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Impact of Nano Materials on Rheological and Physical Properties of Asphalt Cement

Saad Issa Sarsam*

Transportation Engineering, Department of Civil Engineering, College of Engineering, University of Baghdad, Baghdad, Iraq

Abstract

In this work, an attempt has been made to prepare the modified Asphalt cement for pavement construction 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). Two different percentages of each of the above-mentioned additives have been tried using continuous stirring and heating at 150 -160 ºC for 45 minutes .The silica fumes percentages were at the range of (1-2) %, with 1 %increments, the hydrated lime was at the range of (10-20)% , with 10 increments. The prepared modified Asphalt cement specimens were subjected to physical properties determination; the penetration, softening point, ductility, elastic strain recovery, and rotational viscosity. The rheological properties of the modified Asphalt cement such as penetration index, temperature of equivalent stiffness, stiffness and penetration viscosity number were calculated. The effect of each ingredient of the modified asphalt cement on physical, rheological, and elastic properties have been studied and discussed. It was concluded that the silica fumes and lime have beneficial effects on the overall properties of the modified Asphalt cement.

Keywords

Asphalt Rheology, Modified Asphalt Cement, Silica Fumes, Lime

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1. Introduction

Modified asphalt is a mixture of asphalt cement, and additives which form a cohesive impermeable mass, solid or semisolid under normal temperature conditions, but sufficiently fluid when brought to a suitable temperature to be spread by means of hand float or by mechanical means without compaction [1].

The additive material may consist of cement, lime, or sulphur; all are in a form of very fine powder (nano size). The additives may also consist mainly of polymers for modification of asphalt cement and other modifiers [2].

All of these materials are combined chemically and physically to compose a material suitable for roofing or pavement joint sealing, and paving. Modified Asphalt should be impervious to water, capable of lasting for the design life of the pavement, and when it is fully confined; it has the same compressive strength as the containing material. It should be elastic enough to resist permanent deformation, have low temperature fracture toughness, and have enough stiffness to prevent drain down; British standard institute.

2. Criteria for Evaluation and Testing Modified Asphalt

When trying to introduce alternative materials (nano materials) in composition with asphalt cement for application in joint sealing or paving, challenges such as inadequate testing and evaluation methods, varying quality must be addressed. Sometimes, acceptable criteria are defined in a standard, but the challenges still arise for which parameter to measure, how to measure them, and what levels of functioning are acceptable. It was felt that modified asphalt may have independent test certificates,[3]. In Iraq, the experience of

* Corresponding author
E-mail address: saadisasarsam@coeng.uobaghdad.edu.iq

using the modified asphalt is usually scarce, and the follow up of field test is often insufficient. Testing and evaluation may be inadequate; it may be difficult to maintain a constant quality when manufacturing the modified asphalt in small plant manually as in the present case in the country.

3. Previous Work

Considerable work has been conducted to measure fundamental properties of Modified Asphalt, but little work has been reported to explain the complicated rheological and physical Behavior [4]; [5]. The effect of hydrated lime as an active filler has been studied by [6]; [7], they concluded that it strongly affects high temperature rheology in certain asphalt, but less in others. The effect of Modified asphalt composition on its mechanical properties was investigated by [1 while the effect of scrap tire rubber and used oil on Asphalt rheology properties has been investigated by [8], they noticed the change in asphalt stiffness, and overall physical properties. [9] studied the effect of additives such as fly ash, fumed silica on asphalt cement properties, it was concluded that when Fumed Silica was implemented as additives to asphalt cement, it was noticed that penetration decreased, softening point increased and the control on the sensitivity to temperature (PI) could be achieved. On the other hand, fly ash improved the elastic properties of 40-50 asphalt cement.

The developments of new testing methods and equipment for better understanding of Modified Asphalt properties were reported by many research works; [10] introduced the tensile creep test to evaluate the elastic properties of asphalt, and the cold bond test to evaluate asphalt cohesion and adhesion at 0ºC using nonstandard testing procedure developed by them. [11] developed the elastic recovery test using modified type of ductility test mold and procedure, the test was considered as standard for Modified asphalt in Oklahoma. [12] performed tensile elongation test on rectangular specimens of Mastic at constant rate of elongation. Some of the above-mentioned new testing methods were incorporated in this work.

4. Materials Characteristics

4.1. Asphalt Cement

Two type of penetration grade asphalt cement were considered, (40-50) and (60-70). The hard type (40-50) was obtained from the Dora refinery. The soft type (60-70) was obtained from Baji refinery. Table 1 shows their physical properties, while table 2 presents the rheological properties.

Table 1. Physical properties of Asphalt cement implemented in the study

Property	Asphalt cement (40-50)		Asphalt cement (60-70)	
	Before aging	After aging	Before aging	After aging
Penetration	44	28	66	51
Softening point	50	54	48	51
Ductility	>100	60	>100	75

Table 2. Rheological Properties of Asphalt cement

Asphalt cement	Penetration-index (PI)	Penetration viscosity number (PVN)	Temperature of equivalent stiffness (TEES)	Viscosity (poises)	Stiffness modulus (N/m^2)
40-50	-1.474	6.09	-22	3690	$1000x10^3$
60-70	-1.27	6.204	-25	2123	5000×10^3

4.2. Hydrated Lime

Table 3. Properties of Hydrated lime

Property	Test result
Specific gravity	2.5
Specific surface area	4404
Percent finer than 75microns	98

It was obtained from Karbala plant in powder form; Table 3 presents its properties. The portion passing sieve 75 microns was implemented in this investigation.

4.3. Silica Fumes

A vapour phase hydrolysis process using chlorosilanes produces silica fumes; it was obtained from local market, some physical and chemical properties are given in Table 4.

Table 4. Physical Properties of Silica Fumes

Maximum sieve size	PH value	Density [kg/m3]	Specific surface area [m2/ kg]
Passing 75, retained on 40 µm	4.5	202	100000

5. Phasing of the Testing Program

The study was concentrated on three main goals, and the testing program was divided into three phases. In the first phase, a total of four modified asphalt mixes have been prepared in the laboratory by the addition of various percentages of lime, and silica fumes.

In the second phase, evaluation of the effect of each of the additives on rheological and physical properties of the produced modified asphalt took place, the cone and needle penetration, softening point, bending beam, and ductility test were conducted. Then the rheological parameters such as penetration index, penetration viscosity number, temperature of equivalent stiffness, viscosity and stiffness modulus were determined using shell nomograph, [13]. In the third phase, the elastic properties of the Modified asphalt such as resilience, elastic recovery, and adhesion and cohesion properties in cold bond test were determined.

The above testing plan was based on the literature survey conducted; it shows a combination of standard physical test on asphalt and recommended test on Mastic by [14]; [15]; [16]; [17].

5.1. Preparation of Modified Asphalt

The asphalt cement was heated in an oven to160^0 C for asphalt cement (40-50), 150°C for asphalt cement (60-70), then, asphalt cement of each type was poured into the containers of 50gm capacity, and positioned over hot plate to maintain the temperature at (150 – 160) ±2. The required amount of Nano material was introduced gradually with continuous mechanical stirring. The stirring process was continued inside the oven for [60 minutes] at the same mixing temperature of [150-160 ± 2 ˚C] to insure homogeneity of the mix. The selection of mixing time and temperature was based on many trials to insure proper distribution of Nano material inside the asphalt cement, and to prevent the possibility of any agglomeration. The stirring process was continued for 45 minutes as a constant blending time. Five percentage of the silica Fumes (1%, to 5%) and of lime (5%-25%) by weight of asphalt cement (40-50) and (60-70) have been introduced based on previous work by [2]. The produced modified asphalt cement was poured inside the testing mold for further testing. Specimens were subjected to physical properties determination before and after aging process using thin film oven test. The various types of the prepared and tested modified asphalt specimens were illustrated in Table 5.

Table 5. Modified Asphalt types prepared and tested

Designation	Ingredient composition
A	Asphalt + 1% Silica fumes
B	Asphalt + 2 % Silica fumes
C	Asphalt +10% Hydrated Lime
D	Asphalt +20% Hydrated Lime

5.2. Testing of Modified Asphalt Specimens

The Modified asphalt specimens were subjected to various physical tests to evaluate elastic, rheological and physical properties, duplicate specimens were prepared and the average values were considered for analysis. Table 6 illustrates various tests conducted on the prepared specimens. The standard test procedures were conducted as per [16]; [17], while, the elastic strain recovery and the cold bond adhesion and cohesion test were conducted as per the procedure by their inventors. Figure 1 shows the elastic strain-recovery test mold, while Figure 2 shows the cold bond test casting-mold.

Table 6. Various tests conducted on Modified asphalt specimens

Designation Symbol	Test	Specifications
T-1	Softening point °C	ASTM D36-83
T-2	Ductility cm	ASTM D491-83
T-3	Penetration by needle	ASTM D5-78
T-4	Penetration by cone	ASTM D3408-78
T-5	Resilience (ball strain recovery) %	ASTM D3407-78
T-6	Oklahoma elastic strain recovery %	OKDOT L-42
T-7	Flow mm	ASTM D3405-78
T-8	Cold bond adhesion and cohesion at 0 °C	Huff & Vallerga-1981

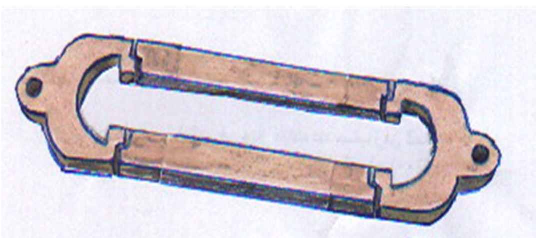

Fig. 1. Elastic strain recovery test casting mold

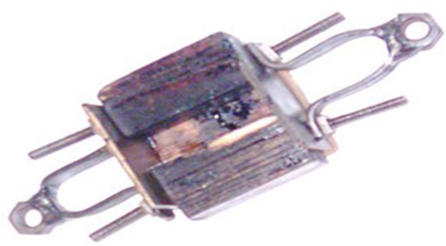

Fig. 2. Cold bond adhesion and cohesion test casting mold

5.2.1. Penetration Test

The penetration test, [17] is an empirical measure of asphalt consistency. In this test, a container of asphalt cement is placed at the standard test temperature (25°C) in a temperature-controlled water bath. A prescribed needle, weighted to 100 grams, is placed on the surface of the asphalt cement, then, allowed to penetrate for 5 seconds. The depth of penetration, expressed in units of 0.1mm, is considered the "penetration" of the asphalt cement.

5.2.2. Softening Point Test

The softening point test, [17] is also used to measure asphalt consistency. The test is performed by confining asphalt samples in brass rings and loading the samples with steel balls. The samples are placed in a beaker of water at a specified height above a metal plate. They are then heated at a specified rate. As the asphalt heats, the weight of the steel ball pushes the sample down toward the plate. When the sample and ball touch the plate, the water temperature is measured, and designated as the (ring and ball) softening point of the asphalt.

5.2.3. Ductility Test

The ductility of a asphalt cement can be defined as the "distance to which it will elongate before breaking when two ends of a briquette specimen of the material, are pulled apart at a specified speed (5cm/min ±5.0%) and at a specified temperature (25±0.50C) [17]. This test method provides measure of tensile properties of bituminous materials and may be used to measure ductility for specific requirements. Ductility is an indicator of flexible Behavior of asphalt under various temperatures.

5.2.4. Thin Film Oven Test

Physical properties of asphalt cement changes with respect to time and temperature. Consequently, the performance of pavement will also witness some changes. To take into account the effects of mixing and compaction temperatures as well as the storage time on the Behavior of asphalt cement and asphalt mixture. All the asphalt samples have been exposed to accelerated aging by heating the samples in an oven for 5 hours at 163°C, [17]. Ductility, Penetration and softening point after thin film oven test have been determined for asphalt cement for all percentage of modifiers that will be used.

5.2.5. Resilience Test

This test was conducted using the standard penetrometer described in Test Method D 5, after replacing the needle by standard penetrometer with a ball penetration Tool [17]. The total weight of the ball penetration tool and penetrometer spindle was 75± 0.01 g .Figure 3 present resilience test.

5.2.6. Cold Bond Adhesion and Cohesion Test

The cold bond test was used to evaluate modified asphalt cohesion and adhesion at 0ºC using ductility device. The apparatus was manufactured at local market, and used for testing. The two sides of the mold are pulled apart at a specified speed (5cm/min ±5 %) [17]; [18].

5.2.7. Cone Penetration, Softening Point and Rheological Properties of Modified Asphalt Cement

The physical properties of asphalt cement changes with respect to addition of modifiers, time and temperature. All the modified asphalt cement samples have been exposed to the penetration, and softening point test. The rheological properties such as penetration viscosity number (PVN), penetration index (PI), and stiffness modulus (SM) have been calculated using the mathematical models and shell nomograph. Table 7 shows the Rheological properties of Modified Asphalt cement.

Figure 3. Resilience test

Table 7. Rheological properties of prepared Modified asphalt

Type of additives	Asphalt cement (40-50)					Asphalt cement (60-70)				
	PI	PVN	TES	SM (N/M^2)	Viscosity poises	PI	PVN	TES	SM (N/M^2)	Viscosity poises
1% silica fumes	-1.443	5.92	23	$1.9x10^6$	8027	0.074	6.17	-35	$1x10^6$	3000
2% silica fumes	-0.931	6.00	24	$1x10^6$	12791	-0.993	6.09	-37	$3x10^6$	4500
10% lime	0.10	6.00	21	$4 x 10^6$	5300	-1.4	6.18	-23	$2 x 10^6$	2379
20% lime	0.03	5.94	20	$4.5 x 10^6$	5470	-2.1	6.12	-21	$5 x 10^6$	2452

6. Analysis and Discussion on Test Results

6.1. Determination of Temperature Susceptibility Parameter

Tables 7 and 8 shows that when Silica fumes was implemented as additives to both types of asphalt cement, it was noticed that penetration decreased, softening point increased and control on the sensitivity to temperature (PI) could be achieved. This may be attributed to the high surface area of silica fumes (100000 m^2/g), and the effect of silica that will reach to (99%) from fumed silica content which could lead to covered grains of silica fumes with asphalt cement and thus control the viscosity of the mixture.

Table 8 illustrates the results of cold bond test for both asphalt cement grades with and without nano additives. The cold bond value of asphalt cement without additives was (<12.7mm), the value of cold bond for all percentage of additives with asphalt cement were (<12.7mm), this was referred as failed in the table. Specimens were failed either in adhesion as the case in silica fume, or in cohesion as the case of lime. Such result correlates with [2]; [19].

As demonstrated in Table 8, the addition of lime, and doubling the lime content, had further increases the elastic strain recovery, and significantly increases the temperature susceptibility of the mix, and makes it more brittle at low temperatures. This may be attributed to the increase in viscosity. It also increases the stiffness of the mastic many folds as shown in Table 7, this was in agreement with [20] findings.

6.2. Effect of Nano Additives on Cone Penetration

Table 8 indicates the results of cone penetration of asphalt cement with and without additives. The cone penetration was 38 for asphalt cement grade 40-50, and 58 for asphalt cement grade 60-70. Two types of nano additives were used with two different percentages for each one. When Silica fumes was added to the asphalt cement, it was noticed that the cone penetration decreases to (20) and 45 respectively for both types of asphalt cement. With further increment of nano additives percentage, the cone penetration decreases. Such Behavior may be attributed to the change of viscosity when adding a nano material, which may practice some chemical reaction with asphalt cement. Silica fumes may practice physical reaction only and disperse in the asphalt cement structure and causes the asphalt to become thicker and start losing its elasticity. Such result correlates with [2].

Table 8. Physical properties of Modified Asphalt cement

Additives Type	Asphalt cement 40-50				Asphalt cement 60-70			
	Cone penetration	Softening point	Resilience %	Cold bond	Cone penetration	Softening point	Resilience %	Cold bond
Control	38	50	91	fail	58	48	93	fail
1% silica fumes	32	55	89	fail	45	49	92	fail
2% silica fumes	30	52	84	fail	37	52	90	fail
10% Lime	34	51	92	fail	55	50	94	fail
20% Lime	30	52	94	fail	49	51	95	fail

6.3. Effect of Nano Additives on Softening Point and Resilience

Table 8 indicates the results of softening point and resilience of asphalt cement with and without nano additives. The softening point value of asphalt cement grade 40-50 without additives was 50°C; when silica fumes and lime were added to the asphalt cement, the softening point was increased to 55°C and 51°C respectively. The softening value of asphalt cement grade 60-70 without additives was 48°C; when silica fumes was added to the asphalt cement, the softening point was increased to 49°C. It shows that it had no significant effect on softening point increment as lime additive increases.

Resilience was 91% and 93% for both types of asphalt without additives and increased when lime was introduced. This could indicate the improvement in elastic properties of asphalt cement due to its possible chemical reaction with such additives. When silica fumes was added to both types of asphalt cement, Resilience was decreased to 89% and 92% respectively. Further increase in such additive percentages shows further reduction in resilience. This may be attributed to the limited physical reaction of this additive with asphalt and high viscosity of the resulted binder. Such Behavior agrees well with [21] work.

6.4. Impact of Nano Additives on Aging Behavior

The impacts of nano additives on physical properties of asphalt cement before and after aging are illustrated in table 9. The asphalt cement of grade 40-50 has retained 40% of its ductility after aging; the softening point was increased by 8%, and the penetration was decreased by 36%. When nano additives were introduced, their impact was variable; the ductility was reduced by a range of (10-60) % based on additive type and percentage. Silica fumes have moderate effect on ductility when it reduces it by 35%. The softening point increases after aging by a range of 6-8% when different percentages and type of nano additives were introduced. The penetration value shows variations by a range of 20-60% based on nano additive type and percentage.

Table 9. Impact of nano additives on aging properties of modified asphalt cement

Nano additive	Asphalt cement 40-50						Asphalt cement 60-70					
	Penetration		Softening point		Ductility		Penetration		Softening point		Ductility	
	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After
1% silica fumes	25	33	53	56	19.5	8	63	52	48	50	27	19
2% silica fumes	32	25	53.5	58	21	19	57	41	54	55	25	16
10% lime	35	28	52	55	85	70	60	49	49	52	110	100
20% lime	30	25	53	56	74	68	55	42	52	55	85	60

On the other hand, the asphalt cement of grade 60-70 exhibit 22% reduction in penetration, 6% increment in softening point and 25% reduction in ductility due to aging. When additives were introduced, silica fumes shows high impact on ductility in the range of 30-50%.

The impact of additives on softening point was in a range of 3-6% for various percentages and type of additives. Silica fumes shows higher impact within a range of 17-30%.

Such behaviour of additives may be attributed to the increase in viscosity due to high specific surface area of silica fumes, and to possible chemical reaction took place in case of lime.

7. Conclusions

Based on the limited testing program, the following conclusions may be drawn:

1. When Silica fumes or hydrated lime were implemented as nano additives to asphalt cement, it was noticed that penetration decreased, softening point increased and the control on the sensitivity to temperature (PI) could be achieved for both grades of asphalt cement.

2- Modified asphalt should be subjected to a combination of testing procedures as those conducted in this work to evaluate the suitability of the mastic for joint sealing, paving or roofing.

3- Lime had reduced the penetration, ductility and had no significant effect on softening point, but doubling the lime content had increased the elastic strain recovery, significantly decreases the ductility, and increases the temperature susceptibility of the mastic.

4- After aging process, for both grades of asphalt, the ductility was reduced by a range of 10-60 % based on additive type and percentage, the softening point increases after aging by a range of 6-8% and the penetration value shows variations by a range of 20-60% based on nano additive type and percentage.

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