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Volumetric Properties and Leaching Effect of Asphalt Mixes with Electric Arc Furnace Steel Slag and Copper Mine Tailings

(Sifat Isi Padu dan Kesan Larut-Lesap Bancuhan Asfalt dengan Sanga Keluli Relau Arka Elektrik dan Tahi Lombong Kuprum)

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ABSTRACT

This study focuses on the potential of electric arc furnace (EAF) steel slag and copper mine tailings as asphalt paving materials with respect to issues of volumetric properties and leaching. In this study, four different asphalt mixes were investigated; each contained EAF steel slag and copper mine tailings of various proportions. Apart from the microstructure analysis of the materials, a toxicity characteristics leaching procedure (TCLP) test was conducted on both the mixes and the aggregates. All the mixes were evaluated by the following parameters: Voids in the mineral aggregates (VMA), voids in total mix (VTM), voids filled with asphalt (VFA), Marshall stability and flow and specific gravity. F-test ANNOVA was used to evaluate the degree of significance of the mixes with each of the evaluated standards. It was observed that the mixes containing either EAF steel slag or copper mine tailings or both gave better results than the control mix. In terms of the TCLP test, none of the detected hazardous elements exceeded the standard limits, which indicates the possibility of using them as construction materials.

Keywords: Asphalt; copper mine tailings; EAF steel slag; TCLP test; volumetric properties

ABSTRAK

Kajian ini memberi tumpuan kepada potensi sanga keluli relau arka elektrik (EAF) dan tahi lombong kuprum sebagai penurapan bahan asfalt yang berkaitan dengan sifat isi padu dan larut- lesap. Dalam kajian ini, empat bancuhan asfalt berbeza telah dikaji; setiap satu mengandungi sanga keluli EAF dan tahi lombong kuprum pada kadar berbeza. Selain daripada analisis mikrostruktur bahan, satu ujian prosedur ciri ketoksikan larut-lesap (TCLP) telah dijalankan ke atas bancuhan tersebut dan agregatnya. Semua bancuhan telah dinilai menggunakan parameter berikut: lompang dalam agregat galian (VMA), lompang dalam jumlah campuran (VTM), lompang yang dipenuhi dengan asfalt (VFA), kestabilan Marshall serta aliran dan graviti tertentu. Ujian-F ANNOVA digunakan untuk menilai tahap signifikasi bancuhan tersebut dengan setiap satu daripada piawai yang dinilai. Adalah diperhatikan bahawa bancuhan yang mengandungi sama ada sanga keluli EAF atau tahi lombong kuprum atau kedua-duanya memberikan keputusan yang lebih baik daripada campuran kawalan. Daripada segi ujian TCLP, tiada unsur berbahaya yang dikesan melebihi had piawai justeru menunjukkan kemungkinan untuk penggunaan bancuhan tersebut sebagai bahan binaan.

Kata kunci: Asfalt; sanga keluli EAF; sifat isi padu; tahi lombong kuprum; ujian TCLP

INTRODUCTION

In Malaysia, the major by-products generated from the metallurgical and mining industries can be classified as steel slag and copper mine tailings respectively. Two types of steel slags are produced were basic oxygen furnace (BOF) and electric arc furnace (EAF). Presently, only EAF steel slags are being produced in the country. Over time, huge amounts of steel slag and copper mine tailings that need to be recycled have accumulated in Malaysia.

During recent years, steel and copper have gained wide applications in many industries. Hence, in order to meet the demand for these materials, the rate of their production has increased; invariably promoting the quantity of copper processing wastes and steel slag. About 115 kg of steel slag and 128 tons of copper tailings were turn out for every 1 ton of steel and copper produced, respectively (Gordon 2002). According to U.S. Geological Survey (2014), Malaysia produced approximately 5612 and 240 metric tons of steel and copper, respectively. Similar to the other metallurgical by-products, the usage of both EAF steel slag and copper mine tailings has been very limited compared to the use of conventional aggregates.

Researchers have studied the utilization of EAF slags and mine tailings as construction materials. Pasetto and Baldo (2012) and Oluwasola et al. (2015a) have investigated the performance analysis of stone mastic asphalts (SMA) with EAF steel slags. They noted that SMA mixes containing EAF slag show good durability. Further, it was reported that EAF steel slag has better mechanical properties than the natural aggregate. In view of this, it has

been recommended as being a reliable highway material (Haritonovs et al. 2012; Sofilic et al. 2011).

Unlike steel slag, the majority of studies on copper mine tailings have been centered on its potential for use in concrete technology. Obinna and Ozgur (2012) studied the consistency, strength and toxic metal immobilization properties of copper tailings as a potential additive in concrete. Interestingly, their studies indicated that the material be used at 5% by mass additional level. In a related work, Novo et al. (2013) and Das et al. (2000) studied the properties of amended copper mine tailings and its potential for use in the production of ceramic tiles respectively. Their studies confirmed tailing as a good aggregate material. Ahmari et al. (2012) and Widojoko (2013) have respectively evaluated copper mine tailings as a filler material in asphalt concrete and as a road base material. Ahmari et al. (2012) found that geopolymerization technology can be utilized to improve the strength of compacted copper mine tailings. From the results of Widojoko (2013), it was indicated that copper tailings reduce the permanent deformation of asphalt mix.

However, far too little attention has been paid to the utilization of copper mine tailings as asphalt material. Presently, there are limited studies on asphalt mixes incorporating both steel slag and copper tailings (Oluwasola et al. 2015b). Hence, this study aimed at investigating the volumetric characteristics of asphalt mix by incorporating EAF steel slag and copper mine tailings as binary base material. In addition, the microstructural analysis and leaching potentials of both base materials and mix design were investigated. The study enhances and contributes significantly to the development of asphalt mix design using metallurgical and mining waste materials and also provides a better alternative material for conventional aggregate. The study also promotes cost- effective solution in managing copper mine tailings and EAF steel slag and controlling environmental pollution.

MATERIALS AND METHODS

The study is basically divided into four major stages as follows. The first stage focuses on the physical and mechanical characterization of the EAF steel slag, copper mine tailings and granites respectively. A microstructure analysis of the aggregates was studied in stage two. The third stage is relative to the leaching potential of the aggregates and the mixes while the mix design of the mixes was evaluated in the final phase. Four HMA mixes were prepared for this study. All types of mixes use asphalt pen 80-100, being one of the bituminous materials recommended for the production of HMA by the Malaysia standard specification for road works (JKR 2008). More so, the pavement temperatures of Malaysia are fairly high and consistent throughout the country and pen 80-100 has high performance characteristics at high temperature. Table 1 summarizes the test results of pen 80 -100 bitumen samples.

The materials used for this study include granite, EAF steel slag and copper mine tailings. EAF steel slags were procured from Antara Steel, Malaysia, while copper mine tailings were supplied by Malaysia Marine and Heavy Equipment (MMHE), Pasir Gudang, Malaysia. Bitumen samples were provided by Shell, Singapore. Both the granite samples and steel slags consisted of all the required particle sizes, whereas the copper mine tailings were predominantly fine with the bulk of its particle size passing through a 1.18 mm sieve size as indicated in Figure 1.

PHYSICAL AND MECHANICAL CHARACTERIZATION

The physical and mechanical properties of the aggregates were determined according to the relevant standard test methods as shown in Table 2. The test results indicate the very low flakiness index of the aggregates and excellent mechanical strength, especially EAF steel slag with average LA value of 5.1%. The specific gravities of the fine aggregate, steel slag and copper mine tailings were 2.585, 3.051 and 3.578, respectively. The high amount of iron (III) oxide in both steel slag and copper mine tailings contributed to their high specific gravity (Singh et al. 2013). Similarly, both copper tailings and steel slag measurements exceeded the specification for water absorption by 94.8 and 108.5%, respectively. The prolonged exposure to agents of weathering and large quantity of fines present in them might have accounted for this trend. In terms of pH, apart

Parameter	Pen 80-100	Standards
Penetration @ 25°C	85	ASTM D5
Softening point	46	ASTM D36
Dynamic viscousity @ 60°C	34.3 Pa.s	ASTM D44
Dynamic viscousity @ 135°C	0.3 Pa.s	ASTM D44
Ductility @25°C	110	ASTM D113
Flash point	252	ASTM D92
Specific gravity	1.0	ASTM D70
Penetration Index (PI)	-2.23	
Penetration viscousity number	-0.78	
Mixing temperature	130°С - 150°С	
Compacting temperature	120°C - 140°C	

TABLE 1. Typical characteristics of Pen 80-100 Bitumen

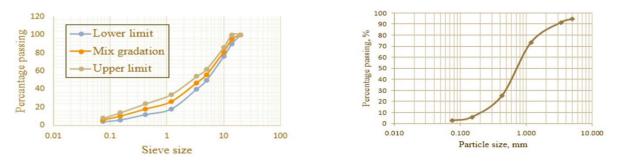


FIGURE 1(a). Particle size distribution of the mix

FIGURE 1(b). Copper tailings gradation

Testing	Standard	Granite	EAF Steel slag	Copper Tailings	Specification
Loss angeles abrasion	ASTM C 131	10.276	5.100	-	≤ 25%
Flakiness	MS 30	7%	5%	-	$\leq 25\%$
Soundness	AASHTO T 104	3.5%	0.71%	-	$\leq 18\%$
Polished stone value	BS 812	52.3%	55.3%	-	\geq 40%
Water absorption	MS 30	0.756%	3.896%	4.17%	$\leq 2\%$
Stripping	AASHTO T 182	>95%	> 95%	-	\geq 95%
Coarse aggregate SG	ASTM C127	2.594	2.816	-	-
Fine aggregate SG	ASTM C 128	2.585	3.051	3.578	-
pН	BS 1377	10.22	11.42	6.42	-

TABLE 2. Physical and mechanical properties of the aggregate

from copper tailings, the aggregates were highly alkaline. This enhances their strong bond with weak acidic asphalt.

MICROSTRUCTURE ANALYSIS

Principally, the asphalt mix comprises of three parts, specifically; the aggregate, the binder and the air voids between the particles. The degree of aggregate porosity is among the parameters that can be evaluated through microstructure analysis. In this study, three microstructure techniques have been adopted, namely: Field emission scanning electron microscope (FESEM) coupled with energy disperse X-ray spectroscopy (EDS), X-ray diffraction (XRD) and X-ray fluorescence (XRF).

The Zeiss supra (model 35 up) coupled with EDS (FESEM + EDS) was used to test the morphology of granite, steel slag and copper mine tailings. The FESEM equipment was operated at 15 Kev and the test was conducted in accordance with ASTM E 2090 (2012) procedures. Figure 2 presents the FESEM micrographs of the aggregates.

Based on the images, the materials show different morphology and texture from one another. Figure 2 illustrates that more pores can be observed on the surfaces of copper tailings and steel slag than on a granite surface. Besides, both steel slag and copper tailings display rougher surface texture than granite. This enhances the adhesion properties of both steel slag and copper tailings, thus, enhanced the volumetric properties and performance of the mixes incorporating both materials.

For XRD, the samples were ground into powder form and the test was performed with Siemens D 5000C X-ray Diffractometer. The XRD patterns were scanned one step of 0.0034° using (Cu=k α) at 40 mA and 40 kV. The diffraction angle was acquired in the 2 θ with angle between 5° and 80° for all the samples. The search matches the International Centre for Diffraction Data, ICDD (2006) with data files being used for proper identification of the mineral phases that were present in the samples. Diffraction data were processed by Philip X'pert software computer program.

Figure 3 indicates the basic mineral composition of the studied aggregates, whilst granite exhibits three peaks in order of quartz, feldspar and mica, respectively; XRD diagram of steel slag identifies meyenite, larnite, lime and periclase as its prominent minerals. Quartz, kaolinite, hematite and calcite dominated the mineral composition of copper tailings. According to Wang et al. 2011, all the identified minerals in the aggregate enhance their alkalinity properties, which increased their bonding strength with the bitumen binders and improved their volumetric properties.

X-ray fluorescence is a non- destructive qualitative tool used for chemical analysis of materials. In this study, S4 Pioneer Binker axs X-ray fluorescence spectrometer was used to analyze the samples. The procedures as stated in the SESDPRDC – 107 standards were followed in performing the test. Table 3 shows the chemical composition of the aggregate type. Granite is principally composed of oxides of silicon and aluminum. Calcium, iron and silicon dominated the oxides present in steel slag. Copper tailing was rich in silicon, iron, aluminum and calcium oxides. The high presence of iron oxide in both EAF steel slag and copper tailing can be attributed to their prolonged exposure to weathering (Singh et al. 2013). Further, EAF steel slag contains oxide of chromium, a potential toxic element in trace quantity. 282



FIGURE 2(a). FESEM micrographs of granite



FIGURE 2(b). FESEM micrographs of EAF steel slag

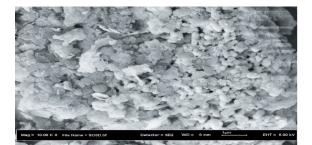


FIGURE 2(c). FESEM micrographs of copper tailings

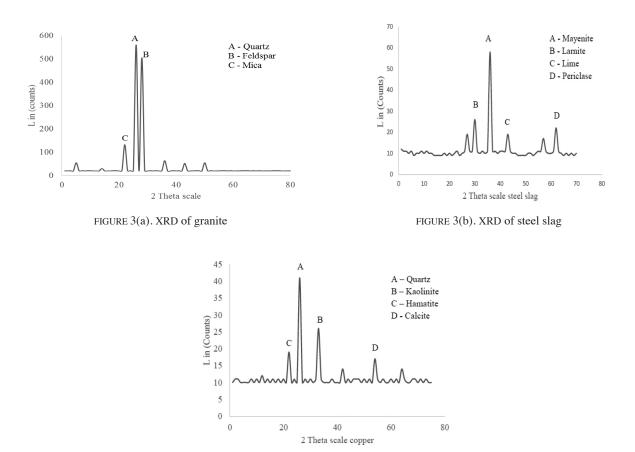


FIGURE 3(c). XRD of copper tailings

TABLE 3. Chemical composition of the aggregate studied

Aggregate type					Oxid	e conter	nt (%)				
Aggregate type	CaO	SiO_2	Fe ₂ O ₃	Al_2O_3	ZnO	MgO	MnO	K ₂ O	Na ₂ O	SO_3	$Cr_{2}0_{3}$
Granite	4.90	64.60	3.80	16.80	-	0.74	-	4.40	3.48	-	-
Steel slag	55.2	10.90	16.80	4.00	3.72	3.06	2.59	0.16	0.75	0.85	0.69
Copper tailings	12.48	44.10	19.00	15.40	0.96	0.87	0.87	1.24	0.46	2.46	-

LEACHING

The toxicity characteristics leaching procedure (TCLP) test developed by the United States Environmental Protection Agency US EPA (1992) was adopted in this study to assess the leaching of heavy metals from the aggregates used as well as the four mixes.

The pH of the sample plays a significant role in choosing the extraction fluid. In this work, all the aggregates and the four mixes have a pH value exceeding 5.0. In view of this and according to the US EPA (1992) method, extraction fluid #2 (5.7 mL glacial acetic acid diluted in IL reagent water) was used. All the aggregate samples were passed through a 0.075 mm sieve while the four mixed samples passed through a 1.18 mm sieve. Approximately, 10 g of the samples were poured into a PYREX conical flask. Thereafter, an appropriate quantity of extraction fluid was added to ensure a 20:1 ratio of liquid to dry sample. The reaction bottles were carefully sealed with aluminum foil and fastened with rubber bands in order to prevent loss in leachate volume. Consequently, the reaction bottles were agitated on a Hotech variable speed orbital shaker (model 722) as shown in Figure 4(b) and operated at 300 rpm for 18 h. The test was performed at laboratory ambient temperature of 26±1°C. This time was sufficient to allow steady - state dissolution and mobilization to take place for fine samples (Hossam et al. 2008).

After the agitation period of 18 h, liquid in each of the reaction bottles was poured into a respective bench top centrifuge (model EBA 21), shown in Figure 4(b). Thereafter, the samples were agitated to 6000 rpm for 10 min. The filtrate was later separated from the residue through a $0.20 \,\mu$ m Agilent Premium syringe filter attached to a 10 mL cellotron syringe. The pH of the filtrate was measured and later acidified for preservation. The concentrations of heavy metal ions were determined after the completion of the process of extraction and filtration of the leachates by inductively coupled plasma - mass spectrometry (ICP - MS).

MIX DESIGN

Four different mixes were designed by using natural and unconventional raw materials. Table 4 reports the aggregate type and composition of the mixture. In preparing the samples, three different aggregate types were used. The granite and EAF steel slag were used as coarse and fine aggregates while the copper mine tailings was only utilized as fine aggregate due to its gradation.

The mixes were designed to have a uniform gradation conforming to gradation limits for the Malaysian asphalt concrete wearing course, ACW 14, JKR (2008). Figure 1 shows the aggregate gradations. The binder used was penetration grade 80-100 asphalt.

The Marshall mix design procedure was adopted for the optimum bitumen content determination with fifteen samples for each mix. The asphalt content range was 4.0-6.0% of the total weight of the mix in 0.5% increments as stipulated in JKR (2008) standards. The specimens for the Marshall test had an average height of 61.6 mm with an approximate diameter of 100.2 mm. The samples were prepared in the laboratory by using an impact compactor and applying 75 blows on each face at the appropriate temperature. The following mix properties: Stability, flow, bulk specific gravity, air voids, voids filled with bitumen (VFB) and voids in mineral aggregate (VMA), respectively, were evaluated.

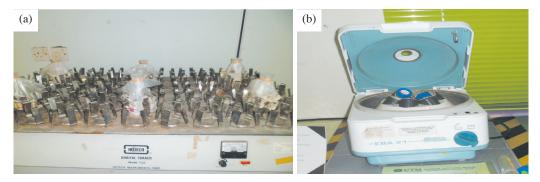


FIGURE 4. (a) Hotech orbital shaker and (b) Centrifuge machine

	Mix 1		Mi	Mix 3		: 3	Mix 4	
Fraction	Granite (%)	Granite (%)	Copper tailing (%)	EAF slag (%)	Copper tailing (%)	Granite (%)	EAF slag (%)	Copper tailing (%)
14.00	6	6	-	6	-	3	3	-
10.00	14	14	-	14	-	7	7	-
5.00	26	26	-	26	-	13	13	-
3.35	9.5	9.5	-	9.5	-	4.75	4.75	-
1.18	21.5	11.4	10.1	11.4	10.1	5.7	5.7	10.1
0.425	8.5	4.5	4	4.5	4	2.25	2.25	4
0.150	8.5	4.5	4	4.5	4	2.25	2.25	4
0.075	4	2.1	1.9	2.1	1.9	1.05	1.05	1.9
Filler (Cement)	2	2		2		2		

RESULTS

VOLUMETRIC PROPERTIES

Voids in the Mineral Aggregates (VMA) VMA was defined by Roberts et al. (2009) as the total volume of voids within the mass of the anticipated aggregate. It affects the performance of asphalt mixture significantly because a mix with low VMA may show durability problems, while stability problems may occur in a mix having large VMA. Based on Figure 5(a), the values of VMA generally increased with bitumen content in the range of 4-6%. This trend is expected because when asphalt content increases, the voids filled with asphalt also increase (Brown et al. 2005). Consequently, this invariably increases VMA. For the samples prepared at OBC, Mix 2 has the least value of 15.4%, while Mix 3 reports the highest value of 18.5%. Clearly, VMA is higher in Mixes 2 and 3 than in Mix 1. The reason behind the minimum VMA requirement is to incorporate the minimum permissible binder content to enhance its durability. Therefore, the minimum permissible bitumen content of Mixes 2 and 3 appears to be higher than the control mix. Mixes 2 and 3 have a higher VMA than control mix due to higher amount of voids in steel slag and copper mine tailings aggregates. Thus, the study infers that by replacing the conventional aggregates with steel slag and copper tailings, the amount of voids filled with bitumen increases and thus enhance its durability. Although the VMA limit was not included in the Malaysian standards JKR (2008), the obtained results

satisfied the recommendation for the VMA proposed by the ASI (1984). The ANOVA results presented in Table 5 shows that all the mixes were desirable and highly significant. The value of R^2 for all the mixes exceeded 0.95 at 95% confidence interval. Also, the F –test has a probability value (Prob > F) of less than 0.05 which also shows the significance of the correlation between VMA and bitumen content (Montgomery 2009).

Voids in Total Mix (VTM) This is the volume of air voids between the aggregate particles of a compacted mix. The Malaysian standards JKR (2008) recommend the VTM limit to lie between 3 and 5% for the laboratory compact mix. Low VTM may result in bleeding, rutting and loss of mixture stability (Roberts et al. 2009). Conversely, high VTM can lead to durability problems and stripping. Brown et al. (2004) also reported that high air voids enhance air and water penetration into the pavement. Thus, the desired VTM content must be approached during construction through the application of compactive effort. As shown in Figure 5(b), the VTM was inversely proportional to the bitumen content, since the increase in asphalt content in the mix reduces the air voids between the aggregate particles. All the mixes satisfied the JKR standard in terms of VTM with Mix 3; Mix 2 had the lowest (3.8%) and the highest (4.7%), respectively, for the mix prepared at OBC (Table 6). In terms of ANOVA statistical analysis, the mixes show a very high significance correlation between VTM and bitumen contents. At a 95% confidence interval, the probability

TABLE 5. ANOVA	results for	r the mixes
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Properties	Mix	r^2	Prob> F	Properties	Mix	r^2	Prob > F
VMA	1	0.9587	0.0487	Stability	1	0.8696	0.0652
	2	0.9637	0.0182	-	2	0.8535	0.0734
	3	0.9543	0.0222		3	0.8155	0.0923
	4	0.9786	0.0107		4	0.7242	0.0793
VFA	1	0.8629	0.0686	VTM	1	0.9611	0.0195
	2	0.8729	0.0635		2	0.9677	0.0162
	3	0.8952	0.0524		3	0.9539	0.0230
	4	0.8666	0.0667		4	0.9901	0.049

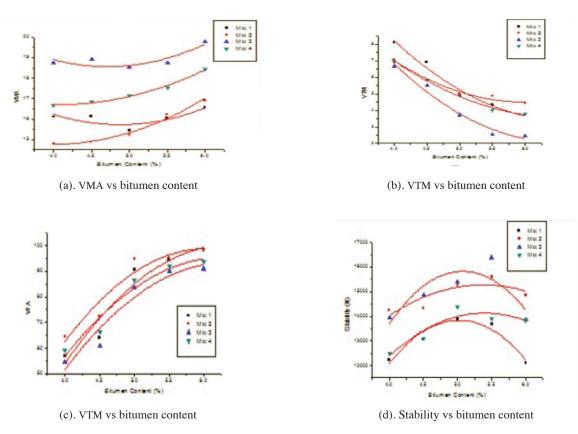


FIGURE 5. Graph of volumetric parameters

value of f – test (prob> 5) were far less than 0.05 and R^2 value greater than 0.95.

Voids filled with Asphalt (VFA) VFA can be described as the percentage of the VMA that is filled with asphalt. VFA increases with the increasing asphalt content as illustrated in Figure 5(c). Like other voids, VFA limits must be maintained during construction in order to avoid any deficiencies in the mix. All the mixes also met the requirements of JKR (2008) in terms of the VFA. Subjecting the results to the ANOVA statistical analysis, the value of R^2 for all the mixes was slightly below 0.95 and the value of probability (prob> F) was slightly higher than 0.5. The significance of the correlation between VFA and bitumen content is not as strong as that of VMA and VTM.

Moreover, the addition of 20% copper mine tailings stiffens the asphalt binder better than the natural aggregate and thus increases the Marshall stability. Also, the large pores present in both tailings and steel slag promote the stability of the mixtures. The statistical analysis of the mixes indicated that the regression models produce a fair correlation for all the mixes with R^2 less than 0.95 and probability value (Prob > F) registering above 0.05, respectively.

TCLP Results Concentrations of five heavy metals: Cu, Cr, Pd, Cd and Ni were measured for all the mixes and the aggregates as presented in Table 7. It was noticed that a TCLP extract of all the aggregates and the four mixes contained the highest concentration of Lead, Pb followed by cadmium, Cd. In addition, very low concentrations of other metals were detected, with the exception of nickel which was not detected in Mixes 3 and 4. However, the concentrations of all the detected metals did not exceed the regulatory limit of TCLP test. Furthermore, it was noticed that the mixing of asphalt (pen 80-100) with the

TABLE 6. Volumetric analysis parameters at optimum bitumen content for the mixes

Parameter	Mix 1	Mix 2	Mix 3	Mix 4	JKR Standard
OBC (%)	5.04	5.06	5.18	5.13	4 - 6
Stability (N)	13700	15150	15750	14100	> 8000
VTM (%)	4.5	4.7	3.8	4.3	3 – 5
VFA (%)	76	77	74	75	70 - 80
VMA (%)	15.6	15.4	18.5	16.8	-
Stiffness (N/mm)	4463	6982	7292	6130	> 2000

			Re	sults (mg/L)			
Parameters	Granite	Steel slag	Copper tailings	Mix 1	Mix 2	Mix 3	Mix 4
Copper, Cu	0.063	0.095	0.146	0.042	0.061	0.075	0.083
Chromium,Cr (5)	0.017	0.024	0.001	0.013	0.019	0.014	0.012
Lead, Pb (5)	1.910	3.476	1.189	1.542	2.961	1.739	2.535
Cadmium,Cd (1)	0.307	0.365	0.602	0.174	0.263	0.377	0.402
Nickel, Ni	0.004	0.002	0.001	0.001	0.0001	ND	ND

TABLE 7. Concentration of heavy metals in leachates

ND - Not detected, TCLP standards are in parenthesis

aggregates had no significant effect on the leachability of the heavy metals.

CONCLUSION

This research investigated the volumetric properties and leaching effect of asphalt mixes incorporating EAF steel slag and copper mine tailings. Based on the results obtained from the laboratory experiments, it can be concluded that apart from high water absorption value, the studied materials met the standard requirements of asphalt materials. EAF steel slag has a very low flakiness index and excellent mechanical strength. The microstructure analysis of the materials shows that they are better quality asphalt material. It was observed that both copper mine tailings and EAF steel slag are more porous and rougher than granite which strengthens their bond with asphalt materials. Furthermore, their mineral content increases their alkalinity property thereby equally confirming their enhanced bond with bitumen. In terms of volumetric properties, all the mixes met the Malaysian standards. Mixes 2, 3 and 4 gave better results than the control sample (Mix 1). The better mechanical properties of EAF steel slag and copper mine tailings might have accounted for this trend. The results of the TCLP test indicated that the concentrations of the five selected heavy metals were far below the TCLP regulatory limit for all the samples and the aggregates. This shows that a leaching process using these materials under field conditions would not present a threat to the environment. Consequently, these findings have proved the possibility of using a mixture of copper mine tailings and EAF steel slag in pavement construction as an alternative material, most especially in terms of hot mix asphalt. Hence, it is envisioned that EAF steel slag and copper mine tailings re promising alternative aggregate to be used as asphalt paving materials.

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