Journal of Engineering and Applied Sciences 11 (8): 1866-1875, 2016
ISSN: 1816-949X


# Modification of Physical and Chemical Properties of Mastic Joint by Waste Oily Bentonite Powder and Sulphur

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**Abstract:** Shrinkage cracks and rutting in pavement occurs in streets due to temperature changes in different seasons. Filling the cracks with elastomeric bituminous sealants is one of the convenient ways to prevent development of cracks. Low strength and flexibility incoherence, low stability, lack of resistant against penetration and expensive ingredient are sealant's problems. Therefore, the aim of this study is the modification of sealant to cut down costs by using waste Sulphur and oily waste bentonite from refining oil factories. The use of waste material in addition to reducing costs, solves the environment problems. In this investigation, several amounts of Sulphur and oily Bentonite was added to basic sealant material (bitumen 60/70, SBS and talc) and then the rheological, physical and mechanical properties of the blend were studied. It is found that addition of Sulphur to basic mastic causes high strength and resistance to penetration, high thermal stability, cross linking and inability to re-melt, promoting G, improving rutting resistance. It is found that the 160°C temperature is not a suitable temperature for SBS modified asphalt with every percentage of sulphur and in this temperature a gel component may be formed. Addition of oily bentonite besides sulphur to mastic causes decreasing density, increasing flexibility, improving G.

**Key words:** Bituminous sealant, sulphur, oily bentonite, physical and rheological properties, flexibility

## INTRODUCTION

The Bituminous-based sealants which have been used for over 60 year reduce the penetration of water into the structure of the road. They also reduce the rate of degradation for asphalt coating (Masson *et al*., 2002). Their broad impact on increasing the life of the seals is apparent (Casey *et al*., 2008). The surveys conducted by the transportation of Ontario (Zhang *et al*., 2010) revealed that sealing the gaps can increase the life of the asphalt by at least 4 year. These seals are also used to fill the deck of the bridges. For this reason and due to the low price of these seals installation easy maintenance, reduced noise and driving quality they have been widely used (Mo *et al*., 2013; Evans *et al*., 1993). The compounds forming gaskets commonly include: bitumen modified with polymers, filler and oil (Masson *et al*., 2002). The compositions comprise of an elastic and adhesive mixture. The reasons for the use of modified bitumen are as follows: their better quality and capability of improving many features of pitch such as thermal sensitivity and high resistance which premature the aging caused by traffic (Zhu *et al*., 2014; Shell, 1995; Shivokhin *et al*., 2012). The problem can be due to failure of the sealant adhesion and cohesion and also adhesion failure caused by cold weather and traffic (Masson *et al*., 2002). Cohesive failure of the sealant material is also due to disruptive factor. On the other hand, a sealant should be of efficient elasticity and adhesion strength at low temperatures, having these characteristics is obtained by correcting pitch) (Qadi *et al*., 2008). One of the useful methods for modifying bitumen is by using polymer modifiers. Polymer of butadiene styrene (Sone of the most commonly used polymers. The main feature of SBS is that it is made of 2 morphology phases including the glass phase made of polystyrene and poly-butadiene and it is one of the rubber compounds connected together. Consequently, the resulting polymer displays a thermoplastic-elastic behavior shows significant elasticity to asphalt and modifies the original features of the bitumen (Navarro *et al*., 2005; Carcer *et al*., 2014). However, due to the poor compatibility of the sealant and

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SBS the low storage stability of bitumen-SBS blend and polymer, a lower-cost modifier with better improving properties is needed. Using sulphur is one of the most effective methods (Zhang *et al*., 2010; Hadidy *et al*., 2011; Zhu *et al*., 2014). In addition, sulphur as an extensive and valuable resource is accessible. Using waste from desulphurization of sulphur in refineries prevents the accumulation of waste and does not contribute to environmental problems. Sulphur is applied to improve the features of the asphalt. Results of the Dah-Lynn revealed that adding Sulphur to bitumen and asphalt mixture increases firmness, elasticity and adhesion of the mixture (which depends on the temperature and sulphur rate) (Lee, 1975). Sulphur reacts with SBS alkene segments and forms cross-links which improve the chemical stability. However, there is no definite theory to explain the reaction mechanism of sulphur mixed with the asphalt-polymer. On the other hand, due to this cross-linking, sulphur addition to bituminous mixture can increase the viscosity factor (Carcer *et al*., 2014; Chen and Huang, 2007). As mentioned, the sealant needs some kind of oil to cause mixed lubrication (Masson *et al*., 2002). Since the use of bentonite in preparation of seals had not been reported yet, so in this article it is used as filler and the resultant oil is used to mixed lubrication.

Due to some seals problems, attempts have been made to improve the quality and to reduce the production costs. To achieve this objective, sulphur wastes are used as soil amendments and refining waste oil purification plant can be used to prevent the accumulation and environmental pollution and to reduce production costs and increase the quality of the final product. Impact of the mixed powder on mastic mixture can cause increased softness and flexibility. Testing the seals according to ASTM standards has shown the impact of these compounds addition to mastic mixture.

## MATERIALS AND METHODS

**Basic bitumen:** Bitumen pitch used in this project is of 60/70 typ e which is the product of Pasargad refinery with its characteristics given in Table 1.

In this project, the SBS Radial type code: Enperen 611 product of the EN CHUAN, a Chinese Company in accordance with the specifications in Table 2 was used.

**Sulphur:** Sulphur has been provided from Bandar Abbas Pasargad Oil Refineries which is a yellow powder with a molecular formula S8.

Table 1: Bitumen 60/70 characteristics

| Sample test | Bitumen 60/70 |
|---|---|
| Softening point | 50 |
| Penetration at 25°C (0.1 mm) | 65 |
| Ductility at 25°C (cm) | +75 |
| Flash Point (°C) | 330 |
| Solubility in T.C.E (wt%) | 99.99 |
| Modulus (kgf/mm$^2$) | 0.0022 |

Table 2: SBS polymer characteristics

| Typical properties | Test method | Enprene |
|---|---|---|
| Structure | - | Radial |
| Butadiene styrene ratio | ASTM D1416 | 30/70 |
| Poly styrene block | | 30 |
| Oil content (PHR) | ASTM D1416 | 0 |
| Toluene solution viscosity (5.0%) CPS | ECCA 116 | 24 |
| Elongation (%) | ASTM D412 | 700 |
| Specific gravity | ASTM D792 | 0.93 |

**Bentonite:** Bleaching soil of non swelling calcium bentonite with the following general chemical formula: (Na, Ca) 0.33 (Al, Mg) $2Si_4O_{10}$ $(O_2H).nH_2O$ which is produced from the waste treatment plant. This compound was obtained from 30% oil content.

**Equipment standards:** Softening point and physical properties of cone penetration grade for bituminous resins, respectively, according to standard ASTM-D36 and D217 ASTM was performed. According to standard, bitumen softening is inversely related to sensitivity to temperature.

To obtain the mastic tensile strength (Tensile) with a constant speed of puller 12 mm min$^{-1}$, the Load cell model 383 with a capacity of 200 kg made by REVERS company and according to America ASTM-D 1985 standard was used. This test is to measure the bituminous mixture particles' agglutination together with the adhesion of the surfaces.

Cold flexibility test was performed using the cold bending machine manufactured by Asia Company equipped with a homogeneous temperature fan capable of cooling to -10°C. This test indicates the resistance level against the bitumen cracking at low temperature.

The amount of material and thermal stability was examined using Fan Oven with heating capacity of 200°C. This test shows the stability of the bitumen and prevents the flow of the bitumen at high temperature. In order to study the rheological properties of bitumen, the following tests were performed.

The Dynamic Shear Rheometer (DSR) test was performed in accordance with: (Anton Paar, MCR300) under the temperature of 40-82 °C and the fixed frequency of 10 srad$^{-1}$ by DSR machine. Also, a stirring device with adjus table bypass was used for stirring the samples.

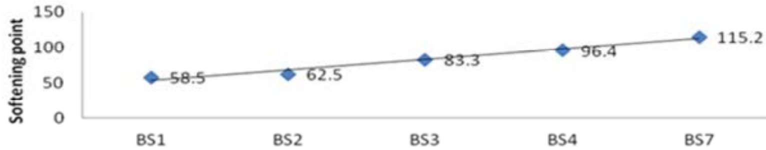


Fig. 1: Softening point results for SBS mixing with bitumen

**Preparation of samples:** Sample preparation is carried out in 3 stages. The first step involves the addition of SBS to bitumen; SBS with bitumen was mixed in different percentages. At all stages, the amount of talc as filler and constant volume, was considered. Bitumen and SBS were prepared using mechanical mixers with adjustable circuit. For this purpose, the bitumen is heated to 180±15 and then the SBS is added slowly. The mixer speed was set on 940 rpm (no bubbles in the mixture of bitumen). For complete liquidation of SBS in bitumen, 90 min was allocated; this period of time will be fixed for all stages of SBS liquidation. Mixing time includes inflammation (swelling) and spread of bitumen to form a homogeneous mixture of polymer. Then the talcum powder is added and 60 min was considered for the mixing stage. The specimens were cast in a mold and then after reaching ambient temperature, they were tested. In the second stage, the temperature was decreased to 150±15°C and different percentages of sulphur were added to the mixture of bitumen and SBS. After 15 min, the mixture was poured into the mold in accordance with the previous stage. The third step, to add powdered bentonite, was divided into 2 phases; different amounts of the bentonite were added to sulphur-SBS-asphalt mixture. Bentonite was added to the mixture 90 min after adding the talcum powder. Then, the powder was added to the mixture and during 60 min the oil content of the powder penetrated into the bitumen to make it soft and flexible. At next stage, the sulphur was added to the mixture.

## RESULTS AND DISCUSSION

Asphalt modification by the polymer SBS Sample's characteristics and test results are given in Table 3 and 4 and changes in the bitumen mix, after adding different amounts of SBS polymers are shown in Fig. 1-5. Penetration results for SBS mixing with bitumen. According to the results reported with respect to Fig. 1-5, adding the SBS polymer to modified bitumen mixture:

- Increases thermal stability
- Decreases temperature flexibility
- Decreases tensile strength
- Reduces the degree of penetration

Table 3: Fixwd mixture of bitumen and SBS with fixed values of 15% talc

| Sample | Bitumen (%) | SBS (%) |
|---|---|---|
| BS1 | 84 | 1 |
| BS2 | 83 | 2 |
| BS3 | 82 | 3 |
| BS4 | 81 | 4 |
| BS7 | 78 | 7 |

Table 4: Test results for SBS mixing with bitumen

| Sample | Softening point (°C) (%) | Degree of penetration | Tensile strength (kg/5 cm) | Cold bending (°C) | Thermal stability (°C) |
|---|---|---|---|---|---|
| BS1 | 5850 | 800 | 10.6 | 15 | In pure material 50 |
| BS2 | 6150 | 800 | 14 | 12 | In 1 cm material 50 |
| BS3 | 8330 | 700 | 17.4 | 10 | In 1 cm material 60 |
| BS4 | 9640 | 700 | 16.23 | 5 | In 4 cm material 60 |
| BS7 | 11520 | 400 | 19 | 0 | In 2 cm material 60 |

Table 5: Bitumen-SBS0-sulphur mixtures with affixed amount of 15% talc

| Sample | Bitumen (%) | SBS (%) | Sulphur (%) |
|---|---|---|---|
| BSS1 | 80 | 4 | 1 |
| BSS2 | 79 | 4 | 2 |
| BSS3 | 78 | 4 | 3 |
| BSS4 | 77 | 4 | 4 |
| BSS5 | 76 | 4 | 5 |
| BSS6 | 75 | 4 | 6 |
| BSS7 | 74 | 4 | 7 |

According to these charts, it can be found that small amounts of SBS do not affect the pitch but considerable amounts can cause significant changes and a review of samples revealed that the standard amount of SBS was 4% the BS4 is a sample of this type.

**Polymer modification by sulphur:** Sample's characteristics and the test results are given in Table 5 and 6.

**Softening point test: samples containing sulphur, bitumen and SBS:** The effect of adding sulphur to the mixture of bitumen and SBS polymer is shown in Fig. 6. Sulphur is chemically mixed with bitumen, reacted with bitumen and polymer components and consequently improved the physical and rheological properties of bitumen. According to the graph, the changes are minor. Sulphur does not have much impact on softening point and compared to the samples containing only SBS and talcum powder in this case, sulphur causes some positive

Table 6: Bitumen-SBS-sulphur mixtures test

| Sample | Softening point (°C) | Degree of penetration (°C) | Tensile strength (kg/5 cm) | Thermal stability (°C) | Cold bending (°C) | Viscosity (Pa $sec^{-1}$) |
|---|---|---|---|---|---|---|
| BSS1 | 81.2 | 3 | 48.75 | Flow: in 70 3 cm | 5 | 920 |
| BSS2 | 83.9 | 6.3 | 58.86 | Flow: in 70 3 cm | 7 | 990 |
| BSS3 | 7190% | 830% | 2620% | Flow: in 60 22 mm | 3 | 900 |
| BSS4 | 6700% | 460% | 5000% | Flow:in 56 mm 4 | 3 | 1060 |
| BSS5 | 6000% | 1100% | 3500% | Flow:in 66 1 cm | 0 | 1135 |
| BSS6 | 8010% | 830% | 1630% | Flow:in 64 1 cm | 5 | 1220 |
| BSS7 | 8770% | 660% | 2280% | Flow:in 64 2 cm | 5 | 1300 |

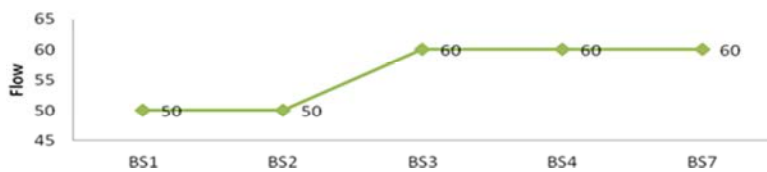


Fig. 2: Flow results for SBS mixing with bitumen

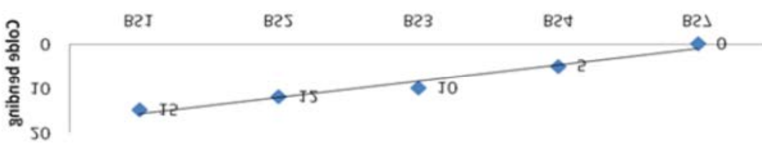

Fig. 3: Cold bending results for SBS mixing with bitumen

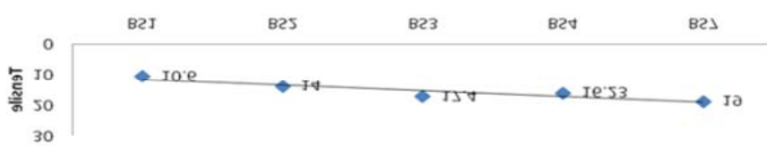

Fig. 4: Tensile results for SBS mixing with bitumen

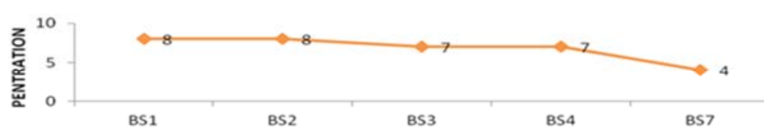


Fig. 5: Penetration results for SBS mixing with bitumen

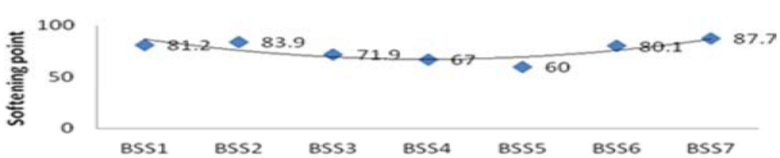


Fig. 6: Softening point result for Bitumen-SBS-sulphur mixtures

changes in softening point but this change will not be too much. Thermal stability test for samples containing bitumen, SBS and sulphur: although, the addition of sulphur increases the thermal stability of polymer-containing bitumen, the impact of sulphur on the thermal stability of polymer-bitumen mixture is not too substantial. For reasons discussed in softening point test section, changes are in low stages. Figure 7 illustrates this trend. Cold bending test for samples containing bitumen, SBS and sulphur: Sulphur has the ability to form very strong cross-bridges and also a high tendency to react with polymer SBS, resulting in high strength and


Fig. 7: Flow result for Bitumen-SBS-sulphur mixtures

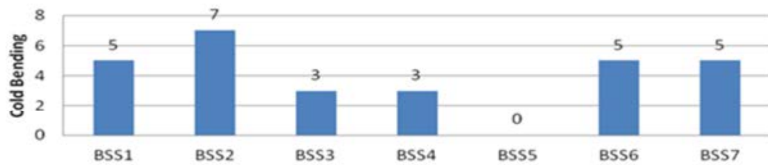


Fig. 8: Cold Bending result for Bitumen-SBS-sulphur mixtures

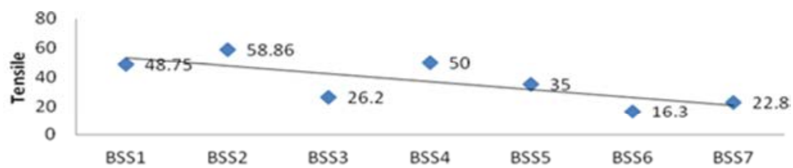


Fig. 9: Tensile result for Bitumen-SBS-sulphur mixtures

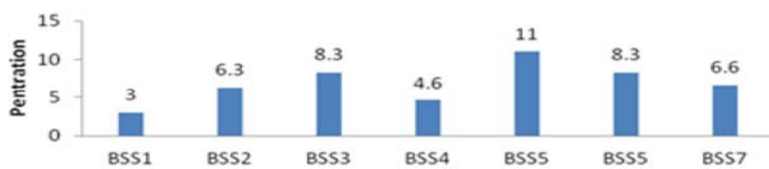


Fig. 10: Penetration result for Bitumen-SBS-sulphur mixtures

sufficient flexibility of mastic mixture at low temperatures. The impact of adding sulphur has been identified in Fig. 8.

Tensile strength test for samples containing bitumen, SBS and Sulphur: Figure 9 shows the impact of adding sulphur to the mastic in the presence of SBS polymer. According to Fig. 9, addition of sulphur decreases tensile strength. Except samples containing BSS4 and BSS5, the tensile strength values compared to samples containing 4% polymer is more. It can be concluded that the tensile strength of mastic depends on the total sulphur additives.

Test to determine the degree of penetration for samples containing bitumen, SBS and sulphur. Test to determine the degree of penetration is a measure of the strength of the mastics against penetration of external objects. Figure 10 shows the changes in the amount of penetration degree for bitumen-polymer samples after the addition of sulphur. By examining the above graph, it is observed that the adding Sulphur initially has tightened the bitumen polymer and has decreased the degree of penetration. This is likely due to the fact that sulphur involves the resin responsible for bitumen softness in the vulcanization process. But as the amount of sulphur increases, the cross-links of sulphur and polymer also become stronger. By developing sulphur and polymeric networks, resin is isolated from the network and consequently the degree of penetration increases. Evaluation and comparison of the results specified that the BSS4 and BSS5 samples have higher flexibility as well as the lower penetration rates. Of the two, BSS4 has higher tensile strength, lower viscosity and thermal stability compared to BSS5. If sulphur is increased to more than 5%, the sealant specified properties will reduce. In these examples, it was indicated that the increased amounts of sulphur have a desired and positive impact on

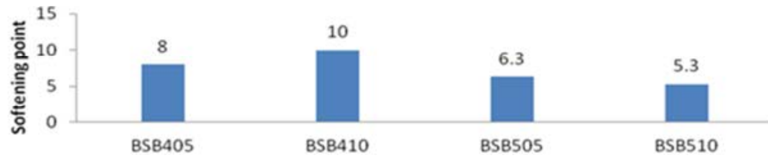


Fig. 11: Softening point test for 4% SBS, 4% sulphur and modified bentonite and also bitumen samples containing 4% bentonite, 5% sulphur and bentonite

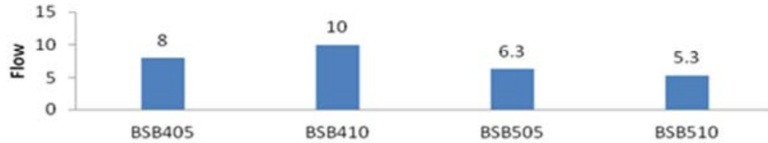


Fig. 12: Thermal stability test for samples containing 4% SBS, 4% sulphur and modified bentonite and also bitumen samples containing 4% bentonite, 5% sulphur and bentonite


Fig. 13: Cold bending tests for samples containing 4% SBS, 5% sulphur and bentonite

the rheological properties of mastic mixture. However, to ensure in this stage of the study both BSS4 and BSS5 were chosen as optimization samples.

Check oil changes after the addition of bentonite powder to the mixture of bitumen-polymer-sulphur: Softening point test: mixture of bitumen-SBS-sulphur-bentonite. Bentonite has a good dispersion in bitumen and mixed with bitumen, it creates a consistent mixture which eventually increases the asphalt softening point rate. Chart evaluation clearly illustrates the impacts of increased amount of bentonite on enhancing the softening point rate. Samples with bentonite <5% did not show any change in softening point rates but samples with >10% bentonite had a very high viscosity rate.

Thermal stability test: (flow) for mixture of bitumenSBS sulphur-bentonite: according to the Fig. 11, 12 increasing bentonite has not changed the thermal stability of the samples containing 4% sulphur and the thermal stability for both BSS405 and BSS410 is equal to that of the BSS4. But in samples with 5% sulphur, thermal stability increased for increasing amount of bentonite. However increased bentonite has eventually increased the thermal stability of the mixture of bitumen-polymer sulphur and as the bentonite content increased from 5-10%, the thermal stability also enhanced. It was observed that for samples with 5% sulphur, the two-phase problem is partially solved.

Cold bending test for mixture of bitumen-SBS-sulphur-bentonite: evaluating the Fig. 13, it is concluded that the addition of 5% bentonite to the mixture of bitumen-polymer-sulphur does not affect the flexibility factor and this is obviously pivotal in both sets of samples (samples containing 4 and 5% sulphur). Increasing the amount of bentonite to 10% only affected the BSS410 and also increased the flexibility (bending) rate of the mixture containing 4% sulphur.

Tensile strength: mixed bitumen-SBS-sulphur-bentonite: according to the graph, we conclude that increased amounts of bentonite have reduced the tensile strength. There is a little correlation between increasing the sulphur and bentonite amount and reduce in tensile strength rate. Actually, greater amounts of bentonite does not reduce the resistance effects of sulphur but yet been able to give more softness and durability to the mastic mixture. On the other hand, it is understood that the two-phase problem for samples without bentonite caused by sulphur 5% additives can be fixed by increasing bentonite amount and in this case both sets of samples containing 4 and 5% sulphur have the same tensile strength.

Tests to determine the penetration degree for bitumen-SBS-sulphur-bentonite mixture: it is clear from the

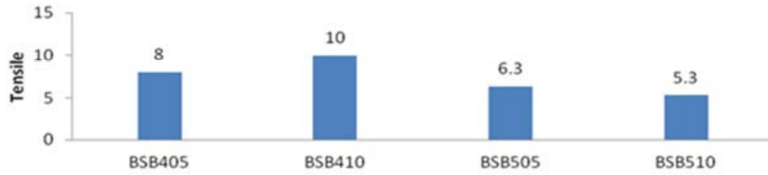


Fig. 14: Tensile strength test for samples containing 4% SBS, 5% sulphur and bentonite

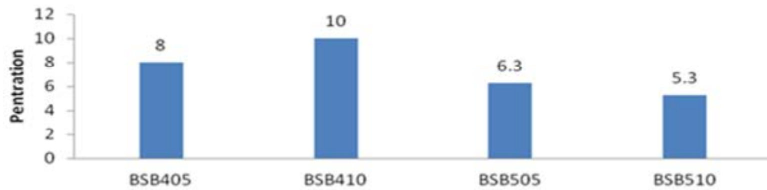


Fig. 15: Tests to determine the degree of penetration for samples containing 4% SBS, 5% sulphur and bentonite

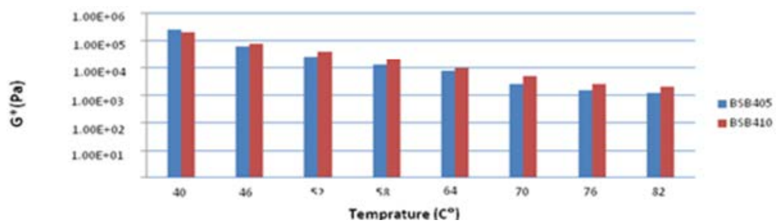


Fig. 16: G* test for samples containing 4% SBS and 4% sulphur (G* pa)

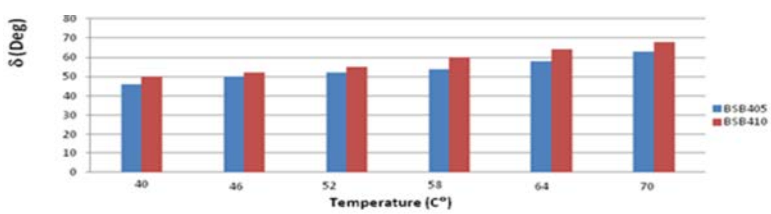


Fig. 17: d test for samples containing 4% SBS and 4% sulphur

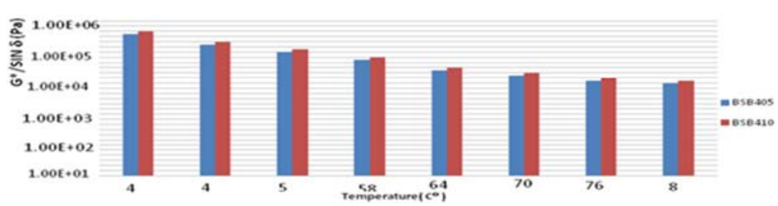


Fig. 18: G* sind test for samples containing 4% SBS and 4% sulphur

figure that for samples with 4% sulphur, bentonite additives has increased the penetration degree compared to that of samples without bentonite. The increased amount of bentonite from 5-10% can create such an effect. However, this trend was reversed in the second series of samples with bentonite and bentonite additives. Increased amounts of bentonite reduced the penetration degree of samples with 5% sulphur compared to samples without sulphur and eventually increased the degree of penetration for these samples (Fig. 16-23).

- Testing Dynamic Shear Rheometer (DSR) for bitumen-SBS-sulphur-bentonite mixture
- Samples containing 4% SBS and 4% sulphur
- Samples containing 4% SBS and 5% sulphur

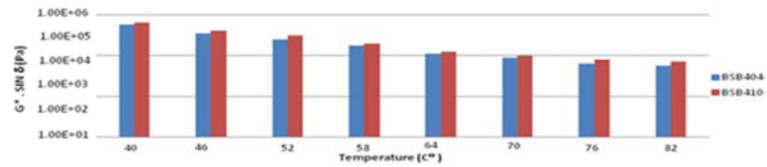


Fig. 19: G* sind test for samples containing 4% SBS and 4% sulphur **(**G* sin**δ** (Pa)**)**

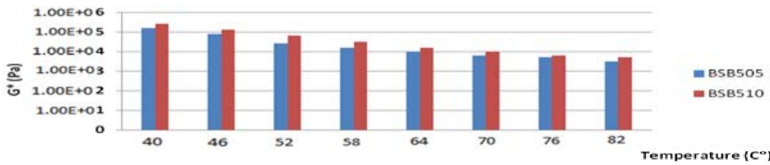


Fig. 20: G* test for samples containing 4% SBS and 5% sulphur (G* Pa)

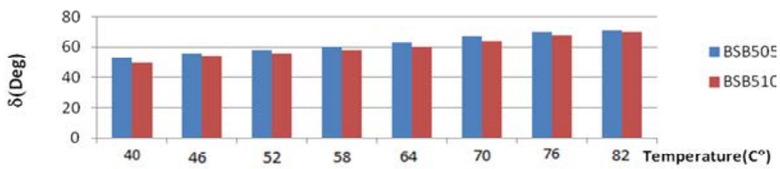


Fig. 21: d test for samples containing 4% SBS and 5% sulphur (**δ** (deg))

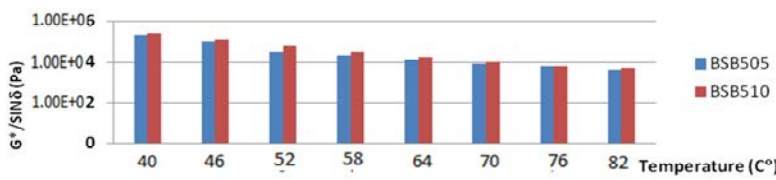


Fig. 22: G*/sind test for samples containing 4% SBS and 5% sulphur (G* sin**δ** (Pa))

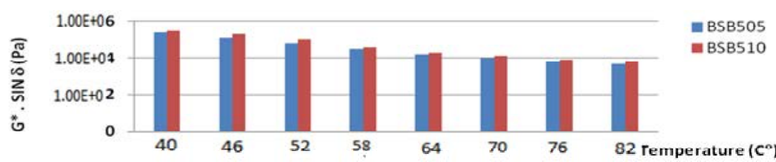


Fig. 23: G* sind test for samples containing 4% SBS and 5% sulphur

Considering all charts in section 23, we can see that the rheological properties of bitumen mastic are improved by addition of bentonite. In graphs of G*, G*/sin d and sin.G*, adding bentonite improves the asphalt resistance and the bitumen mastic resistance against track disruption. However, due to the great increases in viscosity at higher amounts of bentonite, adding >10% can cause the loss of bitumen mastic mixture. This section gives a comparison of the chart results which reveals that increasing the oil powder around <5% does not really affect the major features of the mastic. Despite the increase in viscosity increasing the oil powder up to 10% saves the thermal stability and improves the flexibility rate. A brief test on effect of pure bentonite on mechanical property of bitumen-SBS-sulphur mixture 10% pure bentonite was added to mixture on behalf of oily bentonite to prove the efficiency of oil on mechanical properties of mastic, then penetration, softening point, tensile and cold bending tests were studied (Fig. 24-27). Comparison between these results and results from Fig. 6-10 showed that the effect of pure bentonite on mechanical properties of blends was negligible.

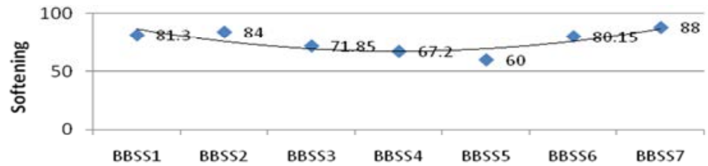


Fig. 24: Softening point result for Bitumen-SBS-sulphur and pure bentonite mixtures

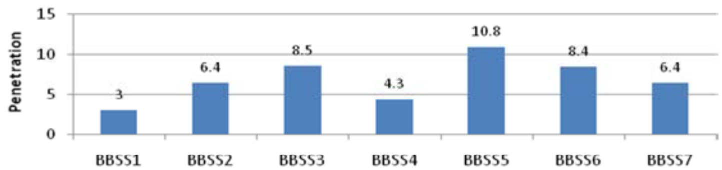


Fig. 25: Penetration result for Bitumen-SBS-sulphur and pure bentonite mixtures

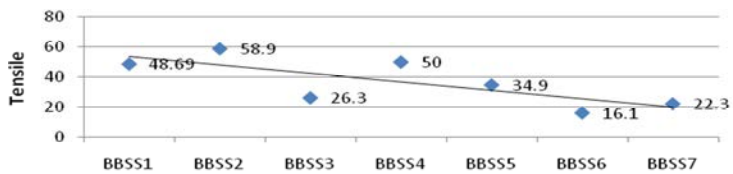


Fig. 26: Tensile result for Bitumen-SBS-sulphur and pure bentonite mixtures

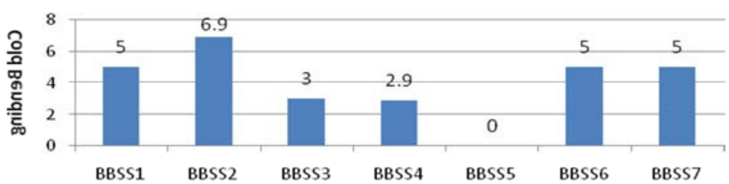


Fig. 27: Cold bending result for Bitumen-SBS-sulphur and pure bentonite mixtures

## CONCLUSION

Sufficient amount of the polymer depends on the type, structure and size of the polymer particles. According to the results, samples containing 4% SBS, have higher softening point, thermal stability (as rutting resistance test), flexibility and higher tensile strength (as cracking resistance test) as well as lower degree of penetration.

Sulphur additives have a great impact on the softening point and thermal stability of bitumen mastic containing talcum powder and SBS polymer. But this is viewed as some minor changes. Impacts of pure sulphur are >3% sulphur. In this study cold bending and tensile tests were done on behalf of cracking resistance. Results shows that adding sulphur to the bitumen mastic mixture can increase the cold bending and tensile strength characteristics and eventually increase the cracking resistance ability.

According to all the tests carried out in the presence of the sulphur, it was concluded that lower amounts of sulphur; <3%, does not affect the bitumen modification process. On the other hand, >5% sulphur can cause the two-phase problem in polymer-sulphur networks. So, BSS4 and BSS5 were selected as the optimal samples.

Bitumen mixtures containing SBS, sulphur and oily bentonite are not capable of re-melting. It is highly desirable to increase the amount of bentonite which increases the softening point and thermal stability and finally increase rutting resistance. Moreover, bentonite eliminated the two-phase problem in samples containing 5% sulphur. Increased bentonite retains the cold bending property of bitumen mastic and it can be concluded that oily bentonite powder positively affects the temperature sensitivity of the bitumen mastic.

Oily bentonite powder keeping the agglutination does not sensitively affect the elastic resistance and has increased the viscosity.

Bentonite added to the samples containing 4% sulphur and increased the degree of penetration but in the presence of 5% sulphur penetration rate reduced and the rheological properties improved.

Evaluating the test results, we can conclude that increasing the bentonite amount to 10% can easily solve the two-phase problem for samples containing 5% sulphur. However, some amounts of bentonite act as filler and increase the volume of bitumen mastic.

**ACKNOWLEDGEMENT**

The researchers of this article would like to thank Mr. Mehdi Nourinejad for their input towards improving this study.

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