Original article

Use of calcium carbonate andaluminium sulphate to decrease Pb, Cu, Cr Mn on waste water laboratory

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Abstract:

Laboratory is one of the supporting units at the higher education institutions that produce waste water containing heavy metals and other dissolved metals in high concentrations and is highly toxic and dumped without proper management. The purpose of this study is done to reduce Pb, Cu, Cr, and Mn using CaCO3 and CaCO3 combination with Al2(SO4)3 through precipitation method.

The results showed a decrease of Pb = 89.92%, Cu=68,42,0%, Cr=99.47% and Mn=98.34%. Statistically this method can reduce heavy metals and other metals significantly but produce sludge that much that require further management by the method according to the characteristics of wastewater laboratory.

Keyword: Waste water chemicals, laboratory, heavy metals and precipitation.

Introduction:

Laboratory waste water is a used material from laboratory practices and research activities that is routinely conducted by students and lecturers in the laboratory. Most of the waste water composition consists of the chemical elements; one of them is a heavy metal. Types of heavy metals which have relatively high toxicity include: Hg, Ag, Cd, Cu, Ag, Ni, Pb, As, Cr, Sn, Zn and Mn (Suprihatin, 2010).Characteristics of laboratory waste water can be categorized as hazardous and toxic waste (B3), because most of the elements are harmful, toxic and has a very low pH value (1.2), thus requiring special handling management. However, waste water in laboratory practice has not managed properly. One of chemical waste water treatments that is often applied is coagulation, flocculation and precipitation. Precipitation method can remove heavy metals in chemical waste water (Rahardjo., PN., 2010).

Laboratory waste water has not well-arranged yet so that its management is still very traditional by separating and collecting all domestic waste, in terms of laboratory waste water including hazardous waste water and toxic because it contains heavy metals elements in it. Efforts which have been made are ineffective and inefficient because the waste water once collected in large quantities and then discarded without processing to the environment that can contaminate surface water and groundwater.The purpose of the research is to find a method that is effective and efficient in reducing Pb, Cu, Cr and Mn by using a combination of CaCO3 and CaCO3 with Al2 (SO4) 3 through a method of coagulation, flocculation and precipitation. This method has been widely used, but its management techniques can be utilized in the treatment of waste water.

Methods :

Tools and materials of the research; the tools used include: Jar Test, measuring cylinder, spectrophotometer, pH meter and other laboratory tools for the analysis of research parameters. While the materials used include: laboratory waste water, limestone (CaCO3) and aluminum sulfate (Al2 (SO4) 3). Working procedures performed three stages, the first observation of the pH value of laboratory waste water. Second, observation toward research variables before treatment. Third, observations of the research variables after treatment (distribution of CaCO3 and combination between CaCO3 and Al2 (SO4) 3). The parameters observed were on the decrease of Pb, Cu, Cr and Mn before and after treatment.

Increasing the pH of the laboratory waste water was conducted as follows:

1. Waste water was diluted with clean water (pH = 7.3, TDS = 150 mg / L) ratio of 1: 1 (500 ml sample of waste water and 500 ml sample of clean water). Check the variable pH, Pb, Cr, Cu, and Mn. See the table of results as shown in Table 4.1. (Data prior to treatment).

2. Waste water was diluted by clean water with 3 repetitions and the ratio 1: 1. Then put 6 grams of $CaCO_3$, 7 g $CaCO_3$, and 8 g of $CaCO_3$. By using Jar Test, all the samples rapidly stirred for 2 minutes at a speed of 120 rpm, 30 rpm and then stirred slowly for 2 minutes. Furthermore, let stand for 20 minutes and it would produce a clear waste water and sludge formed in an average thickness of 210 ml. If it was precipitated for 60 minutes, it would produce a clear waste water and sludge formed in an average thickness of 100 ml-120 ml.

3. Waste water was diluted by clean water. The ratio is 1: 1 by doing 3 times repetitions. Put 6 g $CaCO_3$, 7 g $CaCO_3$, and 8 grams of $CaCO_3$, then by using Jar Test stirred rapidly for 2 minutes at a speed of 120 rpm, and then 30 rpm slow stirring for 2 minutes. Then let stand for 20 minutes. After that, separate between the clear waste water with sludge formed. The next process was separating waste water after treatment into three Becker glass as much as 1000 ml per glass and put 1.5 gr $Al_2(SO_4)_3$, then using Jar Test stirred rapidly for 2 minutes at a speed of 120 rpm, 30 rpm and stirred slowly for 2 minutes. Then let stand for 20 minutes. After that, take the measurement result as shown in table 1.2.

4. Waste water was diluted by clean water. The ratio is 1: 1 by doing 3 times repetitions. Put 6 g $CaCO_3$, 7 g $CaCO_3$, and 8 grams of $CaCO_3$, then using Jar Test stirred rapidly for 2 minutes at a speed of 120 rpm, and then 30 rpm slow stirring for 2 minutes. Then let stand for 20 minutes, after that separate between the clear waste water with sludge formed. The next process was separating waste water treatment into three Becker glass as much as 1,000 ml and 1.5 gr put $Al_2(SO_4)_3$, then using Jar Test stirred rapidly for 2 minutes at a speed of 120 rpm, 30 rpm and stir slowly for 2 minutes. Then let stand for 20 minutes. After that, take the measurement result as shown in table 1.2.

Results:

Chemical waste water sources in this research came from the results of some of the practical activities of students from 10 study programs.

The waste consisted of organic waste (fats, proteins, ethanol, cellulose), acid waste (H_2SO_4), base waste (KOH, NaOH), and heavy metals which were collected, mixed and placed into a plastic drum. The research variables studied include Pb, Cr, Cu and Mn. Pre examination was carried out to the chemical waste water before investigation to see the state of the initial pH value. The pH value is one of the parameters that affect precipitation (precipitation) in chemical waste water treatment (Said, 2002).

1. The dilution process of chemical waste water

Chemical waste water as a brown samples with pH = 0.9. Then it was diluted by qualified clean water, pH = 7.3 and TDS = 150 mg / L. A dilution ratio is 1: 1 (500 ml sample of waste water and 500 ml clean water). Repetition is done 3 times. After it was diluted and observed, yellow waste water samples and the results of all the variables of waste water in this study can be seen in Table 1.1

Table 1.1

Distribution of Chemical Waste water Variables Before Treatment

Variables	Unit	The Result Before Treatment			
		1	2	3	Avarage
pH	-	1,2	1,3	1,1	1,2
Pb	mg/L	5,95	6,1	5,85	5,95
Cu	mg/L	0,95	1,0	0,86	0,95
Cr	mg/L	2,08	1,8	1,95	2,08
Mn	mg/L	13,23	11,8	12,6	13,23

Table 1.1showsthatall the variablesof chemical waste water areveryacid in pHandother variables are above waste water quality standards(KLH MenNo.5of 2014).

2. The Process of pH Value Enhancement

Metcalf&Eddy(1991) states thatawaste watercontainingmetalswitha low pHcannot be coagulated andflocculated. Furthermore, tobe able toperformcoagulationandflocculationof the waste water,pH value must be increased first.In this researchtoraise the pH ofthe coagulantusedCaCO3. Furthermore,the treatmentis doneby usingCaCO3tothreerepetitionsused as much as6 g, 7g and8g. And the combination ofCaCO3+Al2(SO4) 3is as much as6 g+1.5g, 7g+1.5g and8g+1.5g.

3. Univariate Analysis

Results ofunivariate analysisshoweda decrease inthe variablerateof waste waterbefore and after treatment, as Table 1.2

Table 1.2

The frequency distributionbefore and after treatmentinchemical waste water

Variables	Pre	Treatment											
		6 gr	%	7 gr	%	8 gr	%	6+1.5 gr	%	7+1.5 gr	%	8+1.5 gr	%
pH	1,2	11,8	-	13,0	-	13,2	-	8,7	-	9,6	-	11,3	-
Pb	5.95	1.07	82.02	1.03	82.69	0.74	87.56	0.6	89.92	0.91	84.71	0.69	88.40
Cu	0.95	0.29	69.47	0.63	33.68	0.83	12.63	0.64	32.63	0.3	68.42	0.33	65.26
Cr	2.08	0.005	99.76	0.008	99.62	0.007	99.66	0.008	99.62	0.012	99.42	0.011	99.47
Mn	13.23	1.44	89.12	1.28	90.33	0.29	97.81	0.7	94.71	0.71	94.63	0.22	98.34

Table 1.2showsthat the pH valueincreasedfrom8.7 to 9.6at a dose of6 gCaCO3+1.5gand7CaCO3+1.5gAl2(SO4) 3. TSSdecreased to77.5% at the dose of7grCaCO3+1.5gAl2(SO4) 3. CODdecreased to99.94% at a dose of8gCaCO3+1.5gAl2(SO4) 3. Pbdecreased to89.92% at a dose of6 gCaCO3+1.5gAl2(SO4) 3. Cudecreased to69.47at a dose of6 grams ofCaCO3. Crdecreased to99.76% at a dose of6 grams ofCaCO3. Mndecreased to98.34% at a dose of8gCaCO3+1.5gAl2(SO4) 3andFedecreased to99.73% at a dose of6 gCaCO3+1.5gAl2(SO4) 3.

4. Bivariate Analysis

a. Anova Test Analysis

The result of Anova Test Analysis shows homogeneity of the data to pH, TSS and Cu more than 0,05:

1) The Influence of pH Enhancement

The pH ofthe chemical waste waterpreviouslyacidicmayincrease tobaseaftertreatmentas in table1.3

Table 1.3

Avarage Distribution of Chemical Waste water pH Value

Variable	Mean	SD	95% CI	*p*
Pre	1,2	0,1	0,9516 – 1,4484	0,000
6 gr	11,8	0,53	10,4855 – 13,1145	
7 gr	13	0,2	12,5032 – 13,4968	
8 gr	13,2	0,4	12,2063 – 14,1968	
6+1,5 gr	8,7	0,2	8,2032 – 9,1968	
7+1,5 gr	9,6	0,1	9,3516 -9,8484	
8+1,5 gr	11,3	0,1	11,0516 – 11,5484	

Table 1.3showsthere isa significant enhancement inpHwith ap-value(0.000) <0.05 and it is verysignificant. pHis adetermining factor in chemical waste water treatment, increasing thepHcan be doneby addingoneormorecoagulants.

2) The Differences of Cu Contain in Waste water

Table 1.4

Avarage Distribution of Chemical Waste water

Variable	Mean	SD	95% CI	*P*
Pre	2,08	0,069	1,9079 – 2,2521	0,000
6 gr	0,005	0,001	0,0025 - 0,0075	
7 gr	0,008	0,0017	0,0037 – 0,0123	
8 gr	0,007	0,001	0,0045 – 0,0095	
6+1,5 gr	0,008	0,002	0,0030 -0,0130	
7+1,5 gr	0,012	0,001	0,0095 – 0,0145	
8+1,5 gr	0,11	0,001	0,0085 – 0,0135	

Table 1.4 shows the differences on Cu level before and after treatment at p value (0,000) < 0, 05. It means that there is a significant difference in Cu level in the decrease of Cu.

b) Analysis of Kruskal-Wallis test

The result analysis of Kruskal-Wallis Test shows that there is data that is not homogeny; Pb, Cr and Mn less than 0, 05:

1) The Differences of Pb Content in Waste water

Table 1.5

Avarage Distribution of Pb in Chemical Waste water

Variable	Mean Rank	*P*
Pre	20	0,004
6 gr	16	
7 gr	15	
8 gr	8	
6+1,5 gr	2	
7+1,5 gr	11	
8+1,5 gr	5	

Table 1.5 shows that there are a significant differences on Pb level before and after treatment at p value p=0,004 < 0,05.

2) The Differences of Cr Content in Waste water

Table 1.6

Avarage Distribution of Cr in Chemical Waste water

Variable	Mean Rank	P
Pre	20	0,007
6 gr	2,33	
7 gr	8,67	
8 gr	6,83	
6+1,5 gr	8,5	
7+1,5 gr	16,33	
8+1,5 gr	14,33	

Table 1.6 shows that there are real differences on Cr levels before and after treatment at p value p = 0.007 <0.05

3) The Differences of Mn Content in Waste water

Table 1.7

Avarage Distribution of Mn in Chemical Waste water

Variable	Mean Rank	P
Pre	20	0,004
6 gr	17	
7 gr	14	
8 gr	5	
6+1,5 gr	9,17	
7+1,5 gr	9,83	
8+1,5 gr	2	

Table 1.7 shows that there are real differences on Mn levels before and after treatment with p value $p = 0.004 < 0.05$

Discussion:

1. The effect of dose of CaCO3 and CaCO3 + Al2SO4 to pH.

The degree of acidity or often called pH is the determining factor of the decline of heavy metals. Based on the survey results revealed the initial pH or pH prior to the addition of calcium bicarbonate showed pH 1.2. This situation raises the metal fraction is dissolved or soluble so the process of precipitation or deposition cannot be done, it is necessary to attempt to raise the degree of acidity by adding CaCO3 as a buffer. The result of the addition of CaCO3 impacts on the increase in pH which can be seen in Figure 1.1 below.

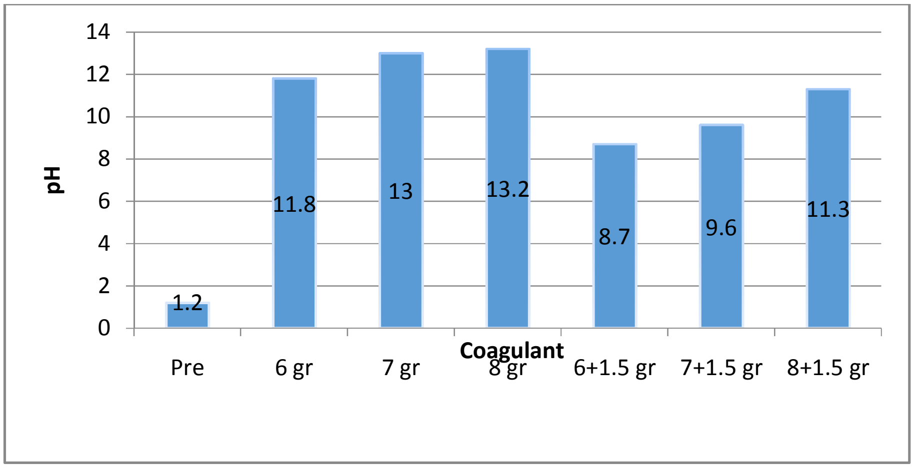

Figure 1.1

Effect of Coagulant on Changes in pH content.

Based on the figure 1.1 it can be seen that the greater amount of CaCO3, the higher amount of pH value. This is due to the number of OH- ions are much more than compared with H + ions. OH- ions derived from CaCO3 + H2O released at water as a solvent. The increase of the highest pH is in treatment to 8 g CaCO3 with the increase in pH to 13.2. While the increase lowest pH occurred in the treatment to 6 g CaCO3 + 1,5 gr Al2SO4, this happens because the initial pH increased become decreased due to the addition of the acidic Al2SO4 resulting in a decrease in pH. Suprihati and Indrasti. NS., (2010), in the research, that to raise the pH in 1 L of waste water from pH 1.4 to 6-14 takes about 600 grams of NaOH. Conversely, to decrease the waste water from pH 14 to pH 7 take approximately 40 mL of H2SO4 1 N. This shows that the use of NaOH is not economical when compared with the use of CaCO3 on the pH adjustment.

2. Effect of Dose Caco3 and Al2 (SO4) 3 to the Decline of metals Pb 2+

Heavy metal is a chemical element that can endanger the lives if its concentration exceeds a predetermined threshold. According to Kim in Suprihati and Indrasti NS., (2010), that heavy metals such as Cr and Pb can be removed easily when the system single ions and these ions have an inhibitory effect on the allowance for other ions. Based on the Metcalf and Eddy (1991), heavy metals will dissolve under normal pH and largely insoluble (insoluble) in alkaline conditions. Furthermore, Metcalf and Eddy (1991), Pb will be deposited to the maximum occurs at pH: 9.4, but based on preliminary test if the pH is conditioned under 10 then the precipitation is not running optimally so it is decided to do a precipitation with pH> 10. Figure 1.2 is known that Pb metal content before processing is 5.95 with a pH of 1.2. In the treatment group, giving CaCO3 decreases Pb metal to the highest level on the addition of 8 g CaCO3 / l while the lowest decline occurred in metals Pb treatment CaCO3 addition of 6 g / l. This shows that more and more CaCO3 affixed the higher the pH value and the higher the decline of the heavy metal content. This is in line with research Ahmad Khalil et al.¸ (2012) decline in metals Pb 2+ using Calcium carbonate produces a high decline reaching 99.94%.

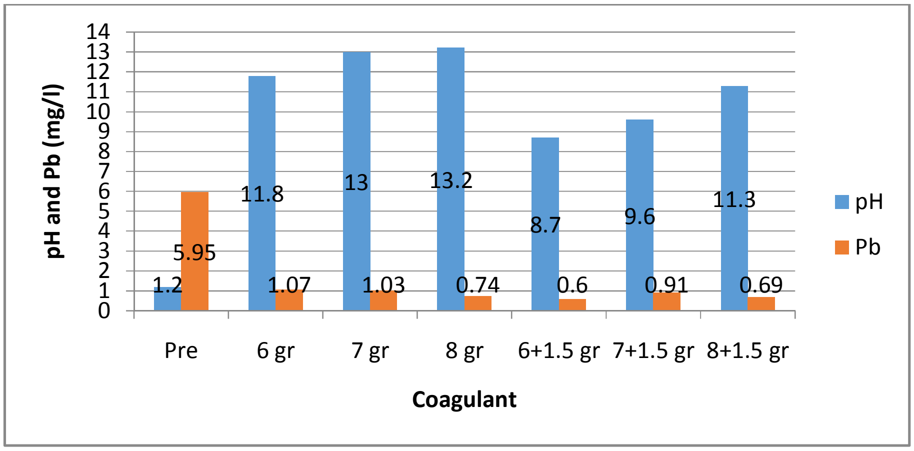

Figure 1.2

The Effect of Coagulant on the Changes of Pb Metal Content

The results showed that CaCO3 is significant in absorbing some heavy metals including Pb 2+. Moi Pang F., (2009) tested several types of coagulants such MgCl2 and NaOH, the result is very satisfactory with the decline more than 99% and the most effective way is to add MgCl2. As we know that mineral Magnesium is the calcium binding, so that if there is Calcium, there will also be Magnesium there.

The addition of coagulant in the form of Al2 (SO4) 3 also influenced to the decrease of Pb, but the addition of Al2 (SO4) 3 gives the impact to the decrease in pH, due to acidic Al2 (SO4) 3. The rate of decline was highest in the combination of 6 grams CaCO3 + 1,5gr Al_2 (SO4) 3 while the lowest decline is in the combination of 7 g CaCO3 + 1,5gr Al2 (SO4) 3, whereas the combination of 8 g CaCO3 + 1,5gr Al2 (SO4) 3 decline back to 0.69 mg / l. Despite the decrease in metal content reached 89.92% Pb cannot meet the requirements of the Minister of Environment No. 5 in 2014 on effluent quality standards. Based on the statistical test by using Benferroni it is not found significant changes in all treatments, either using CaCO3 + Al2 (SO4) 3 and CaCO3 single coagulant.

3. The Effect of Dose NaHCO3 and Al2 (SO4) 3 to the decline of metal Cu 2+heavy Cu has also become one of the objects of research, although it is not found in great numbers, but the presence of Cu can harm the environment if it is not addressed well. Based on Metcalf and Eddy (1991), Cu will be deposited to the maximum occurs at pH 8.9, but based on preliminary test if the pH is conditioned under 10 then the precipitation is not running optimally so that it is decided to conduct a precipitation with pH> 10. The results of the observations are described in the following figure:

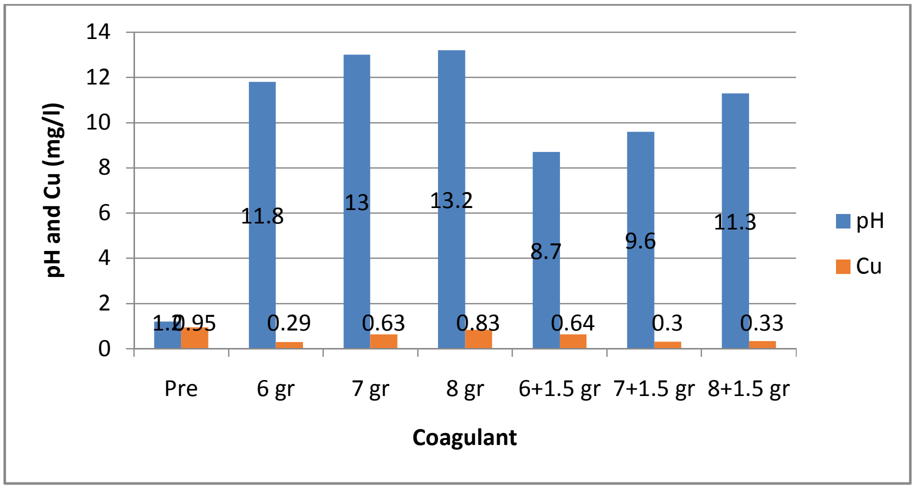

Figure 1.3

The Effect of Coagulant on the Changes of Cu metal content.

Based on Figure 1.3 it is known that Cu metal content is 0.95 mg / l. After processing by adding CaCO3 and Al2 (SO4) 3 showed the highest decrease in treatment 6 g CaCO3 by 0.29 mg / l. whereas at the group CaCO3 + Al2 (SO4) 3 obtained the highest decline at treatment 7 g CaCO3 + 1,5gr Al2 (SO4) 3¬ at 0.3 mg / l.

Research conducted by Ahmad Khalil et al.¸ (2012) doing absorption used CaCO3 to some heavy metals, one of them is metal Cu 2+ and obtain significant results. From the research results, it is known that the decline of heavy metals Cu 2+ in industrial waste water reached 99.96%. This indicates that the researchers did research in line with what is done by Ahmad Khalil. According to P. Venkateswarlu, et. al, (2007) the paper presented in this study used adsorbents to remove industrial waste chromium. Study equilibrium systematically carried out in a batch process, which includes a variety of parameters including the metals Cu 2+ which includes the time of agitation, the adsorbent and the size of the dose, initial chromium concentration, solvent volume and pH. Absorption process is done to reduce several metals and showed a significant achievement.

Handoko C.U.T., Yanti T.B., Syadiyah H., Marwati S. (2013) showed that 5% of calcium solvent can also reduce levels of Cu significantly when compared with the initial sample. The decline percentage in Cu content for the sample A is 99.99% and for the sample B of 99.9958%. This indicates that this method is effectively used to reduce levels of waste Cu silver industry. Based on the results statistically using Bonferroni method, it is not found a significant decrease in Cu content in all treatments. If the terms of the Regulation of the Minister of Environment no. 5 of 2014 concerning effluent standards that all treatments have met the standards set.

4. Effect of Dose NaHCO3 and Al2 (SO4) 3 To the Decrease of metal CR2 + Metal Cr acted as the research object because Cr can harm the environment if it is not managed properly. Metcalf and Eddy (1991), Cr will be deposited to the maximum occured at pH 8.5, but based on preliminary test if the pH is conditioned under 10 then the precipitation is not running optimally so that it is decided to conduct a precipitation with pH> 10. The results of the observations are described in the following figure:

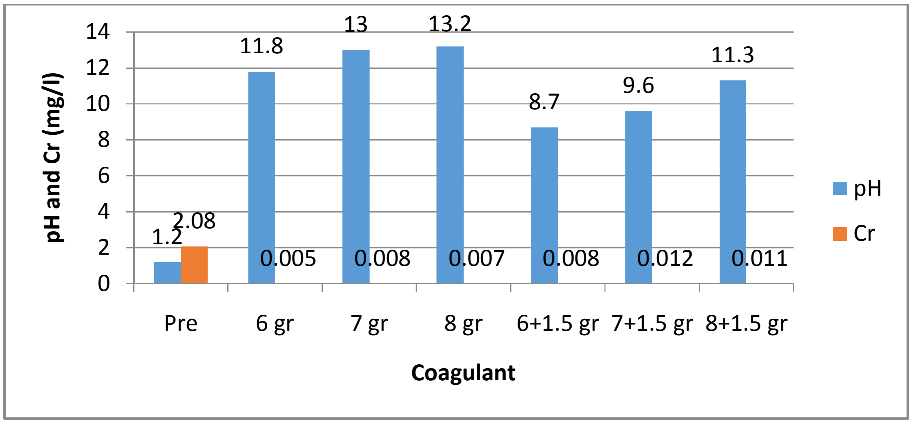

Figure 1.4

The Effect of Coagulant on the Changes of Cr metal content

Figure 1.4 is known that before the treatment, Cr metal content is 2.08 mg / l and after treatment there are various kinds of decline such as the highest decline occurred in the addition of calcium as much as 6 grams with the reduction reached 0.005 mg / l or 99.76%. While the decline in metal content of Cr were lowest in 8 gr treatment $CaCO_3$ + 1.5 g $Al_2(SO_4)_3$ it is equal to 0,012 or 99.42%.

The addition of calcium resulted in the increase on the number of OH^- ions that cause a solution under alkaline conditions. This theory is in line with the statement of Metcalf and Eddy (1991) that Cr would effectively settle at pH 8.5. Every time the calcium added, there is an increase on pH and if there is an increase in the pH, the metal used to be insoluble become soluble. It also happens on the addition of calcium and alum.

Giacinta M., (2014) conducted a study on the processing of heavy metals chromium (Cr) in the waste water of leather tanning industry with coagulation, flocculation and precipitation process and got the result that the amount of calcium which has a big influence in decreased levels of metals in the waste water of tanneries is 6.4 gr / l. whereas alum dosage of the most effective use is on 0.4 gr / l. This is consistent with what the researchers did by adding a dose of calcium with a variation of 6, 7, and 8 g / l. Dose was obtained from the calcium treatment using Jar test.

Suprihatin and Indrasti NS, (Th.2010), stated in the preliminary research on heavy metals from laboratory waste water with precipitation and adsorption methods get results that Cr elimination rate as high as 97% achieved at pH 10, the adsorption then lowering the levels of Cr in the filtrate precipitation results. The efficiency rate of decrease in concentration and minimum concentration that can be achieved depends on the type of metal and a dose of activated carbon.

Based on the statistical results using the Bonferroni method was not found a significant correlation between the increase in pH to the decrease of Cr metal content in the waste water. When compared with the Regulation of the Minister of Environment No. 5 In 2014 about Standard waste water treatment that all treatments have fulfilled the requirements to discharge to the environment because its value is below a specified threshold.

5. Effect of Dose $NaHCO_3$ and $Al_2(SO_4)_3$ to the Decline of metals Mn^{2+} Metals content of Mn contained in the laboratory waste water is quite high, reaching 13.23 mg / l,

after treatment by raising the acidity found a decrease in Mn metal content as shown below:

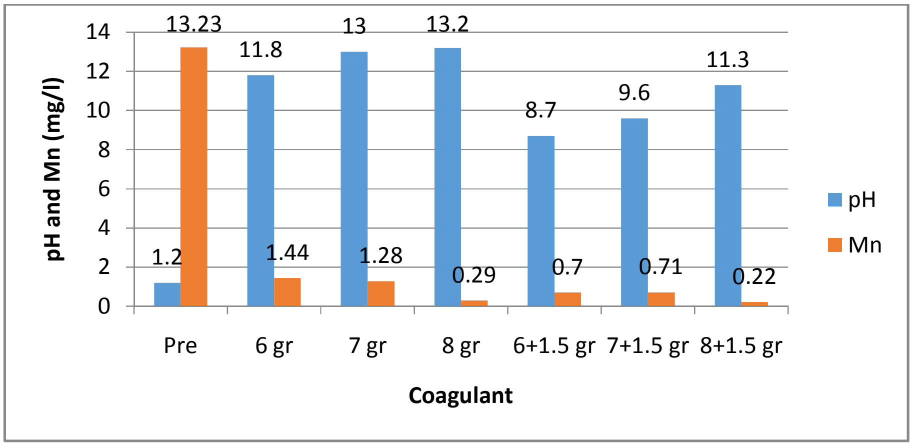

Figure 1.5

The Effect of Coagulant on the Changes of Mn metal content

Figure 1.5 the decline is quite high, from 13.23 mg / l to 0.22 mg / l. This incident along with statements Metcalf and Eddy(1991) that the solubility of metals is influenced by the degree of acidity, The lower the pH, the more dissolved metals in the water as well as contrary. The highest decline in the group occurs in the addition of calcium at doses of 8gr / l with a pH level of increase to 13.2 originally a pH of 1.2. While the decline found in the lowest dose of calcium at 6 g / l with Mn metal decline rate of 89.12%. Whereas in the group a combination of calcium and alum got the highest decrease in the dose of 8 grams of CaCO3 and 1.5 grams of Al2 (SO4) 3. This is due alum or aluminum sulfate has the ability to precipitate heavy metals, including Mn, so the results obtained high drop reaching 98.34%. When compared with the Regulation of the Minister of Environment No. 5 In 2014 about waste water treatment standard that all requirements meet effluent standards for a maximum of 2 mg / l.

Conclusions:

1. Coagulation flocculation by using calcium carbonate and aluminum sulfate can reduce levels of Pb 89.92% at doses of 6 + 1.5 g and test results Kruskal-Wallis test showed significant differences with $p = 0.004 < 0.05$ before and after treatment.
2. Coagulation flocculation by using calcium carbonate and aluminum sulfate can reduce levels of Cu 69.47% at doses of 6 grams and ANOVA test results showed significant differences with $p = 0.000 < 0.05$ before and after treatment.
3. Coagulation flocculation by using calcium carbonate and aluminum sulfate can reduce levels of Cr 99.76% at doses of 6 grams and test results Kruskal-Wallis test showed significant differences with $p = 0.007 < 0.05$ before and after treatment.
4. Coagulation flocculation by using calcium carbonate and aluminum sulfate can reduce levels of Mn 98.34% at a dose of 8 + 1.5 g and test results Kruskal-Wallis test showed significant differences with $p = 0.004 < 0.05$ before and after treatment.
5. Design of waste water treatment laboratories Poltekkes Kemenkes Bengkulu with P = 1 m, L = 2 m and H = 1 m.

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