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(Research Article)

Monitoring the Rheological Properties of Asphalt Cement after Digestion with Micro and Nano Size Additives

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Abstract

The Asphalt binder usually practices ageing throughout its service life due to the environment. The asphalt binder will practice changes in the rheological properties and loses the required flexibility, its adhesion to the aggregates and cohesion. Implication of Micro and Nano size additives can enhance the quality of the binder and extend its useful life. In the present investigation, two types of asphalt binder with penetration grade (40-50 and 60-70) were digested with micro size additive (Fly ash, and hydrated lime) and Nano size additive (Fumed silica and silica fumes). The treated binders were subjected to the rheological properties determination. It was observed that for (40-50) and (60-70) binders, the penetration viscosity number declines by (2.8, 0.3, 1.4, and 1.4) % and (1.7, 1.2, 2.1, and 0.6) % for (Fly ash, fumed silica, silica fumes, and hydrated lime) treated binder respectively. For (40-50) and (60-70) asphalt binder, the viscosity increases by (5.8, 33.4, 54.4, and 43.6) % and (102.5, 135.5, 112, and 12) % for (Fly ash, fumed silica, silica fumes, and hydrated lime) treated binder respectively. For (40-50) and (60-70) binders, the creep stiffness increases by (5, 25, 35, and 32.5) % and (6.2, 12.5, 18.7, and 25) % when fly ash, fumed silica, silica fumes, and hydrated lime were implemented. It can be revealed that implementation of Nano size additives exhibits higher creep stiffness and lower temperature susceptibility when compared with Micro size additives.

Keywords: Asphalt binder, Rheology, Susceptibility, Micro, Creep, additives, Nano material

1. Introduction

Adding Nano particles like Nano-clay, Nano-silica, and Nano tubes in asphalts normally increase the viscosity of asphalt binders as stated by Yanga and Tigheb, [1]. Various physical properties of asphalt cement such as stiffness and tensile strength, tensile modulus, flexural strength, and thermal stability can be enhanced when it is modified with small amounts of Nano-clay as revealed by Jahromi and Khodaii, [2]. Adding Bentonite clay and organically modified bentonite had significantly improved the rheological properties at low temperature and the resistance to cracking of asphalt as reported by Golestani et al., [3]. Hui et al., [4] stated that with the addition of Nano-silica to the asphalt binder, the viscosity values of Nano modified asphalt binder decreases slightly indicating that a lower compaction temperature or lower energy consumption of the construction process will be achieved. Raufi et al., [5] assessed the modification impact of three different types of Nano materials, including Nano-$CaCO_3$, Nano-Bentonite, and Zyco Therm, on the properties of asphalt binder. Conventional and rheological tests on asphalt binders were conducted to identify the influence of Nano modification. The research findings revealed that Nano material can potentially enhance the high-temperature susceptibility resistance, and rheological properties of asphalt binder samples. Bhat and Mir, [6] presents a laboratory assessment of the rheological and physical properties of Nano silica-modified asphalt binders. It was revealed that Nano silica can be produced from rice husk and silica fumes and therefore it is environment-friendly and cost-effective asphalt modifier. Three concentrations were utilized for each additive. The results of the study revealed that the incorporation of additives into the asphalt binder showed an improved elevated temperature performance and enhanced the resistance against fatigue cracking. Zafari et al., [7] investigated the merit of application of Nano-silica as an anti-ageing additive. Different percentages of Nano-silica were added to the asphalt binder. The modified Asphalt binder was then exposed to short-term ageing. It was concluded that Nano-silica can improve the ageing resistance of the asphalt binder and significantly increases the complex modulus and viscosity of the asphalt binder. This in turn can improve the rutting resistance of the pavement. Li et al., [8] stated that Nano material exhibits specific characteristics due to its nature of large surface area and small size, compared to the common material to be applied in the field of asphalt pavement as an additive. Some conventional test results including viscosity, dynamic modulus, and rheological properties were employed to

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characterize the performances of Nano-modified asphalts binder. It was concluded that the addition of Nano materials could dramatically enhance the properties of an asphalt material such as visco-elasticity, high temperature properties, and the resistances to aging. Shafabakhshet al., [9] revealed that the binder modification with Nano-SiO2 results in increased viscosity, reduced ductility, increased softening point, and increased elastic modulus. According to the study by Ashish et al., [10], the most known important Nano material that can improve the rheological and mechanical properties of binder in asphalt mixtures are SiO_2, TiO_2, and ZnO. According to recent study by Farag et al., [11], the addition of Nano-calcium oxide (CaO) in asphalt binder produces reduction of the penetration value by 7%, which directly relates to resistance to high temperature, and an increase of softening point's value by 45% while the resilience modulus value increased by 1.7 times that of control binder. Sarsam and AL-Azzawi, [12] revealed that the addition of Nano materials (fly ash, silica fumes, and phosogypsum powder) to asphalt cement at different percentages causes poor adhesiveness and poor wetting, and the behavior of the modified binder changes from hydrophilic to hydrophobic when Sessile Drop method was adopted. However, the change in contact angle was fluctuating and not significant when Wilhelmy technique was adopted. Golestani et al. [13] evaluated Performance of modified asphalts binder by Nano composite. The results showed that Nano composite could improve the physical properties, rheological behaviors, and the stability of the bitumen. The micro and Nano scaled materials (such as silica fume) have been found to improve the properties of the binder due to high surface area as mentioned by Khodary, [14].Yao et al., [15] investigated the applicability of carbon micro-fiber and Nano clay in asphalt binders and evaluated the performance of micro- and Nano-modified asphalt binders in terms of rheological properties. It was found that the dynamic shear modulus of asphalt binder can be improved by the selected micro- and nonmaterial. Yao et al., [16] evaluated the rheological properties and chemical bonding of Nano-modified asphalt binders blended with Nano silica. The Nano silica was added to the control asphalt at contents of 4% and 6% based on the weight of asphalt binders. It was found that implementation of Nano silica had a positive effect on anti oxidation, while it slightly decreased the viscosity of the control asphalt binder and maintained low dissipated work per load cycle. Sarsam, [17] digested asphalt cement with two types of Nano materials, (fly ash and silica fumes), and studied the possibilities of improving rheological, mechanical, and physical properties of the asphalt binder. It was found that Nano materials shows positive influence on asphalt cement by reducing its temperature susceptibility, while it exhibits variable effect on the viscosity of the binder, silica fumes increase the viscosity, while fly ash reduces the values. However, Silica fumes increases the softening point of the binder significantly, while fly ash increases it marginally. Sarsam, [18] prepared the modified Asphalt cement in the laboratory by adding Nano materials (silica fumes, and hydrated lime) to each of the two-penetration grade Asphalt cement (40-50 and 60-70). It was revealed that the softening point increased while the penetration decreased, and the control on the sensitivity to temperature (PI) could be achieved for both grades of asphalt cement. Lime had reduced the ductility, penetration, and had increased the elastic strain recovery, significantly, and increases the temperature susceptibility of the modified binder. The mechanism of the effect of Nano ZnO on the resistance of asphalt mixtures against moisture damage has been investigated by Hamedi et al., [19]. The results showed that the ratio of wet/dry values of indirect tensile strength for the mixtures containing Nano ZnO for two types of aggregate were higher than the control mixtures. Sarsam, [20] addressed the positive influence of Nano size additive (silica fumes) on the flexural strength of asphalt concrete. It was concluded that such additive may be implemented as a partial substitute of mineral filler in the asphalt concrete mixture to enhance its physical properties.

The aim of the present investigation is to assess the behavior of asphalt binder after digestion with Micro size additives (fly ash and hydrated lime) and Nano size additives (fumed silica and silica fumes) and evaluation of the changes in rheological properties of the modified binder.

2. Materials and Methods

The micro and Nano size additives are locally available; Figure 1 demonstrates the implemented additives.

Figure.1 The implemented Nano and micro size additives

2.1 Fumed silica: Fumed silica is produced by a vapor phase hydrolysis process using chlorosilanes such as silicon tetrachloride in a flame of hydrogen and oxygen. Fumed silica is supplied as a white, fluffy powder, and obtained from local market. It has a specific surface area of 200000 m2/kg, which is considered to have a Nano size. The physical properties of fumed silica are listed in Table 1. The Chemical compositions of fumed silica are listed in Table 2.

Table 1. Physical properties of fumed silica as supplied by the manufacturing plant

Physical properties	Test result
Specific surface area m²/kg	200000
Density kg/m³	202
SiO_2 %(when firing at 1000˚c for 2 hours)	> 99.8
Loss of weight% when drying at 1000˚c for 2 hours	< 2
Loss of weight% when drying at 105˚c for 2 hours	< 1.5
PH	4.1
% Retained on 40 µm sieve	< 0.04
Moisture %	0.82

Table 2. Chemical components of fumed silica

Oxide	Percent
SiO_2	99.1
Fe_2O_3	35.0 ppm
Al_2O_3	< 0.035
TiO_2	< 0.006
CaO	0.03
MgO	52.0 ppm
SO_3	< 0.07
Loss on ignition	0.7

2.2 Silica fumes: A grey-colored densified silica fumes is an ultra-fine powder which has a high content of amorphous silicon dioxide and consists of very fine spherical particles. The specific surface area was 100000 m2/Kg, which is considered to have a Nano size. It was obtained from local market. Table 3 exhibits the physical properties of Silica Fumes, while Table 4 presents its chemical composition.

Table 3. Physical properties of silica fumes

Property	Test value
Bulk specific gravity	2.134
Specific surface area (m^2/ Kg)	100000
PH value	4.5

Table 4. The chemical composition of silica fumes

Chemicals	Percent
SiO_2	90%
Al_2O_3	% 3
CaO	% 1.2
Fe_2O_3	% 1.0
MgO	< 6%
Loss on ignition	1%

2.3 Hydrated lime: Hydrated lime is a derivative of burnt lime. It is produced by reacting burnt lime with water in a continuous hydrator, during this process large amounts of heat are given off. Hydrated lime is light and fluffy and has 4404 m²/kg specific surface area which is considered to have a micro size. This material is obtained from local market. The physical properties of hydrated lime are listed in Table 5. The chemical composition of hydrated lime is listed in Table 6.

Table 5. The physical properties of hydrated lime

Chemical formula	Density gm/cm^3	Specific surface area m^2/kg	% Passing sieve no. 4	Melting point °C
$Ca(OH)_2$	2.211	4404	100	580

Table 6. Chemical components of hydrated lime

Oxide	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Loss on ignition
Percent	0.74	0.19	0.5	64.23	1.17	29.94

2.4 Fly ash: The fly ash class F was obtained as a by-product of coal combustion from local market, Table 7 demonstrates its physical properties. This fly ash has specific surface area of600 m²/Kg which is considered to have a micro size. The Chemical components of fly ash are listed in Table 8.

Table 7. Physical properties of fly ash

Maximum sieve size (mm)	% Passing	Specific gravity	Specific surface area (m^2/ kg)
0.075	98	2.016	600

Table 8. Chemical components of fly ash

Chemicals	Percent	ASTM requirement C618, [21] %
SiO_2	61.95	---------
Fe_2O_3	2.67	---------
Al_2O_3	28.82	---------
SiO_2+ Fe_2O_3+ Al_2O_3	93.44	70.0 min.
Na_2O	0.26	1.5 max.
CaO	0.88	----------
MgO	0.34	5.0 max
SO_3	< 0.07	5.0 max
Loss on ignition	0.86	6.0 max

2.5 Asphalt cement binder: Two types of Asphalt cement binder of penetration grade (40-50) and (60-70) were obtained from Dourah oil refinery. Such types of binder are usually implemented in asphalt pavement construction in Iraq. Table 9 presents the rheological properties of asphalt binders. Table 10 demonstrates the physical properties of the asphalt binders.

Table 9. Physical properties of asphalt binders

Property	ASTM, [21] specification	Asphalt cement (40-50)	Asphalt cement (60-70)
		Test results	Test results
Penetration at 25˚C,100gm,5sec, (0.1mm)	D-5	44	66
Softening Point, ˚C	D-36	50	48
Ductility at 25 ˚C, 5cm/min,(cm)	D-113	+100	+100
Resilience (ball strain recovery) %	D-5329	98	99
Specific gravity	D-70	1.042	1.030
After thin film oven test			
Penetration at 25°C,100gm,5sec, (0.1mm)	D-5	33	51
Loss in weight	D-1754	0.17	0.24
Ductility at 25 °C, 5cm/min,(cm)	D-113	73	90
Softening point, ˚C	D-36	54	51

Table 10. Rheological properties of asphalt cement binders

Asphalt cement type	Penetration grade (40-50)	Penetration grade (60-70)
Penetration-index (PI)	-1.474	-1.370
Penetration viscosity number (PVN)	6.09	6.204
Temperature of equivalent stiffness (TES)	-22	-25
Viscosity (poises)	3690	2123
Stiffness modulus (N/m²)	2000 x10^3	1000×10^3

2.6 Preparation of modified asphalt binder: Modified asphalt binder was prepared by using the wet process. In the wet process, asphalt cement was heated to a 150 °C and then blended with the micro or Nano size additives with the specified percentage. The blending was conducted at a blending speed of 1300 rpm and constant for 30 minutes to promote the possible chemical and physical bonding of the components. During the blending process, the asphalt cement swells or softens and exhibit bubbles as an indication of possible reaction. The degree of dispersion of the Micro and Nano additives was controlled by the mixing temperature, blending duration, and blending speed. Similar precautions were followed by Li et al., [8]. Various percentages of Micro or Nano additives have been implemented. The range of Micro size additives was (5-20) % with 3 % increment, while the range of Nano additives was (1-5) % with 1 % increment. The optimum additive content was selected based on the significant change in physical and rheological properties of the modified binder. Details of mixing and obtaining the optimum additive could be referred to Sarsam, [22]; Sarsam, [23]; Sarsam and Lafta, [24], and Sarsam, [25]. The summary of the optimum additive content for Micro and Nano size is listed in Table 11.

Table 11. Optimum additives content

Micro size additive		Nano size additive	
Fly ash	Hydrated lime	Silica fumes	Fumed silica
5 %	10 %	2 %	3 %

2.7 Testing of modified binder: The prepared asphalt binder samples were subjected to some of the traditional physical properties' determination such as (Cone penetration, ring and ball softening point, resilience %) and the rheological properties determination such as (Penetration index PI, penetration viscosity number PVN, viscosity, creep stiffness, and predicted stiffness modulus of asphalt mixture SM). The various testing follows the recommended testing procedures by ASTM, [21], AASHTO, [26], Shell Nomograph, [27], and other procedures recommended by the literature.

3. Results and Discussions

3.1 Influence of additives on temperature susceptibility parameters of the binder: In the present investigation, the temperature susceptibility was determined based on the penetration index (PI) and penetration viscosity number (PVN). Norhidayah et al., [28] revealed that the penetration index, PI, is a measure of the temperature susceptibility of asphalt binder that can be derived mathematically from the penetration and softening point test values. Temperature susceptibility is the rate at which the consistency of asphalt cement changes with a change in temperature. Asphalt binder with penetration index (PI) above (+2) has less temperature susceptibility and is less brittle at low temperature. However, asphalt binder with PI value between (-2) and (+2) exhibits normal susceptibility to temperature. In addition, the negative sign of (PI) means that asphalt cement is more susceptible to temperature. Figure 2 demonstrates the PI values of the modified asphalt binder; it can be noted that the Nano additives and hydrated lime exhibits significant reduction in the PI values regardless of the binder type, however, the fly ash additive shows higher negative values of PI indicating more susceptibility to the temperature variation. It can be revealed that the Nano additives and lime can improve the resistance of the binder to the variation in temperature. For (40-50) binder, the penetration index declines by (25.7, 36.8, and 25.3) % when fumed silica, silica fumes, and hydrated lime were implemented as compared with the control binder while the PI values increases by 72.4 % when fly ash was implemented. On the other hand, for (60-70) binder, the penetration index declines by (21.5, and 27.5) % when fumed silica, and silica fumes, were implemented respectively as compared with the control binder while the PI values increases by (3, and 72.9) % when hydrated lime and fly ash were implemented respectively.

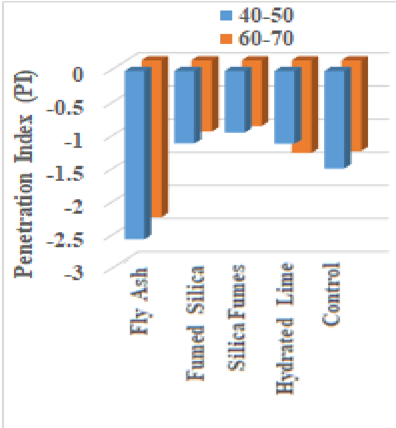

Figure 2. Influence of micro and Nano additives on penetration index of asphalt binder

The penetration viscosity number PVN as demonstrated in Figure 3 is based on penetration at 25° C and viscosity at 135° C which are usually specified as a requirement for paving asphalt binders. The lower the PVN values of an asphalt binder, the higher its temperature susceptibility. Most asphalt binders have a PVN between + 0.5 and – 2 as revealed by Gatoto et al., [29]. It can be noticed from Figure 2 that the PVN values declines after implementation of additives regardless of the binder or additive type. This can indicate an increment in the temperature susceptibility of the binder after digestion with Micro and Nano additives.

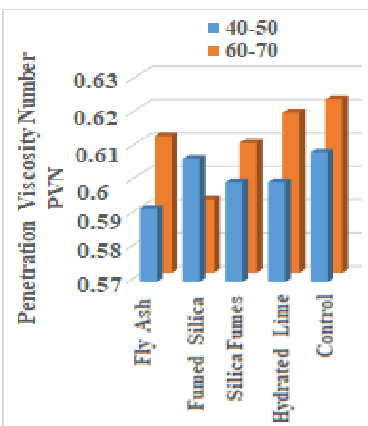

Figure 3. Influence of micro and Nano additives on penetration viscosity number of asphalt binder

For (40-50) and (60-70) binders, the PVN declines by (2.8, 0.3, 1.4, and 1.4) % and (1.7, 1.2, 2.1, and 0.6) % for (Fly ash, fumed silica, silica fumes, and hydrated lime) treated binder respectively. Lower values of PI and PVN indicate higher temperature susceptibility, and asphalt concrete mixtures containing binders with lower temperature susceptibility are expected be more resistant to cracking and rutting as stated by Fazaeli et al., [30]. One notable difference between PI and PVN is that the PI change due to aging process (during mixing and subsequently in service), whereas the PVN value remains substantially the same.

The viscosity of the control binder as exhibited in Figure 4 increases after digestion with Micro and Nano additives regardless of the additive or binder type.

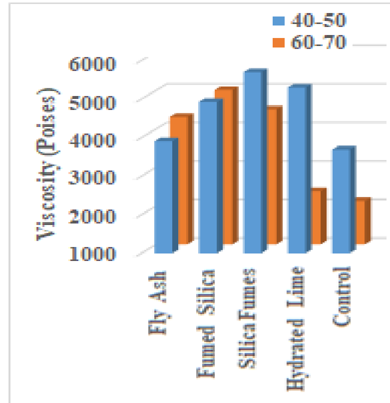

Figure 4. Influence of micro and Nano additives on the viscosity of asphalt binder

This may be attributed to the physical and possible chemical reaction between the additives and the components of the binder. It can be noticed that the Nano additives exhibit the highest viscosity as compared with the Micro size additives. This could be related to the high specific surface area of Nano additives. For (40-50) and (60-70) asphalt binder, the viscosity increases by (5.8, 33.4, 54.4, and 43.6) % and (102.5, 135.5, 112, and 12) % for (Fly ash, fumed silica, silica fumes, and hydrated lime) treated binder respectively. Such behavior agrees with Khairuddin et al., [31].

3.2 Influence of additives on creep stiffness of the binder: Creep stiffness is a measure of the impact of thermal stresses in the asphalt binder resulting from thermal contraction. If these stresses are too great, cracking will occur. Higher creep stiffness value indicates higher thermal stresses. To determine the stress relaxation properties of an asphalt binder, creep stiffness calculations are made at 8, 15, 30, 60, 120 and 240 seconds of loading with the aid of bending beam rheometer BBR. These loading periods were chosen because they are about equally spaced on a logarithmic time scale. For each time, the asphalt binder creep stiffness is calculated and plotted. The creep stiffness measured at 60 seconds should be equal or less than 300 MPa for normal asphalt binder as demonstrated in Figure 5, the creep stiffness declines with the loading period regardless of the binder or additives type. For (40-50) binder type, implementation of Micro and Nano additives exhibit an increment in the creep stiffness as compared with the control binder. A sharp reduction in the creep stiffness could be noticed after the start of loading while the rate of reduction changes to gentle after 120 seconds.

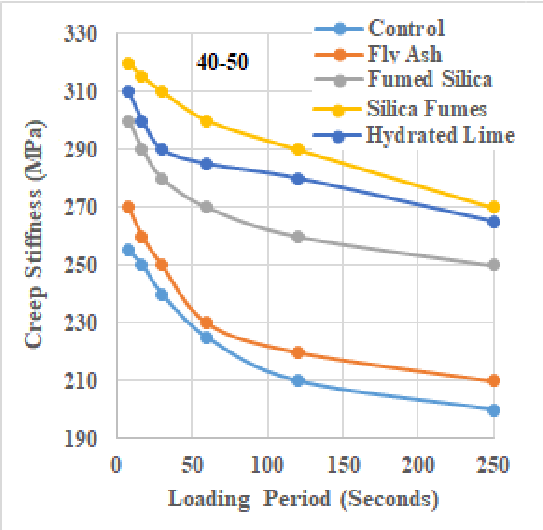

Figure 5. Influence of micro and Nano additives on creep stiffness of (40-50) asphalt binder

The creep stiffness increases by (5, 25, 35, and 32.5) % when fly ash, fumed silica, silica fumes, and hydrated lime were implemented. For 60-70 binder type, Figure 6 exhibits the influence of implementation of Micro and Nano additives on the creep stiffness of the binder. An increment in the creep stiffness could be detected as compared with the control binder. A sharp reduction in the creep stiffness could be noticed after the start of loading while the rate of reduction changes to gentle after 120 seconds. The creep stiffness increases by (6.2, 12.5, 18.7, and 25) % when fly ash, fumed silica, silica fumes, and hydrated lime were implemented. It can be revealed that implementation of Nano additives exhibits higher creep stiffness when compared with Micro size additives or with the control binder. Similar findings were reported by Sarsam and Lafta, [24].

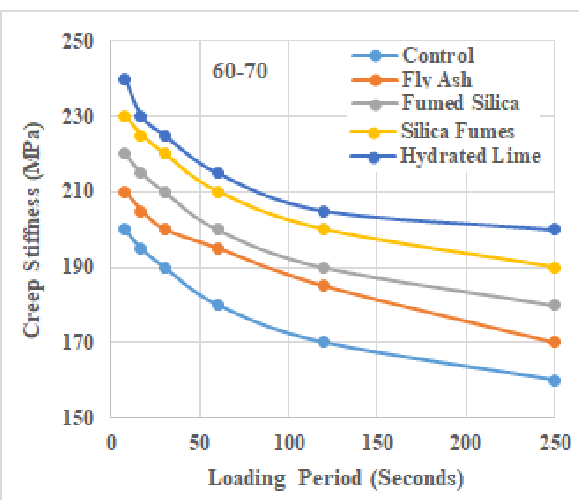

Figure 6. Influence of micro and nano additives on creep stiffness of (60-70) asphalt binder

3.3 Influence of additives on temperature of equivalent stiffness TES: The temperature of equivalent stiffness TES is the temperature at which the stiffness of asphalt is 138 MPa at 2.77 hours of loading time. Figure 7 demonstrates the influence of Micro and Nano additives on TES of asphalt binder. It can be detected that digestion of the binders with the additives except hydrated lime exhibits an increase in the TES. For (40-50) binder type, the temperature of equivalent stiffness declines by (27.2, 9, and 4.5) %when fly ash, fumed silica, and silica fumes were implemented respectively. However, the TES increases by 4.5 % when hydrated lime was introduced as compared with the control binder. For (60-70) binder type, the temperature of equivalent stiffness declines by (20, 40, and 48) %when fly ash, fumed silica, and silica fumes were implemented respectively. However, the TES increases by 8 % when hydrated lime was introduced as compared with the control binder. Such behavior agrees with Sarsam, [17].

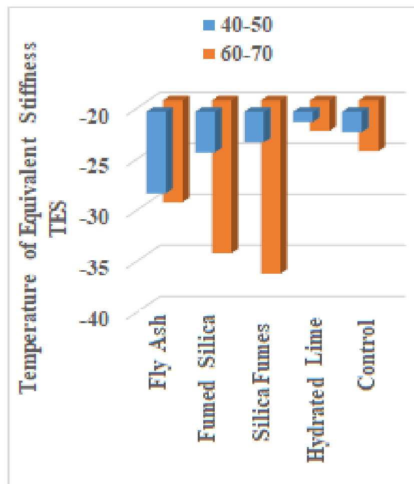

Figure 7. Influence of micro and Nano additives on TES of asphalt binder

4. Conclusions

Based on the testing conducted and the limitations of materials, the following conclusions could be addressed.

- For (40-50) and (60-70) binders, the penetration viscosity number declines by (2.8, 0.3, 1.4, and 1.4) % and (1.7, 1.2, 2.1, and 0.6) % for (Fly ash, fumed silica, silica fumes, and hydrated lime) treated binder respectively.
- For (40-50) and (60-70) binders, the creep stiffness increases by (5, 25, 35, and 32.5) % and (6.2, 12.5, 18.7, and 25) % when fly ash, fumed silica, silica fumes, and hydrated lime were implemented.
- It can be revealed that implementation of Nano size additives exhibits higher creep stiffness and lower temperature susceptibility when compared with Micro size additives.

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