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Research Article

Improving Upland Acid Soil Properties And Increasing Maize Yield By Phosphate Rock Application With Organic Acids

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Problems encountered in the management of upland acid soils, besides soil pH, are also low in phosphorus (P) content and less available to plants. The addition of organic acids (OAs) to the direct application of phosphate rock (PR) has not been widely studied yet and is expected to improve upland acid soil properties. The research aims to determine the effect of adding OAs on the direct application of PR to improve upland acid soil and increase maize yield. It was a greenhouse experiment using a randomized complete design in a factorial arrangement with three replications. The treatments consisted of four types of OA (citric, humic, succinic, and oxalic acid) and five OA concentration levels (0, 25, 50, 100, and 200 ppm). The hybrid maize (*Zea mays* L) was used as the test crop. The parameters observed were soil properties (pH, exchangeable H (H^+), exchangeable Al (Al^{3+}), exchangeable Ca (Ca^{2+}), cation exchange capacity (CEC), available P (as P-Bray1)) and maize growth (plant heights and yield). The results show that the types of OA treatments had no significant effect on soil properties and maize yield; in contrast, the concentrations of OA levels were able to improve soil properties as indicated by a significant effect on pH, H^+, Al^{3+}, Ca^{2+}, CEC, available P, and increased maize yield. An increase in soil pH due to the addition of OAs to the direct application of PR could increase Ca^{2+} and CEC and decrease Al^{3+} and H^+, while increasing available P for plants. Although not significantly different, oxalic acid was able to improve the observed soil properties and maize yield, followed by citric, humic, and succinic acids. The maximum OA concentration was 126.9 ppm. We can say that the main finding of this research is that oxalic, citric, humic, and succinic acids at a concentration of 126 ppm can be applied to phosphate rock. In Indonesia, humic acid has been widely applied by farmers because it is easy to obtain. Therefore, it can be applied to phosphate rock.

1. Introduction

Acid soil upland area with a pH < 5.5 covers about 107.36 million ha or 74.31% of 144.47 million ha of the total dry land in Indonesia. According to the parent material and climatic conditions, most acid-dry lands are in Kalimantan, Sumatra, and Papua [1]. Based on soil taxonomy, red yellow podzolic soils dominate dry acid soils, subdivided into Ultisols, Oxisols, and Inceptisols [2]. Acidic soils have acidic pH, high Al content, and P fixation since soluble inorganic phosphorus is fixed by Al and iron (Fe) [3, 4]. P nutrient is one of the essential macronutrients for plant growth and is often a limiting factor for plant production. It is a problem for soil fertility; besides the total P content in the soil being low, it is also not available to plants and is easily converted into insoluble compounds. The low total P content of the soil is related to parent material and advanced weathering [5, 6].

Manufactured water-soluble P fertilizers such as superphosphate are generally recommended to correct P deficiency [7, 8]. In addition, the intensification of agriculture requires additional P inputs to increase crop production and improves the P status of the soil to prevent further soil degradation. Therefore, it is essential to explore alternative P inputs. Under certain soil and climatic conditions, direct application of PR is an agronomic, economic, and environmental alternative to the more expensive superphosphates in the tropics [9–11]. Szilas et al. [12] found that granulated PR in the direct application can replace TSP as a P fertilizer on acid soils low in available P and exchangeable Ca in subhumid to humid in Tanzania.

Phosphate rock is the only important raw material for fertilizer P, and the general formula for phosphate is $Ca_{10}(PO_4)_6(X)_2$, where X is F^-, OH^-, or Cl^-. PR is not only a source of P but also Ca, and it contains several essential nutrients such as magnesium (Mg), sulfur (S), iron (Fe), copper (Cu), zinc (Zn), and other heavy metal elements [13, 14]. The solubility in the soil solution will be better when the soil pH, exchangeable Ca, and P concentration in the soil solution are low [15]. Good quality PR is often called reactive PR, which contains more than 12.5% P and has high reactivity with P levels dissolved in 2% citric acid more than 6%. Meanwhile, PR with P 9.81% can substitute a chemical P fertilizer [13, 15].

In upland acid soils requiring a lot of P, direct application of PR is considered more effective and cheaper than other forms. This is because PR in acidic soils is more reactive and cheaper than superphosphate [16, 17]. The direct application PR has low solubility compared with inorganic fertilizers. An effort is needed to increase their solubilities, such as the use of manure/organic or microbial materials, thereby improving the chemical properties of soil and crop yield [18–25]. OAs such as citric, malonic, oxalic, tartaric, and ketogluconic acids released by the roots can chelate Al and Fe ions to release P into the available form by replacing the position of the phosphate anion. Combined with cations Ca, Al, and Fe, they form complex compounds that are difficult to dissolve [26, 27]. Some of the OAs produced by bacterial and fungal activity are citric, malic, fumaric, succinic, pyruvic, tartrate, oxaloacetic acids, 2-ketogluconate, lactosalt, propionate, and butyrate [28–31].

Many researchers have described the importance of OAs in improving soil chemical properties and increasing crop productivity. Al toxicity is a major factor limiting crop productivity. The Al-induced secretion of OAs anions, mainly citrate, oxalate, and malate, from roots is the best-documented mechanism of Al tolerance in higher plants [32, 33]. OAs could improve P availability by enhancing the release of inorganic P in the soil. Oxalic and malic acids more effectively and least effectively released Pi than citric acid [34], while according to Tumbure et al. [35], oxalic acid was more effective at solubilizing P from the direct application of PR than citric and acetic acids. Low molecular weight OAs (LMWOAs) secreted by plant roots enhanced the release of inorganic P (Pi) and organic P (Po) into the soil solution and thereby increased plant-available Pi in soils. Inorganic P and organic P were released by LMWOAs, followed by oxalic acid > citric acid > malic acid if the substrates of P and LMWOAs were enough. LMWOAs can significantly promote P availability in acidic soils [34, 36–38]. The P removal efficiency was worst affected by citric acid in the optimal pH range of 9.0–10.5, followed by succinic acid and acetic acid [37]. In addition, Teixeira et al. [39] reported that the coating with OAs reduced the water solubility of the P fertilizers-mono ammonium phosphate (MAP), providing a more gradual release of P over time. The agronomic efficiency (AE) of P applied as the coated-slow-release fertilizers were 3 to 13% greater than that of MAP. Furthermore, Ch'ng et al. [3] reported organic amendments altering soil chemical properties increase phosphorus availability.

The abovementioned results show that OAs could improve the soil properties and increase crop productivity, through the increasing pH, and chelation of Al, Fe, and Ca ions to release P to the available form. However, the types and the most influential concentrations of OA for PR application increase the availability of P nutrients and maize yield are unknown. Departing from the background of the problem, a study is needed to determine the type and concentration of OA treated by the PR application affecting the soil properties, and growth and yield of maize in upland acid soil. The research objective was to determine the effect of adding organic acids to the direct application of PR on changes in the properties of upland acid soil for maize.

2. Materials and Methods

2.1. Methods. The research was carried out at a greenhouse of Indonesia Soil Research Institute in Bogor by using Typic Plinthudults from South Lampung Regency, Lampung Province, between June and October 2021. The experiment used a factorial complete randomized design (RCD) consisting of two factors (Table 1). The treatments consisted of four types of OA (A1: citric acid, A2: humic acid, A3: succinic acid, and A4: oxalic acid) and five concentrations of OA levels (D0: 0, D1:25, D2: 50, D3: 100, and D4: 200 ppm), so that 20 treatment combinations were obtained and each was repeated three times; it needed 60 pots. The PR was given to the entire experimental pots, and the hybrid maize (*Zea mays* L) variety of Pioneer 2 was used as the indicator crop.

Soil material of 15 kg absolute dry weight was placed in a pot, mixed with PR, and incubated for 10 days. After completion of the incubation, 3 maize seeds were planted in each pot. OAs were given by inserting them into the maize planting hole. After the plants were one week old, they were selected and two plants were allowed to grow. Subsequently, urea equivalent to 400 kg·ha^{-1} was given three times, namely, 1/3 of each at 10 days after planting (DAP), 21 DAP, and 35 DAP. KCl equivalent to 150 kg·ha^{-1} was given at planting; then, the plants were maintained until the maize was harvested at 105 DAP.

Observations of plant growth were carried out at 14, 28, 42, and 56 DAP. The parameters observed for plant growth were plant height. The heights of the two plants were measured from the base to the tip of the leaf; then, the results were averaged. After the plants were harvested at 105 DAP, the observed parameter was the dry weight of the seeds. Corn kernels were peeled and dried to a moisture content of 14%.

Table 1: Combination of the treatments in a factorial complete randomized design.

No.	Factor 1: concentrations of OA levels	Factor 2: types of OA			
		Citric acid (A1)	Humic acid (A2)	Succinic acid (A3)	Oxalic acid (A4)
1	0 ppm (D0)	A1D0	A2D0	A3D0	A4D0
2	25 ppm (D1)	A1D1	A2D1	A3D1	A4D1
3	50 ppm (D2)	A1D2	A2D2	A3D2	A4D2
4	100 ppm (D3)	A1D3	A2D3	A3D3	A4D3
5	200 ppm (D4)	A1D4	A2D4	A3D4	A34D4

Soil samples were carried out at 7 days after planting (DAP), 14 DAP, 28 DAP, and at harvest (105 DAP) by taking soil samples from each pot by using a soil drill. Soil samples were taken in each treatment pot from 5 points around the plant, then composited so that there were 60 samples from each observation. After that, the soil samples were dried and ground to be analyzed for chemical properties in the laboratory.

The chemical properties of the soil observed were pH, exchangeable acidity ($Al^{3+}$and H^{3+}) by KCl 1M, Ca^{2+} and CEC by NH_4OAc 1M, and available P by the Bray 1 method [40]. Plant growth was observed at 14, 28, 42, and 56 DAP, and at harvest time, the dry weight (DW) of seeds was observed. This experiment used PR from Morocco and used organic produced by the factory.

To determine the effect of OAs application on maize growth and changes in soil nutrients, an analysis of variance (ANOVA) was carried out using the SPSS software (version 25; SPSS Inc.). Analysis with an accuracy level of 5% was conducted based on the Duncan multiple range test (DMRT) to obtain the differences between treatments [41].

The maximum concentration of OA was calculated using the procedure suggested by Heady et al. [42]. The maximum concentration is determined by (a) calculating the quadratic polynomial regression equation of the maize yield to OA concentration ($Y = a + b\ x - c\ x^2$) and (b) determining the maximum concentration of OA by calculating the first derivative of the regression equation ($Y' = 0$).

3. Results

3.1. Soil Characteristics and Phosphate Rock. The physical and chemical property analyses showed that soil from Jati Agung, South Lampung Regency, was classified into Typic Plinthudults or Podsolik Plinthic [2, 43]. The physical properties of the texture belong to the dusty clay class. In contrast, the chemical properties are consistent with reference [40], where the soil is classified as acid soil (pH H_2O was 4.8), with high Al saturation (30.37%) and exchangeable Al of 1.38 $cmol_e^+ \cdot kg^{-1}$ and moderate C-organic content. The potential P content (as P-HCl) should be relatively moderate (28 ppm), with a low to medium available P of 7.67 ppm and low CEC at 7.77 $cmol_e^+ \cdot kg^{-1}$ with low exchangeable Ca (2.25 $cmol_e^+ \cdot kg^{-1}$). This shows that the productivity is low, and one of the reasons is the P content of the soil. The PR contains 20% P_2O_5, 17% Ca0, 6.39% P_2O_5, and 2% citric acid. The particle size mesh at 25% and 80% is 87.08% and 41.76%. Based on the analysis results, the PR used can be classified as reactive [13].

3.2. The Application of Organic Acids Improves Soil Properties. The changes in the soil chemical properties on average from 4observations are shown in Tables 2–7. In general, the treatment of OA types had no significant effect on pH H_2O, H^+, Ca^{2+}, CEC, Al^{3+}, and available P. On the other hand, the treatment of OA concentration levels had a significant effect on all parameters.

Table 2 presents the effect of OAs application on PR to the soil pH H_2O. The treatment of OA types had no significant effect on the soil pH H_2O, in contrast to the treatment of OA concentration levels, which showed a significant effect. Although not significantly different, oxalic and citric acids increased soil pH higher than humic acid. Citric and oxalic acids have lower molecular weights than humic acid, so they have higher mobility and greater acidity. With these properties, the ability to form cation exchange complexes is greater. This cation exchange increased in –OH ions in the soil solution so that pH H2O increased.

PR application increased soil pH H_2O, as indicated by an increase in soil pH from 4.8 (soil pH H_2O before applying the treatment) to 4.95 (D0) or an increase of 0.15 units. The increase in soil pH was mainly due to the hydrolysis of carbonates contained in PR. The use of H^+ ions in the soil solution by the anions resulting from the hydrolysis of Al-Fe would reduce the amount of H^+ in the soil solution. The use of H^+ could be interpreted as a decrease in the level of H^+ in the soil solution. On the other hand, by increasing the level of OH^- in the soil solution, by increasing the presence of OH^-, the pH would increase. Chien [10] reported that the effect of PR on changes in soil pH varies greatly depending on the amount and reactivity of PR, as well as free carbonate content. The amount of free carbonate increases carbonate hydrolysis. The use of H^+ ions in the soil solution by anions resulting from hydrolysis of Al-Fe due to the addition of PR is indicated by a decrease in H^+ ions as presented in Table 3, where H^+ ions decreased from 0.31 to 0.11 or decreased by 64.5%.

The increase in soil pH was also caused by an increase in the concentration of OA levels. Soil pH H_2O increased from 4.95 (D0) to 5.25 (D4) or increased by 0.30 units. The addition of OA concentration to the soil would cause a ligand exchange reaction between organic anions and free -OH. The increased concentration of OA levels will be followed by an increase in cation exchange capacity (CEC) due to the carboxyl group of OAs capable of binding cations. This cation exchange increased -OH increased so that the soil pH H_2O increased. Ch'ng et al. [3] reported that organic amendments increased soil pH and reduced exchangeable acidity, exchangeable Al, and exchangeable Fe.

Table 2: Effect of organic OA application on phosphate rock to pH H_2O average from 4 observation times.

No.	Concentration of OA (ppm)	Types of OA A1: citric acid	A2: humic acid	A3: succinic acid	A4: oxalic acid	Average
1	D0: 0	4.97 ± 0.08	4.90 ± 0.09	4.92 ± 0.05	5.01 ± 0.08	4.95 ± 0.04a
2	D1: 25	5.03 ± 0.08	4.91 ± 0.08	4.97 ± 0.04	5.05 ± 0.08	4.99 ± 0.04a
3	D2: 50	5.06 ± 0.08	4.96 ± 0.08	4.94 ± 0.08	5.05 ± 0.08	5.00 ± 0.04a
4	D3: 100	5.26 ± 0.08	5.21 ± 0.08	5.16 ± 0.07	5.21 ± 0.08	5.21 ± 0.04b
5	D4: 200	5.22 ± 0.08	5.25 ± 0.08	5.25 ± 0.04	5.30 ± 0.08	5.25 ± 0.04b
6	Average	$5.11 \pm 0.04p$	$5.05 \pm 0.04p$	$5.04 \pm 0.04p$	$5.12 \pm 0.04p$	(—)

Data are presented as mean values with standard error of the mean followed by the same letters are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

Table 3: Effect of OA application on phosphate rock to H^+ average from 4 observation times.

No.	Concentration of OA (ppm)	Types of OA A1: citric acid	A2: humic acid	A3: succinic acid $cmol_e^+$	A4: oxalic acid	*Average*
1	D0: 0	0.31 ± 0.01	0.30 ± 0.01	0.28 ± 0.01	0.37 ± 0.01	0.31 ± 0.01d
2	D1: 25	0.20 ± 0.01	0.26 ± 0.01	0.29 ± 0.01	0.23 ± 0.01	0.25 ± 0.01c
3	D2: 50	0.22 ± 0.01	0.24 ± 0.01	0.25 ± 0.01	0.19 ± 0.01	0.23 ± 0.01c
4	D3: 100	0.21 ± 0.01	0.21 ± 0.01	0.22 ± 0.01	0.18 ± 0.01	0.21 ± 0.01b
5	D4: 200	0.11 ± 0.01	0.10 ± 0.01	0.08 ± 0.01	0.14 ± 0.01	0.11 ± 0.01a
6	Average	$0.21 \pm 0.01p$	$0.22 \pm 0.01p$	$0.23 \pm 0.01p$	$0.22 \pm 0.01p$	(—)

Data are presented as mean values of three replicates standard error of the mean (SEM) followed by the same letters are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

Table 4: Effect of OAs application on PR to Ca^{2+} average from 4 observation times.

No.	Concentration of OA (ppm)	Types of OA A1: citric acid	A2: humic acid	A3: succinic acid $cmol_e^+\ kg^{-1}$	A4: oxalic acid	Average
1	D0: 0	2.97 ± 0.09	2.93 ± 0.09	2.93 ± 0.09	2.97 ± 0.09	2.95 ± 0.05a
2	D1: 25	3.21 ± 0.09	3.12 ± 0.09	3.21 ± 0.09	3.20 ± 0.09	3.19 ± 0.05b
3	D2: 50	3.39 ± 0.09	3.42 ± 0.09	3.38 ± 0.09	3.41 ± 0.09	3.40 ± 0.05c
4	D3: 100	3.47 ± 0.09	3.46 ± 0.09	3.46 ± 0.09	3.54 ± 0.09	3.49 ± 0.05c
5	D4: 200	3.72 ± 0.09	3.67 ± 0.09	3.63 ± 0.09	3.70 ± 0.09	3.68 ± 0.05d
6	Average	$3.35 \pm 0.04p$	$3.32 \pm 0.04p$	3.32 ± 0.04a	$3.37 \pm 0.04p$	(—)

Data are presented as mean values of three replicates standard error of the mean (SEM) followed by the same letter are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

Table 5: Effect of OAs application on phosphate rock to CEC average from 4 observation times.

No.	Concentration of OA (ppm)	Types of OA A1: citric acid	A2: humic acid	A3: succinic acid $cmol_e^+\ kg^{-1}$	A4: oxalic acid	Average
1	D0: 0	7.03 ± 0.07	7.14 ± 0.07	7.39 ± 0.07	7.24 ± 0.07	7.20 ± 0.04a
2	D1: 25	7.39 ± 0.07	7.23 ± 0.07	7.37 ± 0.07	7.56 ± 0.07	7.39 ± 0.04b
3	D2: 50	8.49 ± 0.07	8.19 ± 0.07	8.11 ± 0.07	8.37 ± 0.07	8.29 ± 0.04c
4	D3: 100	8.28 ± 0.07	8.21 ± 0.07	8.28 ± 0.07	8.19 ± 0.07	8.24 ± 0.04c
5	D4: 200	8.44 ± 0.07	8.50 ± 0.07	8.13 ± 0.07	8.17 ± 0.07	8.34 ± 0.04c
6	Average	$7.93 \pm 0.03p$	$7.85 \pm 0.03p$	$7.86 \pm 0.03p$	$7.93 \pm 0.03p$	(—)

Data are presented as mean values of three replicates standard error of the mean (SEM) followed by the same letters are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

The effect of OAs with PR on Ca^{2+} is presented in Table 4. The application of PR increased Ca^{2+} from 2.25 (before applying the treatment) to 2.95 $cmol_e^+ \cdot kg^{-1}$ (D0). Cations such as Ca^{2+}, Mg^{2+}, K^+, and Na^+ were adsorbed electrostatically on the surface of the soil colloid, and these cations were exchangeable. Upland acid soil has a net negative charge, so it can adsorb cations such as Ca, Mg, K, and Na, as well as other cations such as Al. The OA

TABLE 6: Effect of OAs application on phosphate rock to Al^{3+} average from 4 observation times.

No.	Concentration of OA (ppm)	Type of OA A1: citric acid	A2: humic acid	A3: succinic acid $\mathrm{cmol_e^+\ kg^{-1}}$	A4: oxalic acid	Average
1	D0: 0	0.82 ± 0.01	0.89 ± 0.01	0.72 ± 0.01	0.83 ± 0.01	0.81 ± 0.01e
2	D1: 25	0.70 ± 0.01	0.72 ± 0.01	0.77 ± 0.01	0.70 ± 0.01	0.72 ± 0.01d
3	D2: 50	0.64 ± 0.01	0.66 ± 0.01	0.74 ± 0.01	0.64 ± 0.01	0.67 ± 0.01c
4	D3: 100	0.60 ± 0.01	0.62 ± 0.01	0.64 ± 0.01	0.59 ± 0.01	0.61 ± 0.01b
5	D4: 200	0.50 ± 0.01	0.53 ± 0.01	0.58 ± 0.01	0.51 ± 0.01	0.53 ± 0.01a
6	Average	0.65 ± 0.01 *p*	0.68 ± 0.01 *p*	0.69 ± 0.01 *p*	0.65 ± 0.01 *p*	(—)

Data are presented as mean values of three replicates standard error of the mean (SEM) followed by the same letters are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

TABLE 7: Effect of OAs application on available P average from 4 observation times.

No.	Concentration of OA (ppm)	Type of OA A1: citric acid	A2: humic acid	A3: succinic acid	A4: oxalic acid	Average
1	D0: 0	8.87 ± 0.12	8.75 ± 0.12	8.67 ± 0.12	8.92 ± 0.12	8.80 ± 0.06a
2	D1: 25	9.23 ± 0.12	9.07 ± 0.12	9.10 ± 0.12	9.27 ± 0.12	9.17 ± 0.06b
3	D2: 50	10.37 ± 0.12	10.09 ± 0.12	10.27 ± 0.12	10.35 ± 0.12	10.27 ± 0.06c
4	D3: 100	10.47 ± 0.12	10.50 ± 0.12	10.83 ± 0.12	10.71 ± 0.12	10.63 ± 0.06d
5	D4: 200	11.50 ± 0.12	11.32 ± 0.12	11.03 ± 0.12	11.30 ± 0.12	11.29 ± 0.06e
6	Average	10.09 ± 0.05 *p*	9.95 ± 0.05 *p*	9.98 ± 0.05 *p*	10.11 ± 0.05 *p*	(—)

Data are presented as mean values of three replicates standard error of the mean (SEM) followed by the same letters are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

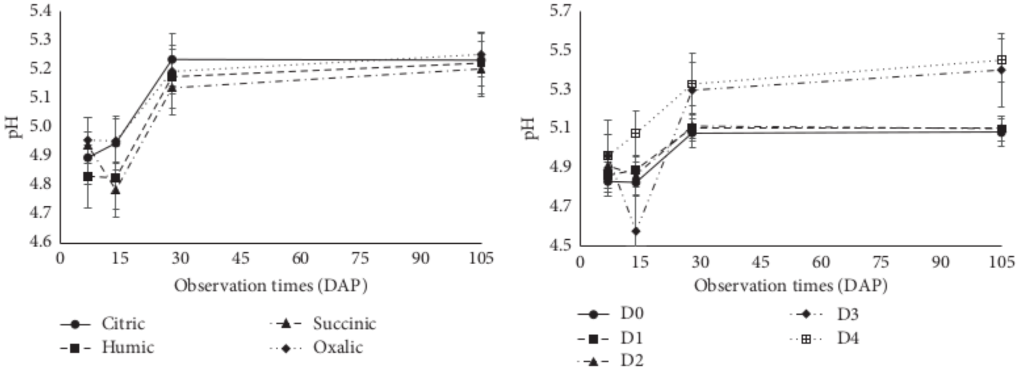
concentration levels had a very significant effect on increasing Ca^{2+} in the soil. The increase in Ca^{2+} occurs due to the hydrolysis of PR, which releases some of the Ca it contains. Ca^{2+} content increased from 2.95 to 3.65 $\mathrm{cmol_e{}^+\cdot kg^{-1}}$ or increased by 23.7%.

The content of exchanged cations such as Ca, Mg, and Na is relatively large amounts on the adsorption surface. This increase in cation displaced Al in the exchange complex, and Al liberated from the adsorption surface will immediately bind to OH- from carbonate hydrolysis, forming $Al(OH)_3$ precipitate. The increase in Ca^{2+} and CEC is shown in Tables 4 and 5.

The effect of OAs application on Al^{3+} is presented in Table 6. The treatment of OA types had no significant effect on Al^{3+}, while the treatment of OA concentration levels had a significant effect on Al^{3+}. The increase in OA concentration levels on PR significantly decreased the Al^{3+}, along with the increase in soil pH and levels of cations such as Ca^{2+} and CEC. An increase in pH and cations caused an increase in the number of cations in the adsorption complex that replaced Al in the adsorption complex, while Al was released into the soil solution. The Al that was released from the adsorption surface would immediately bind to the OH^- from the carbonate hydrolysis. Increasing the pH of H_2O would shift Al^{3+} to form $Al(OH)_3$ precipitate. Therefore, it can be concluded that the addition of PR could increase soil pH and Ca content and suppress Al solubility. The decrease in Al content was also caused by chelation by functional groups of OAs, as shown in Table 6, where Al^{3+} decreased from 0.81 to 0.53 $\mathrm{cmol_e{}^+\cdot kg^{-1}}$ or decreased by 34,3%.

The effect of OAs application with PR on available P is presented in Table 7. Soil available P before the treatment was 7.67 ppm. Application of PR increased available P to 8.80 ppm (D0). Increasing concentrations of OA levels significantly increased available P from 8.80 to 11.29 ppm (D4) or increased by 28,3%. Upland acid soil rich in Al and Fe caused the phosphate delivered by PR into the soil solution to be partially determined by Al and Fe into a form available to plants. OAs were able to increase metal cations such as Al and Fe so that they had nutrients, especially P, that became available. OAs passed through groups that bind ions such as Al and Fe in soil solution so that the phosphate chelated by Al and Fe would function and become available P. As reported by Park et al. [33], Ch'ng et al. [3], and Xie et al. [44], the addition of OAs will increase the available P. Furthermore, Zhu et al. [38] reported an increase in P inorganic and P organic soils with the addition of OAs followed by oxalic acid > citric acid > malic acid. According to Zhu et al. [38], oxalic acid and malic acid more effectively and least effectively released Pi than citric acid, respectively.

Figure 1(a) shows that due to OA types application, soil pH increased from 7 DAP to 14 DAP, then increased sharply to 28 DAP before sloping down to 105 DAP. By applying some OA concentration, soil pH increased sharply to 28 DAP and then sloped down to 105 DAP, except for 100 ppm (D3) and 200 ppm (D4) (Figure 1(b)). A sharper increase was shown by concentrations of D3 and D4 compared with 25 ppm (D1) and 50 ppm (D2). A sharp increase in pH from 4.80 to 5.30 units was due to the addition of OAs that resulted in a very large increase in carboxylic groups, which was able to increase CEC and resulted in an increase in OH^- ions in the soil solution so that the pH of H_2O increased sharply. After this stage, an anion-cation exchange reaction begins, which leads to equilibrium.



(a)

(b)

Figure 1: Effect of OA types (a) and OA concentration levels (b) on soil pH at some observation times.

Exchangeable Al^{3+} decreases sharply from 7 to 14 DAP, then leveled off, before decreasing to 105 DAP with almost the same degree of slope for all OAs as shown in Figure 2(a). For OAs, at all concentrations, Al^{3+} decreased sharply to 14 DAP and then increased to 28 DAP before decreasing to 105 DAP. At a concentration of D4, it decreased more sharply than other concentrations to 14 DAP (Figure 2(b)). The decrease in Al^{3+} during observation is caused by the occurrence of chelates, namely, organic compounds bonded to metal cations such as aluminum (Al), iron (Fe), and manganese (Mn). Metal elements that adsorb P (such as Al) lose their bonds with P and change bonds with OAs in the soil solution. This bond/chelation indicates the interaction between metals and OAs. The impact of the formation of this metal chelate will reduce the binding of phosphate by oxides and clay.

Due to OAs type application, soil CEC ($cmol_e^+ \cdot kg^{-1}$) increased sharply from 28 DAP and then decreased to 105 DAP (Figure 3(a)). In contrast, the application of some OA concentrations, in general, soil CEC increased to 105 DAP, except for D0 CEC decreased from 14 to 28 DAP before increasing to 105 DAP (Figure 3(b)). The increase in CEC during observations showed that there was an increase in the ligand exchange reaction between OAs and -OH. At the initial stage, the cation exchange was quite intensive, then gradually decreased towards equilibrium, as indicated by an increase in CEC, which was increasingly flat at 105 DAP.

Due to OA type application, in general, soil available P increased to 105 DAP (Figure 4(a)). Citric acid and oxalic acid increased more sharply than humic acid and succinic acid. For the OA concentration level treatment for D0 and D1, available P was increased to 105 DAP, but at the treatment of D3 and D4, the increase was more than D1 and D2 (Figure 4(b)). This indicates that the longer the observation time, the available P would increase due to increasing soil pH, decreasing Al^{3+}, and increasing soil CEC as described in the previous paragraph, where P which was adsorbed by metal cations (Al) loses the bond with P and switches bonds with OAs.

3.3. Effect of Organic Acids Application on PR on The Growth of Maize Plants.

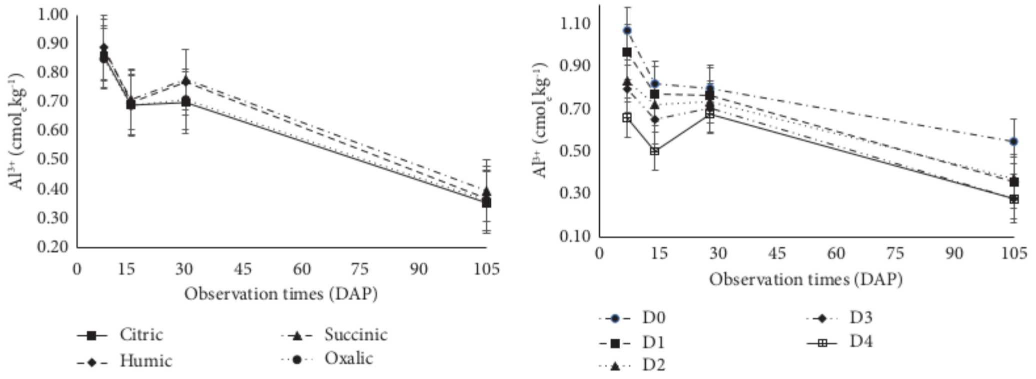
The effect of the application of OAs with PR on the growth of maize plants is presented in Figure 5. Observations on height carried out at 14 DAP, 28 DAP, 42 DAP, and 56 DAP showed no significant difference in the application of OAs type and concentration. The highest plant height up to 56 DAP for OA types was achieved by the application of oxalic acid, which was 202 cm, and further achieved by applying humic, succinic, and citric acids, which was 199 cm (Figure 5(a)).

Levels of OA concentration show almost the same growth in each period of plant vegetative growth. Figure 5(b) shows that plant growth from 14 DAP to 56 DAP increased by 144–148 cm. Furthermore, the concentration of OAs D2 reached the highest plant height was 201 cm, followed by concentrations of D3, D1, D0, and D4 were 201, 200, 199, and 197 cm, respectively.

The effect of OA application with PR on the maize yield is shown in Table 8. The treatment of the OA type was not significantly different from the maize yield at $p < 0.05$ of the DMR test. The highest was achieved by oxalic acid application, which was 43.8 g $plant^{-1}$, followed by citric, humic, and succinic acid applications which were 43.5 g $plant^{-1}$, 43.1 g $plant^{-1}$, and 42.7 g $plant^{-1}$, respectively.

The concentration of OAs treatment significantly affected the maize yield at $p < 0.05$ of the DMR test. The highest was achieved by treatment of D3 concentration, which was 45.6 g $plant^{-1}$, then followed by the application of D2, D4, D1, and D0 which were 44.2 g $plant^{-1}$, 42.3 g $plant^{-1}$, 42.2 g $plant^{-1}$, and 38.1 g $plant^{-1}$, respectively.

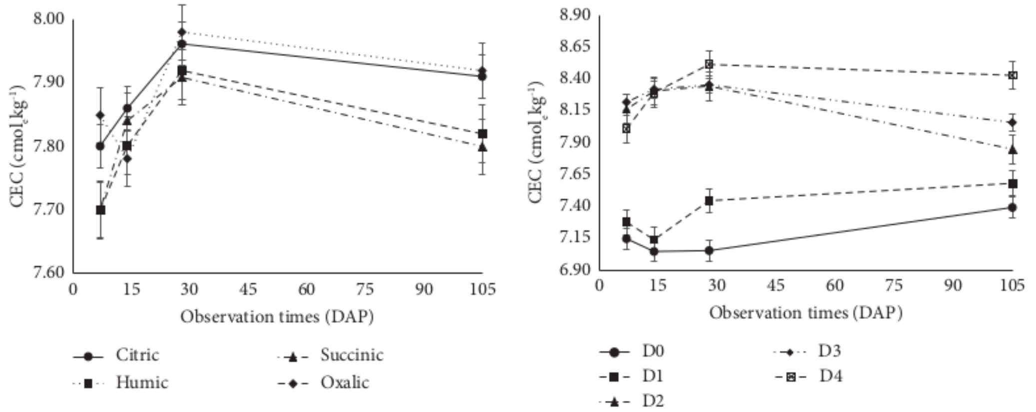
The maximum concentration of OA is determined by calculating the quadratic equation of plant response indicated by the yield of maize to the application of several concentrations with the equation $Y = a + bx + cx^2$ then calculating the first derivative of the regression equation ($Y' = 0$). The polynomial regression equation was $Y = -0.0005X^2 + 0.1269X + 38.385$, $R^2 = 0.9772$ with its derivative $Y' = -0.001X + 0.1269$. The maximum recommended concentration of OA was 126.9 ppm (Figure 6).



(a)

(b)

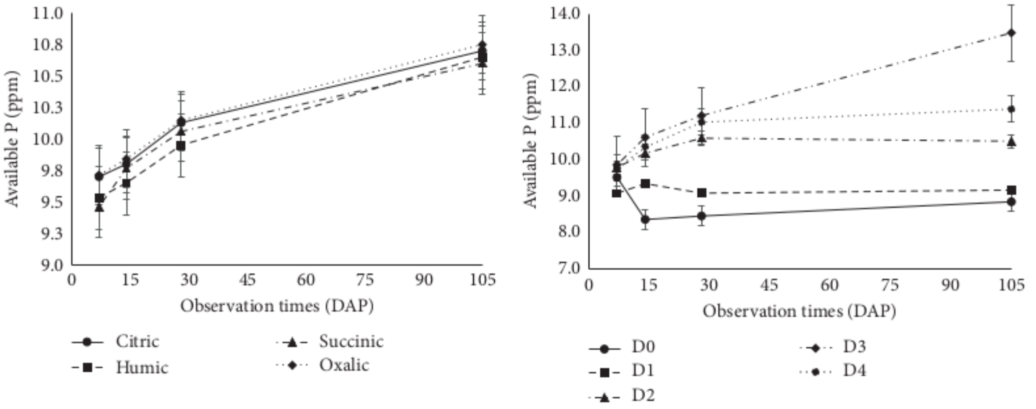
Figure 2: Effect of OA types (a) and OA concentration levels (b) on soil Al^{3+} at some observation time.



(a)

(b)

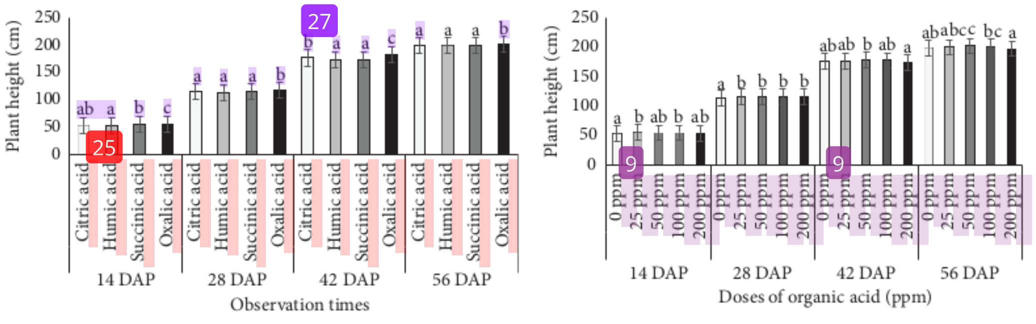
Figure 3: Effect of OA type (a) and OA concentration levels (b) on soil CEC at some observation times.



(a)

(b)

Figure 4: Effect of OA types (a) and OA concentration levels (b) on soil available P (ppm) at some observation time.



(a)

(b)

Figure 5: Effect of OAs application on PR on plant height of maize. At the same time observation (DAP), data followed by the same lowercase letters indicate that was no significantly different among the treatments on OAs type and OAs concentration at $p \geq 0.05$.

Table 8: Effect of OAs application on yield of maize.

No.	Concentration of OA (ppm)	Type of OA				
		A1: citric acid	A2: humic acid	A3: succinic acid	A4: oxalic acid	Average
1	D0: 0	35.7 ± 1.4	41.7 ± 1.4	38.4 ± 1.4	36.5 ± 1.4	38.9 + 1.1a
2	D1: 25	38.3 ± 1.4	41.9 ± 1.4	42.8 ± 14	44.7 ± 1.4	42.8 + 1.1b
3	D2: 50	40.6 ± 1.4	44.10 ± 1.4	43.1 ± 1.4	44.3 ± 1.4	43.9 + 1.1bc
4	D3: 100	43.9 ± 1.4	46.8 ± 1.4	46.0 ± 1.4	47.0 ± 1.4	46.8 + 1.1c
5	D4: 200	40.5 ± 1.4	44.1 ± 1.4	43.1 ± 1.4	44.1 ± 1.4	43.9 + 1.1bc
6	Average	43.5 ± 1.0*p*	43.1 ± 1.0*p*	42.7 ± 1.0*p*	43.8 ± 1.0*p*	(—)

Data are presented as mean values of three replicates standard error of the mean (SEM) followed by the same letters are not significantly different (Duncan at $p \geq 0.05$), (—): no significant interaction.

The types and levels of OA concentration had not significantly affected the plant height's vegetative growth and maize yield. This showed that soil P change only significantly affected the generative growth of maize, as indicated by the maize yield. Kim et al. [45] reported that OAs increased water-soluble phosphorus and available phosphate. At the level of 10 mM, the effectiveness of OAs for water-soluble P was citric acid (44%) > oxalic acid (32%). Citric acid of 1 mM can potentially improve the availability of phosphate and the healthy growth of corn.

4. Discussion

Direct application of PR is expected to be able to overcome the problem of the availability of P in acid soils due to the slow release of PR. The addition of OAs to PR application was expected to increase the soil pH and the available P and suppress Al toxicity to plants.

This research showed that the direct application of PR increased soil pH, Ca^{2+}, CEC, and available P and decreased H^+ and Al^{3+}; when OAs were added to PR, the types of OA treatments had no significant effect on all parameters; in contrast, the levels of OA concentration treatments significantly increased soil pH, Ca^{2+}, CEC, and available P, and decreased H^+ and Al^{3+}. Soil pH significantly increased during the observation times due to OAs application, which decreased in H^+ and Al^{3+}, and increased in Ca^{2+}, CEC, and available P in line with the studies below.

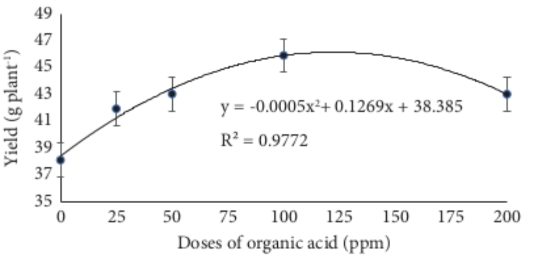


Figure 6: Polynomial regression equation of OAs concentration application on maize yield.

Soil pH profoundly affects the amount and manner in which soluble phosphates become adsorbed. Phosphorus adsorption occurs at very low and high soil pH, and in alkaline soils, the dominant P ion species are $H_2PO_4^-$ and PO_4^{3-} [4, 46]. Adsorption of phosphorus by iron and aluminum oxides also declines with increasing pH due to reduced solubility of Fe and Al ions [5, 6]. At lower pH, adsorption results from the reaction with iron and aluminum and their hydrous oxides. Above pH 7.0, ions of calcium and magnesium and their carbonates cause P precipitation [36, 47, 48]. The maximum amount of phosphate is available at a slightly acidic to neutral pH, where Fe, Al, and Ca phosphate solubility is simultaneously high [49, 50]. The P adsorption maxima in soils containing high contents of oxides of Fe and Al might be due to the

Figure 7: Training on making humic acid with water hyacinth (*Eichhornia crassipes*) as raw material for farmer groups in East Java, Indonesia (source: Imelda Safitri).

formation of their respective metal phosphates [36, 48]. The dissolving of P precipitates with free $Fe(OH)_2$ and $Al(OH)_3$ causes P to form insoluble complexes, especially at $pH < 5$. In addition, P can precipitate with Ca^{2+} ions at $pH > 6.5$, and the sorption of inorganic phosphate in soils with a pH less than 7.0 is closely related to the amount of reactive Fe and Al compounds [51].

Organic acids play a role in releasing P that is absorbed to increase the availability. The increase in soil P availability was caused by changes in the chemical properties of the soil, as indicated by an increase in the pH of H_2O and a decrease in the activity of Al and Fe as metal elements. Welch et al. [52] reported that the chelation or complex bonding between metals and OAs occurs when OAs are added to the soil, causing a ligand exchange reaction between organic anions to free -OH. The occurrence of this cation exchange increased in -OH ions to increase the pH-H_2O value. Increasing the pH-H_2O value decreases the activity of metals in the soil, such as Al and Fe, and the functional groups of OAs will bind to free Al and Fe to form a complex/chelating bond. Furthermore, OAs contribute to lowering solution pH as they dissociate in a pH-dependent equilibrium into their respective anion(s) and proton(s). According to Traina et al. [53], at lower OAs concentrations, the principal reaction scheme is the exchange of OH^- by an adsorbed polymerized hydroxy–Al^{3+} species, followed by a cation exchange reaction between an exchangeable Al–OA complex and Na^+ to promote Al-phosphate. Since OH/Al molar ratios >2 are typically in adsorbed hydroxy-Al polymers, OH– (aq) is a net production. At high OA concentrations, the principal adsorbed Al^{3+} reaction with the ligand OA is the formation of a soluble Al–organic complex and the production of H^+ (aq) when OA is protonated.

The effect of OAs on phosphate adsorption is related to the pH value of the solution. The minimum reduction is present at a specific pH value of OAs solution ranging from 2 to 10 [54]. Initial solution pH had a much more substantial effect on P release from PR than iron phosphate [55]. Meanwhile, the ability to solubilize PR is attributed to acidification, chelation, and exchange reactions ([Omar in 4]). The presence of OA increases the dissolution of carbonates and phosphates [56]. OA anions, with oxygen-containing hydroxyl and carboxyl groups, can form stable complexes with Ca^{2+}, Fe^{2+}, Fe^{3+}, and Al^{3+} [57]. The increased acidification of PR appears to be a function of acidification by OAs and the chelating ability of Ca, Fe, and Al (Omar in [58]). Citric acid has the most obvious effects on the P removal efficiency and rate, followed by succinic acid and acetic acid. Under different solution pH values, the influence of OA on the P removal efficiency decreased with increasing pH values [37].

The major mechanism of mineral phosphate solubilization is the release of microbial metabolites such as OAs, which takes place through their hydroxyl and carboxyl groups chelating the cations bound to phosphate, thereby converting them into soluble forms [59]. Kpomblekou and Tabatabai [60] reported that the release of P from the PRs to acids was affected by the chemical structure, type, and position of the functional groups of the ligands and the concentration of the acids. Citric acid up to 100 ppm concentration increases the tolerance of oil palm seedlings to the stress of Al, becoming complex compounds that are difficult to dissolve [61]. In this research, 100–200 ppm was a concentration of OA that could provide lower soil exchangeable Al compared with other OA concentrations. The application of oxalic and citric acid significantly affected maize, which was supported by increasing soil available P. Furthermore, P was significantly available for plants to increase crop yield [62]. Oxalic acid was more effective at solubilizing total P from the direct application of PR. Meanwhile, three sequential extractions at pH 3 using oxalic and citric acids were able to solubilize a total of 46 and 22% P, respectively [35, 48]. Citric acid, especially oxalic acid, is the most efficient for dissolving PR. The strategy proposed can contribute to advances in the large-scale development of phosphate fertilizer production using OAs [63]. Oxalic acid was more effective at solubilizing total P from PR, providing twice as much P in solution as citric acid at the same molar ratio [64].

Types of OAs were not significantly different in terms of changes in the soil properties and maize yield. Therefore, the application of humic acid has the potential to be applied with PR in the field. In Indonesia, humic acid has been widely applied by farmers because it can be obtained in farm shops or made by farmers themselves. Farmers make humic acid soil improvers through the process of composting organic matter (straw, manure, and water hyacinth (*Eichhornia* *crassipes*)) (Figure 7). In general, farmers apply humic acid. In general, farmers apply liquid humic acid by mixing it with water and then pouring it on the ground.

5. Conclusions

The types of OA treatments with PR did not significantly affect pH, Al^{3+}, Ca^{2+}, CEC, and P-Bray 1, as well as the maize yield. Subsequently, the application of oxalic and citric acids improved soil properties and maize yield higher than humic and succinic acids.

The concentration of OA level treatments significantly affected soil pH, Al^{3+}, Ca^{2+}, CEC, available P, and the maize yield. The highest soil pH, CEC, available P, and maize yield, as well as the lowest Al^{3+} achieved by a concentration of 100 ppm (D3) or 200 ppm (D4) treatment.

Increasing soil pH due to the application of OAs would decrease soil H^+ and Al^{3+}, which in turn increased Ca^{2+}, CEC, and available P. The maximum OA concentration was 126.9 ppm, and after this concentration, the maize yield was reduced.

Although not significantly different, oxalic acid was able to improve the observed soil properties and maize yield, followed by citric, humic, and succinic acids. In Indonesia, humic acid has been widely applied by farmers because it is easy to obtain. Therefore, it can be applied to phosphate rock.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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