

The Mixture of Limes in Acid Mine Drainage Treatment

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Abstract In this study the mixture of limes was used to treat acid mine drainage (AMD) or acid rock drainage (ARD). The results showed that AMD can be treated by a mixture of limes (CHL). CHL was a mixture of carbide and hydrated limes. Arsenic (As) was decreased after reaction with CHL. 0.5 g of CHL with an interval time of 20 mins was chosen as the best parameter for treating AMD. pH value was increased from 3.29 to 6.69 and as concentration was decreased from 3.273 mg/L to 0.002 mg/L. Both of the results obtained after the reaction complied with Standards A and B of the Environmental Act 1974.

Keywords: Limes, acid mine drainage, pH, arsenic.

Introduction

The lime neutralisation method is widely used in acid mine drainage (AMD) treatment. The main objective of using lime is to bring the pH of AMD to a point where the heavy metals can no longer dissolve. Certain heavy metals such as zinc (Zn), iron (Fe) and copper (Cu) are precipitated at a pH of around 9.5. Other heavy metals such as cadmium (Cd) and nickel (Ni) need a higher pH of 10.5 to 11 to precipitate in hydroxides formed [1]. The method that used lime as a neutralisation agent can be considered an active treatment. In this study, hydrated and carbide limes were mixed and used in treating AMD. Using quicklime or hydrated lime is suitable for treating highly acidic water and is very simple to handle [2, 3]. Nevertheless, the main problem of using lime is the large amount of sludge produced which consequently caused difficulties in dewatering sludge, high cost to dispose of sludge and blockage of equipment and pipelines [4, 5]. The objective of this study is to show that the mixture of limes is capable of treating AMD by increasing the pH and reducing arsenic (As) content in AMD. CHL is a mixture of carbide and hydrated limes.

Limestone is suitable to be used to neutralise acidic water (H_2SO_4) and removed Fe(III), Fe(II) and Al(III). While lime is suitable to be used to treat heavy metals such as Mn, Cu and Zn [6]. Lime such as hydrated lime ($Ca(OH)_2$) can precipitate heavy metals but it cannot completely reduce Cu from AMD. The Sulphidisation is combined with precipitation to optimise in removing Cu from AMD [7]. The previous study showed that at pH 5.5 and 7.0, Cu was completely removed from acidic water using the combination method of precipitation and sulphidisation [8]. Most of the heavy metals precipitate as hydroxide at pH 6 to 9 except ferrite iron, which can precipitate at around pH 3.5. Meanwhile, ferrous iron can precipitate at $pH \geq 8.5$. Al can precipitate as hydroxide at $pH \geq 5.5$ but would be dissolved back at pH 9. Mn can be precipitated from an acidic solution at pH 9-10 [9]. Previous work had also reported the complete removal of Mn from AMD at pH 10.5 [10].

Hydrated lime is the most common alkaline material that is being used in treating AMD compared to other alkaline materials. More than 90 % of the countries that encountered AMD problems use hydrated lime for treatment [10]. Table 1 shows the common chemical reagents used in AMD treatment [10, 11, 12, 13, 14, 15, 16, 17, 18].

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Table 1. The chemical reagents used in AMD treatment

No.	Common Name	Chemical Name	Formula	2010 Cost (\$/ton or gal)	2023 Cost (\$/ton or gal)
1	Limestone	Calcium carbonate	CaCO ₃	60/ton	100/ton
2	Hydrated lime	Calcium hydroxide	Ca(OH) ₂	275/ton	200-300/ton
3	Pebble quicklime	Calcium oxide	CaO	355/ton	100-500/ton
4	Soda ash	Sodium carbonate	Na ₂ CO ₃	840/ton	1000/ton
5	Caustic soda (solid)	Sodium hydroxide	NaOH	1240/ton	1140-1280/ton
6	Liquid caustic 20%	Sodium hydroxide	NaOH	0.95/gal	4.73/gal
7	Liquid caustic 50%	Sodium hydroxide	NaOH	2.35/gal	60/gal
8	Ammonia	Anhydrous ammonia	NH ₃	1000/ton	1237/ton

A common by-product produced after the reaction between hydrated lime with AMD is gypsum (CaSO₄.2H₂O) as shown in Equation (1). The reaction can occur between heavy metals such as nickel with ion hydroxide (OH⁻) as shown in Equation (2):

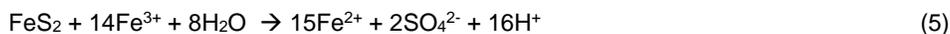
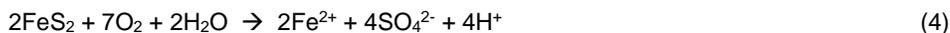


Carbide lime (CL) is considered a solid waste and it is produced as a by-product of acetylene gas [19]. Besides being used as a material to treat wastewater, it can be applied as a starting material to produce synthetic calcium carbonate (SCC) or precipitated calcium carbonate (PCC) [20]. The reaction equation for the production of acetylene gas and CL (by-product) is shown in Equation (3).



AMD is also known as acid rock drainage [21]. It is produced from active and abandoned mines [22, 3]. It contains a high concentration of heavy metals and sulphate. The heavy metals present in AMD are As, Fe, Mn, Cd, Cr, etc. The sulphate concentration in AMD is about several thousands (mg/L) [23, 3]. Acidic water with a pH of around 2.5 to 5.0 can be considered AMD [24]. The geological conditions of the mine and the presence of minerals are the main factors that can affect the components of AMD [25]. Pyrite (FeS₂) is one of the sulphide minerals that can cause AMD [26]. Other sulphide minerals that can cause AMD are marcasite (FeS₂), arsenopyrite (FeAsS), chalcopyrite (CuFeS₂), chalcocite (Cu₂S), galena (PbS), millerite (NiS), sphalerite (ZnS) and cinnabar (HgS) [27, 28]. Table 2 shows the sources of AMD [21, 29]. In Malaysia, certain mines encounter AMD issues, especially occurring in abandoned mines [30, 31, 32, 33, 34]. Table 3 shows the mines in Malaysia that have issues with AMD. [30, 31, 32, 33, 34]. A Previous study had shown that commercial quicklime and CL were used to treat AMD from an active tailing pond in Perak, Malaysia [35]. Generally, the formation of AMD can be classified into three stages [36, 37]:

- i) Oxidation of iron sulphide and enhancement of sulphide minerals by ferric iron as shown in Equations (4) and (5), respectively.



- ii) Oxidation of ferrous iron as shown in Equation (6):



- iii) Hydrolysis and precipitation of ferric iron as shown in Equation (7):



Table 2. The sources of AMD [21, 27]

No.	Primary Source	Secondary Source
1	Mine rock dump	Treatment sludge pond
2	Tailing impoundment	Rock cuts
3	Underground and open pit mine workings	Concentrated load-out
4	Pumped/nature-discharged underground water	Stockpiles
5	Diffuse seeps from replaced overburden in rehabilitated areas	Concentrate spills along roads
6	Construction rock is used in roads, dams, etc.	Emergency ponds

Table 3. The active and abandoned mines that encounter issues of AMD [30, 31, 32, 33, 34]

No.	Name of Mine	Status of Mine	Type of Ore
1	Old Repas Dam, Bentong, Pahang	Active/abandoned	Iron
2	Tasik Chini, Pahang	Abandoned	Iron/manganese
3	Bukit Besi, Dungun, Terengganu	Abandoned	Iron
4	Bukit Ibam, Rompin, Pahang	Abandoned/active	Iron
5	Sungai Lembing, Kuantan, Pahang	Abandoned	Tin
6	Selinsing, Raub, Pahang	Active	Gold
7	Mamut Copper Mine, Ranau Sabah	Abandoned	Copper
8	Pengkalan Hulu, Perak	Active	Iron

The method for AMD treatment can be classified into two: active method and passive method. The active method involves a precipitation process with alkaline chemicals such as quicklime, hydrated lime, CL, limestone, magnesium hydroxide, alkaline tailings, etc [38]. Figure 1 shows a typical method in the treatment of AMD using lime as the neutralisation agent [14]. The passive method involves biological treatment with constructed wetlands and together with limestone drains [2, 3]. For example, the combination of anoxic limestone drain (ALD), anaerobic wetland, aerobic wetland and oxidation have been used for the construction of passive treatment systems in Korea as shown in Figure 2 [39].

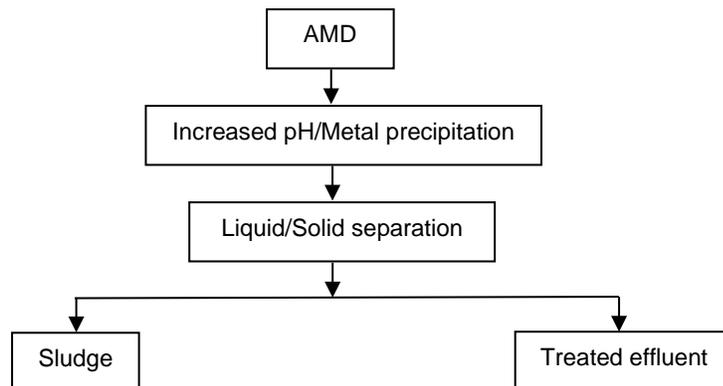


Figure 1. A typical method in the treatment of AMD using lime as the neutralisation agent

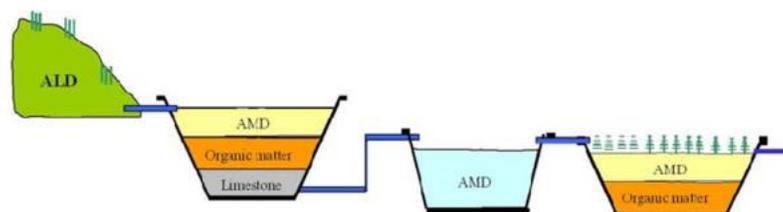


Figure 2. A schematic diagram of the passive treatment system in AMD

Materials and Methods

Materials

The chemical reagent used was hydrated and carbide limes. Hydrated lime used was obtained from a lime company in Simpang Pulai, Perak. CL used was obtained from a local acetylene gas factory in Taiping, Perak and AMD sample was obtained from a tin tailing pond in Perak.

Instrumentation

A portable pH meter (A329, Thermo Scientific, Indonesia) was used to measure the pH value in the AMD sample, X-ray Fluorescence (XRF-1700, Shimadzu, Japan) was used to determine the presence of oxide metals in CHL and Inductive Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (Optima 5300 DV, Perkin Elmer, USA) was used to determine As content in AMD sample before and after reaction. Field Emission Scanning Electron Microscopy (FESEM) (Supra 40VP, Zeiss, Germany) to obtain micrographs of CHL before and after reaction with AMD.

Methods

In this study, CHL was used to treat 0.25 L of AMD by using the Jar Test method. Six different weights of CHL were used to treat AMD. Six different weights of CHL (0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g) were in this experiment. Each weight was placed in a beaker that contained 0.25 L of AMD sample and was stirred at 200 rpm using a jar test equipment. The pH value was recorded at 10 mins intervals for 60 mins. Figure 3 shows a jar test that was carried out in the laboratory before and after the reaction. The pH values were recorded before and after the reaction using a pH meter. The content of heavy metals before and after the reaction was determined using ICP-OES.

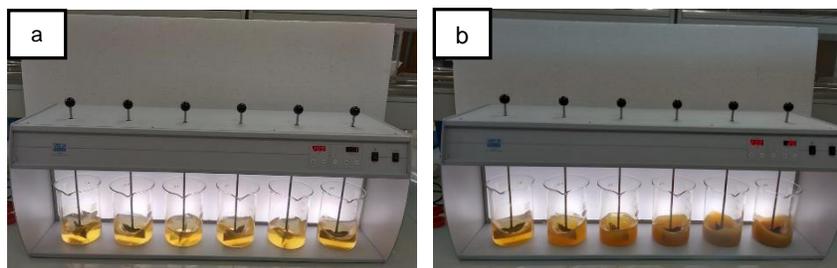


Figure 3. A jar test was conducted on AMD samples before (a) and after (b) reaction with CHL

Results and Discussion

XRF Results of CL and CHL

Table 4 shows the XRF results of CL and CHL with the major elemental oxide being CaO, respectively. Others were SiO₂, Al₂O₃, Fe₂O₃, MgO and SO₃. The percentages of CaO in CL and CHL were 95.71 % and 93.84 %, respectively indicating that both of them were suitable used as a material to treat AMD. CaO is capable to increase pH and reduce certain heavy metals such as manganese (Mn) [40]. Other materials that contain CaO are flying ash, limestone, dolomite, etc.

Table 4. XRF results of CL and CHL

Elemental Oxide	Percentage (%)	
	CL	CHL
CaO	95.71	93.84
MgO	0.39	3.44
SiO ₂	2.48	1.50
Fe ₂ O ₃	0.64	0.63
Al ₂ O ₃	0.66	0.47
SO ₃	0.12	0.12
Total	100.00	100.00

FESEM Results of CHL Before and After the Reaction

Figure 4 shows the CHL before and after the reaction with AMD and it shows the change of colour from greyish to brown-yellowish. Figure 5 shows the FESEM micrographs of CHL before and after reaction with AMD at 1000 and 15000 magnifications, respectively. The morphologies of particles have an almost rounded and irregular shape (Figure 5 (a, b)). After the reaction, the morphologies of the particles showed a less rounded and irregular shape (Figure 5 (c,d)). This is due to the neutralisation process that occurred between CHL and AMD. The process can cause rapid precipitation of iron hydroxide and gypsum [41] and heavy metals such as As, Cd, Cr, Mn, Al, etc.

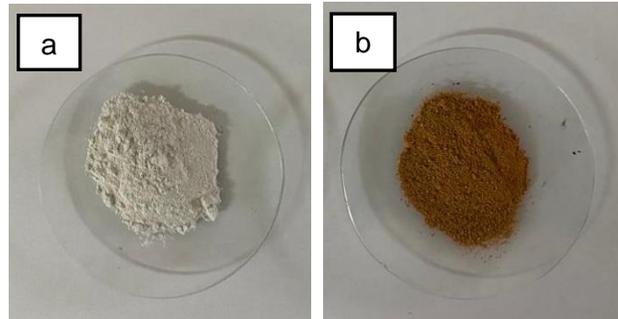


Figure 4. CHL before (a) and after (b) reaction with AMD

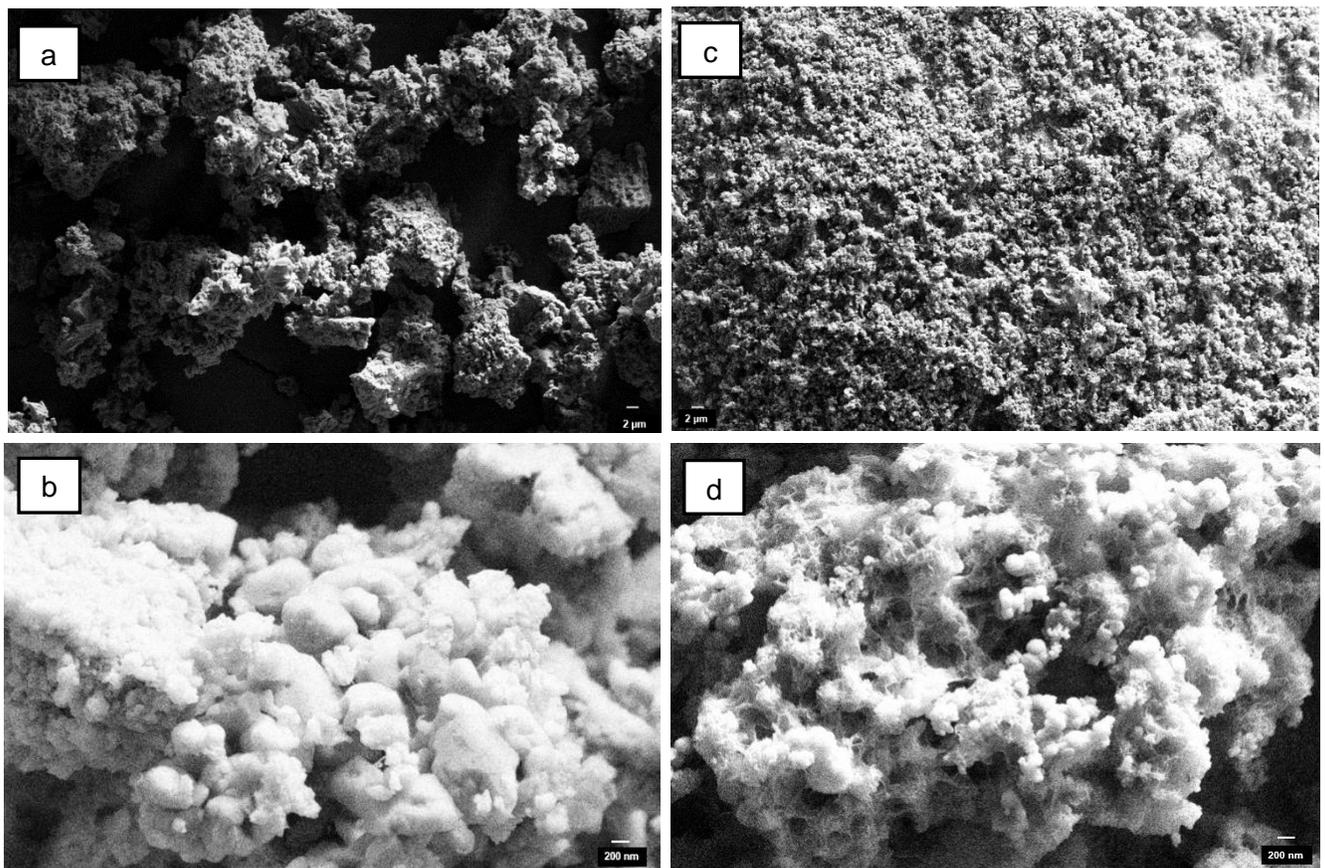


Figure 5. Micrographs of CHL before (a, b) and after (c, d) reaction with AMD at 1000 and 15000 magnifications, respectively

pH Values in AMD

Table 5 and Figure 6 show the pH value of AMD before and after the reaction with CHL, respectively. The results show the increase in pH value after the reaction. As observed in Figure 6 (a), using 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g of CHL at the interval time of 30 mins, the pH values increased from 3.29 to 3.47,

3.29 to 4.13, 3.29 to 4.79, 3.29 to 6.33, 3.29 to 9.50 and 3.29 to 10.78, respectively. Profile of the graph in Figure 6 (b) indicates that by using 0.1, 0.2 and 0.3 g of CHL, pH values had shown slight increment after 10 minutes, respectively. pH values for 0.2 and 0.3 g of CHL also showed a slight increment after 30 mins. As shown in Figure 6 (b), pH values recorded a constant trend after 30 mins of reaction between AMD with CHL. The pH values became constant due to the less hydroxide ion (OH⁻) having reacted with hydrogen ion (H⁺) and heavy metals to produce water (H₂O) and metal hydroxides. The obtained metals precipitated are separated by sedimentation and filtration [42]. Equations (8) and (9) show the dissolved of hydrated lime in water and the reaction occurred between OH⁻ and H⁺ to produce H₂O, respectively. The chemical formula of CHL is equal to hydrated lime (Ca(OH)₂). The less quantity of OH⁻ can reduce the efficiency of CHL as a neutralisation agent in increasing the pH value of AMD. The formation of gypsum (CaSO₄) also can reduce the effect of CHL after calcium ion (Ca²⁺) reacted to sulphate ion (SO₄²⁻) [43] as shown in Equation (10) [1]. From the results, it can be concluded that the best parameter chosen was 0.5 g of CHL at an interval time of 20 mins which recorded a pH value of 6.69 that complied with Standard A and Standard B of the Environmental Act 1974. pH values for Standards A and B are 6.0-9.0 and 5.5-9.0, respectively [44]. Standard A refers to the discharges of activities and industries located near catchment areas and Standard B refers to the discharge of industries and development activities nationwide [45]. The parameter (0.5 g of CHL at an interval time of 20 mins) was chosen because it can increase pH value and reduce heavy metals concentration such as Al, As, Cd, Cr, Cu, Fe, Ni, Pb and Zn except for Mn that complied with both standards compared to other parameters.



Table 5. pH of AMD sample before and after reaction with different weighs and interval times of CHL

Weight of CHL (g)	Interval Time (min)	pH	
		Before Reaction	After Reaction
0.1	10	3.29	3.44
	20	3.29	3.49
	30	3.29	3.47
	40	3.29	3.38
	50	3.29	3.38
	60	3.29	3.34
0.2	10	3.29	3.81
	20	3.29	3.87
	30	3.29	4.13
	40	3.29	5.32
	50	3.29	6.69
	60	3.29	9.39
0.3	10	3.29	3.47
	20	3.29	4.40
	30	3.29	5.15
	40	3.29	7.37
	50	3.29	9.81
	60	3.29	10.73
0.4	10	3.29	5.09
	20	3.29	5.32
	30	3.29	6.33
	40	3.29	5.96
	50	3.29	6.28
	60	3.29	6.68
0.5	10	3.29	6.01
	20	3.29	6.69
	30	3.29	9.50
	40	3.29	8.79
	50	3.29	9.28
	60	3.29	9.04
0.6	10	3.29	8.90
	20	3.29	9.39
	30	3.29	10.78
	40	3.29	10.54
	50	3.29	10.54
	60	3.29	10.36

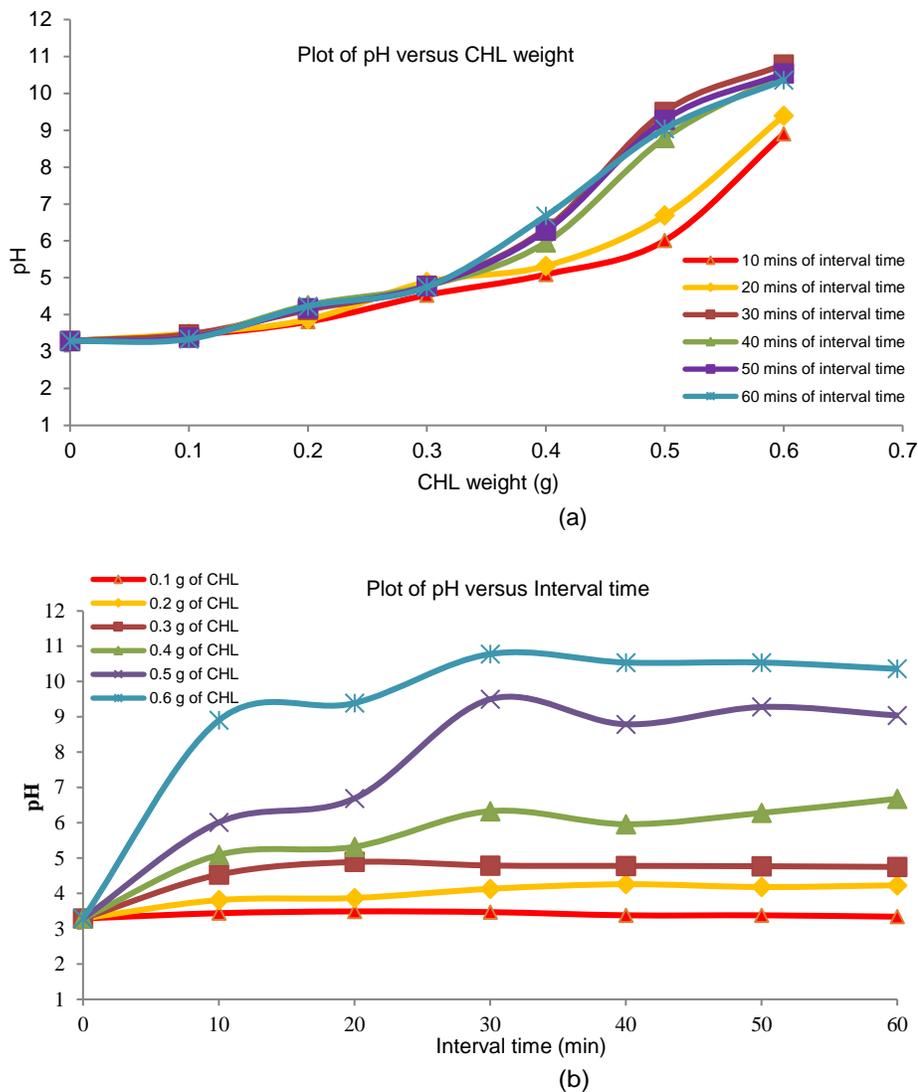


Figure 6. (a) Change of pH versus CHL weight; (b) Change of pH versus interval time

Heavy Metals Content in AMD

Table 6 shows the heavy metals content in AMD before and after the reaction with CHL. The results indicated that the heavy metals detected by ICP-OES were Al, As, Cd, Co, Cr, Cu, Fe, Mn, Ni, and Zn, while Sn was not detected. All heavy metals content obtained in the AMD sample after reaction with 0.5 g of CHL at 30 mins of interval time until 0.6 g of CHL at 60 mins of interval time complied with Standards A and B. Nevertheless, the pH values obtained from the parameters were around 9.50 to 10.36 which did not comply with Standards A and B. The best parameter chosen in this study was 0.5 g of CHL at an interval time of 20 mins which complied with both Standards for pH value and heavy metals content but only the Mn concentration obtained did not comply with Standard A which recorded 0.507 mg/L. Standards A and B for Mn are 0.20 and 1.00 mg/L, respectively. The precipitation process occurred when heavy metals such as Cu, Ni and Zn [1] reacted to OH⁻ to form metal hydroxide [46] as shown in Equations (11), (12) and (13), respectively.



Table 6. pH of AMD sample before and after reaction with different weighs and interval times of CHL

Weight of CHL (g)	Interval Time (min)	Heavy Metal Concentration (mg/L)																							
		Al	As	Cd	Co	Cr	Before Reaction		Mn	Ni	Pb	Sn	Zn	After Reaction											
							Cu	Fe						Al	As	Cd	Co	Cr	Cu	Fe	Mn	Ni	Pb	Sn	Zn
0.1	10													98.943	0.410	0.138	4.986	0.276	50.553	108.600	32.863	6.736	0.364	ND	8.368
	20													10.173	0.031	0.015	0.597	0.030	5.442	10.730	4.668	0.820	0.045	ND	1.078
	30	101.390	3.273	0.221	5.952	0.368	56.457	297.871	46.190	8.194	0.523	ND	10.960	10.435	0.041	0.016	0.602	0.030	5.621	10.256	4.631	0.822	0.047	ND	1.080
	40													10.657	0.055	0.016	0.608	0.031	5.703	10.244	4.696	0.837	0.049	ND	1.090
	50													10.177	0.060	0.016	0.593	0.031	5.486	10.970	4.605	0.812	0.047	ND	1.059
	60													10.607	0.077	0.017	0.611	0.030	5.671	10.305	4.786	0.840	0.047	ND	1.095
0.2	10													91.513	0.081	0.128	4.841	0.133	47.783	14.457	32.903	6.543	0.249	ND	7.895
	20													9.723	0.009	0.015	0.596	0.013	5.378	5.587	4.672	0.815	0.031	ND	1.042
	30	101.390	3.273	0.221	5.952	0.368	56.457	297.871	46.190	8.194	0.523	ND	10.960	8.729	0.006	0.015	0.575	0.005	5.189	0.064	4.332	0.787	0.017	ND	1.015
	40													8.691	0.006	0.015	0.579	0.005	5.248	0.057	4.438	0.799	0.021	ND	1.011
	50													9.032	0.009	0.015	0.583	0.006	5.219	0.068	4.515	0.800	0.023	ND	1.025
	60													8.646	0.008	0.015	0.590	0.004	5.225	0.064	4.597	0.811	0.021	ND	1.031
0.3	10													52.833	0.049	0.120	4.596	0.013	38.907	0.791	31.977	6.238	0.094	ND	6.650
	20													13.380	0.048	0.114	4.340	0.010	5.263	0.473	4.676	5.936	0.052	ND	1.053
	30	101.390	3.273	0.221	5.952	0.368	56.457	297.871	46.190	8.194	0.523	ND	10.960	9.413	0.042	0.113	3.988	0.008	5.176	0.019	4.653	5.287	0.029	ND	1.035
	40													2.885	0.005	0.016	0.591	0.002	4.527	0.005	4.660	0.799	0.008	ND	1.246
	50													2.572	0.006	0.014	0.558	0.002	4.196	0.010	4.374	0.766	0.010	ND	0.936
	60													2.383	0.005	0.014	0.563	0.001	4.166	0.005	4.398	0.775	0.008	ND	0.949
0.4	10													0.162	0.004	0.010	0.423	0.001	1.422	0.714	3.778	0.586	0.003	ND	0.609
	20													0.048	0.005	0.011	0.475	0.001	0.995	0.416	4.219	0.656	0.003	ND	0.674
	30	101.390	3.273	0.221	5.952	0.368	56.457	297.871	46.190	8.194	0.523	ND	10.960	0.001	0.002	0.002	0.097	0.001	0.018	0.002	2.493	0.115	0.001	ND	0.036
	40													ND	0.001	0.008	0.365	0.001	0.019	ND	3.454	0.502	0.001	ND	0.302
	50													ND	0.002	0.003	0.128	0.001	0.011	ND	2.479	0.168	ND	ND	0.184
	60													ND	0.003	0.001	0.065	0.001	0.008	ND	2.222	0.075	ND	ND	0.019
0.5	10													ND	0.001	ND	0.004	ND	0.002	ND	0.590	0.007	0.001	ND	0.002
	20													ND	0.002	ND	0.004	ND	0.002	ND	0.507	0.004	0.001	ND	ND
	30	101.390	3.273	0.221	5.952	0.368	56.457	297.871	46.190	8.194	0.523	ND	10.960	0.001	0.001	ND	0.001	ND	0.001	ND	0.031	0.136	ND	ND	ND
	40													0.001	0.001	ND	ND	0.001	ND	0.001	0.148	0.001	0.001	ND	0.007
	50													0.004	ND	ND	ND	ND	0.001	ND	0.013	ND	0.001	ND	ND
	60													0.004	0.002	ND	ND	ND	0.001	ND	0.043	ND	0.001	ND	ND
0.6	10													0.012	0.003	ND	ND	ND	ND	ND	0.059	ND	ND	ND	0.001
	20													0.010	0.001	ND	ND	ND	ND	ND	0.021	ND	0.001	ND	ND
	30	101.390	3.273	0.221	5.952	0.368	56.457	297.871	46.190	8.194	0.523	ND	10.960	0.030	0.003	ND	ND	ND	0.001	0.001	0.004	ND	ND	ND	ND
	40													0.015	0.002	ND	ND	ND	ND	ND	0.002	ND	ND	ND	ND
	50													0.022	0.003	ND	ND	ND	ND	ND	0.001	ND	ND	ND	ND
	60													0.021	ND	ND	ND	ND	ND	ND	0.001	ND	0.001	ND	ND

Abbreviation: ND, not detected

Arsenic Content in AMD

Figures 7 (a and b) shows the arsenic content in AMD before and after reaction with CHL. The results show that As concentration had decreased constantly that complied with Standards A and B started at 0.2 g of CHL with 20 mins of interval time until 0.6 g of CHL with 60 mins. For example, the concentration of As had decreased from 3.273 mg/L to 0.004 mg/L, 3.273 mg/L to 0.005 mg/L, 3.273 mg/L to 0.002 mg/L, 3.273 mg/L to 0.002 mg/L and 3.273 mg/L to 0.003 mg/L by using 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 g of CHL respectively at an interval time of 30 mins. The best parameter chosen was 0.5 g of CHL with 20 mins of interval time which recorded arsenic concentration of 0.002 mg/L, pH and other heavy metals that complied with Standards A and B except Mn complied with Standard B only. Standards A and B for arsenic are 0.05 and 0.10 mg/L [44], respectively. Compared to other chemicals such as NaOH, using lime is cost-effective and produces non-toxicity waste such as CaSO₄ [33]. Previous studies had shown that hydrated lime can reduce As to 44.9 % at pH 12 [47]. The combination of hydrated lime and iron (III) chloride can reduce As concentration to 98.6 % at pH 8 [47]. In this study, As was reduced to 99.9 % at pH 6.69 using 0.5 g of CHL with an interval time of 20 mins.

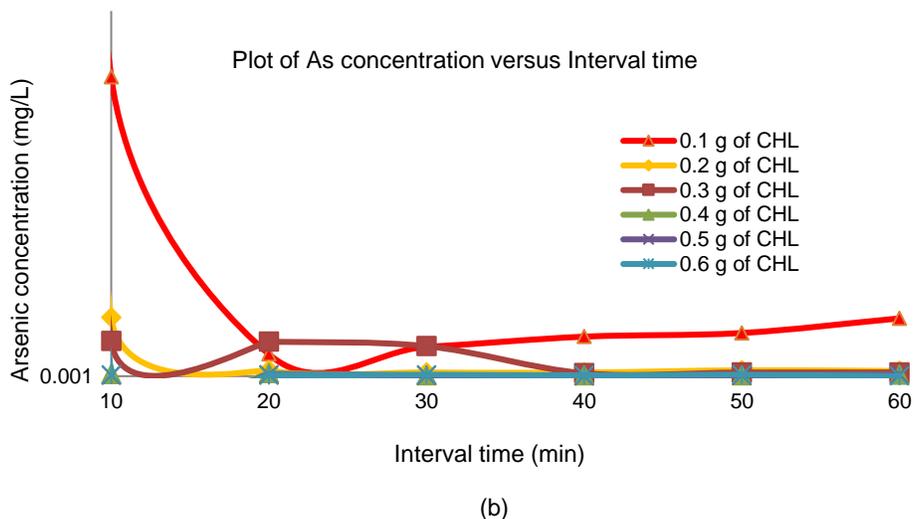
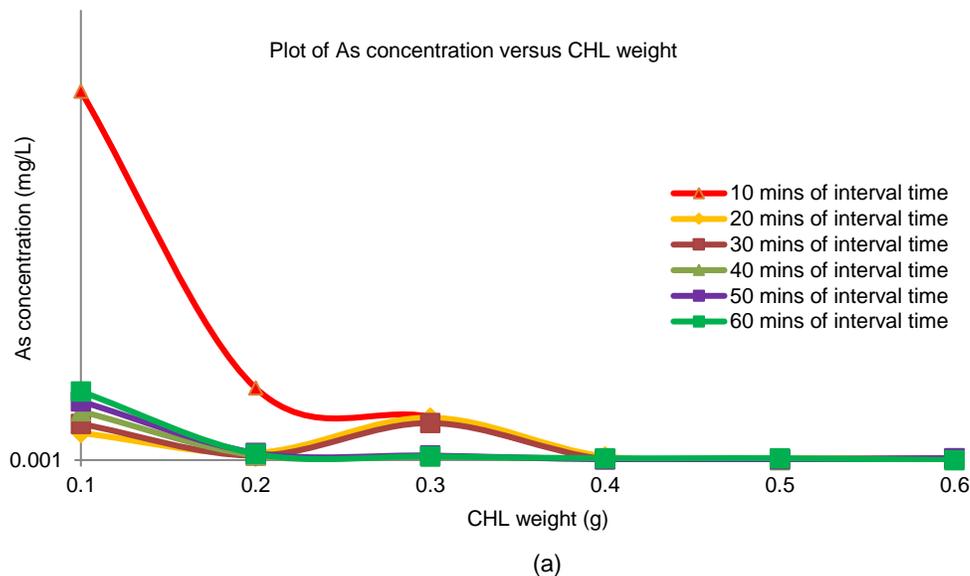


Figure 7. (a) Graph As concentration versus CHL weight; (b) Graph As concentration versus interval time

Conclusions

This study demonstrates that using 0.5 g of CHL is suitable to be used in AMD treatment because the pH value obtained was 6.69 at an interval time of 20 mins, which complies with the Environmental Act 1974 for Standard A (6 to 9) and Standard B (5.5. to 9). The arsenic concentration was also obtained at

0.002 mg/L using the parameter and complied with Standards A and B of the Environmental Act 1974. The efficiency of CHL to reduce As concentration was around 99.9 % at pH 6.69. Other heavy metals concentration that complied with both standards were Al, Cd, Cr, Cu, Fe, Ni, Pb and Zn. The standard limit of cobalt (Co) did not mention in Environmental Quality Act 1974. While the concentration of Mn obtained using the parameter did not comply with Standard A. CHL is suitable for treatment in the Malaysian mining industry. The materials used in a mixture of CHL are easy to access, economical and high efficiency for As treatment.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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