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FORMULATION OF BITUMEN FROM INDUSTRIAL WASTE

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ABSTRACT

Bitumen is a mixture of organic liquids which is black, high viscosity and it is sticky materials where it can be applied in several of application. Waste sludge can be used as another alternative to formulate the bitumen by manipulating ratio needed. Waste sludge consists of mineral oil solid waste, which includes oily sand, tank bottoms and other three kinds of sludge from refineries such as dissolved air flotation scum, excessively activated oily sludge and bottom sludge of oil from pools. Waste sludge is the major source of pollution produced in the process of oilfield production and development. In order to formulate the bitumen, other material that is needed include mineral oil, waste sludge and crumb rubber. The form of crumb rubber is form from discarded tire. Generally, the tire rubber is ground to a particulate or crumb prior to adding it to bitumen. This form of the tire rubber is called Crumb Rubber and the mineral oil is used as the medium to heat up the crumb rubber until it is melt and dissolved. The main objective in this experiment is to formulate and identify the best ratio of the bitumen produced. The method that is used to formulate is heating and mixing process which is being conducted inside the fume hood. So that it can absorb the fume released when the process is conducted. The key parameter during the process is weight of the sample, temperature, time and the speed of the mixer (rpm). Once it is produced, then the sample need to analyze based on the density 15 ^{0}C using ASTM D70 method, viscosity test by using ASTM D2170, penetration @ 25 ^{0}C test which by using ASTM 5 method and softening point test which is by using ASTM D36 method. The best ratio by far is (1:2:1.2) formulation by having viscosity of 93 centipoise, density of 1.0398 Kg/L, softening point which is at 62 ^{0}C and penetration which is at >40 mm of penetration. The sample that is formulated were then being compared with the actual bitumen sample.

Keywords: bitumen, waste sludge, tire rubber.

INTRODUCTION

Bitumen plays an important role as it benefit to the society in terms of infrastructures which is widely used in the world. Bitumen is a naturally occurring mixture of various organic liquids, it is black, highly viscous and sticky material which is the first oil product that utilized by humans due to its adhesive and cohesive properties [1,2]. It is also can apply in several of application that includes road construction, roofing application, paving roads, waterproofing products, building material and etc. It is estimated that the current world use of bitumen is roughly about 102 million tons per year. About 85% of all the bitumen produced is used as the binder in asphalt for roads. It is also used in other paved areas such as airport runways, car parks and footways [3]**.** Typically, the production of asphalt involves mixing sand, gravel and crushed rock with bitumen, which acts as the binding agent. Other materials, such as polymers, may be added to the bitumen to alter its properties according to the application for which the asphalt is ultimately intended. A further 10% of global bitumen production is used in roofing applications, where its waterproofing qualities are invaluable. The remaining 5% of bitumen is used mainly for sealing and insulating purposes in a variety of building materials, such as pipe coatings, carpet tile backing and paint [4].

Based on industry survey, there is a lot of method to formulate bitumen such as by refining crude oil using fractional distillation, solvent extraction, hydrodesulphurization and hydrogenation. The most common method and widely used in industry to form bitumen are by using fractional distillation where the bitumen is obtained by vacuum distillation or vacuum flashing of atmospheric residue from the vacuum distillation column [5,6]. However by performing this process required highly cost and somehow it effecting the environment where the fumes and vapor that forms in the distillation process can cause physical effects such as irritation of the eyes, nose, and respiratory tract in animals and humans. In simplicity bitumen can also being produced and formulate by using waste sludge where it also requires crumb rubber and mineral oil.

Waste sludge

Waste sludge is a substance that consists of mineral oil solid waste, which includes oily sand, tank bottoms and other three kinds of sludge from refineries such as dissolved air flotation scum, excessively activated oily sludge and bottom sludge of oil from pools [7,8,9]. Generally, the oily sludge accumulates in large quantities as a dumped waste. This mixture of oil, solids, and water deposited at the storage tank bottom is known as waste oily sludge [10]. It is removed during tank cleaning operations and sent for further treatment or disposal [11]. Both the upstream and downstream operations in petroleum industry can generate a large amount of oily wastes. The upstream operation includes the processes of extracting, transporting, and storing crude oil, while the downstream operation refers to crude oil refining processes. The oily waste generated in petroleum industry can be categorized as either simple oil or sludge depending on the ratio of water and solids within the oily matrix [12].

Simple waste oil generally contains less water than sludge that is highly viscous and contains a high percentage of solids. Stable water-in oil (W/O) emulsion is a typical physical form of petroleum sludge waste [13]. In the upstream operation, the related oily sludge sources include slop oil at oil wells, crude oil tank bottom sediments, and drilling mud residues [14]. A variety of oily sludge sources exist in downstream operation, including (a) slop oil emulsion solids; (b) heat exchange bundle cleaning sludge; (c) residues from oil/water separator, waste sludge from storage tank can be a very useful waste material due to, it contains of hydrocarbon that can be used in a wide area of application. By reused this waste sludge it can benefit the society as well as the environment and also it can reduce the amount of cost in a particular research. It is actually a mixture of bituminous hydrocarbons which consists of clay, sand, inorganic matter, heavy metals, water and etc. Industrial sludge is produced from the processing of energy related to raw materials, chemical products, water, mined ores, sewerage, and man-made products. It is estimated that 3.2 billion of oil produced per year, an over 3.2 million tonnes enter the marine environment [15]. A lot of researches have been done in order to identify the best condition to overcome the oil pollution. According to [16], the Load on Top and the Crude Oil Washing (COW) were implemented and has led to a drastic decrease in oil pollution. COW can be reduced the amount of oil spilled during tanker operations since the oil tanks are washed with crude oil that dissolves the residues but this technique is not fully covered the slop .So, in order to reduced tanker slop to zero a modern tanker design such as segregated ballast tanks (SBT) was applied because they are considered eco-friendly vessel which is when the refineries receive a product from this tanker, the oil will not contaminated with chemicals, seawater, or oil residues. Since the tanks are washed using crude oil.

According to [17] that purified refinery sludge can be a substitute for processing oil in natural as well as synthetic rubber compounding. It is also found that 17 % of lighter oil fraction can be recovered from this sludge [18]. Properties in table 1.1 shows the residue left after the removal of lighter oils was converted into different grades of industrial bitumen by heat treatment at temperatures ranging from 200 to 250 ^{0}C with aluminium chloride ($AlCl_3$) catalyst for time periods ranging from 2 to 3 hours. According to [19] stated that the waste sludge contains approximately 70% hydrocarbons mostly paraffin and asphaltene together with clay, sand, inorganic matter, heavy metals, water, etc. Based on study of [20] the ratio content approximately 25% water, 5% inorganic sediments like sand, clay, scales, etc., and the rest 70% hydrocarbons.

Crumb rubber

Crumb rubber is a product of waste rubber; it is in form of powder with black colour and being widely used in sports turf medium for alleviating soil compaction. Furthermore, it is a solid waste which is an unwanted material presence, where can basically found on tires. It can be obtained from truck tyres or automobile tyres or both. Truck tyres contain 80 percent more rubber hydrocarbons than automobile tyres and also contain significantly higher amounts of natural rubber [21]. Crumb rubber is produced from discarded tyres by two methods which are grinding at ambient temperatures and grinding cryogenically cooled tyre rubber. In the latter process, the tyre rubber is chilled by liquid nitrogen. The resulting brittle rubber is then easily fractured in a hammer mill and then ground. Grinding at ambient temperatures produces sponge-like crumb rubber particles with high surface area.

In terms of environmental wise, this vehicle tires will eventually end up in landfills and this objects made of rubber can causes serious environmental problems. More than one billion of vehicle tires in the world are discarded every year. People prefer to throw it away because the tires have a material that is not easily biodegradable. When reviewed in chemical, waste consists of organic chemical compounds and inorganic compounds with a certain concentration and quantity, so we need the handling of waste tires. Destruction effort by burning them in the usual evidently produces harmful effects of pollution for it affects to human health [22]. There are two main ways to treat the used tires i.e.: recycle and reuse rubber tires and rubber reclaiming raw materials [23]. An attempt to reuse the abandoned tires by grinding them into small particles (crumb rubber) and use in sports field, pavement and construction materials can be an important approach to reduce waste rubbers in large quantities [24,25].

The annual total global production of waste rubber material, which amounts to more than 10 million tons, approximately 60~70% is used in tires [26]. After they have been worn-out during their limited service life, millions of used tires are discarded and disposed every year. In the last year, over three million tons of waste tires were generated in the Europe states [27]. China is a large country for rubber consumption. Where the annual production of waste rubber amounts to 2 million tons, 80 million scrap tires were produced in 2002, and with 12% of growth rate every year [28,29].

In this research, the method that is used to formulate the bitumen is by heating and continuously mixing process which is being conducted inside the fume hood. So that it can absorb the fume released while the sample is being heated. The main problem with this method is that to identify the suitable ratio to formulate the bitumen which is to manipulate the amount weight of crumb rubber, waste sludge and mineral oil. Therefore, in this research the suitable ratio will be determined and discussed.

During the heating and continuous mixing process, the idea of using mineral oil is as a medium to heat and melt the crumb rubber so that it will begins to degrade (devulcanize and depolymerize) causing the viscosity of crumb rubber to decrease [31]. Therefore the temperature during the process of melting and dissolve the crumb rubber must be at melting point of crumb rubber

which is at 190 ^{0}C. The speed of mixer is also playing an important role as it will stirred or mixed the mixtures until it is homogenously mixed and the crumb rubber will thus dissolved and settling down. At this stage of mechanism, the reaction is not a chemical process but rather a diffusion process that includes the physical absorption of aromatic oils from the bitumen into the polymer chain of the rubber. The rubber particles swell as they absorb oils [32], which cause the viscosity of the crumb rubber to increase during the first hour [33].

MATERIALS AND METHOD

Materials

Waste sludge was collected from the bottom of crude oil tank in an oil refinery plant located at Gebeng, Kuantan. Crumb rubber was obtained from recycling factory at Shah Alam while mineral oil with 99% purity was obtained from Shah Alam

Experimental set-up

Pre-treatment starts by placed waste sludge in separating funnel. The pre-treatment need to perform two days before the experiment began, to ensure that the component of waste sludge is fully separated. Methodology that is used called liquid-liquid extraction, where the mechanism depends upon the difference in solubility of a compound in various solvents. In this experiment, basically it will result in three layer of fluid with a difference of density of the component. According to the study of [19] the ratio content approximately 25% water, 5% inorganic sediments like sand, clay, scales, etc, and the rest 70% hydrocarbons. In this research, the bottom waste sludge were used due to it is highly concentrated compared to the other layer of the waste sludge and lesser oil content. Next, the desired amount of crumb rubber, mineral oil and waste sludge were placed in weighing boat and beaker respectively for weighting purposed. After that the hot plate and mixer were placed into the fume hood and the fume hood is switched on.

Experimental procedure

Firstly, the desired mineral oil was placed into 1000 ml stainless steel cup and were heated up to 120 ^{0}C by using hot plate for 10 minutes. Once it start to vaporized, ¼ quarter of the total weight of crumb rubber were added. The mixture then were heated at 190 ^{0}C and continuously mixed at 1500 rpm for 30 minutes. This procedure was repeated until all crumb rubber was used for 30 minutes respectively. Next once the mixture is mixed homogenously until it producing a paste form where the crumb rubber were dissolve and completely melt, the bottom product of waste sludge was added into the paste. The temperature was reduced to 100 ^{0}C and the speed of mixture was also reduced to 1000 rpm. Finally, the sample that was formulated then will be analyzed to compare the actual standard of bitumen. The analysis test includes viscosity test, density test, penetration test and softening test.

Analysis procedure

Kinematic viscosity

Viscosity of bitumen was determined by using American Standard Testing Methods ASTM D2170. The apparatus used include vane spindle, hot plate, thermometer and viscometer. The vane spindle was choosing due to it can used for paste like materials with suspended particle. The viscometer unit is switched on and the machine starts up by performing automatic calibration. After that, the spindle number is selected at the control menu. The speed of spindle was set at 100 rpm. The sample was heat using hot plate at temperature of 120 ^{0}C. Next, the spindle was dipped into the beaker that containing sample of bitumen. The spindle then, is start to revolving and the viscosity of bitumen was determined. In order to obtain a better result, the viscosity is run about 3 times to obtain the average viscosity of bitumen [30].

Density test

Density of bitumen was determined by using American Standard Testing Methods ASTM D70. The apparatus used in this experiment include pycnometer, Oven, balance refrigerator bath and thermometer. The sample is filled in a calibrated pycnometer at about 3/4 of the volume. The pycnometer and sample are weighed. The remained volume is filled with water. The filled pycnometer is brought to the test temperature using refrigerator bath. After at least 30 minutes in the bath the filled pycnometer is weighed. The density of the sample is calculated from the mass of water displaced by the sample in the filled pycnometer.

Softening point test

Softening point of bitumen was determined by using by using American Standard Testing Methods ASTM D36. The apparatus used in this experiment include shouldered ring, ball centering guide, ring holder and assembly, bath (beaker), thermometer and electric heater. The heated sample was poured slightly excess into two shouldered ring and allow to cool in ambient temperature for at least 30 min. When the specimen has cooled, the excess bitumen is cut away using slightly hot knife. The apparatus with rings, ball centering guides and thermometer in the bath was assembled. The ball was placed in each ball centering guide. Next, the bath is heated from below using heater at controlled rate. The softening point, where mean of the temperature which two disks soften enough to allow each ball envelope in bitumen to fall at distance of 250 mm/ 1 inch.

Penetration test

Penetration of bitumen was determined by using American Standard Testing Methods ASTM D5. The apparatus used include water bath, penetrometer with needle and timer, sample container and thermometer. The melted sample was poured into the sample container and allows cooling in air for 45 min to 1.5 hour. The sample was placed in the water bath at the prescribe temperature

and allow for the container to remained for 45 min to 2 hour. The needle was positions until it tip and just make contact with the surface of the sample. The needle holders then were released for the specific period (5 seconds) and adjust the instrument to measure the distance in tenths of millimeter (mm).

RESULT AND DISCUSSION

Formulation of bitumen

The result shows three different formulation of sample bitumen produced of different ratios of crumb rubber, mineral oil and waste sludge. The sample then being observed according to physical properties include colour, solubility of crumb rubber, phase and liquid content. As a result, according to observation by means of physical properties sample C have quite similar bitumen physical properties compare to the other samples. Table-1 shows the standard grade of bitumen that is obtained from Kemaman Bitumen and Figure-1 shows the best sample by far being formulated which is sample C having a ratio of (1% of mineral oil: 2% crumb rubber: 1.2% of waste sludge).

Table-1. Physicals properties of sample formulated.

Sample (crumb rubber to mineral oil and waste sludge ratio)	Color	Solubility of crumb rubber	Phase	Liquid content
A (8:3:1)	Black	Low	Liquid	High
B (2:1.7:1)	Black	Moderate	Liquid	Moderate
C (1: 2: 1.2)	Pure black	High	Semi Liquid	Low
Standard Bitumen*	Pure black	-	Semi solid	-

Table-2. Viscosity of sample bitumen.

Viscosity @120 ^{0}C (cP)	Samples			
	A (8:3:1)	B (2:1.7:1)	C (1: 2:1.2)	Standard Bitumen*
1st	34	51	95	298
2nd	29	43	87	337
3rd	31	49	91	328
4th	30	57	99	313
Average	31	50	93	319

Characterization of Bitumen sample

Characterization of bitumen sample is important before further investigation can be performed in order to understand the nature and behavior of the sample. Determination of sample of bitumen characterization which includes viscosity test, penetration test, density test and softening point test has been done using ASTM D5-97 [34].

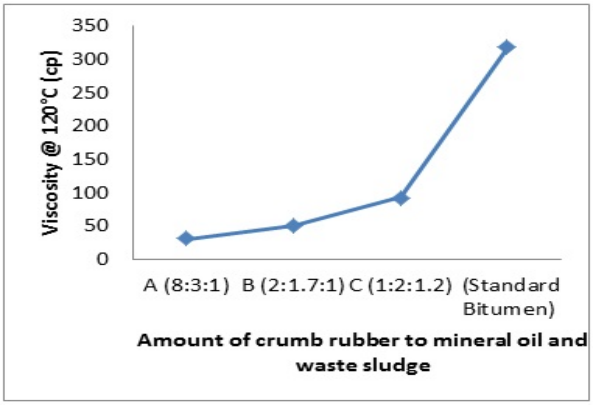

Figure-1. Viscosity of bitumen at 120 ^{0}C (cp) for several different compositions.

From the graph, the result obtained in sample C is highly viscous compare to sample A and B. However, the standard bitumen is far more viscous compared to the other sample that is formulated. This is because the amount of crumb rubber presence in the formulation sample is lesser. Therefore, the needs of increasing the ratio of crumb rubber is a must so that, the sample that is formulated is not fully liquid. The viscosity of bitumen is its internal resistance to flow or measure of it resistance to deformation by either shear stress or tensile stress. The deformation characteristics vary not only with load, but also with time rate of load application and temperature. It is neither elastic nor viscous in behavior. At low temperature it exhibits elastic behavior while at high temperature it exhibits viscous behavior [35].

Table-3. Characteristics of several compositions of bitumen.

Parameters	Method	Samples			
		A (8:3:1)	B (2:1.7:1)	C (1: 2:1.2)	Standard Bitumen*
Average Viscosity @120 ^{0}C (cp)	ASTM D2170	31	50	93	319
Density @ 15 ^{0}C (Kg/L)	ASTM D70	1.0443	1.0411	1.0398	1.0366
Softening Point (^{0}C)	ASTM D36	-	-	62	48.5
Penetration @ 25 ^{0}C (0.1mm)	ASTM D5	-	-	>40	9.3

Sample C having a higher value of softening point with 62 ^{0}C compared to standard bitumen which is 48.5 ^{0}C. Generally, having a high softening point it indicates lower temperature susceptibility and is preferred in hot climates. It same goes to the penetration where the sample C is greater than standard bitumen with having more than 40 mm compared to standard bitumen that is having 9.3 mm. In this experiment, the temperature was

set at room temperature which is at 25 ^{0}C. The idea of having penetration test is to examine the consistency of a sample of bitumen by determining the distance in tenths of a millimeter that a standard needle vertically penetrates the bitumen specimen under known conditions of loading, time and temperature (ASTM, 1998). Penetration test is a commonly adopted test on bitumen to classify the material in terms of its hardness.

The density of the sample can be calculated by using below formula

$$\text{Density}=\frac{(C-A)X\ Density\ of\ water1}{(B-A)-(D-C)} \quad (1)$$

where:
A = mass of pycnometer plus stopper
B = mass of pycnometer filled with water
C = mass of pycnometer partially filled with sample
D = mass of pycnometer plus sample plus water

From Figure-2, it is demonstrate that the sample A having a high density compared to sample B, sample C and standard bitumen. This is because the liquid content which includes the mineral oil and waste sludge content in sample A is greater compared to the other samples. The density of bitumen is greatly influenced by its chemical composition. Therefore, the increase in aromatic type mineral impurities such as crumb rubber will causes an increase in specific gravity.

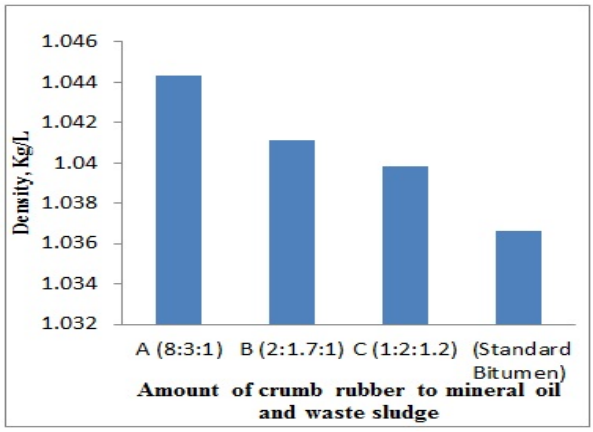

Figure-2. Density of bitumen, Kg/L for several different compositions.

Stability test

Stability test is performed to analyze the stability of the sample being formulated and compared with the actual standard of bitumen. In this experiment, each of the samples were heated up at heated at 190 ^{0}C and continuously mixed at 1500 rpm until it forms fully liquid phase. Once it starts produced, the sample was then being settling down for 5 minutes at room temperature. Next, the stainless steel cup was then placed perpendicularly and the stopwatch were started to observe the movement of the fluids.

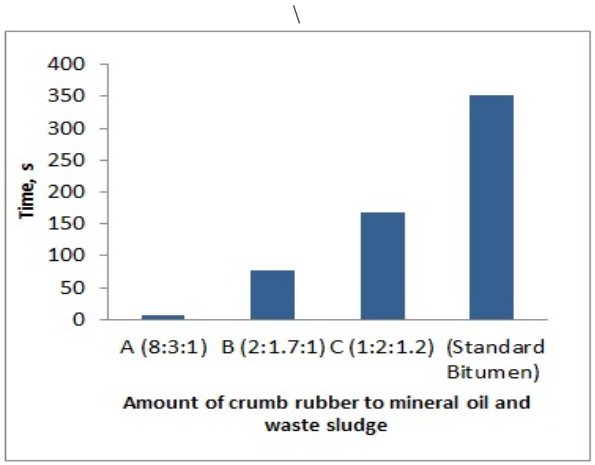

Figure-3. Stability test graph.

CONCLUSIONS

Based on result of this study, the best formulation bitumen sample is sample C with ratio of ratio (1% of mineral oil: 2% crumb rubber: 1.2% of waste sludge. The formulation of sample C produced is pure black, semi liquid in phase, crumb rubber is fully dissolve in the mixture at 190 ^{0}C where it settling down and the liquid content in the mixture is low since the amount of crumb rubber presence in the mixture of this sample is more than waste sludge and mineral oil. The sample C having viscosity of 93 centipoise, density of 1.0398 Kg/L, softening point which is at 62 ^{0}C and penetration which is at >40 mm of penetration. while the sample of standard bitumen in other hand, is having a different amount of viscosity, density, softening point and penetration which are 319 cp, 1.0366 Kg/L, 48.5 ^{0}C and 9.3 mm respectively. The result is slightly different due to the crumb rubber presence in the mixture is perhaps not fully degrade (devulcanize and depolymerize) during the heating process.

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