more effective. This indicate that functional group (COOH) enrichment of HS can be done using EDTA or synthetic acetic acid.

### Exchangeable Aluminum

The HS treated with 5 mM EDTA or acetic acid significantly reduced exchangeable aluminum (exch-Al) in Ultisol. Addition of 5 mM EDTA reduced soil exch-Al drastically to zero value at one WAI and at two WAI with acetic acid (Figure 4). Its mean that EDTA was stronger than acetic acid in neutralizing exch-Al, and all exch-Al neutralized between 1 to 2 WAI. Its might because EDTA have more functional group per molecular weight unit than acetic acid.

Table 3. Effect of combination between HS with EDTA and acetic acid to the soil pH change.

Treatment combination	pH increase compare to initial soil pH (unit)				
	0 WAI	1 WAI	2 WAI	3 WAI	4 WAI
5 mM EDTA	0.40	1.29	1.44	1.62	1.62
5 mM EDTA + 1,000 mg kg <sup>-1</sup> HS	0.49	1.39	1.53	1.69	1.69
5 mM EDTA + 2,000 mg kg <sup>-1</sup> HS	0.83	1.41	1.57	1.73	1.76
5 mM EDTA + 5,000 mg kg <sup>-1</sup> HS	1.25	1.45	1.63	1.80	1.86
5 mM Acetic acid	-0.21	0.15	0.78	1.42	1.54
5 mM Acetic acid + 1,000 mg kg <sup>-1</sup> HS	-0.12	0.24	0.88	1.52	1.66
5 mM Acetic acid + 2,000 mg kg <sup>-1</sup> HS	-0.05	0.50	1.03	1.56	1.74
5 mM Acetic acid + 5,000 mg kg <sup>-1</sup> HS	0.27	0.86	1.25	1.63	1.83

Note: WAI= week after incubation; initial soil pH= 4.8.

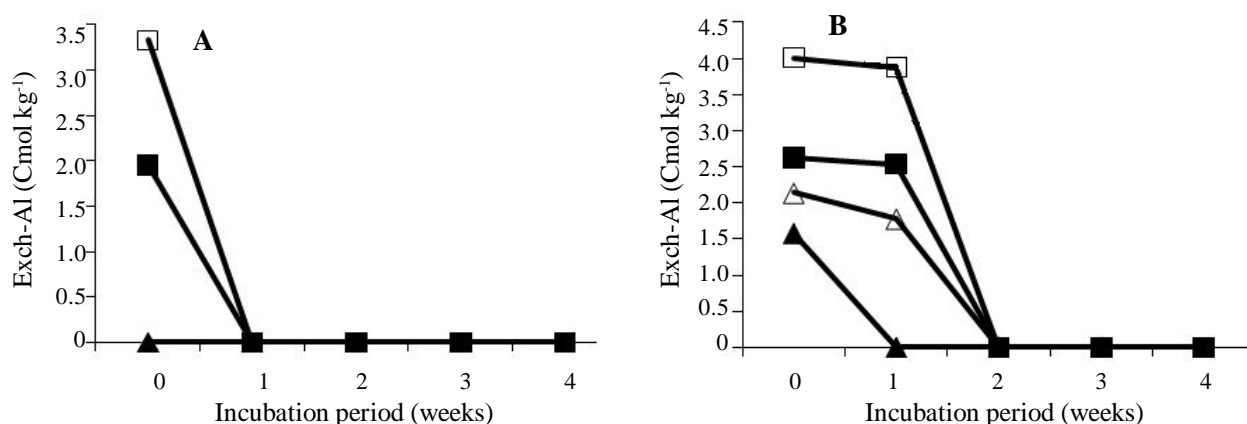


Figure 4. Effects of HS and 5 mM EDTA (A) or 5 mM acetic acid (B) addition on exch-Al during four weeks incubation.  $\square$  = without HS,  $\blacksquare$  = + 1,000 mg kg<sup>-1</sup> HS,  $\blacktriangle$  = + 2,000 mg kg<sup>-1</sup> HS, and  $\blacktriangle$  = 5,000 mg kg<sup>-1</sup> HS.

Ethilendiamintetraacetic acid treated with the HS at concentration of 2,000 mg kg<sup>-1</sup> reduced exch-Al from 6.02 Cmol kg<sup>-1</sup> (initial soil) to very low level (until undetectable) at 0 WAI, but need more time with acetic acid. Combining 5 mM EDTA with 1,000 mg kg<sup>-1</sup> HS had similar effect in neutralizing exch-Al with treatment 5 mM acetic acid combined with 5,000 mg kg<sup>-1</sup> HS (Figure 4). This result indicated that the HS addition at concentration 1,000 to 5,000 mg kg<sup>-1</sup> could make EDTA and acetic acid more effective in neutralizing exch-Al.

Reduction of exch-Al in Ultisol treated with HS, EDTA and acetic acid as such correlated with pH increment. The relationship between pH and exch-Al at 0 and 1 WAI presented in Figure 5, while at 2 to 4 WAI did not calculate since exch-Al reached zero value. There was quadratic relationship between exch-Al and pH at 0 WAI as well as at 1 WAI with equation  $Y_0 = 18.78 X^2 - 186.62 X + 464.85$  ( $R^2 = 0.92$ ) and  $Y_1 = 17.76 X^2 - 199.84 X + 562.32$  ( $R^2 = 0.98$ ), respectively. The

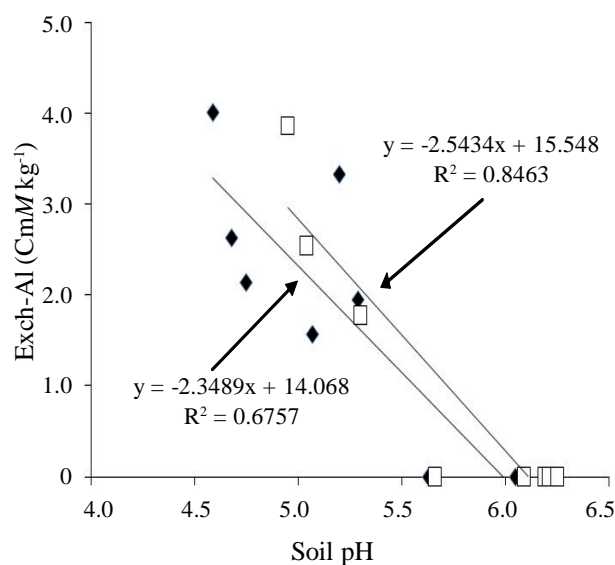


Figure 5. Relationships between pH and exch-Al during one week incubation.  $\blacklozenge$  = 0 week and  $\square$  = 1 week.

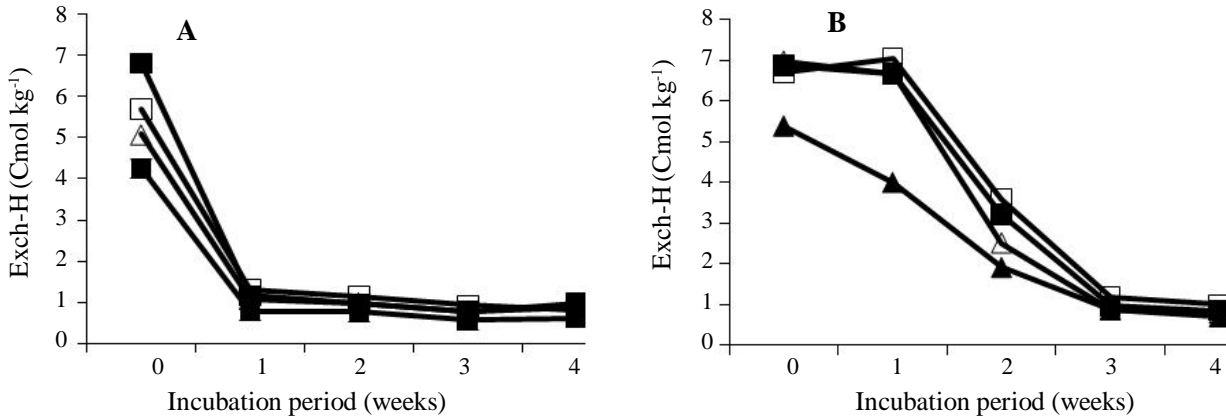


Figure 6. Effect of HS and 5 mM EDTA (A) or 5 mM acetic acid (B) addition on exchangeable H<sup>+</sup> ion during four weeks incubation.  $\square$  = without HS,  $\blacksquare$  = + 1,000 mg kg<sup>-1</sup> HS,  $\blacktriangle$  = + 2,000 mg kg<sup>-1</sup> HS, and  $\bullet$  = 5,000 mg kg<sup>-1</sup> HS.

Figure 5 indicated that exch-Al will netralize at pH around 5.5. This result was comparable with some researches as reviewed by Lindsay (1979). According to Loboda and Wolejko (2006)

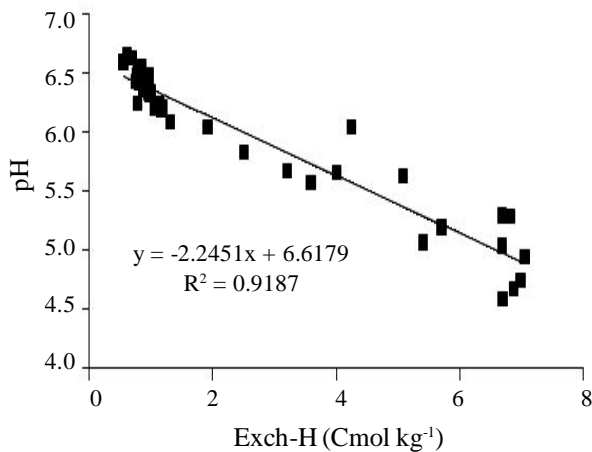


Figure 7. Relationship between exchangeable H<sup>+</sup> ion concentration and pH.

phosphorus and magnesium content in dry matter of the leaves barley decreased if Al concentration in growing medium increased.

**Exchangeable H<sup>+</sup> (Exch-H)**

Exchangeable Al is one of major ion H<sup>+</sup> buffer on acidic soil. In the soil solution system, Al ion (Al<sup>3+</sup>) surrounded by six H<sub>2</sub>O molecules that can dissociate and release six H<sup>+</sup> ions each one Al atom. The H<sup>+</sup> ion dissociation depends on concentration and pH (Hillel *et al.* 2004). Figure 6 indicates that HS, EDTA and citric acid treatments reduced H<sup>+</sup> ion concentration, and EDTA individually or in combination with HS gave more effect in reducing H<sup>+</sup> ion than acetic acid. In acidic soil, exch-Al and H<sup>+</sup>, and pH are interrelated each other, hence change in pH will affect H<sup>+</sup> ion (Figure 7) and exch-Al, and so the reverse.

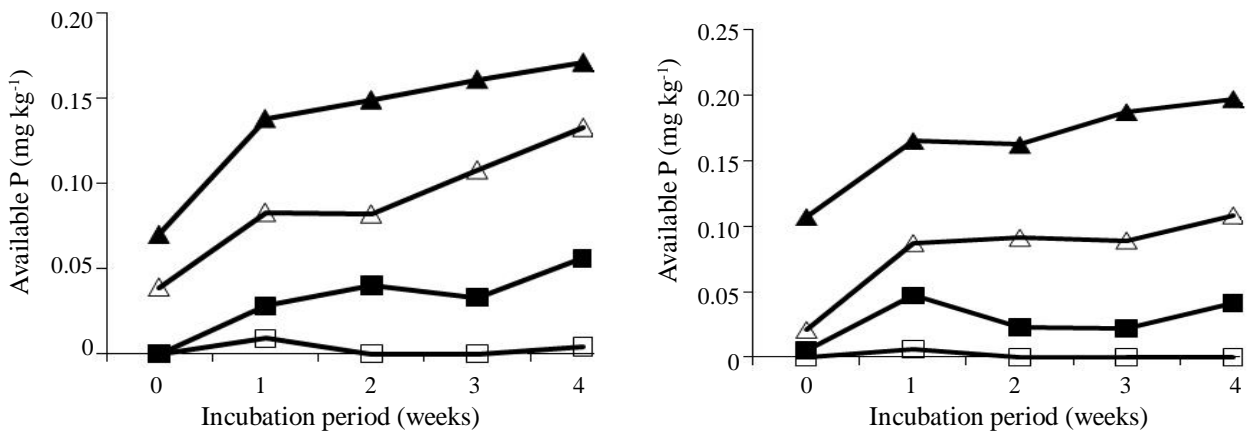


Figure 8. Effects of HS and 5 mM EDTA (A) or 5 mM acetic acid (B) addition on available P during four weeks incubation.  $\square$  = without HS,  $\blacksquare$  = + 1,000 mg kg<sup>-1</sup> HS,  $\blacktriangle$  = + 2,000 mg kg<sup>-1</sup> HS, and  $\bullet$  = 5,000 mg kg<sup>-1</sup> HS.

## P Availability

Ethilendiamintetraacetic acid or acetic acid treatment at concentration of 5 mM could not increase available P, but when they combined with HS at various concentration as such increased P availability (Figure 8). Availability of P higher on HS treatment combined with acetic acid than with EDTA at all incubation periods, especially at HS concentrations of 2,000 and 5,000 mg kg<sup>-1</sup> (Figure 8). Figure 8 also indicates that P availability increased as incubation period increased from 0 to 4 WAI. It means that HS can effectively desorb P from adsorption site, and desorption is higher when it is combined with acetic acid than with EDTA. Exch-Al is high at low soil pH hence increase its activity and it can desorb P and causing low P availability. Addition of HS, EDTA or acetic acid reduced exch-Al and therefore increased P availability. Hua *et al* (2008) reported that addition of 0.5 g and 2.5 g HS to soil containing 0.31 P kg<sup>-1</sup> increased concentration of water extractable-P by 70% and 360%; and labile-P (HCO<sub>3</sub>-extractable) by 400% and 540%, respectively compared to without HS addition.

## CONCLUSIONS

Ethilendiamintetraacetic acid or acetic acid treatment at concentration of 5 mM effectively increased soil pH and decreased soil exch-Al, but it did not increase P availability during 4 weeks incubation periods. Ethilendiamintetraacetic acid was more reactive and having higher capability in neutralizing soil pH and detoxifying soil exch-Al than acetic acid. Humic substance extracted from composted rice straw at concentration ranging from 1,000 to 5,000 mg kg<sup>-1</sup> effectively increased soil pH, decreased soil exch-Al, and increased P availability during 4 week incubation periods. This effect increased according to its concentration. Combining HS with EDTA or acetic acid gave more effect to increase soil pH, decrease exch-Al, and increase P availability. Effect of HS in Al-detoxification and P desorption P in acid soil can be boosted by addition of EDTA or acetic acid synthetic.

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