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Study the Effect of Nano-Kaolinite on the Properties of Bitumen and Asphalt Mixtures

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ملخص البحث

أصبحت تكنولوجيا النانو من أهم المجالات التي اقتحمت كثيراً من المجالات منها الفيزياء والكمياء والهندسة والطب وغيرها. أيضا قام الباحثين في مجال الأسفلت باستخدام هذه التكنولوجيا في تحسين خواص الاسفلت والخلطات الاسفلتية. في هذا البحث تم إستخدام الكاولين كمحسن لخواص الأسفلت والخلطة الأسفلتية بعد تحويله الي حجم النانو باستخدام الطريقة الميكانيكية عن طريق تكسيره بجهاز الطحن ذو الكرات (Ball Milling) لتحويل الكاولين الي جزيئات يتراوح قطرها بين 15 نانو متر الي 50 نانو متر. تم خلط الاسفلت بنسب محددة من النانو كاولين (Nano-Kaolinite) هذه النسب هي 2٪ و4٪ و6٪ و8٪ من وزن الاسفلت ودراسة تأثير إضافة النانو كاولين بهذه النسب على خصائص الاسفلت المستخدم في الخلطات الاسفلتية. إتضح من التجارب المعملية أن القيمة المثلي لإضافة النانو كاولين هي 4٪ من وزن الاسفلت. أظهرت النتائج تحسن ملحوظ في خواص الاسفلت حيث تقل قيمة الاختراق مع زيادة نسبة النانو كاولين. أيضا زادت درجة التطرية مع زيادة نسبة النانو كاولين. أيضا زادت قيمة اللزوجة الكينماتيكية عند درجة حرارة 135 درجة مئوية مع زيادة نسبة النانو كاولين بالبيتومين المستخدم. ولتحديد تاثير النانو كاولين علي الخلطات الاسفلتية تم تصميم الخلطة الاسفلتية وتحديد النسبة المثلي للبيتومين بالخلطة. تم استعمال البيتومين الغير معالج والبيتومين المعالج بالنانو كاولين بنسب 2٪ و 4٪ و6٪ و 8٪ من وزن البيتومين المستخدم بالخلطة. تم تشكيل خمسة عينات من كل نسبة لاخذ المتوسط وتحديد قيمة الثبات والانسياب باستخدام جهاز مارشال. وجد قيمه الثبات تزداد بزيادة نسبة النانو كاولين بالبيتومين. ولإختبار سلوك الخلطة الأسفلتيه على المدى الطويل كان من الضرورى إختبار الخلطة الأسفلتية بجهاز الشد الغير مباشر. ولذلك تم عمل ثلاثون عينه باستخدام الاسفلت الغير محسن والاسفلت المحسن بالنسبة المثلي من النانو كاولين وتحديد قيمة اجهاد الشد الغير مباشر عند ثلاث درجات حرارة مختلفة هي 5م و 25م و 40م ووجد ان قيمة اجهاد الشد الغير مباشر زادت بنسبة 19.3٪ و 21.9٪ و 18.5٪ عند درجة حرارة 5م و 25م و 40م علي التوالي.

1. ABSTRACT

The Nanotechnology has become one of the most important and exciting forefront fields in Physics, Chemistry, Engineering, and Biology. Also, many researchers have tried to use Nanotechnology to improve the properties of bitumen and asphalt mixtures. In this investigation, a new application of Kaolinite will be used as an additive to improve the properties of bitumen. The methodology of this work is based on converting the Kaolinite to Nano size using a mechanical method. The Ball Milling machine was used to convert Kaolinite to Nano size. The product Nano-Kaolinite particle size was in the range of 15 to 50 nm. The Nano-Kaolinite was added to bitumen with percentages 2%, 4%, 6% and 8% by the weight of bitumen. The effect of the Nano-Kaolinite was studied with these percentages on the properties of bitumen that was used in the asphalt mixtures. The results indicated that the penetration of modified bitumen decreased with increasing the percentage of Nano-Kaolinite. The softening point and kinematic viscosity increased with increasing the percentage of Nano-Kaolinite. Also, the results indicated that the optimum Nano-Kaolinite content was 4% by the weight of bitumen. To determine the effect of Nano-Kaolinite on the properties of asphalt mixtures, the

Marshall Test was used to determine the optimum asphalt content. The Marshall Test was used to determine the stability and flow for the fabricated specimens. At the optimum Nano-Kaolinite content, the stability increased by 24.9%. To study the effect of Nano-Kaolinite additives on the long term performance of the mix, 30th asphalt mix samples was evaluated by the Indirect Tensile Test using unmodified and modified bitumen at optimum Nano-Kaolinite content at three different temperatures (5^{o}C, 25^{o}C, and 40^{o}C). At the optimum Nano-Kaolinite percentage, the indirect tensile strength value increased by 19.3%, 21.9%, and 18.5% at temperature 5^o C, 25^o C, and 40^o C respectively.

2. Introduction

Bitumen plays a very important role in determining the properties of asphalt mixtures. Therefore, many researchers around the world tried to improve the properties of bitumen using different additives such as different types of polymer. With the widespread use of Nanotechnology around the world, pavement researchers used this new technology to improve the properties of bitumen and asphalt mixture. The Nano material that were used as additives to improve the properties of bitumen and asphalt mixtures include Nano Silica, Nano Hydrated Lime, Nano Clay and Nano Calcium Oxide (CaO). *Hui Yao and et al, 2013,* studied the effect of Nano Silica on the rheological properties and chemical bonding of asphalt. The results indicated that the viscosity values of modified asphalt with Nano Silica decreased slightly, the dynamic modulus of modified asphalt mixture had a significant increase, and the rut depth of modified asphalt mixture decreased significantly. Also, *Saad Issa Sarsam, 2015,* studied the impact of Nano Silica fumes and Nano Hydrated Lime on the rheological and physical properties of asphalt cement. The results indicated that when Nano Silica fumes or Nano Hydrated Lime were added to asphalt cement, the penetration values decreased and the softening point increased. After aging, the ductility was reduced by a range of 10% to 60% based on the Nano additive types and percentages. The softening point increased after aging by a range of 6% to 8% and the penetration value showed variation by a range of 20% to 60% based on the percentages and types of Nano additives. *Farag Khodary, 2015,* studied the effect of Nano Clay on the properties of modified bitumen by using Styrene Butadiene Styrene (SBS). The results indicated that the penetration was decreased and the softening point increased with the increase of the percentage of Nano Clay. *Abolfazl Zare-Shahabadi and et al, 2010,* studied the effect of Nano Clay on the properties and rheological characterization of asphalt binders. The results indicated that the values of the softening point and viscosity increased with the increase of the percentage of Nano Clay. *Farag Khodary and et al, 2015,* suggested using CaO to improve the properties of bitumen and asphalt mixtures. The results indicated that the CaO Nanoparticles improve the penetration properties of bitumen. The value of penetration decreased by 28.9%, and the softening point increased by 24.5% when the Nano CaO percentage increased by 5%. *M. Farmarzi et al, 2015,* studied the effect of Carbon Nanotubes (CNT) on the asphalt binder. The results indicated that the use of CNT as an additive presented improvement in the rheological properties of asphalt binder. Similarly to the these materials that the previous researchers used, Nano-Kaolinite has the potential to improve the properties of bitumen and asphalt mixtures. Therefore, this paper focuses on the effects of Nano-Kaolinite on the properties of bitumen and asphalt mixtures.

3. Material Characterization

3.1 Bitumen Properties

The bitumen used in this study was 60/70 penetration grade, which was obtained from Suez Nasr Petroleum Company (NPC). The bitumen and asphalt mixtures were tested at The General Authority of Roads and Bridges and Land Transport (GARBILT) laboratory. Table 1 shows the conventional physical properties of the base asphalt.

Table 1: Conventional Physical Properties of the Base Asphalt.

Binder grade	Penetration @25°C (mm)	Softening Point, (C°)	Viscosity @135°C	Flash point, (C°)
60/70	63	48	443	270

3.2 Aggregate Properties

The crushed siliceous stone was used in this research as coarse aggregate. The coarse aggregate was a combination of: course aggregate A with specific gravity 2.429, course aggregate B with specific gravity 2.408, and course aggregate C with specific gravity 2.403. The sieve analysis for course aggregate and fine aggregate, as well as the filler were done according to AASHTO T (27-06) and T(37-07), respectively. Table 2 shows the gradation of course aggregate, fine aggregate and filler.

Table 2: The Gradation of Course Aggregate, Fine Aggregate and Filler.

Sieve size (mm)	P% CA (A)	P% CA (B)	P% CA (C)	P% fine sand	P% filler
1"	100				
3/4"	66	100			
1/2"	5	48			
3/8"	3	17	100		
#4	1	1	21	100	
#8			1.4	90	
#16			1.3	75	
#30			1.2	52	100
#50			1.2	35	100
#100			1.1	24	95.5
#200			1	13	75

3.3 Nano-Kaolinite

The process of preparing Nano-Kaolinite was done in the powder metrology and Nano technology laboratory at the Faculty of Engineering, Al-Azhar University using the High-Energy Ball Mill apparatus. The Kaolinite was grained in a Ball Mill apparatus for 24 hours. The morphology of Nano-Kaolinite was analyzed using a Transmission Electronic Microscope (TEM) in the National Research Center. The TEM image (Figure 1) shows that the Nano-Kaolinite diameter was in range of 15 to 50 nm. Also, the X-Ray Fluorescence (XRF) was done, to give information about the full chemical composition. Table 3 shows the chemical composition for Nano-Kaolinite.

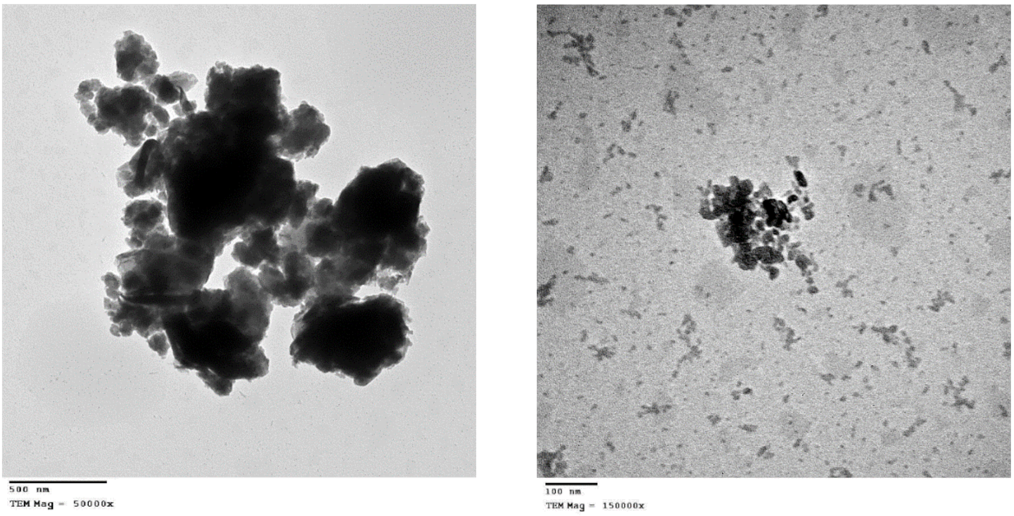

Figure 1: TEM Image of Nano-Slag.

Table 3: Chemical Analysis of Nano-Kaolinite.

Oxides	Weight %	Oxides	Weight %
SiO_2	45.01	TiO_2	2.55
Al_2O_3	40.30	Fe_2O_3	0.89
MnO	0.004	MgO	0.15
CaO	0.12	Na_2O_3	0.16
K2O	9.59		

4. Preparation of Modified Bitumen

To prepare the modified bitumen, firstly it was heated alone to 130ºC±5ºC, and then the specific percentages of Nano-Kaolinite were added to the bitumen. The percentages of Nano-Kaolinite are 2%, 4%, 6% and 8% by the weight of bitumen. The mixing temperature of Nano-Kaolinite with bitumen was between 120 to 130ºC. The Low Shear Mixer with a constant speed of 600 rpm was used for 30 minutes to ensure the production of a homogeneous mixture**.**

5. Preparation of Modified Asphalt Mixtures

To determine the optimum asphalt content, trial Marshall specimens were prepared at asphalt contents of 4.5%, 5%, 5.5%, 6% and 6.5%. The Marshall Test results indicated that the optimum asphalt content is 5.80%. The unmodified bitumen was used at optimum asphalt content to prepare 3 specimens. Also, the modified bitumen with 2%, 4%, 6% and 8% Nano-Kaolinite was used to prepare 5 specimens for each percent. The prepared specimens were used to determine the effect of Nano-Kaolinite on the properties of the mixtures (Marshall Stability and Marshall Flow).

6. Laboratory Data Analysis

6.1 Penetration Test

The Penetration Test (ASTM D5-97) was used to determine the effects of adding Nano-Kaolinite on the penetration grade of bitumen. Five modified samples were prepared to determine the distance (tenths of a millimeter) that a standard needle penetrates the bitumen sample at 25ºC. The results indicated that adding Nano-Kaolinite to bitumen decreased the penetration value, as shown in Figure 2. The value of the penetration decreased from 63 for the unmodified bitumen to 52 at adding 4% Nano-Kaolinite, which means that adding 4% Nano-Kaolinite to bitumen can reduce the penetration value by about 17.5%. This reduction in penetration value is preferable for paving in hotter climates. At 6% Nano-Kaolinite, there are no more significant effects on the penetration value, this means that a 4% additive can be considered as the optimum Nano-Kaolinite content to obtain the lowest penetration value.

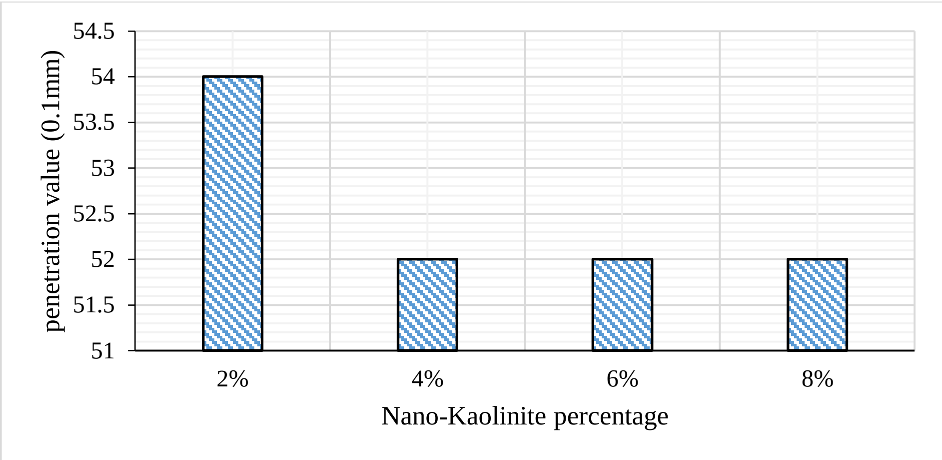

Figure 2: Results of Penetration Test.

6.2 Softening Point

The Ring and Ball Method (ASTM D 36-95) was used to determine the softening point of modified bitumen by Nano-Kaolinite. The results indicated that the use of Nano-Kaolinite to modify the bitumen increased the softening point. This increase in softening point indicated that the asphalt mixtures have more resistance to high temperatures. Figure 3 shows the results of the Softening Point Test. The softening point was increased by 8.3% when 4% Nano-Kaolinite was added to bitumen. At 6% Nano-Kaolinite, there was no change in the value of the softening point. Therefore, 4% Nano-Kaolinite can be considered as the optimum additive content to obtain the maximum softening point.

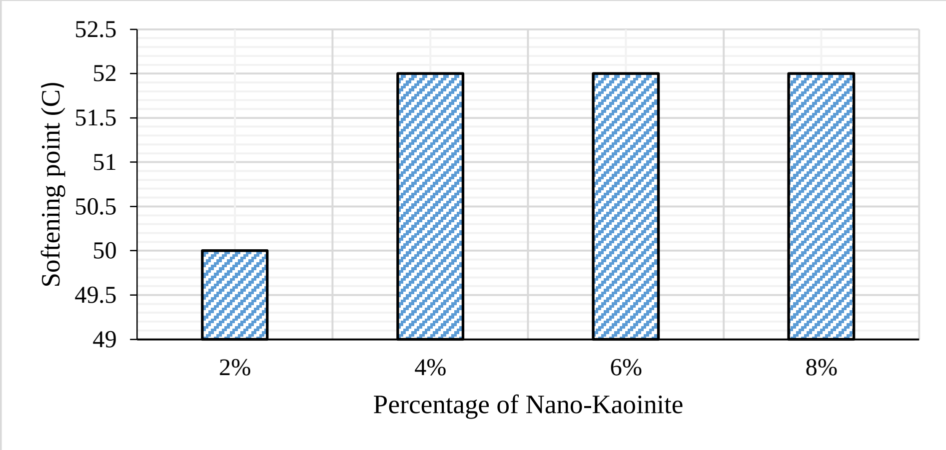

Figure 3: Softening Point Test Results

6.3 Kinematic Viscosity

The Kinematic Viscosity Test at 135ºC was used to determine the effect of Nano-Kaolinite on the viscosity of bitumen. Figure 4 shows the results of the Kinematic Viscosity Test. The results indicated that adding 4% Nano-Kaolinite to bitumen will increase the kinematic viscosity by about 22%. At 6% Nano-Kaolinite the kinematic viscosity will decrease again, thus, 4% Nano-Kaolinite can be considered the optimum percentage to obtain highest kinematic viscosity.

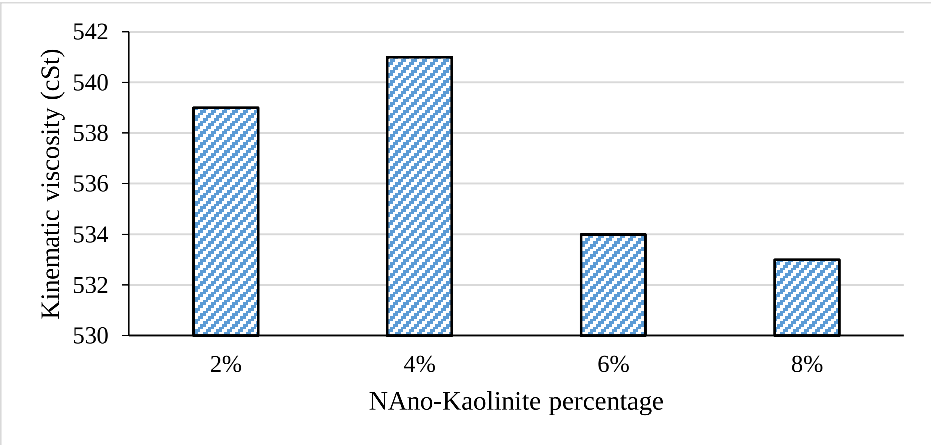

Figure 4: Kinematic Viscosity Test Results

6.4 Marshall Test

The Marshall Test was used to determine the stability and flow for modified and unmodified asphalt mixtures. Table 4 shows the results of the Marshall Test. The results indicated that the stability of the asphalt mixture increased with the increase of the percentage of Nano-Kaolinite from 2% to 4%. At 6%, stability began to decrease again, while there is no effect on the flow results. At 4% Nano-Kaolinite, the stability increased about 23.8%. The results indicated that the optimum Nano-Kaolinite content is 4%. These results can be shown in Figure 5.

Table 4: Marshall Test Results

No. of Specimen	Percent of Nano Slag	Stability Results (kg)	Mean Stability (kg)	Flow (mm)	Mean Flow (mm)
N-1	0%	1700.8	1665	3.9	3.9
N-2		1659.8		3.9	
N-3		16634.9		3.8	
MK2-1	2%	2009.7	1977	4	3.9
MK2-2		1968.6		3.9	
MK2-3		1976.7		4	
MK2-4		1991.8		3.9	
MK2-5		1935.7		3.7	
MK4-1	4%	2032.0	2062	3.9	3.9
MK4-2		2044.3		4	
MK4-3		2080.8		3.9	
MK4-4		2069.3		3.8	
MK4-5		2074.6		4	
MK6-1	6%	2000.4	1970	4	3.9
MK6-2		1963.3		3.9	
MK6-3		1974.9		4	
MK6-4		1951.8		3.9	
MK6-5		1958.0		3.9	
MK8-1	8%	1859.2	1876	4	3.9
MK8-2		1877		3.8	
MK8-3		1907.3		3.9	
MK8-4		1859.2		4	
MK8-5		1877.1		3.8	

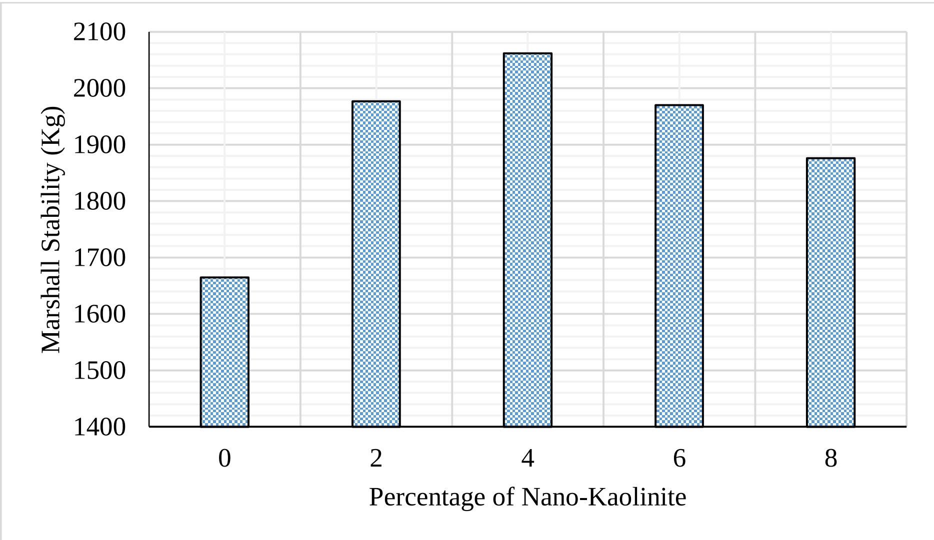

Figure 5: Marshall Stability Results

6.5 Indirect Tensile Test (IDT)

Fatigue cracking is one of the major distresses in hot mix asphalt. To examine the long term performance of the bituminous mixtures, the Indirect Tensile Test was used to determine the tensile properties of the mix, which relates to pavement fatigue cracking. A greater tensile strength means stronger fatigue cracking resistance.

To determine the indirect tensile strength, the following equation was used.

$$ITS = 2P_{max} / (\pi d * h)$$

Where:

P_{max} is the maximum applied failure load (kg) of the specimens under diametric compression, *d* and *h* are average values of the diameter (cm) and height (cm) of the specimen.

The Indirect Tensile Test was conducted at three different temperatures (5^{o}C, 25^{o}C, and 40^{o}C). Figure 6 shows the results of the IDT for asphalt mixtures using unmodified and modified bitumen at optimum Nano-Kaolinite content that determined by 4% by the weight of bitumen at different temperatures.

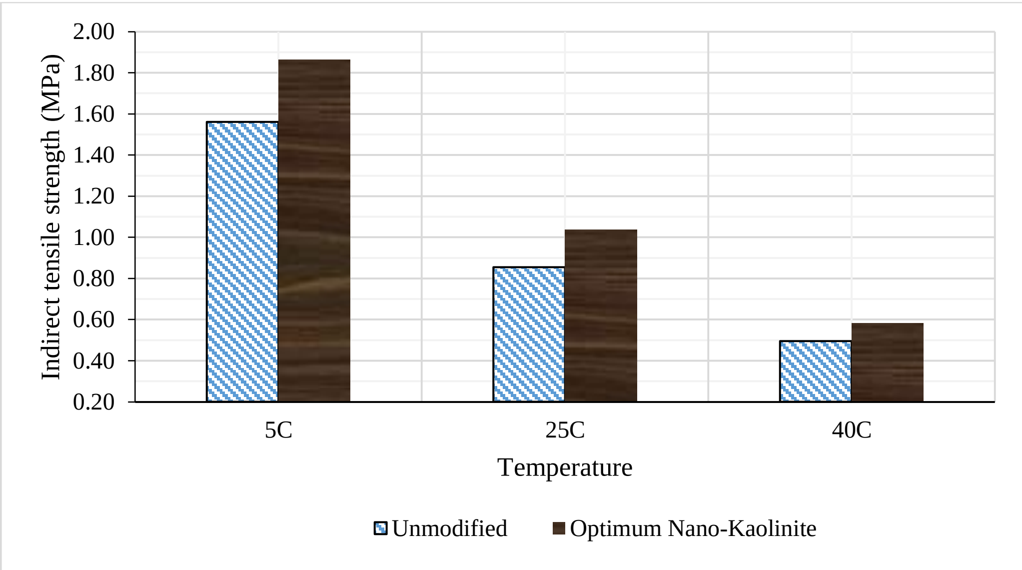

Figure 6: The results of IDT for asphalt mixtures using unmodified and modified bitumen at optimum Nano-Kaolinite content at different temperatures.

The Nano-Kaolinite additive had a significant effect on the tensile strength of asphalt mixtures. At 5^{o}C the tensile strength increased by about 19.3%. At 25^{o}C the tensile strength value increased by about 21.9%. Also at 40^{o}C this value increased by about 18.5%.

7. Conclusion

Nano-Kaolinite has a significant effect on the properties of bitumen. The results indicated that the use of Nano-Kaolinite as a modifier to bitumen can significantly improve the bitumen properties to resist high temperatures.

The results indicated that the optimum Nano-Kaolinite content is 4% by the weight of bitumen. At this percentage, the penetration value decreased by about 17.5%. Also, at the optimum content of Nano-Kaolinite, the softening point temperature increased by about 8.3%. The decreasing of the penetration value and the increasing in the softening point make the bitumen more suitable for use in hot climatic regions. The results indicated that the kinematic viscosity also increased by about 22.0% at the optimum content of Nano-Kaolinite. Additionally, the Nano-Kaolinite had a significant effect on the properties of asphalt mixtures. Using Nano-Kaolinite increased the stability of asphalt mixtures by 24.9%, and the indirect tensile strength increased by 19.3%, 21.9%, and 18.5% at temperatures 5^oC, 25^oC, and 40^oC respectively. The increasing in the indirect tensile strength means an increase in the fatigue resistance. Therefore, the long term performance of bituminous mixtures is positively affected through the addition 4% Nano-Kaolinite content by the weight of bitumen.

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