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Development of a Shear Thickening Fluid and its Use as a Modifier in Asphalt Binder

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Abstract- This study presents development of a Non-Newtonian fluid, also referred to as a Dilatant or Shear Thickening Fluid (STF), and its use as an additive/modifier for improvement of different visco-elastic properties of bitumen. Dosage optimization of STF was done with the help of Superpave performance grading (PG) technique and other conventional asphalt binder tests. Storage stability test was carried out to ensure the stability of the new STF modified bitumen samples. Binder samples were prepared using different dosages of STF and then different conventional and rheological tests were used to study the effect of STF on the properties of modified binders. From the results of investigations, an optimal dose of 4% STF by weight of the binder is selected for upgrading the binder properties. The addition of 4% STF helped to improve the high-temperature visco-elestic properties of the asphalt binder which is a major requirement of the local pavement industry in Pakistan. STF improved the high-temperature range of the binder under study from Superpave PG 52 to PG 64.

Keywords-Shear Thickening Fluid, Performance Grading (PG), Complex Shear Modulus, Modified Bitumen

1. **Introduction**

Bitumen is a very complex material, exhibits both viscous and elastic response at the same time, and these responses are highly dependent on both the traffic loading and the temperature[1], [2]. Experts, engineers and researchers have used a variety of additives and modifiers to upgrade the performance of asphalt materials in different loading and environmental circumstances. The modifiers include styrene butadiene styrene (SBS) [3], [4], crumb rubber [5], [6], styrene-butadiene-rubber (SBR) [7], [8], waste fibres [9], ethylene glycidyl acrylate (EGA) terpolymer[1] and waste tire rubber[10].

During the past decade nano-technology has grabbed the attention of the material industry; it has been extensively used in various fields. Asphalt binder has been modified with different nano-materials such as nano-Silica, Nano-clay, Single-wall nano-tube (SWNT), and with nano-fibers of carbon. The developed binder samples are subjected to different laboratory investigations including rheology (DSR), fatigue and visco-elastic characteristics, and their mixing procedures are also investigated. Results from these investigations showed that nano-materials helped improve different parameters such as rut resistance, complex shear modulus (G^*), viscoelastic-plastic behaviour of the binder, and fatigue life [11]–[13]. Upper temperature performance of the binder can also be improved by using nano-clay as an additive [14]. Addition of carbon microfibers in bitumen also helped enhancing the resistance against moisture susceptibility [15].

As discussed above nano-technology is emerging very rapidly and showing its usefulness in many fields. Shear thickening Fluid (STF) is one of the best emerging applications of nano-technology which is also called Dilatant. A Dilatant is composed of nano-particles that are suspended in a solvent (Carrier Fluid). Dilatant (STF) is a non-Newtonian fluid [16] whose viscosity dramatically increases with the increase in shear rate above a certain threshold value. When the energy from the impact is dissipated the material reverts back to a liquid [17]. Particle size, concentration, distribution, shape, particles dispersions and particle to particle interaction are all the factors that affects the commencement of shear thickening effects [18]. Numerous studies have been made on the Dilatant and generated vast quantity of patent filings due to its extra ordinary potential since their discovery. The most common uses and suitability of Dilatant that are discovered till now includes its use in personal protection material, sports and in body armour [19]. Although several studies have been made to investigate uses of the dilatant but still a lot of efforts are required to further explore potential uses of this material.

As discussed earlier STF is an emerging technology and showing it's applications in many fields but this material was never used before for asphalt modification so, through this study, an attempt has been made to check the effects of STF on high temperature performance of the bitumen. The STF used in this study was prepared from Nano-Silica dispersed in ethylene Glycol because silica nanoparticles are one of the very best material for a shear thickening fluid [18]. The STF was added to virgin bitumen because it could improve different rheological and conventional properties of the asphalt binder especially for high temperature applications. High temperature performance of asphalt pavements is a major concern of local pavement industry in Pakistan, as flexible pavements fail prematurely when exposed to high summer temperatures and excessive loading. Local pavements fail due to excessive permanent deformation of the asphalt mix far before reaching the end of its fatigue life. To address these problems several modifiers have been used to improve binder performance. The most commonly polymers (SBS, SBR) and acids, especially polyphosphoric acid. The PMBs have certainly improved the performance of asphalt binder but, the issue with PMBs is that of storage stability and are unstable to be stored for a longer time. On the other hand, PPA modified asphalt binders have been found adequate in their performance but still lack in some properties such as difficulty to handle during mixing, higher temperature requirement for mixing, due to higher viscosity and instability at elevated temperatures. "So by using STF as modifier we have made an attempt to develop such a modifier which enhance the viscoelastic performance of the binder, which is environmental friendly as it will require less heat and time for mixing, easy in handling and storage stable also". The STF was added in the base binder in different concentrations by weight of the base binder and then for the rheological and performance evaluation of the modified samples different asphalt binder tests were performed such as the shear thickening test of modifier, bitumen conventional tests, and dynamic shear rheometer (DSR) investigations. Storage stability of the modified samples was also checked. Details of the experiments and discussions of outcomes from the results obtained are presented in the subsequent sections of the manuscript.

2. Experimental Work

2.1Materials

Control asphalt binder of pen grade 80/100 was obtained from Attock Refinery Limited (ARL), Pakistan. Nano-Silica was imported from Dalian Fuchang Chemicals China and Ethylene Glycol was imported from Honeywell Germany. Tables1, 2 and 3 presents the properties of these materials respectively.

Table 1: Properties of Control Binder

Test	**Value**	***Standard***
Softening Point (◦C)	*48*	ASTM D36
Penetration Value (0.1 mm)	*95*	ASTM D5
Ductility mm (25 ◦C)	*108*	ASTM D113

Table 2: Properties of Nano-silica

IngredientName	**Purity**	**Size**	**Specific area**	**Melting point**
Silicon Dioxide	*>99%*	*12 nm*	*200* m^2/g	*>300 °C*

Table 3 Properties of Ethylene Glycol

Material Name	**Purity**	**Boiling Point**	**Freezing Point**	**Density**
Ethylene Glycol	*>99%*	*197 °C*	*-12 °C*	*1.11* g/cm^3

2.2 Sample Preparation

The sample preparation is divided into two parts. In the first part a Shear Thickening Fluid (STF) or Dilatant is prepared while in the second step the STF is mixed with base binder to produce STF Modified Bitumen.

2.2.1 Preparation of Shear Thickening Fluid (STF)

STF is composed of two components the particles and a carrier fluid to fabricate them. STF prepared in this study was a high concentrated mixture of 40% nanoparticles and 60% Ethylene Glycol as carrier. As it was a high concentrated mix so, to get a homogenous mixture particles were added to the solvent in small increments and the blender was used to mechanically stir the mixture for 25 minutes after every incremental addition [19] and this process was continued until the exact ratio of 40:60 by weight was achieved. In order to get a homogenous result the mixture was stirred for 2 hours after the addition of the complete amount of the nanoparticles. To eliminate bubbles from the mixture the prepared STF was kept in open air for 24 hours. Mixing was done using high shear mixer at 3000 rpm.

2.2.2 Preparation of STF Modified bitumen

According to literature, different additives are mixed in the base binder with the help of high shear mixer, and the selected temperature range is 130 ºC to 165ºC, while the proposed duration of mixing range from 30 minutes to 60 minutes [21]. The prepared STF in this study was added to the base binder in three proportions (2%, 4% and 6%). Proportioning was made by weight of the binder. To get a uniform concentration the STF was introduced to the bitumen and was continuously mixed for 45 minutes at 130ºC using a high shear mixer at 3000 rpm. This procedure was carried out for each of the modified sample.

2.3 Shear thickening test of Dilatant (STF)

Steady state test was used to check the thickening behaviour of the modifier. In the steady state test the sample (40% of nano-silica in 60% ethylene glycol; by weight) were rotated in DSR by providing varying shear rate as input from 0.1 to 1000. Figure *1* represents viscosity as a function of shear rate for the prepared STF.

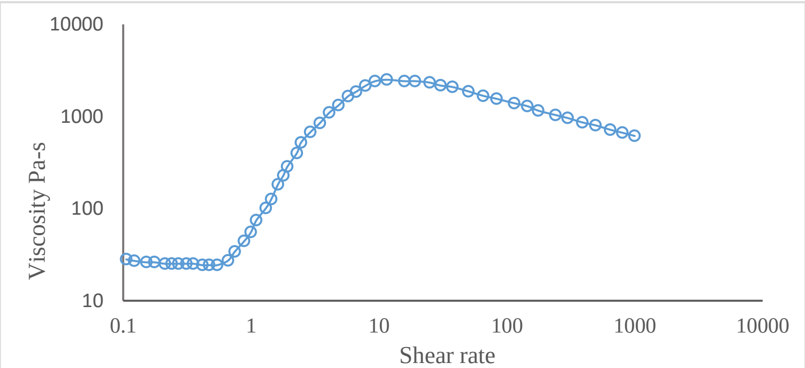

Figure 1: Viscosity as a function of shear rate for STF

From Figure 1it can be concluded that 40% concentrated STF has a clear shear thickening (non-newtonian) effect such that at low shear rate the viscosity gradually decreased and then the viscosity began to increase dramatically when the shear rate was above a critical value until it reached the peak viscosity value. After the shear thickening region, the viscosity decreased again[19].

2.4Stability investigation of STF Modified Bitumen

To check the stability of modified bitumen the following test was conducted.

2.4.1Storage Stability

Results of storage stability test verified the consistent diffusion of STF in the binder. Table 4 shows the results of the test. Stability was evaluated from the difference in top and bottom softening points, and the stability is considered as acceptable because the softening difference from top to bottom is less than 2.2ºC.

Table 4: Storage Stability of Base and STF Modified Asphalt Binders

Sample ID	**Softening Point**		
	Top	**Bottom**	**Top – Bottom**

Base Binder	48.2	48.5	0.3
2% STF Modified	50.4	50.9	0.5
4% STF Modified	54.3	55.2	0.9
6% STF Modified	57.6	58.8	1.2

2.5 Conventional Binder Tests

The conventional physical properties tests on the modified and base binder Such as penetration, softening point and ductility were performed according to ASTM D5, ASTM D36 and ASTM D113 respectively. These tests were executed to examine the changes after the addition of STF in the binder.

2.6 Performance Grade (PG)

The PG grade of bitumen describes the low and high temperature at which the binder shows suitable behaviour and perform satisfactorily. As in this study we are mostly concerned with high temperature so, MCR101 Dynamic shear Rheometer (DSR) with parallel-plane geometry, 25mm dia and 1 mm gap was used to determine the required High PG of the base and modified binder. This test was performed according to AASHTO T315.

2.7 Rheological properties

For the rheological characterization DSR model MCR 101 by Anton paar with plane-parallel geometry, 25mm dia and 1 mm gap was used to perform the corresponding Frequency Sweep test. This test was performed to estimate the shear deformation performance and temperature sensitivity of the binder. Test was performed according to superpave criteria i-e with constant strain of 10% and ranged frequency from .1 to 10 HZ, furthermore these tests were performed on different temperatures from 10°C to 82°C with 12 °C difference.

3. Results and Discussions

3.1. Conventional binder Properties tests

Error! Reference source not found. enlists the effect of STF on the softening, Ductility and penetration values of improved/modified and Base binder. The results show that by adding STF into the base binder there is an increase in the softening point and decrease in ductility and penetration values was recorded. By the addition of 2%, 4% and 6% STF to the base binder 7%, 18% and 22% reduction in penetration and 7%, 15% and 19% increase in softening point was recorded respectively. Softening point can be used as indicator to illustrate the stiffness of binder. From the observed results it can be concluded by the fusion of STF in Bitumen stiffness has been increased and the high temperature susceptibility has been reduced [22], [23].

Table 5 Results of conventional tests

Blends description	Penetration (1/10 of mm)	Softening(°C)	Ductility (cm)
Standard	**ASTM D5**	**ASTM D36**	**ASTM D113**
Base binder	96	48	112
2% STF	89	51	107
4% STF	79	55	99
6% STF	75	57	93

3.2. Performance grade

Results of PG test are presented in Figure 2which clearly demonstrates a substantial improvement in PG of binder when it was treated with different dosage of STF. With the increase in PG we can say that resistance to permanent deformation also increases in other words with the increase in PG of the binder resistance to rutting also improves. Hence we can say that by the addition of STF to the binder resistance to permanent deformation also increases, which is a main cause of road failure in Pakistan. From the results in the Figure *2* it can be seen that bitumen having PG 52 was valued to binder of PG 64 by the addition of both the dosage 4% and 6% STF, but 4% STF as modifier was selected as optimal dosage on the basis of PG test. We can conclude that Dilitant (STF) has the affinity to improve high temperature performance of the binder without compromising the lower temperature performance.

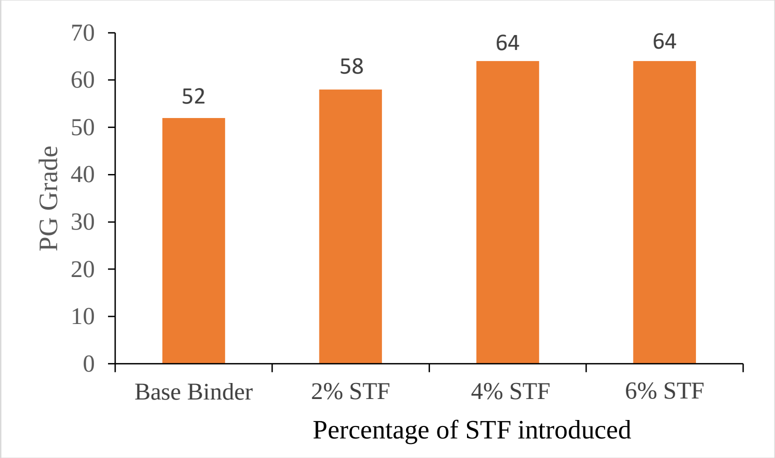

Figure 2 Effect of STF dosage on performance grading

3.3 Rheological properties

Figure 3represents the interdependency of reduced frequency and G^*that is the complex shear modulus of the binder. From the investigations it was concluded that by the addition of 4% STF there is a 45% increase in the G^*at frequency of 10 HZ, which means that the modifier is resulting in the stiffness of the binder. Lower left portion of the Figure *3* represents high temperature and low frequency zone, a significant increase can be clearly seen in that area which means that STF modified binder is more appropriate for high-temperature areas. Meanwhile it was also noted that upper right portion of the Figure *3* remained almost unchanged which represents low temperature and high frequency, so we can say that we have improved the high temperature properties without compromising low temperature performance.

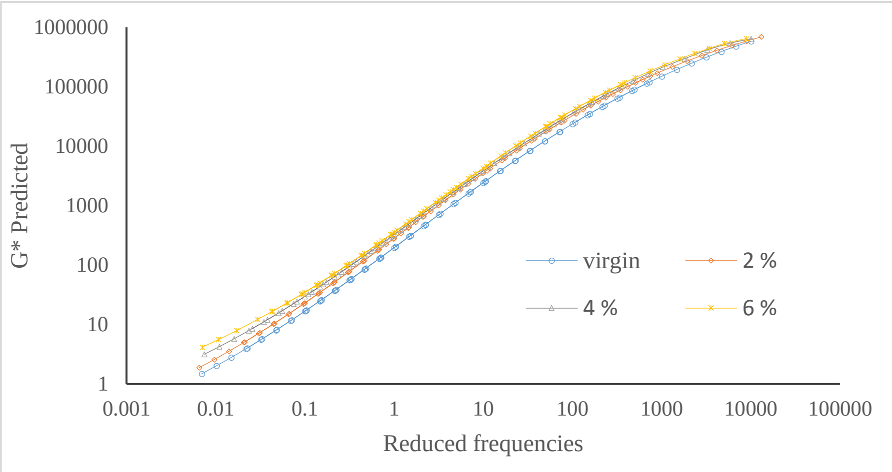

Figure 3 Relation between G* and reduced frequency.

4. Practical applications the work

In the regions where summer season prevails and during the summers temperature reaches to very extreme which increases the surface temperature of the pavement due to which pre mature failure of the pavement may occur in the following two way:

1. It may lead to bleeding of the binder which effects the skid resistance of the pavement badly and
2. It may lead to heavy rut potential due to high surface temperature which is favoured by heavy axle loading

So by using STF as modifier the above mentioned failure mechanism can be highly reduced which will help the local pavement industry to maintain good and durable road network.

5. Conclusions

The following conclusions are made based on the test results performed on STF-modified binder

- A good STF can be obtained by mixing of nano-silica and ethylene glycol in the proportion of 40:60 by weight
- From Storage stability test we can say that STF was properly dispersed in binder and has produced a stable modified binder
- The optimum dosage of 4% STF was selected, with the addition of this 4% STF the base binder of PG 52 was improved and modified to PG 64
- With the addition of 4% STF the Penetration value has decreased by 18% and increased the softening point by 15% as as the ductility was reduced by 12%.
- The complex shear modulus (G^*) of the binder enhanced meaningfully which means the elastic behaviour and stiffness of binder has been improved with the intrusion of STF in the binder
- STF promoted high temperature performance without degrading the low temperature response

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