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Formation of Phosphate Crystals from Cow Urine Using Aeration System Batch Reactor

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ABSTRACT

The waste generated from the livestock sector is cow urine containing nitrogen, sulfur, phosphate, ammonium, sodium, manganese, iron, silica, chlorine, magnesium, and calcium. The aim of this research is to reduce phosphate levels, which will impact the environment caused by cow urine, which contains phosphate and ammonium and forms phosphate crystals using a batch reactor with an aeration system. The wastewater treatment results using a batch reactor produce precipitate in the form of phosphate crystals, which can be used as fertilizer for plants. This study used a completely randomized design (CRD) with two repetitions, namely with a comparison of the rate of aeration (airflow) 1 Lpm, 1.5 Lpm, and 2 Lpm with the time used, namely 0 minutes, 60 minutes, 120 minutes, 180 minutes and 240 minutes. Based on research that has been done, the highest aeration rate (airflow) in phosphate removal in cow urine waste is at 1 Lpm air flow with a time of 240 minutes and a phosphate removal efficiency of 84.8822%. Meanwhile, at an airflow of 1.5 Lpm with a time of 60 minutes and a phosphate removal efficiency of 95.4315%. At an airflow of 2 Lpm with a time of 240 minutes, the removal efficiency can only be 34.3421%. The content of phosphate crystals obtained from the XRF results was 3.173%. At an airflow of 2 Lpm with a time of 240 minutes, the removal efficiency can only be 34.3421%. The content of phosphate crystals obtained from the XRF results was 3.173%. At an airflow of 2 Lpm with a time of 240 minutes, the removal efficiency can only be 34.3421%. The content of phosphate crystals obtained from the XRF results was 3.173%.

1. Introduction

Waste results from various activities that are no longer used physically, chemically, or biologically and have no economic value. Waste can be divided into three types (i.e., liquid, solid, and gaseous waste). Suppose the waste treatment is ineffective and is just released into the environment. In that case, it will cause various problems and environmental pollution, starting from water, air, and soil pollution. It can interfere with human health and damage the ecosystems of living things around it. Environmental problems that often occur due to the livestock industry are liquid waste directly discharged into river bodies without any prior processing [1].

Cattle farm waste is waste generated from business activities in cattle farming, either solid waste, liquid waste, or animal feed residue. One of the wastes produced on cattle farms is cow urine. Cow urine is the remaining liquid from feed that has been digested and metabolized by the cow's body, which contains nitrogen, sulfur, phosphate, ammonium, calcium chloride, silica, minerals, lactose, enzymes, hormones, and acids [2]. Based on research conducted by [3], in a day, one cow can produce approximately 20 liters of urine. Struvite and calcium oxalate crystals are often found in cow urine [4]. About 56.67% of cattle breeders dispose of waste into river bodies without prior management. This causes environmental pollution caused by waste released by livestock (i.e., feces, urine, and leftover feed).

Phosphate is a material that has an important role in human life but is present in limited quantities and cannot be renewed. Some of the important parts of phosphate play a role in plant growth and the formation of human DNA. One of the elements that form phosphate is phosphorus (P). Phosphorus is an important nutrient needed by plants, which plays a role in photosynthesis, using sugar and starch, and transferring energy in plants [5]. Element P in plants cannot be replaced with other elements, so plants must get the element phosphorus for growth and development.

If the waste is released into the environment without treatment, high phosphate and ammonia content can cause environmental problems such as eutrophication. Eutrophication is an environmental problem caused by phosphate waste that occurs in aquatic ecosystems with the emergence of excessive nutrients in aquatic ecosystems. One of the methods to reduce the impact of decreasing quality on the environment caused by PO_4 and NH_4 is to treat waste containing PO_4 and NH_4 ions.

Phosphate crystals are rocks formed from precipitation (crystallization) containing phosphate minerals. In addition, phosphate crystals can be used as a slow-release fertilizer for plants because of the mineral content plants need [5]. The formation of struvite crystals occurs via several phases, generally beginning with nucleus (nidus) formation, more concentrated urine, and deposition of minerals such as phosphorus, which is the origin of struvite, phosphate excretion, pas a predisposing factor for the formation of struvite crystals and phosphate calculi [6]. Several ways have been carried out in forming phosphate crystals: BNR (Biological Nutrient Removal), electrolysis, and adsorption methods.

Researchers have conducted several studies on forming phosphate crystals using several reactors. Formation of phosphate crystals using a fluidized bed reactor equipped with a stirrer impeller and using a batch system, it was obtained that the P removal efficiency was 97-98%. Still, there were obstacles; there was no circulation flow, and the energy needed during the process was quite large due to the use of impellers [7]. The impeller or stirring causes the particles to collide with each other so that the particle size becomes finer and has the possibility of re-dissolving in the solution.

In addition, there are several modifications to the reactor used by some researchers, namely, using a reactor with a continuous system equipped with circulating flow. This flow serves to reuse the mixture that has formed precipitate and is suspected to still contain P levels in the mix. The size of the continuous reactor used in this research is designed to be longer to maximize the crystallization process. Different from the several studies carried out by the researchers above, this study was created using a batch reactor equipped with a recycle flow and an aerator equipped with various variants of airflow.

2. Research Methodology

2.1. Materials

The tools and materials used in this study were a batch reactor with recycle, aerator, universal pH paper, analytical balance, XRF, filter, sample bottle, spectrophotometer, cuvette, 125 mL Erlenmeyer, volumetric flask (100 mL, 250 mL, and 1000 mL), measuring cups (25 mL and 50 mL) 10 mL measuring pipettes, volumetric pipettes (5 mL, 10 mL, and 20 mL), 1000 mL beaker glass, 250 mL beaker glass, 50 mL beaker glass, 10 mL beaker glass and dropping pipette.

The materials used in this study were cow urine obtained from cow pens at the Lampung State Polytechnic, $MgCl_2$, distilled water, NH_4Cl (1 N), KOH solution, H_2SO (4.5 N), potassium antimony tartrate solution ($K(SbO)C_4H_4O_6.1/2H_2O$), ammonium molybdate solution ($(NH_4)_6MO_7O_{24}.4H_2O$), ascorbic acid solution $C_6H_8O_6$ (0.1 M), anhydrous potassium hydrogen phosphate (KH_2PO_4).

2.2. Procedures

The phosphate removal process of cow urine waste used a batch reactor with an aeration system using a Completely Randomized Design (CRD) with variations, namely the flow rate of the aerator used and the variation in the length of reaction time with two repetitions of the experiment. The experimental design can be seen in Table 1.

Table 1. Experimental Design Completely Randomized Design (CRD)

Airflow (AF) (Lpm)	Time (Menit)	Ulangan	
		1	*2*
	0	AF1t0 (1)	AF1t0 (2)
	60	AF1t1 (1)	AF1t1 (2)
1	120	AF1t2 (1)	AF1t2 (2)
	180	AF1t3 (1)	AF1t3 (2)
	240	AF1t4 (1)	AF1t4 (2)
	0	AF1,5t0 (1)	AF1,5t0 (2)
	60	AF1,5t1 (1)	AF1,5t1 (2)
1,5	120	AF1,5t2 (1)	AF1,5t2 (2)
	180	AF1,5t3 (1)	AF1,5t3 (2)
	240	AF1,5t4 (1)	AF1,5t4 (2)
	0	AF2t0 (1)	AF2t0 (2)
	60	AF2t1 (1)	AF2t1 (2)
2	120	AF2t2 (1)	AF2t2 (2)
	180	AF2t3 (1)	AF2t3 (2)
	240	AF2t4 (1)	AF2t4 (2)

1) Tool Design

The equipment used in this study is a batch reactor with an aeration system made of conical acrylic, which is equipped with an aerator as a stirrer to help form phosphate crystals with a device capacity of 1.5 L, which is fitted with a centrifugal pump and valve for the recycle flow. Schematic of the aeration system batch reactor can be seen in Fig. 1.

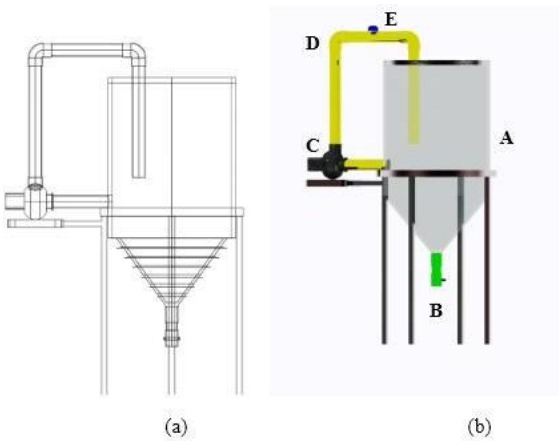

Fig. 1. Design reactor aeration system batch in a capacity of 1.5 L

Where:

A : Reactor

B : Sample output valve

C : Recycle pump

D : Recycle hose

E : Valve recycle

In a reactor that is designed not to use stirring because the stirred reactor has several weaknesses, namely, the crystals formed will be larger attached to the stirrer impeller. However, using a stirrer by increasing the stirring speed can consume greater power [8], [9]. Therefore, a reactor design was formed, as shown above, with a conical shape that can slow down the flow pattern at the top of the column. This flow pattern will help the crystal formation process by maintaining the particle size [10].

2) Cow Urine Sample Preparation

This research was started by mixing cow urine with the reactant, namely $MgCl_2$. Cow urine obtained from farms is filtered first using filter paper to separate solid particles or impurities in cow urine. The volume is 1 L, measured using a measuring cup.

3) Reactant Preparation

The reactant used is technical $MgCl_2$, which is mixed with cow urine. The $MgCl_2$ used was weighed first as much as 71.25 grams using an analytical balance, then mixed with filtered cow urine, and a sample volume of 0.25 was measured; a MAP (Magnesium Ammonium Phosphate) solution was produced, then the pH value was checked using universal pH paper.

4) Phosphate Removal Reaction Process

A total of 71.25 grams of $MgCl_2$ was weighed using an analytical balance, dissolved in 0.25 liters of cow urine, and the pH of the MAP (Magnesium Ammonium Phosphate) solution using universal pH paper or a pH meter. Before use, ensure the reactor is clean, pumps, and valves are in good condition and ready for use. Make sure the aerator or compressor has been installed with a flow meter.

The solution was put into the reactor and flowed with air according to the experimental design (i.e., 1, 1.5, and 2 Lpm). The airflow rate is regulated using a flow meter or valve on the compressor. The recycle flow pump in the reactor is connected to an electric current. The valve is opened to flow the recycled material to be regulated to control the flow rate of the incoming recycle solution. The size of the recycle valve opening is set at 0.75 liters/minute of the flow of material entering the reactor.

The length of reaction time was set according to the experimental design (i.e., 0, 60, 120, 180, and 240 minutes) and taken out every 60 minutes. When sampling all pumps used, ensure they are turned off to make it easier to collect solids at the bottom of the reactor. Using a spectrophotometer, liquid samples were tested for P levels, and phosphate crystals were analyzed for composition using XRF.

3. Results and Discussion

3.1. Air Flow on Phosphate Removal

Removal of phosphate from cow urine waste using a batch reactor with an aeration system begins with taking a sample of cow urine from the farm, after which, before reacting the sample, an analysis of the initial phosphate content in the cow urine is carried out using a spectrophotometer with the procedure used referring to SNI - 06 - 6989.31 – 2005 so that the initial phosphate level in cow urine is 4.3554 mg/L. Before the reaction process was carried out using a batch reactor, the sample was prepared by filtering it. The filtering process is carried out to remove solid particles or organic solids originating from the urine sampling site. Cow urine waste is reacted in the reactor and then analyzed for phosphate levels before and after the reaction using a spectrophotometer with the SNI 06-6989 reference method. 31-2005. Before the analysis, a linearity test was carried out by making a calibration curve. A calibration curve or linearity test is done by making a series of standard working solutions with a concentration of 0.0, 0.2, 0.4, 0.8, and 1.0 mg P/L. One drop of PP indicator was added, and 8 mL of the previously prepared mixed solution was measured using a spectrophotometer with a wavelength of 880 nm. Based on these results, the equation $y = 0.588x$ is obtained with an R^2 value of 0.9988, where y is the absorbance in nm and x is the concentration of the working solution. This equation calculates the P content in cow urine waste with the average results presented in Table 2.

Table 2. P levels in cow urine waste

Airflow(Lpm)	**Time (minutes)**				
	0	***60***	***120***	***180***	***240***
1	4.4	2.1	2.1	1.1	0.7
1.5	4.4	0.2	0.6	0.9	1.3
2	4.4	3.5	3.5	3.5	2.8

Based on Table 2, it can be seen that the maximum decrease in phosphate levels in cow urine waste occurs in the airflow variation of 1 Lpm at 240 minutes, which is 0.6584 mg/L, and the slowest decline in phosphate elements, namely in the air flow variation of 2 Lpm and time 60 minute. Phosphate removal is proportional to the time the reaction is used; the longer the reaction time, the more phosphate removal will increase [11].

Airflow or aeration rate and length of reaction time are used to reduce phosphate levels in cow urine wastewater. In general, the stirring speed will affect phosphate removal efficiency; the higher the stirring speed, the more effective it will be [12]. The function of aeration used in this study, apart from being an agitator in removing phosphates, has another function, namely the addition of oxygen from aeration, which functions to form precipitates or crystals. This study presents the results of P levels in cow urine waste in Fig. 2.

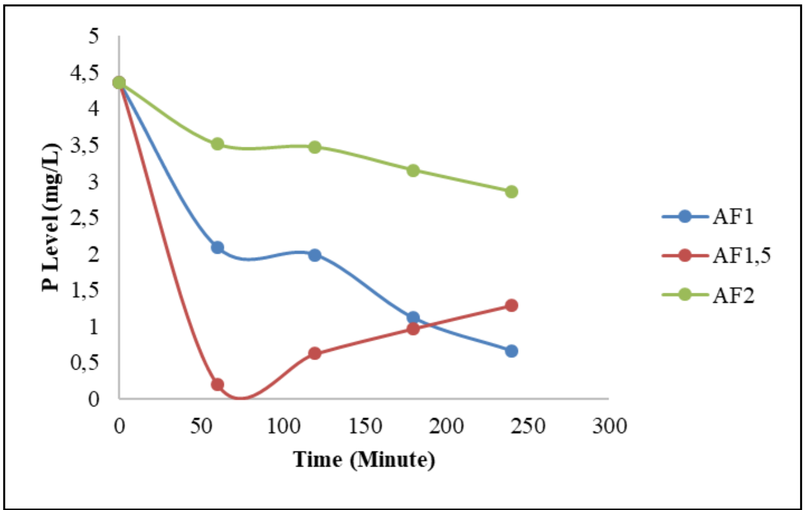

Fig. 2. P Levels in cow urine waste with in combination airflow 1, 1.5, and 2 Lpm

Fig. 2. presents a graph of the P content contained in cow urine waste using an airflow of 1, 1.5, and 2 Lpm. The longer the reaction time used, the higher the level of phosphate reduction in cow urine waste. Phosphate removal at 1 Lpm air flow is the maximum flow rate that can be used to remove phosphate levels in cow urine waste with a phosphate removal efficiency of 84.8822% with a time usage of 240 minutes. The test sample with a flow rate of 1.5 Lpm could significantly reduce the phosphate levels in cow urine waste from the initial P level of 4.3554 mg/L to 0.1990 mg/L with the required time of 60 minutes. The lowest P content data can be achieved at an airflow rate of 2 Lpm of only 2.8597 mg/L with the required time of 240 minutes. This is due to the high flow rate, which increases the stirring speed. The aeration rate that is too large affects the speed of stirring in the reaction process; this fast stirring will cause the precipitate that has been formed to break up so that it will dissolve again in the cow urine waste.

Aeration as a stirrer can increase the acidity (pH) of the MAP (Magnesium Ammonium Phosphate) solution used. Aeration can increase the pH by releasing the CO_2 present in the solution into CO_2 gas. One of the factors that can affect the increase in pH with the aeration method is the bubble size. Aeration using microbubble aeration can increase the pH value by 1 level greater than the aeration method using macrobubble aeration. The effect of bubble size is more significant in increasing the pH than the influence of the aeration rate used [13]. Aeration to raise the pH using free air can increase the pH value to 8.53 [14]. The increase in the pH value in this study is presented in the graph in Fig. 3.

In this study, the cow urine was alkaline with a pH of 9. After mixing with MgCl, a MAP (Magnesium Ammonium Phosphate) solution was obtained in neutral conditions (pH 7) and then added with 1 N KOH solution to reach an alkaline pH. Based on the graph in Figure 16, the pH increase is not too significant, but the higher the flow rate used, the higher the pH of the solution in the reactor. This is in line with research conducted by [12], where the higher the flow rate, the higher the pH value. The pH increase was insignificant because this study used aeration with macro bubble size.

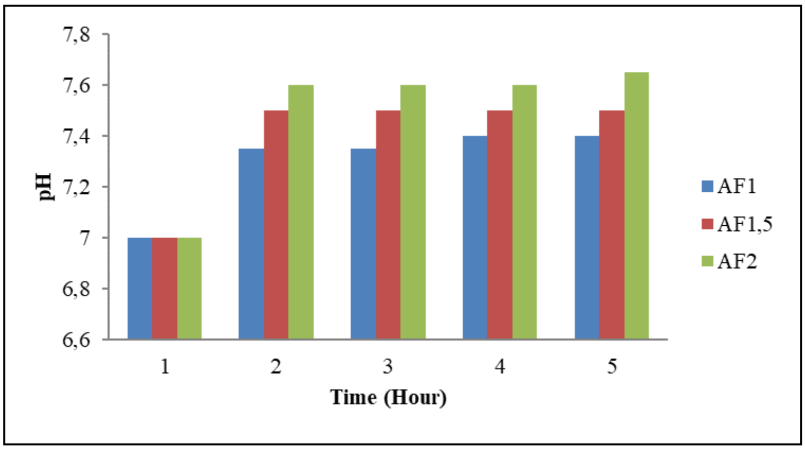

Fig. 3. Effect of airflow (i.e., 1, 1.5, and 2 Lpm) on pH value

The degree of acidity is an essential factor in the removal of phosphate. Precipitation can occur when the waste is in an alkaline state, which has a pH ranging from 6.5-10; apart from adding chemicals, an increase in pH can occur due to aeration of the wastewater [15]. This study's maximum flow rate obtained for reducing P levels and crystal formation was 1 Lpm, with an average pH of 7.4.

3.2. Phosphate Content in Phosphate Crystals

Phosphate reduction efficiency in percent (%) can be seen that the decrease in the concentration of P levels in the sample experienced a fluctuating increase and drop from the initial concentration of the sample with the treatment of air flow rate and the length of reaction time used with pH seven and a large recycle flow rate of 0.75 m^3/minute. Fig. 4. presents a graph of the efficiency of reducing P levels in percent (%).

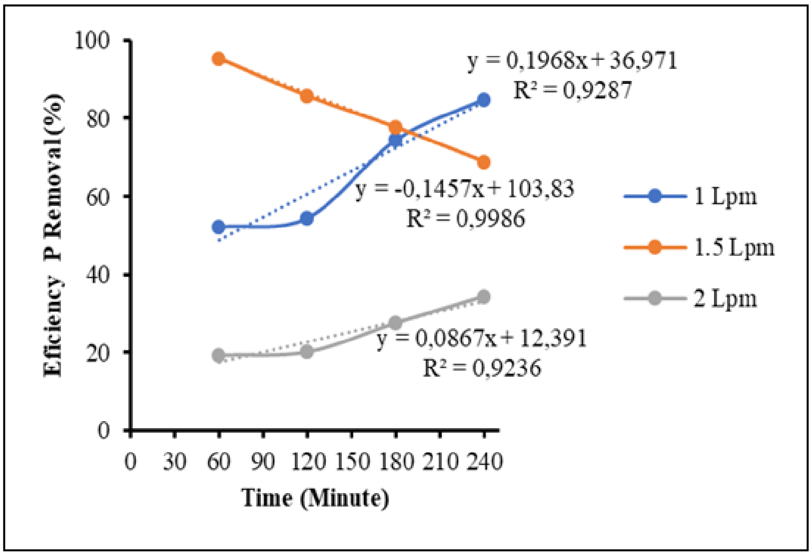

Fig. 4. Efficiency P removal on combination airflow of 1, 1.5, and 2 Lpm

From the treatment applied to the samples, it was obtained that the maximum phosphate removal efficiency was 84.8822% with the air flow rate used at 1 Lpm and the reaction time was 240 minutes. In this study, not all experiments produced sediments in the form of rocks or crystals, only a few treatments formed rock deposits. The reaction that takes place lowers the P content in cow urine waste and, in this reaction, produces a slurry-shaped precipitate, which, when filtered, will be shaped like a cake. The existence of this precipitate is suspected because the cow urine used still contains solids or organic solids that come from the sample collection and is not filtered first using a vacuum pump.

3.3. XRF (X-Ray Fluorescence) Value

After removing the phosphate from cow urine waste using a batch reactor with an aeration system using variations in airflow and time to obtain the results of cow urine waste after phosphate removal, they check the phosphate levels in the cow urine waste using a spectrophotometer. In addition to the liquid waste that has gone through the reaction process, it also forms crystalline precipitates and other precipitates in slurries during the phosphate removal process. Not all treatments formed or produced crystal precipitates during the phosphate removal process on the samples used twice. Treatments that produce crystal deposits include 1 Lpm air flow and 1.5 Lpm airflow.

The samples used to determine the composition or content of phosphate crystals, namely samples produced from 1 Lpm airflow, were analyzed using the X-Ray Fluorescence (XRF) method with the results shown in Table 3.

Table 3. Phosphate crystal XRF from cow urine waste

Element			Geology			Oxides		
Compound	***Conc.***	***Units***	***Compound***	***Conc.***	***Units***	***Compound***	***Conc.***	***Units***
Al	0.757	%	Al_2O_3	1.285	%	Al_2O_3	1.282	%
Si	0.901	%	SiO_2	1.685	%	SiO_2	1.681	%
P	3.173	%	P_2O_5	6.224	%	P_2O_5	6.212	%
Cl	15.883	%	Cl	12.607	%	K_2O	2.354	%
K	2.611	%	K_2O	2.362	%	CaO	73.6	%
Ca	74.043	%	CaO	73.892	%	TiO_2	0.012	%
Ti	0.01	%	Ti	0.007	%	V_2O_5	0.014	%
V	0.012	%	V	0.008	%	MnO	0.536	%
M N	0.631	%	M N	0.417	%	Fe_2O_3	0.404	%
Fe	0.429	%	Fe_2O_3	0.406	%	ZnO	0.095	%
Zn	0.117	%	Zn	0.077	%	Aug_2O	0	%
Aug	0	%	Aug	0	%	in_2O_3	1.186	%
In	1.36	%	in	0.985	%	BaO	0.05	%
Ba	0.072	%	Ba	0.045	%	eu_2O_3	0	%
Eu	0	%	eu	0	%	cl	12.573	%
Re	0.001	%	Re	0.001	%	Re	0.001	%

Based on the component analysis of phosphate crystals carried out using the XRF method, a table of phosphate crystal components and a graph of XRF results were obtained. Based on the component table, it can be seen that the crystals formed are not completely pure phosphate crystals; there are still many other elements contained in these crystals. One of the elements contained in phosphate crystals and has a high percentage is element calcium (Ca), with a percentage of 74.043 %. The element Ca contained in the crystals is thought to have come from urine cattle contained in the food consumed by cattle.

Based on the XRF analysis test results, the P content of phosphate crystals was 3.173%, meaning that the crystals formed from the removal of phosphate from cow urine waste can be used as fertilizer to meet the nutrients needed by plants for optimum growth [5]. The crystals or precipitate formed does not have a large enough phosphate content but has a high percentage of Ca, which is 74.043%. In addition to the P nutrient needed by plants, calcium content is also needed by plants for growth. Calcium is an important nutrient after the essential nutrients (N, P, and K), which supply plant nutrition. Besides that, the function of calcium in plants is to build cell walls, control soil pH, and play a role in protein formation and carbohydrate movement [16]. The need for Ca in each plant will vary depending on the type of plant and the needs of the plant [17]. Based on the opinions and results of this research, it means that calcium (Ca) is one of the elements needed by plants, so the crystals obtained with a Ca content of 74.043% can be used as fertilizer to meet the needs of phosphate in plants, but can also be used as fertilizer to meet calcium needs Ca in plants.

The graph in the XRF analysis itself is data from sample measurements using XRF in the form of a dimension and spectrum source where the x-axis is the energy (keV), and the y-axis is the portion or intensity of the x-rays produced from each element. Each element has a different spectrum and energy; in this study, the composition of the material in the sample is shown in a graph where the higher the percentage of elements present in the sample, the peaks on the graph will be higher.

3.4. Phosphorus Value

The research resulted in the removal of phosphate levels and crystal formation with the influence of the amount of airflow and the reaction time for reducing phosphate using a spectrophotometer with the reference procedure of [18]. It was carried out twice in repetition according to the experimental design used. The results of the analysis of P levels using a spectrophotometer can be seen in Table 4.

Table 4. P concentration with combination airflow (i.e., 1, 1.5, and 2 Lpm) and time (minute)

Air Flow (Lpm)	Time (Minute)	Concentration (mg/L)	Standard Deviation
1	0	4.3554	0.000
	60	2.0830	0.012
	120	1.9827	0.050
	180	1.1125	0.001
	240	0.6584	0.014
1.5	0	4.3554	0.000
	60	0.1990	0.022
	120	0.6222	0.000
	180	0.9674	0.049
	240	1.3535	0.095
2	0	4.3554	0.000
	60	3.5094	0.033
	120	3.4725	0.049
	180	3.1545	0.020
	240	2.8597	0.008

Based on the analysis results obtained, it was then processed using SPSS IBM 26 software. After testing the normality of the data obtained, it was found that the data was normally distributed; because the data was normally distributed, the processing of the data obtained used the two-way Analysis of Variance (ANOVA) test method. Table 5 presents the results of the analysis test.

Table 5. ANOVA using IBM SPSS application 26

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	61.377a	14	4.384	3506.814	0.000
Intercept	163.715	1	163.715	130954.77	0.000
Air_Flow	20.749	2	10.374	8298.422	0.000
Time	31.172	4	7.793	6233.581	0.000
Air_Flow * Time	9.457	8	1.182	945.528	0.000
Error	0.019	15	0.001		
Total	225.111	30			
Corrected Total	61.396	29			

a R Squared = 1.000 (Adjusted R Squared = .999)

Based on Table 5, the results of the ANOVA with the treatment of the effect of using airflow and time on reducing P levels in cow urine waste. Based on the results obtained on the airflow variable, the Sig. 0.000 <0.05, meaning that the data is significant and acceptable. A significant value means that there is an influence from the use of flow rate or air flow on reducing P levels in cow urine waste. Using aeration as a stirrer can increase the pH of the solution during the reaction and will affect the phosphate removal during the reaction [12]. Stirring will increase the chance of interaction between the reactants used, increasing the possibility of a reaction [19]. The variable time or reaction time used in this study obtained the value of Sig. 0.000. This value is less than 0.05, meaning the data is acceptable and significant. Data with a significant value means this variable reduces P in cow urine waste.

After the analysis test was carried out using ANOVA, significant results were obtained or significantly compared between the variables used. Obtained ANOVA results that were significantly comparable, then continued with further tests using DMRT (Duncan). The table of Duncan's test results can be seen in Appendix 7. The table shows that most of the treatments used were significantly different from one another, but the use of 1 Lpm air flow with a time of 240 minutes (AF1t4) was not significantly other from the AF1, 5t2 variation; the same thing occurred between AF2t2 and AF2t1 variations. Based on the table from the Duncan test results, the best combination for phosphate removal in wastewater is the AF1t4 combination (1 Lpm air flow and 240 minutes time). The phosphate level will decrease in this combination as the reaction time is used.

4. Conclusion

Based on the research that has been done, namely the removal of phosphate from cow urine waste using a batch reactor with an aeration system to form phosphate crystals with the highest aeration rate (airflow) in phosphate removal from cow urine waste, namely at 1 Lpm air flow with 240 minutes and phosphate removal efficiency of 84.8822%. Meanwhile, at an airflow of 1.5 Lpm with a time of 60 minutes and a phosphate removal efficiency of 95.4315%. At an airflow of 2 Lpm with a time of 240 minutes, the removal efficiency can only be 34.3421%. The maximum aeration rate used for phosphate removal in cow urine waste is 1 Lpm with a reaction time of 240 minutes, which can reduce phosphate levels by 84.8822%. The phosphate content in the phosphate crystals can be seen from the analysis of the crystal components carried out using XRF. Based on these results, the phosphate content in the crystals was obtained at 3.173%.

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