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Potency of Agroindustrial Wastewaters to Increase the Dissolution of Phosphate Rock Fertilizers

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ABSTRACT

The used of agroindustrial wastewaters are not maximum yet in Lampung Province**,** althought it can be used as an acid solvent because of its acidic properties. This study was aimed to explore the most potential agroindustrial wastewaters in dissolving phosphate rock through acidulation in the laboratory scale. The experiment was arranged in a factorial. The first factor was origined of phosphate rock (Sukabumi, west Java and Selagailingga, central Lampung) and the second factor was solvent types (agroindustrial wastewaters which were pineapple, tapioca, tofu industry, and palm oil as well as conventional acid solvents which were HCl, H_2SO_4, and CH_3COOH). The incubation processes were 0, 1, 2, and 3 months. The results showed that agroindustrial wastewaters that have the highest potency to solubize phosphate rock was industrial tofu wastewaters and followed by industrial wastewaters of tapioca, palm oil, and pineapple. Both the conventional acid and agroindustrial wastewaters solvent had a big potency to solubilize phosphate rock, however, its highest soluble P-value did not match with the ISO criteria for phosphate fertilizers Quality I (SNI) because it did not reach the solubility of 80% of its total P_2O_5, but it has been qualified as a fertilizer both the quality phosphate A, B, and C (SNI).

Keywords: Acidulation, agroindustrial wastewater, P fertilizer, phosphate rock

INTRODUCTION

Natural phosphate rocks (PR) are an important natural material traditionally used for the production of P (phosphorus) fertilizers. Traditional P fertilizer is produced based on chemical processing of a insoluble high-grade mineral phosphate which includes an energy intensively with sulfuric acid at a high temperature (Van Straaten 2002). This process is environmentally undesirable, it is not least because of the release of contaminants into the main product, gas streams and by-products.

Natural PR's have been recognized as a valuable alternative for P fertilizers and as a cheaper source of phosphate fertilizer for crop production (Ghosh 1999). Phosphate rock is not available for plant where the soil pH is greater than 5.5-6.0 and even when conditions are optimal, plant yields are lower than those obtained with soluble phosphate (Khasawneh and Doll 1978). The use of PR as fertilizer is determined by the mineral properties, solubility, grain size, free carbonate content, total P_2O_5 content in PR, and type of deposit. Although PR can be used directly on the soil, but its availability to plants is very slow (Zapata and Zaharah 2002) and the effectiveness of PR as a fertilizer is limited by its low dissolution rate (Hamdali *et al.* 2010), so it needs to be processed chemically to accelerate its solubility. Phosphate rock is generally found in the form of calcium phosphate [$Ca_3(PO_4)_2$] or fluorapatite [$3Ca_3(PO_4)_2CaF_2$], these compounds are generally slow release (Havlin *et al.* 2005). Commonly, the phosphate fertilizers are produced by acidulation (Budi and Purbasari 2009).

One alternative to accelerate the solubility of PR is by utilized wastewaters as a byproduct of various industrial treatments of agro-products of tapioca, palm oil processing industry, pineapple canning industry, and the tofu industry. Several studies have shown that these wastewaters have a low pH. Vassilev and Vassileva (2003) reviewed that there was a potency of agro-industrial waste to solubilize PR, besides they have low pH, they also release some organic acids produced by microorganisms.

Based on the possibility of acid wastewaters to have high potency for acidification of potentially PR with low cost, it will be able to overcome the environmental pollution. Lampung Province also has deposits of PR that is located in the Region

Selagailingga, Central Lampung, and many agroindustrial wastewaters by product can be created as a cheaper alternative for dissolution of PR as the basis for the fertilizer manufacture. The establishment of local PR can be used to overcome the problem of high price for importing PR from abroad.

The purpose of this study was to find the potency of local agroindustrial wastewaters to solubilize local PR in order to create P-fertilizer and for find the optimum incubation time for solubilizing P_2O_5 from PR.

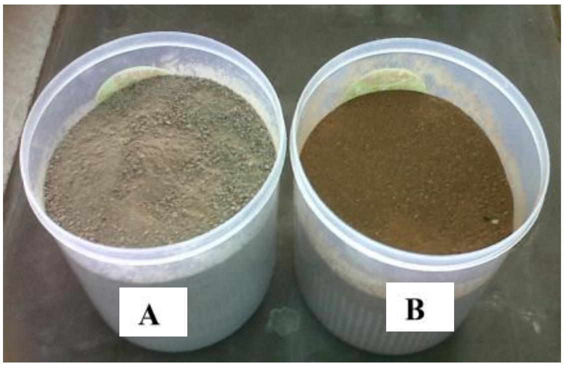

Figure 1. Texture differences between phosphate rock from Selagailingga, Central Lampung (A) and Sukabumi, West Java (B).

MATERIALS AND METHODS

Phosphate Rock and Agroindustrial Wastewaters Uses

Phosphate rocks were taken from Sukabumi District, West Java and Selagai Lingga, Central Lampung District, Lampung Province. The performances of PR used are shown on Figure 1. Agroindustrial wastewaters were taken from several locations of agroindustrial factory such as center of tofu industry in Bandarlampung, CV Bumi Waras for tapioca and palm oil agroindustrial wastewaters, and PT Great Giant Pineapple for pineapple industrial wastewaters. The PR and agroindustrial wastewaters characteristics are shown in Table 1 and 2.

Research Design

Treatments were design by a factorial in a randomized block design. The first factor was kinds of solvent, consist of three conventional solvents *i.e.* hydrochloric acid (HCl), sulfuric acid (H_2SO_4), and acetic acid (CH_3COOH) as well as agroindustrial wastewaters of tofu, palm oil, tapioca, and pineapple caning. The second factor was origin of PR *i.e.* from Sukabumi and Selagai Lingga. Every treatment was incubated by soaking the PR with acid solvent in 0, 1, 2, and 3 month. Every treatment was done by three replications.

Tabel 1. Phosphate rock characteristics.

Origin of phospate rock	pH	Total-P (%)	Soluble-P (%)
Sukabumi	5.76	14.24	3.78
Selagailingga	5.96	25.63	7.08

Phosphate Rock Incubation

Phosphate rocks were milled and then sieved with a 3 mm siever. Selected agroindustrial wastewaters were analyzed its pH, total-N, total-P, BOD (biological oxygen demand), and COD (chemical oxygen demand). Five hundreds gram of the powder of rock phosphate (less than 3 mm in diameter) were submerged with every fresh agroindustrial wastewaters as well as with conventional acid solvent until all PR's were drowned. Submerged rockposphate were covered tigthly and incubated according to the treatments *i.e.* one day, one-, two-, and three-month. All PR were incubated at room temperature.

Tabel 2. Characteristics of agroindustrial wastewaters used for asidulating phosphate rock.

Variable	Selagailingga Agrondustrial wastewaters			
	Tofu	Palm oil	Tapioca	Pineapple canning
pH	3.76	4.0	4.27	3.08
COD (mg L^{-1})	9,900	34,720	10,525	27,850
BOD (mg L^{-1})	925	21,280	743	1,295
P-total (%)	0.00054	0.0008	0.0025	0.00037
N-total (%)	0.07	0.03	0.03	0.03

Sampling and Analyzing Incubated Phosphate Rock

Samples were taken by spatula in five replications from five different sites in the incubated container, and then it were mixed as a composite sample. Samples were taken in accordance to incubation times. Incubated wet samples were dried up to one week with room temperature until dry. For further analysis, the incubated PR after oven were milled and analyzed. Every sample were analyzed the characteristic of its available-P (citric acid 2%), total-P (HCl 25%), and pH.

Statistical Analysis

Data obtained were analyzed by analysis of variance (ANOVA) and separation of main differences were tested by Least Significance Difference test at 5% level. Correlation between variable was done for study the relationship between variable.

RESULTS AND DISCUSSION

Changes of Phosphate Rock pH after Acidulated

One-day submerged. After one day incubation, pH of PR's from Sukabumi were significantly higher than that from Selagailingga when submerged with H_2SO_4 or CH_3COOH (Table 3). All of PR submerged with conventional solvent had significantly lower pH than that all agroindustrial wastewaters. Almost the same thing was obtained in the PR of Selagailingga and oil palm wastewater which had a higher potency in decreasing pH than tapioca and pineapple wastewaters as a solvents of phospahte rock.

One-Month Submerged. After acidulation of PR by acid solvent or agroindustrial wastewaters one month, the increasing of pH were higher compared to one days incubation (Table 3). in dissolution of PR with conventional acid solvent and agro-industrial wastewaters PR from Sukabumi had a higher pH than Selagailingga excepted by solvent from palm oil and pineapple canning wastewaters. The same as the submerged of one day, the H_2SO_4 and CH_3COOH solvent had the highest acidity in submerged PR that was indicated by the low pH compared to the other solvents.

Two-Month Submerged. The pH value of PR in the two month submerged had the same value as one month submerged altought the pH value of the PR from Sukabumi was higher than Selagailingga, except when it had dissolved by a palm oil and pineapple canning wastewaters. Phosphate rock from Sukabumi had a greater potency for acidifying by palm and pineapple canning wastewaters, while the pH of phosphate rock from Selagailingga did not significantly decrease by agroindustrial wastewaters solvent.

Three-Month Submerged. After 3 months submerged by various solvents, pH of PR from Sukabumi and Selagai Lingga showed no significantly difference with LSD test, whereas other results were the same as submerged at 1 and 2 months (Tables 3).

Variation in pH during Incubation

During incubation of 1 day to 1 month pH significantly increased pH and afterwards (at 2 and 3 months) were also likely to increase, but with a small gradient (data not shown). Figure 2 also shows that the pH of the PR solution from Sukabumi had a higher pH compared to PR from Sukabumi. Conventional acid solvent, H_2SO_4, had a very significantly higher than the other solvents in dissolving P.

Changes in Soluble-P from Phosphate Rocks

One-day submerged. Results of one day incubation showed that soluble PR from Sukabumi had significantly higher pH than the original PR of Selagailingga (Table 4). Acidulation with H_2SO_4 significantly increased P-soluble compared to other solvents for both PR's. At day one, agroindustrial wastewaters solvent had the ability to dissolve phosphate as good (not significantly different) as conventional acid solvent CH_3COOH *i.e.* industrial tofu wastewater. From these data it can be presumed that the tofu wastewater had a better potential than other wastewaters.

One-Month Submerged. After acidulation of PR by acid solvent or wastewaters agro-industry during the first month, there was an increase compared to soluble P at one day submerged (Table 4). Dissolution P of PR from Sukabumi was significantlly lower than the original PR of Selagailingga. Dissolution of PR with H_2SO_4 conventional solvent, remains soluble P-value was higher than the other unless the acid CH_3COOH with wastewaters industry that it was not significantly different from the same as in the acidulation 0 months.

Two-Month Submerged. Improved dissolution of PR was relatively low even nearly equal to one month well submerged in PR from Sukabumi and Selagailingga (Table 4). Dissolution of PR with conventional solvent H_2SO_4 still provided higher soluble P value more than the CH_3COOH with agro-

Table 3. Interaction effect of phosphate rock that were acidulation by agro-industrial wastewater and acid solvent on the of pH value.

Type of phospate rock solvent	Incubation time							
	1-day		1-month		2-month		3-month	
	PR Sukabumi	PR Selagailingga	PR Sukabumi	PR Selagailingga	PR Sukabumi	PR Selagailingga	PR Sukabumi	PR Selagailingga
Conventional Acids	.... pH							
HCl	2.56 a (C)	2.36 a (C)	6.20 a (B)	3.52 b (B)	7.48 a (AB)	4.2 b (B)	7.57 a (AB)	4.25 a (B)
H_2SO_4	2.00 a (D)	1.21 b (D)	3.35 a (C)	2.03 b (C)	4.48 a (C)	2.27 b (C)	4.52 a (C)	2.30 a (C)
CH_3COOH	2.10 a (D)	1.45 b (D)	3.46 a (C)	2.49 b (C)	5.24 a (C)	3.21 b (BC)	5.30 a (C)	3.24 a (BC)
Agroindustrial wastewaters								
Tofu	4.27 a (B)	4.31 a (B)	7.37 a (A)	6.03 b (A)	8.26 a (A)	6.55 b (A)	8.35 a (A)	6.63 a (A)
Tapioca	4.98 a (AB)	5.13 a (A)	7.32 a (A)	6.16 b (A)	8.39 a (A)	6.58 b (A)	8.48 a (A)	6.66 a (A)
Palm oil	4.65 a (A)	4.57 a (B)	6.66 a (AB)	5.74 a (AB)	7.77 a (AB)	6.67 a (A)	7.86 a (AB)	6.75 a (A)
Pineapple canning	5.03 a (A)	5.17 a (A)	6.38 a (B)	6.2 a (A)	6.56 a (B)	6.57 a (A)	6.63 a (B)	6.65 a (A)
LSD 0.05	0.32		0.98		1.28		1.30	

Median values followed by the same letter in the same row and column are not significantly different at the level of 5% LSD. Lowercase was read vertical lyand capital letter (in parentheses) was read horizontally.

Table 4. Interaction effect of phosphate rock that were acidulation by agroindustrial wastewater and acid solvent on soluble-P under different incubation times.

Type of phospate rock solvent	Incubation time 1-day PR Sukabumi	1-day PR Selagailingga	1-month PR Sukabumi	1-month PR Selagailingga	2-month PR Sukabumi	2-month PR Selagailingga
Conventional Acids	........................,,,,,,,,,,,,,,,,,,...... Soluble P (%)					
HCl	4.23 a (B)	9.78 b (C)	6.27 a (C)	12.26 b (D)	5.59 a (AB)	12.48 b (C)
H_2SO_4	5.60 a (C)	10.22 b (D	6.61 a (C)	14.70 b (D)	6.88 a (CD)	14.52 b (D)
CH_3COOH	4.36 a (B)	9.86 b (C)	6.00 a (BC)	12.97 b (C)	6.73 a (BC)	12.90 b (C)
Agroindustrial wastewaters						
Tofu	8.35 a (A)	6.63 a (A)	6.57 a (C)	11.75 b (B)	6.50 a (BC)	10.37 b (B)
Tapioca	8.48 a (A)	6.66 a (A)	5.02 a (A)	9.89 b (A)	4.76 a (A)	9.78 b (B)
Palm oil	7.86 a (AB)	6.75 a (A)	5.11 a (AB)	9.05 b (A)	4.96 a (A)	7.95 b (A)
Pineapple canning	6.63 a (B)	6.65 a (A)	4.72 a (A)	9.09 b (A)	4.61 a (A)	8.29 b (A)
LSD 0.05	1.30		0.96		0.96	

Mean values followed by the same letter in the same row and column are not significantly different at the level of 5% LSD. Lowercase was read vertical and capital letter (in parentheses) was read horizontal.

industrial wastewaters that was not significantly different from the same as one-day acidulation. Of the three kinds of conventional solvents, H_2SO_4 remains as the best solvent while HCl and CH_3COOH were equally lower potency than H_2SO_4. In addition, from Table 4 it can be seen that the industrial tofu and tapioca wastewaters had high potency as a solvent for PR acidulation compared to oil palm industry and pineapple canning wastewaters.

Three-Month Submerged. At three months of submerged there was no interaction effect between the origin of PR and solvent for P-soluble acid (Tables 5 and 6). P-soluble from PR origin of Selagailingga was significantly higher than Sukabumi. While conventional acid solvent H_2SO_4 was significantly higher than the other solvents in dissolving P.

Table 5. Soluble P from two kind of phosphate rock after 3 months incubation.

Phosphate rock origin	Soluble-P (%) 3 months incubation
Sukabumi	5.81 a
Selagai Lingga	10.62 b
LSD 0.05	3.18

Values followed by the same letter are not significantly different at the level of 5% LSD.

Variation of Soluble-P during Incubation

Figure 2 shows maximum values of soluble-P generally occured at 1-month after incubation. The soluble-P increased from one-day submerged up to one-month, then stable and tend to decreased until

Table 6. Soluble P from two kind of phosphate rock after 3 months incubation.

Conventional solvent	Soluble-P (%) 3 months incubation
HCl	8.50 a
H_2SO_4	12.71 b
CH_3COOH	9.10 a
Agroindustrial wastewaters	
Tofu	8.25 a
Tapioca	6.65 a
Palm oil	6.21 a
Pineapple canning	6.07 a
LSD 0.05	3.12

Values followed by the same letter are not significantly different at the level of 5% LSD.

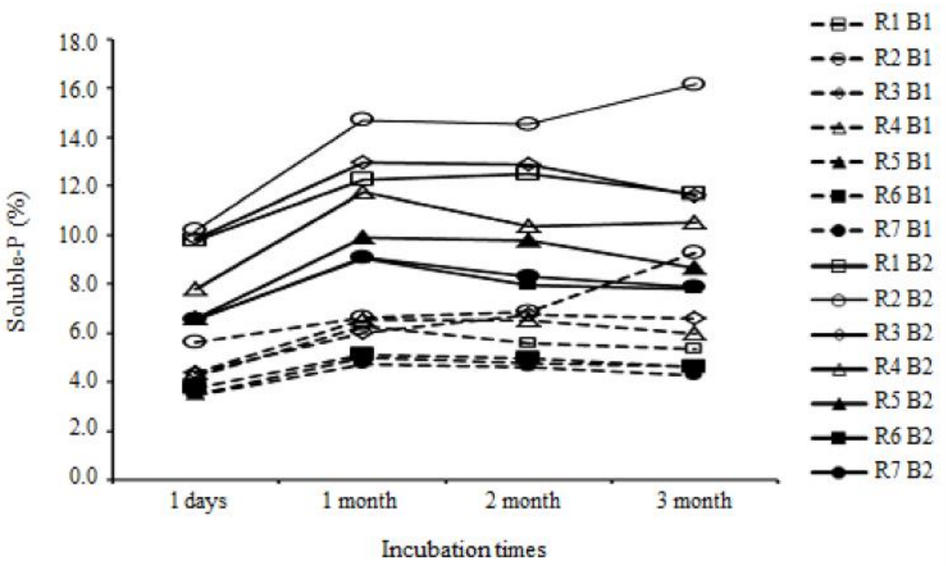

Figure 2. Changes in soluble-P during the incubation periods from two kinds of phosphate rocks. B_1= phosphate rocks of Sukabumi, B_2= phosphate rocks of Selagailingga, R_1 = HCl, R_2= H_2SO_4, R_3 = CH_3COOH, R4 = tofu wastewaters R5 = palm oil wastewaters, R6 = tapioca wastewaters and R7 = pineapple canning wastewaters.

three-months. It indicated that equilibrium reaction between PR with solvent was already stable.

Changes in Total-P Phosphate Rocks after Acidulation

One day submerged. Table 7 shows that the PR from Sukabumi had a lower P-total than that of Selagailingga. While the PR from Sukabumi had the highest P-total with pineapple canning wastewaters and the lowest by HCl solvent.

One- and Two-Month Submerged. Tables 8 shows that the total-P was depending on the origin of the PR. It is very clear that the P-total of PR Selagailingga was significantly higher than of Sukabumi.

Three-Month Submerged. Table 7 shows that the PR Sukabumi had lower total-P than of Selagailingga. Furhtermore, total-P remaining after acidulation by pineapple canning and tofu industrial wastewaters was high as at one-day incubation.

During three-month incubation, total-P was changed in which it would increase after one-month incubation (Figure 4). It was in line with maximum P-solubilization occured during one-month incubation.

Discussion

Based on the results obtained, acidulation of PR with agro-industry wastewater and conventional acid solvent would have highest dissolution P after one-month of incubation. After one-month, P-soluble would decline, except in the treatment of PR Selagailingga and H_2SO_4 acid solvent that was still a bit up to two-months. This is likely due to (1) the availability of H^+ ion to dissolution PR increased and mixed rock phosphate released monovalent $H_2PO_4^-$ in concomitant with increasing of pH and divalent HPO^{-2} occured (2) the reaction equilibrium dissolution of PR has been achieved so that the reaction product in the form of $H_2PO_4^-$ and HPO^{-2} has reached the maximum. Different with research of Al-Oud (2011) when PR mixed with calcareous soil and acid solvent such as Organic manure, citric acid, oxalic acid, or EDTA would increase in concomitant with 90 days incubation.

Interaction for dissolution phosphate between origin of PR and industrial wastewater was occurred in the one-month incubation. The combination of PR Selagailingga and H_2SO_4 produced the highest soluble-P and combined with a solution of HCl or CH_3COOH produced soluble P-values above 10% so that the combination qualify *Standar Nasional Indonesia* (SNI) in quality category A, where P-value of at least 10% soluble. Combination of PR Selagailingga and tofu wastewater produced P-soluble of 11.75% (SNI category A). Combination of PR Sukabumi and CH_3COOH, HCl, or H_2SO_4 produced soluble-P at 6.27%, 6.32%, and 6.34%, respectively. It had been produced soluble-P with which is almost qualify SNI by category C. While in combination with a mixture of PRSukabumi wastewaters knew generate P-soluble at 6.57% also qualify SNI combination in phosphate C. Fertilizers

Table 7. Interaction effect of phosphate rock that were acidulation by agro-industrial wastewater and acid solvent on total-P under different incubation times.

Type of phospate rock solvent	Incubation times			
	1-day		3-month	
	PR Sukabumi	PR Selagailingga	PR Sukabumi	PR Selagailingga
Conventional Acids	....Total-P (%)	....		
HCl	15.09 a (A)	19.22 b (B)	17.15 a (B)	22.42 b (A)
H_2SO_4	13.56 a (A)	21.03 b (A)	16.62 a (B)	21.66 b (A)
CH_3COOH	14.53 a (A)	19.69 b (A)	14.98 a (A)	21.98 b (A)
Agroindustrial wastewaters				
Tofu	13.45 a (B)	20.28 b (A)	14.68 a (A)	24.21 b (B)
Tapioca	15.03 a (A)	19.13 b (B)	18.12 a (B)	20.87 b (A)
Palm oil	14.47 a (A)	20.36 b (A)	16.84 a (B)	24.50 b (B)
Pineapple canning	13.48 a (B)	20.32 b (A)	15.66 a (A)	23.77 b (B)
LSD 0.05	1.57		2.65	

Mean values followed by the same letter in the same row and column are not significantly different at the level of 5% LSD. Lowercase was read vertical and capital letter (in parentheses) was read horizontal.

Table 8. Total-P of phosphate rock after one-and two-months incubation

Phosphate rock	Total-P (%)	Total-P (%)
	1-month incubation	2-month incubation
Sukabumi	15.61 a	15.89 a
Selagai Lingga	21.76 b	22.25 b
LSD 0.05	2.47	2.48

Values followed by the same letter are not significantly different at the level of 5% LSD.

quality must meet the requirements in accordance with the SNI, when the fertilizer product will be sold.

Significantly increasing in pH during incubation was occured. On the solubility of P from PR Selagailingga, which was caused by the presence of Ca^{2+} increasing the pH became alkaline, so it did not dissolved P anymore. As in soil, Kanabo and Gilkes (1986) and Soelaeman (2008) reported that increasing of soil pH resulted in decreased dissolution of PR and contact period affected in dissolution.

Of the four types of wastewater used, each had a potentially for dissolution P from PR, all of the acids wastewater had lower pH. However, from the results of research conducted, dissolution of P by tofu wastewater was not comparable with the acid solvent. This is due to the pH of the agro-industrial wastewater was higher than the acid solvent. Of the seven types of the acid solvent, H_2SO_4 was the highest in dissolution of PR. Although solvent H_2SO_4 was better as solvent for PR, but it was not qualified as a Phosphate Fertilizer Quality A according to SNI 2005 (BSN 2005).

At one-day incubation dissolution of PR by all solvent have occurred (Figure 2). This happens because of shortly after submerged, chemical reaction was action by protons released from wastewater. In accordance with Sauvé and McBride (1998) that activity of phosphate have relationship with pH where highest solubility at low pH and gradual decreased with increasing pH. In

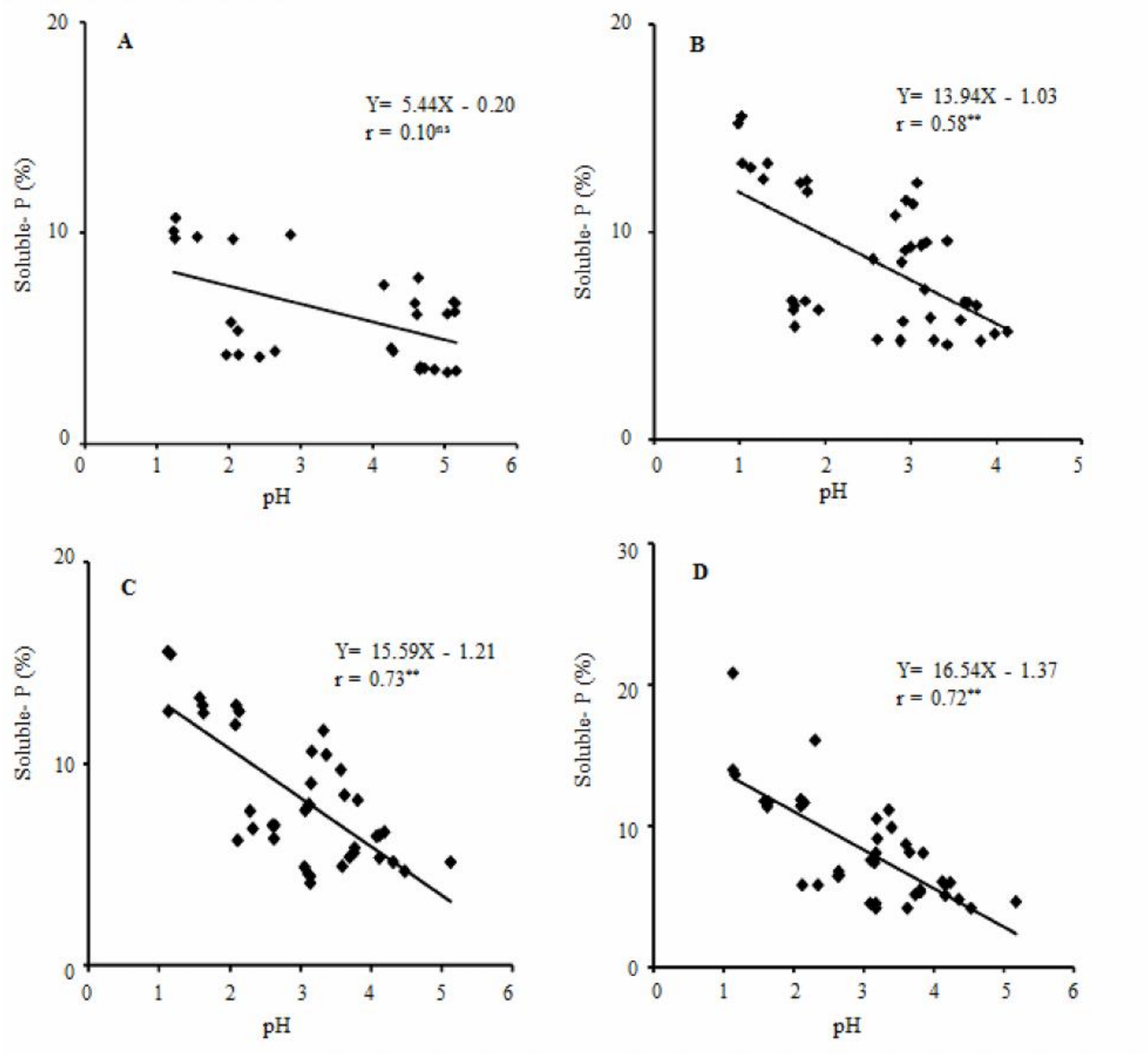

Figure 3. Relationship between phosphate pH and soluble P during time of incubation of phosphate rock submerged by agroindustrial wastewaters. A = 1-days after incubation, B = 1-month after incubation, C = 2-month after incubation, and D = 3-month after incubation.

addition Vassilev *et al.* (2006) reported that beside of agroindustrial waste have low pH, it also released some organic acid by the activities of microorganism.

Soluble-P from PR Selagailingga were higher than that from Sukabumi. This is because the initial percentage level of P_2O_5 in PR Selagailingga was 25.63% where it was higher than the PR Sukabumi which was only 14.24% (Table 1). It is proved that the levels of P_2O_5 in PR to be one of the factors for dissolved P. Shipitalo *et al.* (2012) mention that different types of materials would produce different yield. This is in line with research reported by Kawusulan (2007), that the high solubility of P is also influenced by the total P content of PR itself. The concentration of P in natural PR that is used will affect the solubility of the phosphate in solution.

CONCLUSIONS

Based on observations it can be concluded that the accidulation of PR from Sukabumi and Selagailingga had maximum value at one-month incubation. The industrial tofu and tapioca had big potencial for accidulation of PR. Both the conventional acid solvent (14.7%) and tofu industry (11.6%) wastewaters that interact with the PR Selagailingga, even the value of the P-soluble was highest, but it was not belong to criteria for quality phosphate fertilizer I (SNI) because it has not reached the solubility of 80% of the total P_2O_5, however it has been qualified as a good quality for natural phosphate fertilizer grade A, B, and C.

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