Physical leaching and toxicity characteristics of PG76 binder modified with Rediset

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ABSTRACT

Asphalt binder, as one of the pavement components, is exposed to heat and rainfall. Polymer modified asphalt binder is a good alternative to withstand the weather in tropical countries. By utilizing warm mix asphalt additive, the high compacting and mixing temperature can be reduced. However, the impact to the environment and its characteristic towards high temperature need to be evaluated before putting it to use. In this study, the physical properties, thermal characteristics, and leaching and toxicity of PG76 incorporated with Rediset LQ were investigated. The Tank Leaching Test was carried out to mimic the worst scenario of flood event effect on the asphalt binder. The results of physical tests denote that the new material has relative consistency, while the thermal analysis indicates that the materials are stable beyond their mixing and compaction temperature. The decomposition starts at temperature > 360°C and ends at temperature <500°C for all samples. The tank leaching test also found that the materials are safe to be used as pavement materials, because the heavy metal elements from the leachate are below the maximum allowable volume by the World Health Organization and the United States Environmental Protection Agency.

Keywords: WMA; Rediset, PG76; Thermal characteristics; Leaching; Toxicity, ICP-MS.

INTRODUCTION

Warm Mix Asphalt (WMA) technologies are beneficial to suppliers and project authorities, as they were developed to reduce carbon emission, which then would contribute to the reduction of global warming factor. These technologies caught many attentions due to the positive feedback received regarding carbon emission, reduced fuel consumption, and providing better working environments for the workers (Kim et al., 2012). Mixing temperature for WMA is around 100 to 140 °C, so it releases lesser carbon content compared to Hot Mix Asphalt (HMA) pavements (Rubio et al., 2012). WMA additives used in WMA technologies can be classified into three groups, which are chemical additives, foamed asphalt binder, and organic additives. Chemical additives added to asphalt mixture can also promote adhesion quality by reducing the viscosity of the mixture, improving workability of the binders, and easing compaction process (Mo et al., 2012). Rediset is one of the additives used to reduce the temperature and viscosity of the mixture. It was first introduced in USA in 2007 and had been used and analyzed ever since, as it has shown a good improvement to the road mixtures (Bonaquist, 2011). Rediset comes in two forms, liquid and solid, depending on the usage and applications (Akzonobel, 2014). Rediset can be added to the asphalt binder directly or put into mixture chamber (Hamzah et al., 2014). Different amounts of Rediset produce different amounts of reduction in mixing and compaction temperatures and also promote different viscosity of the mixture (Arega et al., 2011; Trujillo, 2011; Sampath, 2010). According to Hanz et al. (2010), warm mix asphalt has three mechanisms: (1) reducing viscosity of asphalt mixture by adding organic additives, (2) hydrophilic additives or water injection added to the mixture to perform foamed asphalt binder, and (3) reducing frictional forces between the aggregates and asphalt binder and promoting easier coating. By using Rediset, it also can promote mitigation of internal friction between the binder and aggregate (Arega & Bhasin, 2012). Due to wide usage of WMA, environmental concern has been on the rise since WMA often associated with various chemical addition in order to achieve favorable results. Though a past study has shown that the additives used in WMA have positive impact in terms of suppressing the emission of pollution gaseous during their manufacture (Abdullah et al., 2016), road pavements are heavily exposed to climate change, extreme temperature changes, and rainfall. In order to improve the durability of the pavements, modification by means of the addition of additives is an option. The addition of synthetic zeolites is used to produce foaming process, which are composed of aluminosilicates of alkali metals (Vidal et al., 2013). Leaching test is often used to simulate a submerging solid in water for a certain period of time (Chai et al., 2009), and the result can be used to estimate the amount leached. The leachate is then analyzed by using latest equipment and tools such as Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (Hiller at al., 1999; Fadzil et al., 2010).

With the availability of recently developed technology, WMA has been involved in mixture with chemical and organic additives (You & Goh, 2008). However, the leachability of heavy metals from WMA technologies is still lacking in depth of information. Leachability is one of the issues apart from polluted gaseous that may increase the risk of the environment pollution regarding road pavement. Afonso et al. (2020) found that permeable asphalt pavement and a conventional asphalt pavement had shown some leaching on Cr, Fe, Mn, Ni, Pb, and Zn. On the other hand, even though Awazhar et al. (2020) studied the leaching of WMA, the study focuses on short-term effect only.

This study was done with the objective of determining the physical characteristics of PG76 with the modification of Rediset addition to gauge the physical abilities through the tests of penetration, softening point, and ductility tests for the modified samples of asphalt binder PG76. Tank leaching test was conducted to simulate heavy rainfall and flash flood of the road as the sample submerged in acidic solution (pH: 4) for a set period of time (0.25, 1, 2.25, 4, 9, 16, 32, and 64) (NEN 7347, 2006: Fadzil et al., 2010) and projected to observe long-term effect. The leachate is then analyzed by ICP-MS instrument, and then, the cumulative results of the heavy metals, Cd, Cu, Zn, Ni, Pb, Cr, Al, and Co, would be tabulated and compared with Department of Environmental guidelines to determine their toxicity.

EXPERIMENTAL DESIGN

Materials and Sample Preparation

In this study, performance grade asphalt binder PG76 (PG76) and Rediset LQ1106 (Rediset) are used. PG76 and Rediset are mixed together as the Rediset is added according to the weight percentage of the binder, which is 1, 2, 3, 4, and 5% of the total binder weight. Even though the manufacturer recommended 0.3-1.0% of binder weight for warm mix asphalt application (AkzoNobel, 2014), this study used 1 to 5% weight to observe the impact of Rediset on PG76 in terms of physical properties of the binder and the contribution of PG76+Rediset to the soil contamination. The two materials are mixed by using rotary shear mill with velocity of 1250 rpm at temperature of 160 °C for 30 minutes. Physical characteristics for PG76 are listed in Table 1.

Table 1. Physical properties of PG76.

Test	Unit	Result	Requirement	Test Standard
Quality specification				
Softening point	°C	93	Min. 70 °C	ASTM D36
Penetration	0.1 mm	46	Min. 45	ASTM D5
Flash Point	°C	343	Min. 260 °C	AASHTO T48
Performance specification				
Viscosity at 135°C	Pa s	2.45	Max. 3 Pa s	ASTM D4402
Dynamic shear, G*/sin δ	kPa	2.10	Min. 1.00 kPa	AASHTO T315
Test temp @ 10 rad/s, 76°C				

Source: Yusoff et al. (2014)

Physical Tests

Penetration test, based on ASTM D5, was conducted to determine the hardness and consistency of the asphalt binder. Softening point test, on the other hand, is the determination of the point of which the binder starts to soften. This ring and ball test was done according to ASTM D36. In ductility test, the ductility value is determined according to ASTM D113-86.

Thermogravimetry Analysis

Thermogravimetric Analysis (TGA) was carried out for this study to assess the weight change of the sample with the function of temperature under controlled conditions. The analysis was done for this study to observe the decomposition state of PG76+Rediset. The changes of weight were observed up to maximum of 600 °C. The analysis was carried out using Shimadzu TGA-50 thermogravimetric analyser in a protective atmosphere of nitrogen, with a flux of 50 ml/min and a heating rate of 10 °C/min. The analyses were carried out with reference to ASTM E 1311.

Tank Leaching Test

This test was conducted to determine the leaching of inorganic components from compacted granular materials as the solid materials submerged under acidic solution. In this study, a total of 49 samples including one controlled sample were tested using acidic solution to simulate continuous heavy rainfall or flash flood making the materials to leach inorganic components. The samples of unmodified PG76 and modified PG76 with addition of 1, 2, 3, 4, and 5% Rediset were submerged in acidic solution in a bottle container with the ratio of 1:20; 1 gram of sample will be submerged in 20 mL acidic solution. These samples were partitioned according to duration of acidic solution exposure (0.25, 1, 2.25, 4, 9, 16, 36, and 64) days before extraction of the solute using filter with porosity of 0.45 μm as they need to be analyzed later using ICP-MS with the samples being sent prepared in 10 mL tube bottle. This test is based on NEN 7345.

Inductively Coupled Plasma Mass Spectrometry

After the extractions of the solutes from the samples from leaching tank test, the solutes were then brought to ICP-MS laboratory at Faculty of Science and Technology, UKM, to be analyzed. ICP-MS is the spectroscopy that uses an inductively coupled plasma in order to ionize the sample. During the analyzing process, it atomizes samples creating atomic and polyatomic ions, which would later be detected. In this study, the heavy metal elements analyzed are Cd, Cu, Zn, Ni, Pb, Cr, Al, and Co, which were later on plotted in the form of graph of cumulative release per unit area (mg/m^2) for each element as a function of time (days). The ICP-MS used is modelled after Perkin Elmer SCIEX Elan 9000.

RESULTS AND DISCUSSION

Physical Properties

From the penetration test result according to the ASTM D5 standard shown in Table 2, the penetration value increase, as well as the increases of percentage of Rediset in PG76 binder. It is reported that penetration value of the PG76 binder with 1%, 2%, 3%, 4%, and 5% Rediset was 42.1, 47.5, 49.9, 55, and 60.8 dmm, respectively, while it was 46 dmm for the control sample. This explained that the higher the percentage of Rediset content in PG76 yield, the softer the asphalt binder behavior. In order to have better resistance towards road damage due to fatigue failure especially in warmer climate region, R0 is the best choice in terms of penetration value obtained, while R5, on the other hand, showing the least favored as it would be best to be applied for a colder climate region. Thus, from the physical tests conducted, penetration test shown could suggest that R1 is better to be used in warmer climate region in terms of penetration value at 42.1 dmm, while R5 can be a choice for colder climate region.

Table 2. Results of physical tests for modified PG76 with 1-5% Rediset addition.

Sample	Tests		
	Penetration (dmm)	Softening Point (°C)	Ductility (cm)
R0	46.0	90.0	140.0
R1	42.1	64.2	110.4
R2	47.5	62.3	102.8
R3	49.9	66.3	140.0
R4	55.0	61.7	108.5
R5	60.8	68.7	120.7

Softening point test was conducted according to ASTM D36. The softening point temperature recorded for the control sample was 90 °C, while 1%, 2%, 3%, 4%, and 5% Rediset samples were 64.2, 62.3, 66.3, 61.7, and 68.7 °C, respectively. The softening point value of the binder R5 is the highest at 68.7 °C compared to the other samples, while R4 is the lowest at 61.7 °C. Temperature drop for the softening point as the content of Rediset increased except for samples of PG76 with 3 and 5% Rediset addition. The softening point temperature and penetration values are interconnected. The lower the softening point temperature, the higher the penetration value of binder.

Ductility test was performed according to ASTM D113 to test the elasticity of modified asphalt binder. The length where the asphalt binder sample starts to break is recorded in centimeter as the ductility value. Minimum ductility value should be at least 100cm. The ductility value of the samples was not constant. The control asphalt binder sample has a ductility value of 140 cm, meanwhile sample for 1, 2, 3, 4, and 5% having 110.4, 102.8, 140, 108.5, and 120.7 cm, respectively. Results of these ductility test exceed the minimum length required for elasticity behavior in asphalt binder. With the increasing of ductility value, it will benefit the pavement in order to avoid cracking (Tejankar and Chintawar, 2016). However, PG76+Rediset showed a decrease in ductility value ranging from 13 % to 26 %, thereby reducing the fatigue life (Miró et al., 2015). However, all the values obtained from all respective samples showed that PG76 binder is suitable to be used practically as it meets the standard requirement of ASTM D113, in which the minimum value is 100 cm. Thus, the Rediset modified asphalt binder is suitable to be used as pavement layer in term of ductility measure.

Thermal Analysis

The findings from the TGA analysis in this study are in line with the study by Miguel et al. (1996). The decomposition of asphalt binders was found to occur in at least three phases, in three temperature ranges, as shown in Figure 1. Overall, the material decomposition took about 66.9% to 91.5% of the total sample weight. All samples were thermally stable until about 300° C except for samples with Rediset content of 4% and 5% of binder weight. Both blends began to change at 100 °C. In the second phase of decomposition at temperatures > 300 °C > 500 °C, the whole samples were substantially decomposed. As the process advances to phase three, all the decomposition processes have reached a steady state. At this stage, the decomposition of the binder is slowing down, and the weight remains only due to the evolution of hydrogen alone (Yu et al., 2016).

Even though the WMA additive added into PG76 makes the penetration value increase, and the softening point and ductility values decrease, the blend of Rediset and PG76 can withstand temperature up to 300 °C. This indicates that the combination of Rediset and PG76 is heat resistant. By not easily decomposing the material, it will sustain its intrinsic behavior and might not be easily fused into the environment.

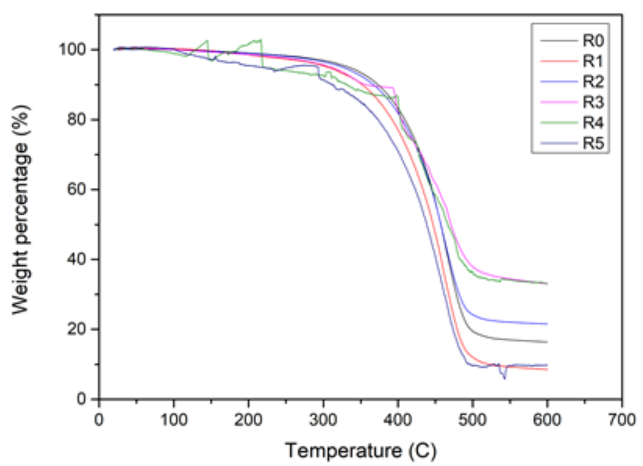
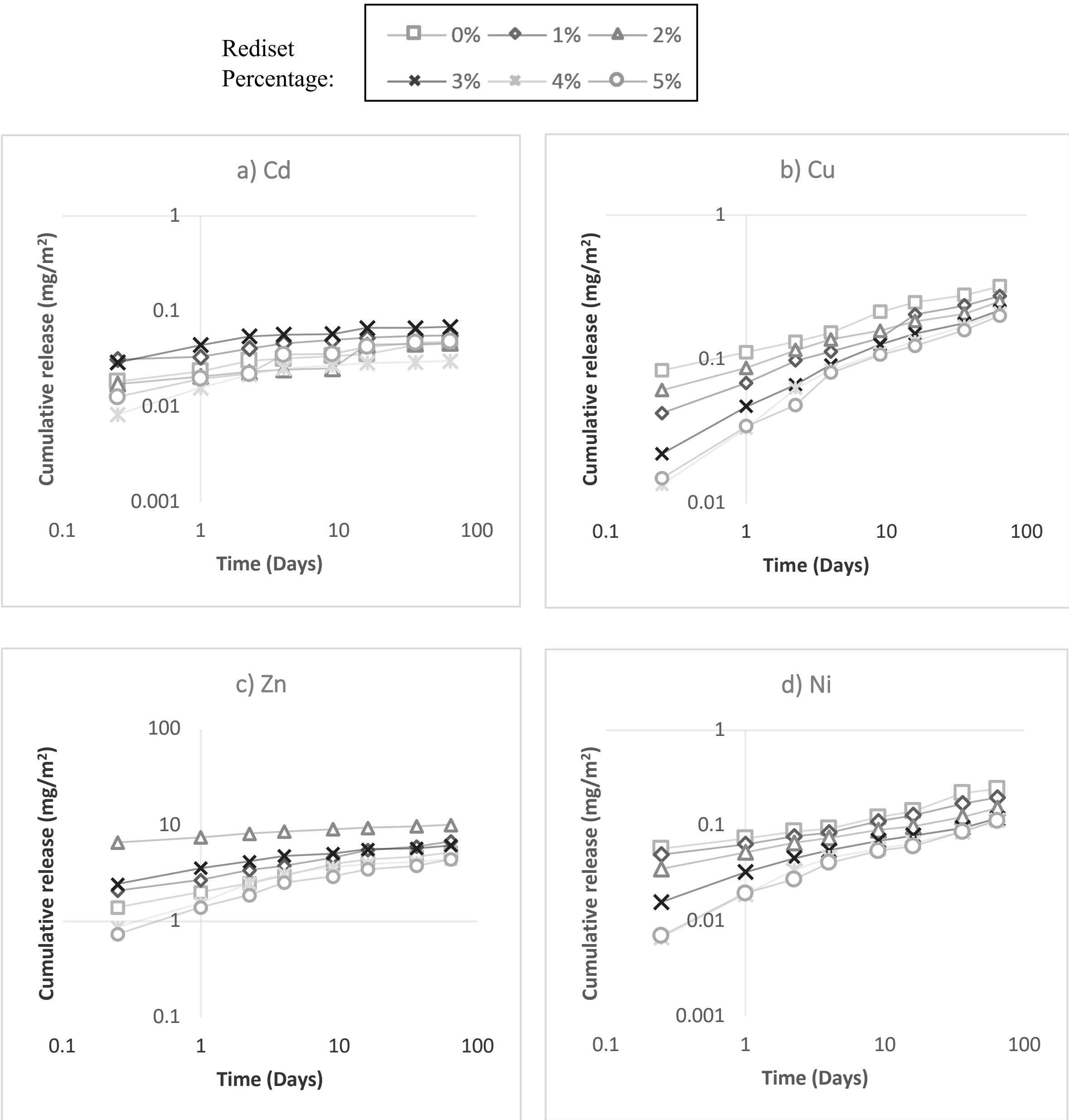


Figure 1. Thermal analysis of all samples.

Cumulative Release of Heavy Metals

By using ICP-MS devices, heavy metal components can be analyzed, as well as the amount obtained as the device through the use of combined inductively coupled plasma source with a mass spectrometer. Heavy metals released are plotted in log graphs as cumulative released per area (mg/m2), in which the area is the area of the surface of the sample (615.75 mm2) exposed to the acidic solution against time period (days) based on the NEN 7345 standard (0.25, 1, 2.25, 4, 9, 16, 36, and 64 days) for unmodified PG76 and Rediset-PG76 (1-5%). The studied heavy metal elements (Cd, Cu, Zn, Ni, Pb, Cr, Al, and Co) are plotted as shown in Figure 2. From the graphs, increments can be seen as the day passed. This indicates that a chemical reaction occurred to the samples. The gradient of the plots for each sample indicates the trend of heavy metal components leached from the samples for respective days. However, it was found that the trend of the curves is not consistent. Each element gives different trend regardless of the amount of Rediset added. This indicates that the amount of Rediset included into PG76 did not determine the release of heavy metal elements leachate from the blends.



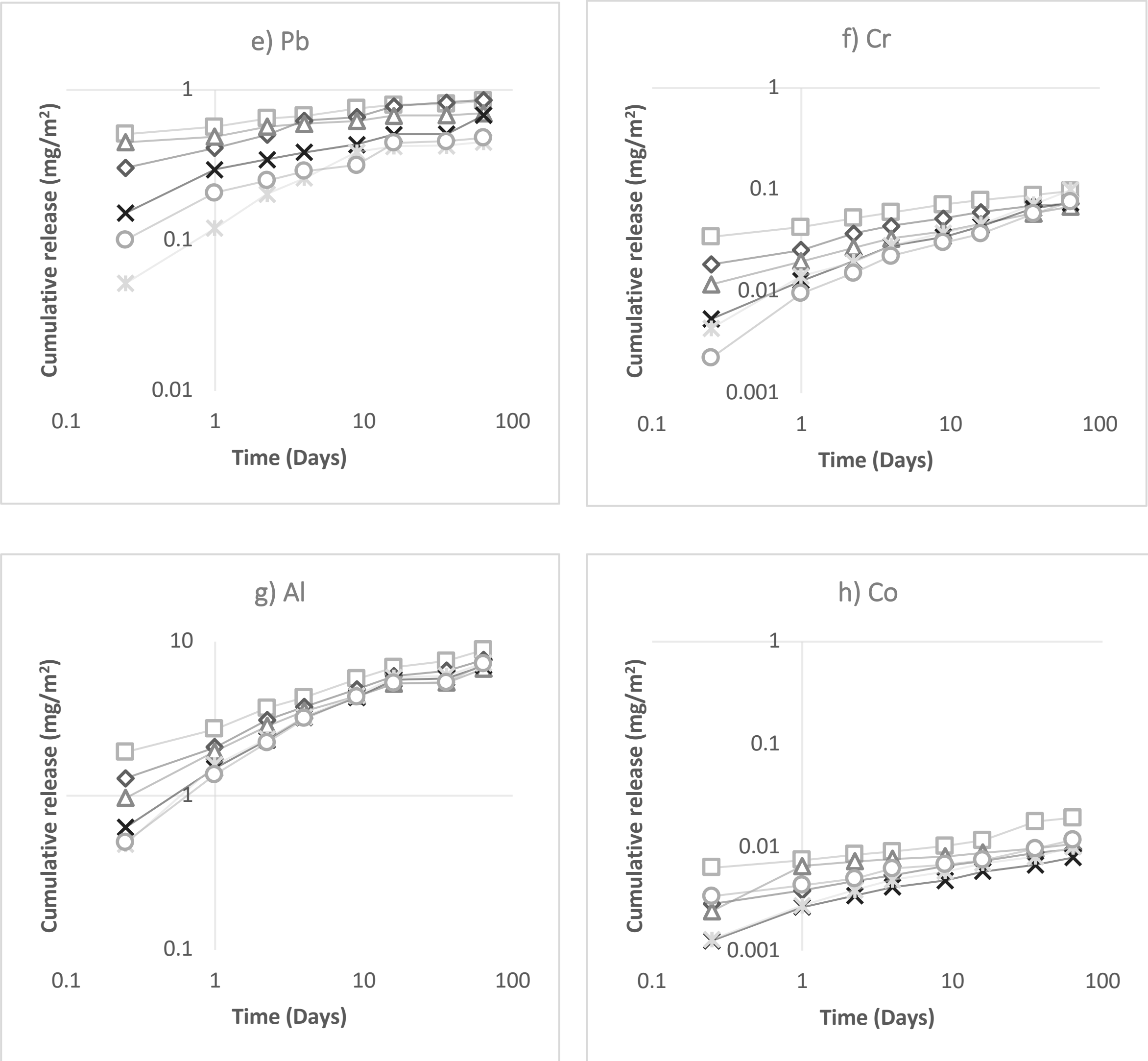


Figure 2. Cumulative release of heavy metals leached from the samples up to 64 days as represented in graphs as a) cadmium (Cd); b) copper (Cu); c) zinc (Zn); d) nickel (Ni); e) lead (Pb); f) chromium (Cr); g) aluminium (Al) and; h) Cobalt (Co) leached from the samples respectively.

Leaching Behavior Analysis

The behavior of each of the elements was investigated by referring to Table 3. The table was based on the phase of time period and the respective gradient obtained from the cumulative release of the graph to determine the appropriate behavior that occurs during that particular period of time adopted from NEN7345.

Table 3. Leaching behavior based on the cumulative released graph gradient.

Time (day)	Gradient		
	≤ 0.35	> 0.35 and ≤ 0.65	> 0.65
0.25–4	Surface wash-off	Leaching	Dissolution or delayed leaching
1–36	Surface wash-off	Leaching	Dissolution
9–64	Wear out	Leaching	Dissolution

Source: NEN 7345 (2006)

For time period of 0.25 to 4 days, for elements Cd, Cu, Ni, Pb, Cr, and Co, the behavior of the samples showed that all samples (unmodified and modified PG76) undergo surface wash-off. Surface wash-off is a behavior limited on the surface of the samples being affected by external factors such as pH value of the liquid mostly acidic that triggers the beginning of the leaching of the samples (Shi & Kan, 2009) as most of the heavy metals reacted with acids. For Zn, all the samples showed that they undergo normal leaching behavior, and for Al, only unmodified PG76 shows normal leaching behavior, while the others undergo dissolution or delayed leaching. Dissolution is a behavior where the samples are observed to be dissolving into acidic solution or exhibit delayed leaching behavior (Shi & Kan, 2009).

For 1 to 36 days, all elements from all samples were shown to have surface wash-off. This indicates that the amount of particles to be leached is only limited to the surface of the samples, and the water does not have enough means to infiltrate deeper into the sample to trigger possible leaching reaction chain. Furthermore, the pH value of the acidic solution is quite mild. Most metallic elements react with acidic solution but in this study, the acidic solution simulating flood or suspended heavy rainfall does not have enough acidic properties to trigger any further reaction completely (Meunier et al., 2006). For 9 to 64 days, all of the samples already showed to be wearing out due to the lesser particles being leached out. Furthermore, the amount of liquid present is limited; thus, the elements particles have limited space for them to be leached out competitively (Fadzil et al., 2010).

Heavy Metal Toxicity Analysis

Heavy metals content leached from any solid suspended in liquid should be analyzed as it can potentially contaminate surrounding and causing many complicated health issues (Elsgaard et al., 2001; Filip, 2002). Heavy metals can contaminate groundwater resources and also the surrounding soils before being absorbed by vegetables that later on will end up consumed by humans (Singh et al., 2010).

Thus, in this study, the cumulative amount of leached heavy metals are being referred to the US EPA soil contamination guidelines and WHO drinking water guidelines that have been published to control the allowable heavy metal released in soils and water resources. Table 4 shows the cumulative value of heavy metal released up to 64 days from this study and the US EPA guideline values provided for each heavy metals in the soil while Table 5 shows the allowable heavy metal content in water resources according to WHO.

From the Table 4, it shows that the cumulative release of all heavy metals studied did not exceed the limit value set by US EPA making both unmodified and modified PG76 safe for environmental soil due to the low amount of heavy metals released accumulated up to 64 days. It can be seen that by the addition of Rediset to produce modified PG76 binder does reduce the amount of heavy metals release for most metals studied.

For drinking water standard set by WHO as shown in Table 5, all of the cumulative release samples were shown to be below the limit except for Pb and Al. The values that exceeded are not too much, but they are still on a level where they need to be concerned. For Pb, though it does exceed the limit set by WHO, it is considered as being in the safe range by India standard drinking water specification as the limit is 0.05 mg/L (Puri & Kumar, 2012). Exceeding lead content in drinking water consumed might cause several health complications such as anemia (Moore, 1988). To deal with the exceeding lead content in drinking water, further treatment is required. The past study was shown to have used the application of absorbents such as zeolites, resins, activated carbon, manganese oxides, and cellulose powder (Sublet, 2003). Watt et al. (2000) stated the usage of programs of water treatment with addition of orthophosphate lime able to maximize the reduction of lead content in water resources.

As for Al, the cumulative released value was shown to be exceeded the limitation set by WHO which is 0.2 mg/L. While the limitation of Al has been set, exposure to Al in drinking water usually is not harmful as there is no apparent proof for it to have serious adverse effect if it is overconsumed (Krewski et al., 2007). According to Vaaramaa and Lehto (2003), Al can be removed by a cation exchanger as regeneration of the exchange bed must be done with sulfuric or hydrochloric acid. Reverse osmosis also can be done to eliminate Al contents up to 98% or more (Davison et al., 1982).

On the other hand, for Co, WHO did not mention its exact limitation in drinking water resources. Co is considered beneficial to humans as it facilitates pregnant woman with anemia in stimulating the blood red cells production and acts as a part of vitamin B12. However, when it is being consumed too much, it could cause adverse effects and may damage human health (Chaitali & Jayashree, 2013). However, New Zealand standard for drinking water has set the limitation of 1 mg/L (Chaitali & Jayashree, 2013). This indicates that the Al content in this study was found to comply with the available admissible limits.

Table 4. Heavy metals cumulative release with US EPA soil guidelines.

Elements	R0	R1	R2	R3	R4	R5	EPA limit (mg/kg)
Cd	0.0289	0.0345	0.0286	0.0425	0.0186	0.0296	7.10
Cu	0.1968	0.1688	0.1547	0.1343	0.1294	0.1231	310.00
Zn	3.2148	4.1994	6.2627	3.7975	2.7460	2.7351	2300.00
Ni	0.1498	0.1211	0.0939	0.0732	0.0698	0.0694	150.00
Pb	0.5221	0.3688	0.4325	0.4183	0.2784	0.2961	400.00
Cr	0.0598	0.0448	0.0420	0.0455	0.0613	0.0469	0.30
Al	5.4409	4.6907	4.1136	4.2920	4.4710	4.4247	$3x10^6$
Co	0.0060	0.0060	0.0067	0.0049	0.0063	0.0073	2.30

Table 5. Heavy metals cumulative release with WHO drinking water guidelines.

Elements	R0	R1	R2	R3	R4	R5	WHO limit (mg/L)
Cd	0.0014	0.0017	0.0014	0.0021	0.0009	0.0015	0.003
Cu	0.0038	0.0084	0.0077	0.0067	0.0065	0.0062	2.00
Zn	0.1607	0.2100	0.3131	0.1899	0.1373	0.1368	3.00
Ni	0.0075	0.0061	0.0047	0.0037	0.0035	0.0035	0.07
Pb	0.0261	0.0265	0.0216	0.0209	0.0139	0.0148	0.01
Cr	0.003	0.0024	0.0021	0.0028	0.0031	0.0023	0.05
Al	0.272	0.2345	0.2057	0.2146	0.2236	0.2212	0.20
Co	0.0006	0.0003	0.0003	0.0002	0.0003	0.0004	N/A

Projection of Approximated Cumulative Heavy Metal Content Release

For future reference, the possible approximation of heavy metal content leached could be predicted by projecting the cumulative released value. In this study, the projection values are set to be up to 100 days and 100 years (Fadzil et al., 2010). The aim of this projection is to predict the leaching content released from the asphalt pavement that used PG76 binder. Tables 6 and 7 show the forecast of approximated values of elements Cd, Cu, Zn, Ni, Pb, Cr, Al, and Co for 100 days and 100 years.

Table 6. Forecasting of approximated value of leached heavy metals for 100 days.

Sample	Amount leached (mg/kg)							
	Cd	Cu	Zn	Ni	Pb	Cr	Al	Co
R0	0.0293	0.1985	3.4132	0.1406	0.5472	0.0616	5.5000	0.0114
R1	0.0371	0.1677	4.2736	0.1157	0.5756	0.0483	4.8258	0.0061
R2	0.0298	0.1525	6.5095	0.0889	0.4560	0.0414	4.2387	0.0071
R3	0.0467	0.1332	4.0939	0.0712	0.4044	0.0447	4.4832	0.0050
R4	0.0216	0.1266	3.0937	0.0660	0.3233	0.0535	4.6668	0.0063
R5	0.0328	0.1190	2.8430	0.0643	0.3237	0.0423	4.4414	0.0070

Table 7. Forecasting of approximated value of leached heavy metals for 100 years.

Sample	Amount leached (mg/kg)							
	Cd	Cu	Zn	Ni	Pb	Cr	Al	Co
R0	0.0475	0.3605	6.0779	0.2637	0.7808	0.1046	10.1632	0.0201
R1	0.0552	0.3243	7.4738	0.2127	0.9650	0.0868	9.1097	0.0109
R2	0.0516	0.2742	8.8377	0.1612	0.6318	0.0781	7.9656	0.0118
R3	0.0718	0.2629	6.4912	0.1359	0.7066	0.0908	8.8226	0.0094
R4	0.0361	0.2555	5.5666	0.1328	0.6154	0.1116	9.2489	0.0121
R5	0.0582	0.2403	5.2872	0.1308	0.5816	0.0889	8.8190	0.0124

From the forecast of the heavy metals for 100 days and 100 years, the heavy metals amount leached are predicted. The cumulative amount of leached heavy metal contents that are forecasted indicates the possible amount of heavy metals that could be leached from the Rediset-PG76 for a long period of time if they are exposed to continuous heavy rainfalls or event flash flood occurrences. For Cd components, it shows that the leached amount ranges from 0.0216 to 0.0328 and 0.0475 to 0.0582 mg/kg for 100 days and 100 years of leaching, which is still under the limit set by US EPA. It is the same for all other heavy metals as the ranges of heavy metals components leached are under control.

From the study, Rediset-PG76 does not exhibit serious heavy metals leaching problems that could disturb the quality of the surrounding soil. Though, it is to be reminded that the cumulative amount of the heavy metal components for Rediset-PG76 is approximated and does not actually represent the amount leached for practical application since leaching behavior, and its factors could be heavily affected by the surrounding conditions, climate, and surface pores of the mixtures that occurred during mixing (Sani et al., 2005).

CONCLUSION

The physical tests comprised of penetration test, softening point test, and ductility indicate that Rediset had some influence on PG76 when the two substances were blended together. Thermal analysis results show that the new materials decomposed at temperature more than 300 °C even though a WMA additive was added in the base binder. Also, tank leaching test and ICP-MS analysis gave out that all the leached heavy metals are complied with the US EPA for soil content. For WHO drinking guidelines, only Pb and Al exceeded the limit stated by WHO drinking water guidelines. However, other countries have their guidelines that might differ from those of WHO. Finally, the results of projection value up to 100 years also inclined to a conclusion that the combination of PG76 an Rediset will not harm the groundwater and soil. Thus, it can be concluded that modified PG76 with the addition of 1 to 5% Rediset did not cause serious problems to the surrounding soil.

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