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INVESTIGATING THE IMPACTS OF CARBON NANO-TUBE ON ASPHALT AND ASPHALT CONCRETE MIXTURES

by

Rahmatullah

May 2015

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APPROVAL PAGE

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ABSTRACT

The Failures of flexible pavements as a huge concern to the roads authorities and users have now became a common scenario across the globe. Researchers and academicians have been evaluating the distresses and looking for the remedy to improve the functional properties of asphalt and asphalt concrete. Various different materials have been used for the mentioned purpose, which have contributed some or not enough. This comprehensive study finds out the optimum amount and impacts of carbon nano-tubes (CNTs), while used in combination with asphalt and asphalt. The investigations were conducted in several categories to determine the pavement characteristics such as resistance to bonding and disintegration (in presence of water) by tensile strength and retained stability tests, which were performed on bituminous mixtures. Penetration, Softening point, RTFO, DSR and BBR were performed on bitumen to determine CNTs contribution on rutting, fatigue and low temperature cracking, also the microsturcture and morphology of fracture surfaces of CNT-modified asphalt samples were studied using the Scanning Electron Microscpy (SEM) analysis. Experimental results have shown that the addition of CNTs has improved both classic (penetration degree and softening point) and performance(fatigue parameter, rutting factor and low-temperature cracking) properties of asphalt as compared to the standard bitumen.

Keywords:Flexible pavement, Additives, Bitumen, Carbon nano tubes, Rutting, Fatigue, Low temperature cracking, Tensile strength, Retained stability Index.

KARBON NANOTÜPLERİN ASFALT VE ASFALT BETONLAR ÜZERİNDEKİ ETKİSİNİN

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Yüksek Lisans Tezi – İnşaat Yapı ve Altyapı Mühendisliği
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ÖZ

Yol kullanıcıları ve ilgili makamlar için büyük bir endişe kaynağı olan esnek yol kaplama bozulmaları dünya genelinde ortak bir senaryo halinegelmiştir. Araştırmacılar ve akademisyenler bozulmaları değerlendirmekte; asfalt ve asfalt betonlarının fonksiyonel özelliklerini iyileştirmek için çareler aramaktadırlar. Bu amaç için çeşitli malzemeler kullanılmakta, bu malzemeleren bazıları kaplama performansına yeterli katkıda bulunmakta bazıları yeterli olamamaktadır.

Bu kapsamda yapılan bu çalışmada karbon nanotüplerin (KNT) asfaltta kullanılabileceği en uygun oran ve etkileri araştırılmıştır. Karbon nano tüplerin üstyapıların çeşitli bağlanma ve suya dayanım karakterleri üzerindeki etkileri bitümlü karışımlar üzerine uygulanan çekme gerilmesi, kalıcı stabilite deneyleri ile belirlenmiştir. Penetrasyon, yumuşama noktası, dönen ince film, dinamik kesme, eğilmeli kiriş deneyleri ise karbon nanotüplerin tekerlek izi, yorulma ve düşük sıcaklık çatlakları üzerindeki etkisini incelemek üzere asfalt numunelere uygulanmıştır. Ayrıca karbon nanotüp – asfalt numunelerin kırılma yüzeylerinin mikrosalyapısı ve morfolojisi Taramalı Elektron Mikroskobik Görüntü Analizi (SEM) ile belirlenmiştir. Deneylerden elde edilen sonuçlar göstermiştir ki karbon nanotüplerin kullanımı klasik (penetrasyon, yumuşama noktası) ve performans (yorulma parametresi, tekerlek izi faktörü ve düşük sıcaklık çatlağı) özelliklerinin her ikisini de önemli ölçüde iyileştirmektedir.

Anahtar Kelimeler: Esnek yol üstyapısı, Katkı maddeleri, Bitüm, Karbon nano tüpler, Tekerlek izi, Yorulma, Düşük sıcaklık çatlakları , Çekme gerilmesi, Kalıcı stabilite indeksi.

This work is dedicated to my whole family and teachers.

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INTRODUCTION

1.1 GENERAL

Flexible pavements are supposed to be built such that they provide a safe and comfortable driving surface to the public. They should be designed and constructed in such a way that they could provide such surface for a long period of time and at the lowest possible costs. This implies that the thickness design and the material selection should be such that the major failure types are under control. In today's world, the road and surface failure of the flexible pavement has become one of the central and attention switching problem. There are numerous failures that can be observed on flexible pavement, such as cracking, deformation, disintegration and wear. These failures take place due to various of causes, that could be traffic load associated, thermal movement, climate change, settlements, swelling soils, frost heave and most importantly the imperfect mixture and low quality of materials used.

As the traffic intensity increases with the passage of time, the pavements start showing distress in the form of minor cracks. Cracks develop in some sections after being overlaid with bituminous surfacing. The rehabilitation of cracked roads by simply overlaying with a layer of bitumen is rarely a durable solution. The cracks gradually propagate through the new overlay. It is a matter of fact that newly overlaid bituminous layer does not possess the inherent property to prevent the propagation of cracks. The phenomenon referred to as reflective cracking is prevalent over many countries around the world. With the current financial crunch the departments involved in road construction are forced to use cost effective solutions.

During past years researchers and academicians around the world are working curiously to pick up the best possible remedial measures in order to improve the functional prosperities of asphalt. For this reason various micro-additives and nano-reinforced materials have been recommended in the past and yet to come, which have contributed some or not enough and at the same time a number of them are still in experimental stages. Nanotechnology deals with the creation and use of functional materials with novel properties and functions that are achieved through the control of matter at the atomic and molecular level. The use of nano-structured materials has shown a tremendous development in recent years, with wide-ranging applications in many engineering fields.

In this study, carbon nanotube (CNT), that perhaps represents the most promising additive among other nano-sized materials has been used as the modifier of bitumen and it's effects on asphalt binders, aggregates and hot mix asphalt are investigated.

1.2 AIM AND OBJECTIVES OF THE STUDY

The aim of the study is to find out the influences of using carbon nanotubes as a modifier or an additive to the asphalt binder, and to find the optimum amount of carbon nanotube through conventional bitumen and asphalt concrete tests.

The above aims and objectives were achieved through performing a number of routine tests:

- The hardness or consistency of asphalt through penetration test at different percentages of CNTs.
- Resistance to rutting at high temperature through softening point test.
- The moisture damage determination by performing the retained stability test at high temperature.
- Investigating the rutting factor through dynamic shear rheometer (DSR) test, which also analyzes the performance grade (PG) at high and intermed temperatures.
- Stripping value of aggregates through CNTs modified bitumen.

CHAPTER 2

LITERATURE REVIEW

2.1 HOT MIX ASPHALT AND PERFORMANCE CHARACTERISTICS

Pavement failure is defined in terms of decreasing serviceability caused by the development of surface distresses such as cracks, potholes and ruts. Before going into the maintenance strategies, we have to think of the causes of distresses of bituminous pavements and find out the major causes and it's combination, which is often a tough task to achieve. Pavement failures may be classified as structural, functional, or materials failure, or a combination of these factors. Structural failure are those associated with the pavement structure " not being able to carry or resist the designed amount of load anymore without corrosion". Functional failure can be the loss of any of the various functions of the pavement such as skid resistance, structural capacity, and serviceability or passenger comfort. Materials failure occurs due to the lack of integration or loss of material distinctiveness of any of the material's component [1,2].

The major kinds of pavement failures can be classified as either deformation failures or surface texture failures. Deformation failures include rutting, depressions, corrugations, potholes and shoving. These failures may be due to either traffic, which are load-associated or environmental influences, which are non load-associated. Surface texture failures include cracking, bleeding, polishing, stripping and raveling. These failures often trigger the rehabilitation of pavement structure although the road pavement may still be structurally sound, but the surface no longer performs the function it is designed to do, which is normally to provide skid resistance, a smooth running surface and water tightness [3].

The failure of any one or more components of the pavement structure develops the waves and corrugations on the pavement surface or longitudinal ruts and shoving. Therefore each one of the layers should be carefully designed and laid in order to maintain the stability of the pavement structure as a whole, each layer should be stable within itself and thus making the total pavement preserved its stability.

Pavement unevenness is also considered as a failure when it is excessive. The subject of pavement failure/distress is considered to be complex as several factors contribute to its deterioration and failure. The aging and oxidation of bituminous films lead to the deterioration of flexible pavement. Detrimental actions in pavement are rapidly increased when excess water is retained in the void spaces of the pavements. The more the distress, the shorter the pavement's life- and at some point, the distresses are so great in intensity (for example, 75% of the wheel path area in project area has cracks) that the pavement is considered to be "failed" or at the end of its design life.

Cracking is one of the most important types of distresses in asphalt pavement and repeated traffic loads causes pavement deterioration of many-sided, sharp-angled pieces, known as alligator cracking. Once initiated, cracking quickly spreads both in severity and extent, one of the main problem with cracks is that they allow moisture into pavement, the intrusion of water decrease the strength in lower layers as well as decreasing the bearing capacity of sub-grade soil by forcing of soil particles through the cracks, which accelerates the corrosion of pavement.Cracks can occur in a wide variety of patterns. They may result from a large number of causes, but generally are the result of either ageing, environmental conditions, structural or fatigue failure of the pavement. The formation of cracks in the pavement surface causes numerous problems such as discomfort to the users, reduction of safety, etc [4,5].

Rutting is said to be the permanent downward deformation of the surfacing within wheel paths and is due to the lateral displacement of material within the pavement layer. It occurs when the structural properties of the compacted pavement are insufficient to resist the stresses forced upon it. It is important to determine which layer has rutting since this will influence the optimal maintenance strategy, although recent studies have shown that rutting is mostly a near-surface phenomenon, affecting only the top 1–3 in. of the asphalt concrete layer, with visible slip surfaces associated with the rutting failure [6],[7],[8] . The worse level of rutting is the higher variation in the transverse profile of road surface. Therefore, ruts interfere with surface run-off patterns and increase the risk of wetting in the upper pavement layers. Rutting can also initiate aquaplaning, and hence have adverse impact on safety.

Potholes, as one type of pavement distresses, are bowl-shaped depressions of various sizes in the pavement surface. Considering their visual impact, they can also be defined as almost egg-shaped pavement parts, which are fully or partially surrounded by a dark shadow (due to depression) and which have a granular and course textural appearance due to disintegration. Based on these visual characteristics they are recognized and evaluated manually within visual inspections of pavement image and video data.Potholes are an indication of structural surface failure. Water entering pavement is often the cause, and could be caused by a cracked surface, high shoulders or pavement depressions ponding water on pavement, porous or open surface, or clogged side ditches. Once water enters pavement layers, the base and/or sub-grade become wet and unstable, and the resultant degradation leads to rapid growth of pothole area and depth. If the potholes are numerous or frequent, it may indicate underlying problem such as inadequate pavement or aged surfacing requiring rehabilitation or replacement [9],[10],[11].

The excess moisture in pavement structure is believed to be the main trigger of distresses in most of the pavement failures cases. The presence of moisture lot changes and reduces the strength and stiffness of pavement materials. Moisture greatly affect the sub-grade materials as compared to sub-base and base. Excess moisture and particularly high degrees of saturation result in significant pore pressures within the material. Depending on the degree of saturation, failure may occur as any of rapid shear or bearing failure, premature rutting, lifting of wearing course due to positive pore pressures, or embedment of cover aggregate due to weak base [12] . It can be seen that for nearly all types of pavement failure, moisture is often the primary or a contributing cause of failure. Moisture entry through the surface may be caused by inadequate pavement surface drainage during construction, exposure of surface to rain during construction, or porous or open graded asphalt.

Moisture entry from the side may be caused by pond age in pits or poorly constructed surface drainage, and lateral movement of water into pavement. Other factors affecting the moisture in a pavement include the general drainage condition, such as the effectiveness of drainage structures, shoulder cross-fall and condition, longitudinal grade, and whether the pavement is constructed on cut or fill [1,13].

2.2 NANOTECHNOLOGY IN ASPHALT MIXTURES

Nanotechnology has been explored to a considerable degree to address the problems in design, construction, and utilization of functional structures with at least one characteristic dimension measured in nanometers [14]. Nanotechnology is widely considered as one of the 21st century’s important technologies, and its economic importance is gradually on the rise. In architecture and the construction industry it has possibilities that are already usable today. Such possibility can already be seen today through many current applications related for instance to surface coatings, self-cleaning capacity, and fire resistance, and others [15].

Nanotechnology can be depicted as the understanding, control, and restructuring of substances on the order of nanometers. It involves the research and development at atomic, molecular, macromolecular or nano levels, i.e., less than 100 nm, to create materials, structures, devices and systems that have novel properties with basically new properties and functions because of their small and/ or intermediate size [16]. Nanotechnology therefore allows the design of systems with high functional density, high sensitivity, special surface effects, large surface area and high strain resistance [14].

The macroscopic mechanical behavior of bituminous materials, which are mainly used on a large scale and in huge quantities for pavement still depends to a great extent on microstructure and physical properties on a micro and nano-scale. Although researchers, material producers, and engineers have explored the potential of nanotechnology for many years, its usage has been limited. New efforts and exploration of the development of nano-materials for pavement application that improve the nano - scale mechanical and physical properties as well as durability of this important group of construction materials provide a considerable prospect [14].

Many researches have been carried out across the globe by various researchers intended to improve the hot mix asphalt (HMA) performance characteristics by investigating the addition effects of nano-family materials. M. El-Shafie, et. al. carried out a laboratory-tests based research study intending to investigate "The addition effects of macro and nano clay on the performance of asphalt binder". They blended nanoclay in an asphalt binder in various percentages (starting from 2% to 8%). The blended asphalt binders were characterized using kinematic viscosity, softening point temperature and penetration and compared with an unmodified binder [16]. By using nanoclay of 2% and 8%, the viscosity at 135 oC increased by an average of 140–236% respectively as compared with the unmodified binder. The kinematic viscosity at 150oC increased by an average of 45% and 102% for asphalt with 2% and 8% unmodified clay respectively as compared with the unmodified binder, while it was ranging from 94% to 160% for asphalt with 2% and 8% modified clay respectively. The maximum increment was found with 8% of nanoclay compared to unmodified binders. The increase in the viscosity value at high temperature is a good property of rutting resistance. Moreover, the softening point temperatures for various percentages of nanoclay were found that nanoclay modified asphalt gives a higher softening temperature by an average of 4–13 oC for 2% and 8% of modified asphalt with clay respectively as compared with the unmodified binder. The nanoclay modified asphalt which seems to be less sensitive to high temperature may be more resistant to plastic deformation (rutting) as compared to unmodified asphalt. In addition to that the results obtained from penetration tests showed that with the addition of nanoclay the penetration values decreased. The decrease in the penetration value for asphalt modified nanoclay ranged from 14% to 28% for 2% and 8% respectively as compared with the unmodified binder. The maximum decrease in the penetration value was recorded with 8% nanoclay content for all modified binder compared with the control binder. Since there is no significant difference between 6% and 8% nanoclay content, and from the practical cost view the ratio of 6% of nanoclay content was found to be the most suitable ratio for nanoclay addition.

Nur Izzi Md. Yusoff, et.al. had an investigation on the performance characteristics of polymer-modified asphalt mixture (PMA) with the addition of nano-silica particles. Polymer-modified asphalt, PG-76, was mixed with nano-silica at concentrations of 0%, 2% and 4% by weight of asphalt binder. Asphalt mixture tests such as moisture susceptibility and resilient modulus were conducted to evaluate the performance of

PMA mixed with nano-silica under various ageing and moisture susceptibility conditions [17].

The moisture susceptibility test was conducted on all mixtures at the Optimum Bitumen Content (OBC) for each individual mix. The Tensile Strength Ratio (TSR) result is an indication that the asphalt mixes are susceptible to moisture damage. Figure (2.1) shows the {TSR} values obtained for each sample. As shown in this figure, all the mixtures met the required minimum 80% {TSR} value specified in AASHTO T283. However it is observed that the PMA mixed with 4% nano-silica is the least susceptible to moisture damage with a TSR of 98.41%, followed by PMA mixed with 2% nano-silica and the control sample (PMA). This finding indicates that the strength of the asphalt mixes increases with the addition of nano-silica particles.

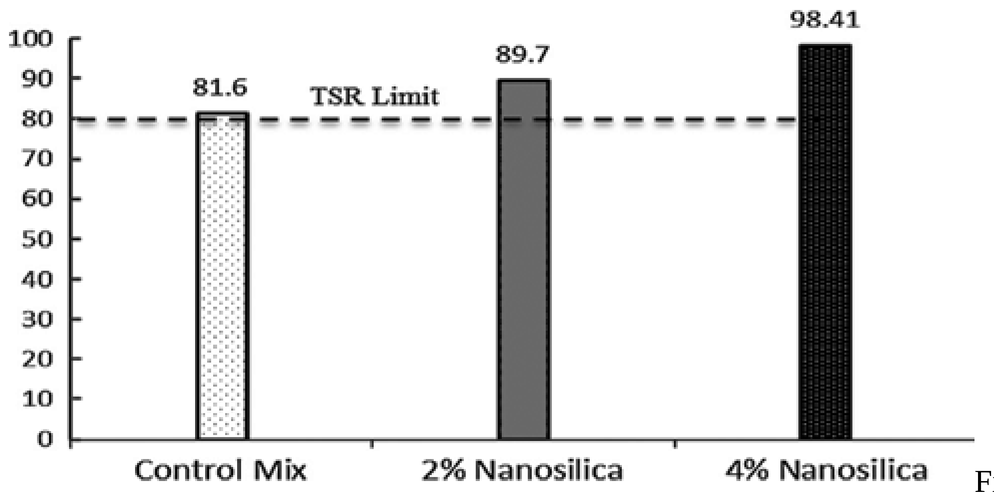

Figure 2.1 The Tensile Strength Ratios (TSR) For Various Percentage of Nano-silica Added To Asphalt Binder [17].

The resilient modulus test was conducted at two temperatures, 25 and 40. At 25oC, the resilient modulus is an indication of the mixture’s resistance to fatigue, whereas the resilient modulus at 40 oC indicates the mixture’s resistance to rutting [17]. The results obtained from their research on the resilient modulus tests are presented in Figure {2.2 and 2.3} for un-aged and aged samples respectively. As shown in figure(2.2) the un-aged PMA mixed with 4% nano-silica shows the least susceptibility to fatigue deformation with the highest resilient modulus of 2037 MPa, followed by PMA mixed with 2% nano-silica and the control sample (PMA). Similar trends can be observed for the samples that were exposed to short-term and long-term ageing. This finding indicates that the addition of nano-silica would improve resistance to fatigue deformation at intermediate temperatures, both for un-aged and aged samples compared to the control sample.

As the temperature increases to 40oC, the difference in resilient modulus is more notable, with a decline in stiffness at 40oC (figure 2.3). At higher temperatures, PMA mixed with 4% nano-silica shows the highest resilient modulus value compared to PMA mixed with 2% nano-silica and the control sample. Considering the difference in the resilient modulus values at higher temperatures, this indicates that PMA mixed with 4% nano-silica is the least susceptible to rutting compared to the control mix. At a pulse period of 100 ms of the resilient modulus test, the 4% nano-silica mixed with PMA shows the highest resilient modulus of 2639 MPa, followed by 2% nano-silica mixed with PMA and the control sample.

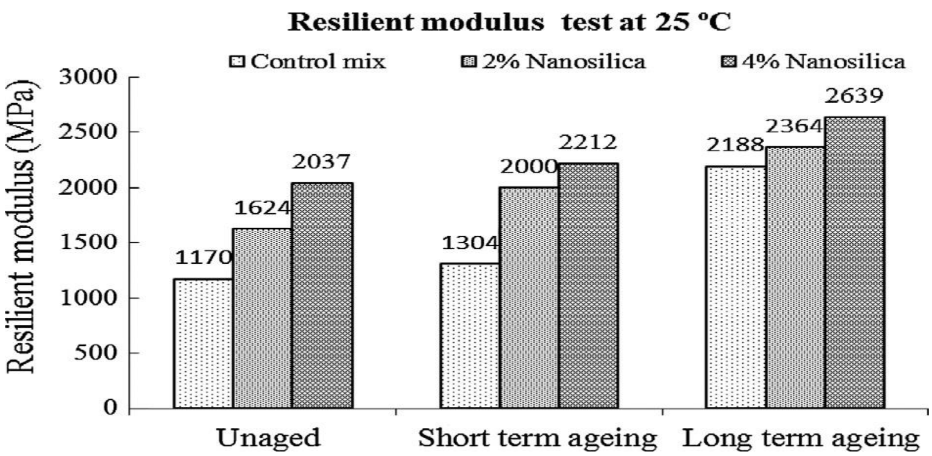

Figure 2.2 Resilient Modulus Test At 25oC.

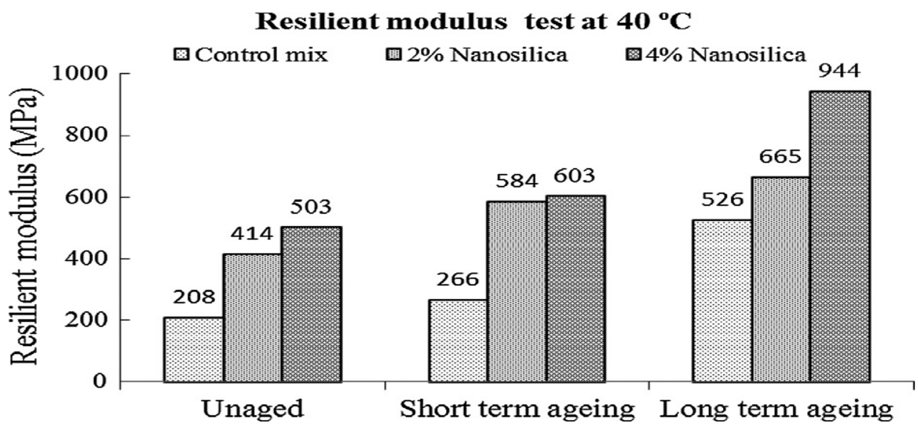

Figure 2.3 Resilient Modulus Test At 40oC[17].

Based on this limited laboratory work done in this study, they concluded that the addition of 4% nano-silica is the optimum content to improve the performance characteristics of PMA in various conditions.

Ghafarpoor et al. carried out comparative rheological tests on bitumen and mechanical tests on asphalt mixtures containing unmodified and nanoclay modified bitumen. Results showed that nanoclay could improve properties of asphalt mixtures such as stability, resilient modulus, and indirect tensile strength [18]. Golestani et al. evaluated performance of bitumen modified with nano-composite. The physical, mechanical and rheological properties of original bitumen, and bitumen modified with nano-composite have been studied and compared. The results showed that nano-composite could improve the physical properties, rheological behaviors and the stability of the bitumen [19]. Vandeven et al. investigated nanotechnology effects on the adhesion of asphalt mixtures. Two different types of nanoclay were used to modify bitumen. In the first case, viscosity of modified bitumen in comparison to original bitumen (70–100) did not change after the addition of 6% of nanoclay, although it was improved its short-term and long-term hardening. In the second case, viscosity of bitumen was increased after adding nanoclay [20].

Ghasemi et al. evaluated the potential benefits of nano-SiO2 powder and SBS for the asphalt mixtures used in pavements. Five bitumen formulations were prepared by using various percentages of SBS and nano-SiO2 powder. Marshall samples were then prepared by the modified and unmodified bitumen. The results of this investigation indicated that the asphalt mixtures modified with 5% SBS plus 1% nano-SiO2 powder could give the best results in the tests [21]. Khodadadi et al. investigated the effect of adding Nanoclay on long-term performance of asphalt mixtures. Indirect tensile test was conducted on cylindrical specimens made of conventional and modified bitumen at the stress levels of 200, 300, 400 and 500 kPa. The results showed that the addition of 1% nanoclay could increase the fatigue life of the asphalt mixtures [22].

Gh. Shafabakhsh, et. al. had a research study on " Evaluation the effect of nano-TiO2 on the rutting and fatigue behavior of asphalt mixtures". In this study effect of nano-Titanium dioxide (TiO2) has been investigated to improve HMA properties.To achieve this goal, mixtures with different content of bitumen and nano-Titanium dioxide were prepared and the effects of these parameters were investigated on the modified mixtures in comparison to conventional asphalt mixtures. The results of penetration, softening point, ductility and RV tests are presented in Figs (2.4 to 2.7). It can be seen from figures that adding of nano-TiO2 has positive effects on the rheological properties of bitumen. The viscosity of bitumen is increased and Penetration of bitumen is decreased by adding the nano-TiO2. Moreover, the softening point of bitumen is improved by decreasing temperature sensitivity of modified bitumen due to adding nano-TiO_2. It is illustrated in Figs (2.6 to 2.7) that ductility and apparent viscosity (in RV test) are significantly increased to 5% nano-TiO2 with improvement of modified bitumen stiffness in comparison to conventional bitumen. The results obtained by the penetration, softening point, ductility, and RV tests for bitumen showed that in 5% nano-TiO_2, penetration is increased and ductility, apparent viscosity, and softening point are decreased. As a result, 5% nano-TiO_2 as modifier of bitumen is an optimal content [23].

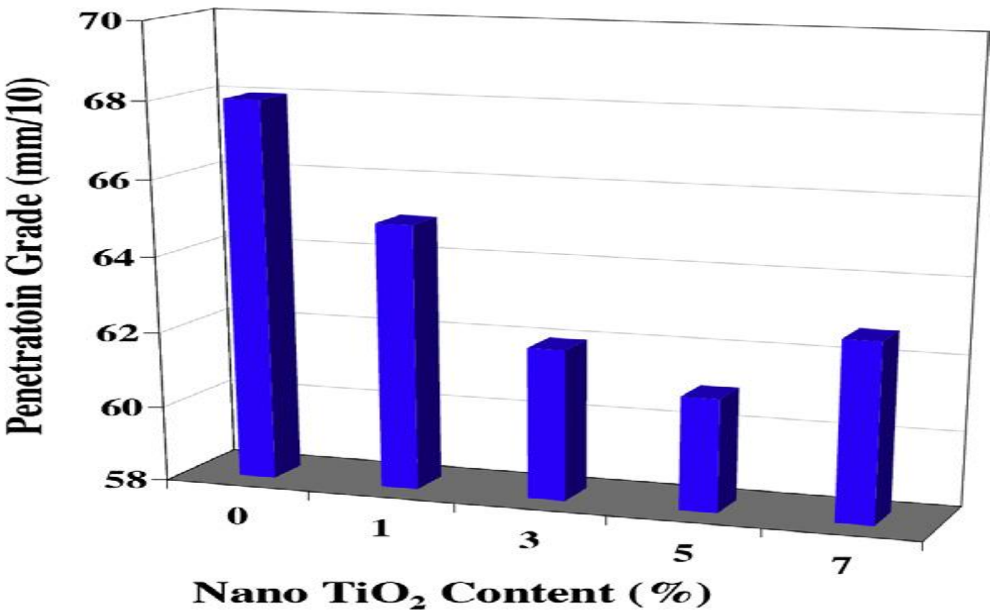

Figure 2.4 Penetration Test Results On Unmodified And Modified Bitumen Samples [23].

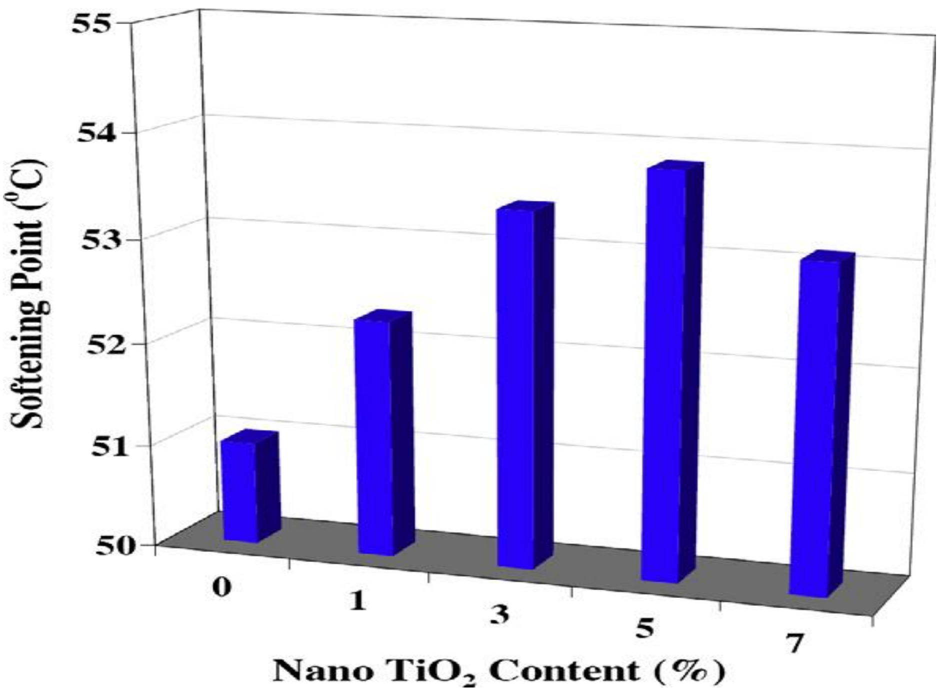

Figure 2.5 Softening Point Test Results On Modified And Unmodified Bitumen Samples [23].

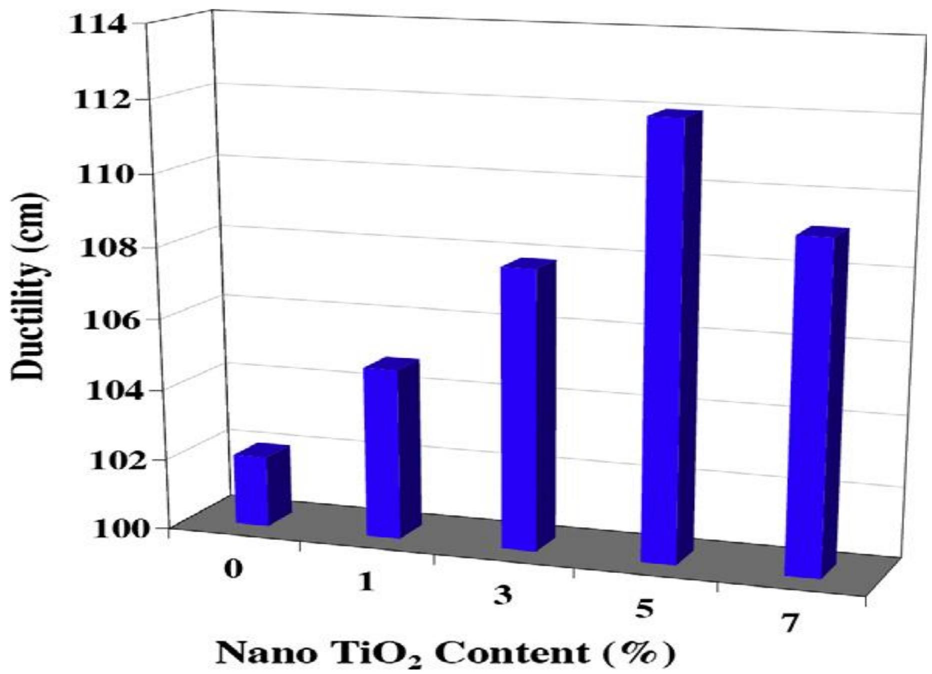

Figure 2.6 Ductility Test Results on unmodified and modified bitumen samples [23].

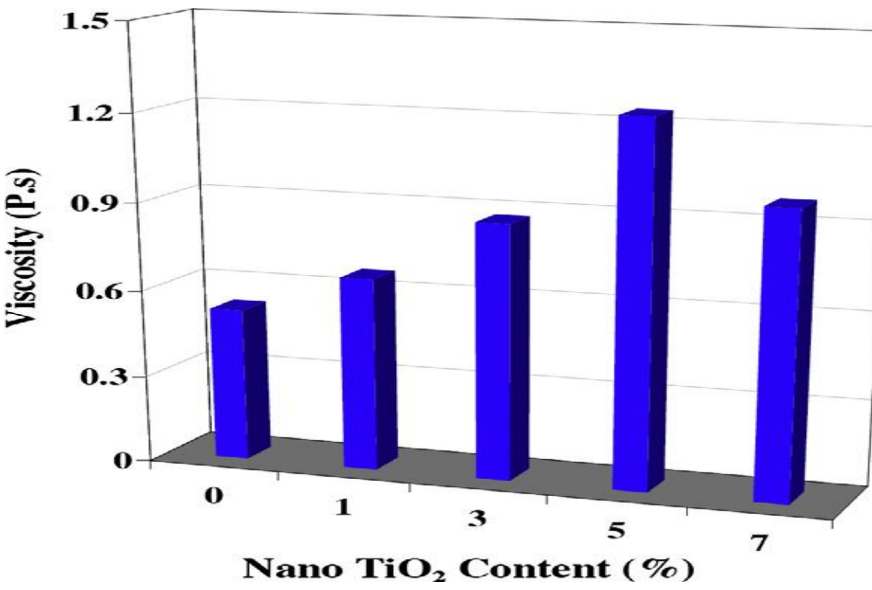

Figure 2.7 Rotational Viscometer Test Results OnUnmodified And Modified Bitumen Samples [23].

2.3 CARBON NANOTUBES (CNTS) IN ASPHALT MIXTURES AND PERFORMANCES

A CNT is a one-atom thick sheet of graphite rolled up into a seamless hollow cylinder with a diameter of the order of one nanometer which were discovered by Iijima in 1991, who first reported the arc-discharge synthesis and characterization of helical microtubules, formed by molecular-scale fibers with structures related to fullerenes.

CNT's has an excellent properties as its high conductivity (being more than copper), elastic deformability, strength (being stronger than steel), surface chemistry, high stability are some of the properties that CNT's provide due to their structure and topology [24,25]. Depending on the radius of the tube, the Young's modulus of a CNT can be as high as 1,000 GPa and the tensile strength can reach 150 GPa.

Two different types of CNT exist respectively in the form of single tubes (called single-wall CNTs) and coaxial tubes (multiple-wall CNTs). Multi-wall CNTs are less expensive and easier to produce but exhibit lower strength and stiffness than single-wall CNTs [26].

Using carbon nano-tubes equals to 0.001 of weight bitumen in asphalt mixtures, in addition to improving asphalt pavement properties, will decrease thickness of under layers and as a result will reduce stone materials consumption. CNTs provide an enhancement of rutting resistance potential and resistance to thermal cracking [27].

When CNT's are mixed with asphalt and concrete, the compressive strength are boosted and increased for a longer duration of time, and not only that but also contribute to the tensile strength by improving the flexural strength. It also reduces the emission of greenhouse gases. Energy consumption, maintenance costs, resistance to moisture are other advantages of using asphalt that contains CNTs [28] .

A group of researchers H. Ziari, et. al. has investigated the impacts of carbon nanotube on bitumen and HMA performance through various classical and functional experiments of pavement and found that the use of CNT improved the performance characteristics of HMA. They selected five different percentages of CNTs (0.3, 0.6, 0.9, 1.2 and 1.5) to be mixed with bitumen. From the results of DSR it was obtained that the complex modulus (G*), phase angle (δ) and G*/sinδ parameters were greatly improved with CNT modified bitumen. It was seen that the G* values were gradually increased with increase in amount of CNT, and they got the highest value of G* for 1.5% of CNT. Similarly the δ values were progressively reduced with increase in amount of CNT, the lowest δ value was obtained for 1.5% of CNT. The material, which exhibit higher G* and lowest δ are believed to be the best.

Furthermore they found that G*/sinδ values were also increased with increase in amount of CNT content, and they got the highest value for 1.5% of CNT, but for all these three parameters it was described that there was not a significant difference between 1.2% and 1.5% of CNT. Therefore, it can be said that 1.2% may be the optimum amount [29].

Moreover, Ezio Santagata, et. al. have investigated the influence of CNT on bitumen. They selected three different percentages of CNTs (0.1%, 0.5% and 1.0%) and carried out the viscosity test, which is a measure of flow characteristics of bituminous binders. It is recommended that binders must be sufficiently fluid at high temperatures so that they can be easily pumped and handled during production and laying of bituminous mixtures paving applications. The results obtained indicated that, the viscosity gains are of the order of 8-9% for the lowest CNT dosage (0.1%), of the order of 23-25% for the intermediate dosage (0.5%) and are significantly higher (reaching values 100 and 200%, respectively for 165 and 135 oC) in the case of blends with 1% CNT [30].

In addition to that, Aemen N. Amirkhanian, et. al. had a research on the impacts of CNT on asphalt binders. They carried out a number of routine tests of pavement (Viscosity, performance Grade and Creep and Creep Recovery) modifying the binder with four different percentages of CNT (0.2%, 0.5%, 1.0% and 1.5%) and evaluated the results. The grade determination feature of the DSR was used to determine the failure temperature for each binder with or without nano particles in the original un-aged state. The results indicated that the addition of nano particle resulted in an increase in failure temperature, especially, as the dosage percentage of nano particles increases from 0.2% to 0.5%, the failure temperature rises remarkably.

In general, a PG grade (6oC) is achieved as the addition of nano particles is greater than 0.5%. In addition to that, based on the values of complex modulus (G*) and phase angle (δ), it also indicated that regardless of at a starting temperature of 64oC or other higher test temperatures, 1.5% of CNT has the highest G*/sinδ value while virgin binder showed the lowest value. Moreover both virgin binder and the binder with 0.2% nano particles had the G*/sinδ value less than 1.000 Kpa at 70oC. However, the G*/sinδ values of binders with 1.0% and 1.5% nano particles is greater than 1.000 Kpa at 76oC. From the above investigation it can be concluded that the addition of nano particle has a significant effect on PG and contributes to an improvement of rutting resistance at a high performance temperature [31].

2.4 BENEFITS OF NANOTECHNOLOGY IN ASPHALT MIXTURES

In general, Nanotechnology produces benefits in two ways – by making existing products and processes more cost effective, durable and efficient and by creating entirely new products. Nanotechnology has the following known benefits [In particular to asphalt and asphalt mixture properties]:

- Improve the storage stability in polymer modified asphalt

- Increase the resistance to UV aging
- Reduce the moisture susceptibility under water, snow and deicers
- Improve the properties of asphalt mixtures at low temperature
- Improve the durability of asphalt pavements
- Save energy and cost
- Decrease maintenance requirements

CHAPTER 3

RESEARCH PROGRAM

3.1 MATERIAL PREPARATION

3.1.1 Aggregates

The type of aggregates used in this laboratory study are basalt, the usage of which is well known in production of asphalt concrete mixtures, and that is because of its excellent and reliable physical properties. Basalt aggregates are extremely hard, free of moisture and with tight grained structure that provides high abrasion and excellent UV resistance. The aggregates were supplied from one of the prominent asphalt construction site in Turkey. With the nominal maximum size of 19.1mm and the minimum of 0.075mm. The physical aggregate properties are given in Table (3.1).

Table 3.1 Basalt Aggregate Test Results.

Tests	Units	Test Methods	Results	Specification-Wear Type--1
Abrasion loss (Loss Angeles) (max)	%	TS EN 1097-2	12	30
Abrasion (max)	%	TS EN 1097-6	1,53	2,0
Magnesium sulphate freezing loss (max)	%	TS EN 1367-2	6,61	16
Flatness index	%	TS 9582 EN 933-3	20	20
Peeling strength, (min)	%	KTS 2006	30-40	50
Bulk specific gravity of coarse aggregates	g/cm^3	TS EN 1097-6	2,986	-
Apparent specific gravity of coarse aggregate	g/cm^3	TS EN 1097-6	2,997	-
Bulk specific gravity of fine aggregate	g/cm^3	TS EN 1097-6	2,998	-
Apparent specific gravity of fine aggregate	g/cm^3	TS EN 1097-6	2,999	-
Apparent specific gravity of filler	g/cm^3	TS EN 1097-7	2,989	-

3.1.2 Aggregate Gradation

The gradation of an aggregate is one of the most significant aggregate distinctiveness in finding how it will perform as a pavement material. In HMA, gradation helps to find nearly all essential properties including stiffness, stability, durability, permeability, workability, fatigue resistance, frictional resistance and moisture susceptibility (Roberts et al., 1996). Therefore, gradation is a principal concern in HMA design and thus most agencies specify allowable aggregate specifications.

The aggregate gradation and grading curve for asphalt mixture were selected in convenience with Turkish Highway Construction Specifications, the details are tabulated in Table (3.2) and Figure (3.1).

Table 3.2 Aggregate Gradation And Specification Limits.

Sieve Opening		**% Passing**		**Design Limit**
Inch	Mm	Min	Max	% Passing
3/4"	19.1	100	--	100
1/2"	12.7	83	100	92
3/8"	9.52	70	90	80
No.4	4.76	40	55	48
No.10	2	25	38	32
No.40	0.425	10	20	15
No.80	0.18	6	15	11
No.200	0.075	4	10	8

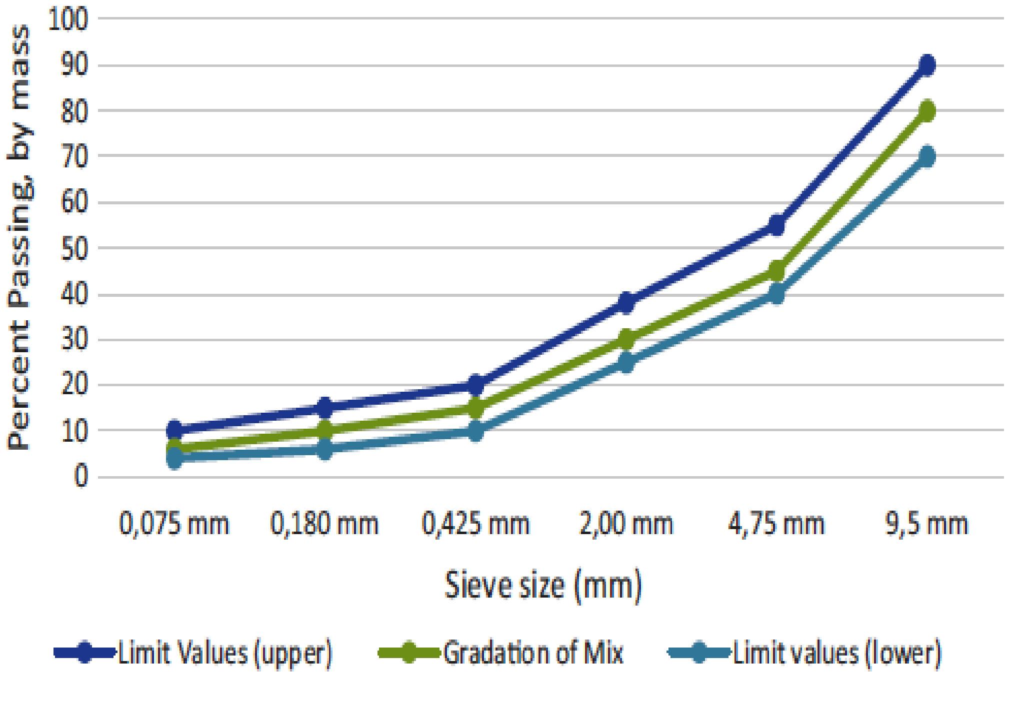

Figure 3.1 Gradation limits.

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3.1.3 Bitumen Binder and Carbon Nanotubes (CNT)

Materials used in the experimental investigation included a neat base binder belonging to the 50/70 penetration grade and a commercially available multiwall CNT obtained by catalytic chemistry vapor deposition. Four different percentages of CNT were chosen to produce bitumen-CNT blends (0.1%, 0.4%, 1.0% and 1.4% by weight of the base binder). A simple shear mixing technique was employed to incorporate CNTs into the base bitumen not only because it is very convenient in laboratory operations, but also because it has the potential of being easily transferred to the industrial scale in hot mix asphalt plants.

The mixing practice followed in the study consisted of two subsequent phases: a first phase in which CNTs were added and manually blended to the bitumen, and a second phase in which the bitumen-CNT blends were mixed with a mechanical stirrer, operating at a speed of 1,550 rpm for a total time of 40 minutes in order to obtain a reasonable dispersion. During both phases of mixing, temperature was kept constant at 160°C.

Properties of bitumen binder such as specific gravity, softening point , penetration and other properties are given in the table (3.3).

Table 3.3 50/70 Grade Bitumen Tests Results.

Tests	**Units**	**Test Method**	**50/70 Bitumen grade**	**Specifications**
Penetration	0,1mm	TS EN 1426	61.0	50 - 70
Softening Point	°C	TS EN 1427	50.4	46 - 54
Frass (max)	°C	TS EN 12593	-12	-8
Flash Point Test (min)	°C	TS EN ISO 2593	332	230
Specific Gravity (d25/25)	g/cm^3	TS EN 15326+A1	1.021	-
Thin Film Heating Loss (Mass Change) (max)	%	TS EN 12607-2	0.3	0.5
RTFOT (Rolling Thin Film Oven Test) (max)	%	TS EN 12607-1	0.5	-

3.2 TEST METHODS FOR EVALUATION OF PERFORMANCE CHARACTERISTICS OF BITUMEN AND BITUMINOUS MIXTURES

3.2.1 Tests On Bitumen Binder

3.2.1.1 Penetration Test

The Penetration value is a measure of hardness or consistency of bituminous material. It is the vertical distance penetrated by the point of a standard needle into the bituminous material under specific conditions of load, time and temperature.

This test is used for evaluating consistency of bitumen. Penetration test is a commonly adopted test on bitumen to grade the material in terms of its hardness. A 50/70 grade bitumen indicates that its penetration value lies between 50 & 70. Grading of bitumen helps to assess its suitability in different climatic conditions and types of construction. The depth of penetration is measured in units of 0.1 mm and reported in penetration units (e.g., if the needle penetrates 8 mm, the asphalt penetration number is 80). The standard test method for penetration of bituminous materials is AASHTO T 49 and ASTM D 5.

As per AASHTO T 49 and ASTM D 5 specifications the material is heated to a pouring consistency at a temperature not more than 90°C and stir until it is homogeneous and free of air bubbles and water.

The sample is cool down in a room temperature between 15° to 30°C for one hour, after that it is placed in the water bath at 25° ± 0.1°C. After conditioning the sample the penetration depth is measured on three different points and under three specific conditions: 100 gm of load, 5 seconds of running time and at temperature of 25°C.

For bituminous macadam and penetration macadam, Indian Roads Congress (IRC) suggests bitumen grades 30/40, 60/70, 80/100. In warmer regions, lower penetration grades are preferred to avoid softening whereas higher penetration grades like 180/200 are used in colder regions to prevent the occurrence of excessive brittleness. High penetration grade is used in spray application works [32].

3.2.1.2 Softening Point Test

This test is performed to determine the softening point of asphaltic bitumen. Softening point is the temperature at which the substance attains a particular degree of softening under specified condition of the test or it is the temperature at which a bituminous sample can no longer resist the weight of a 3.5 gm steel ball. Two horizontal disks of bitumen, cast in shouldered brass rings, are heated at a controlled rate in a liquid bath while each supports a steel ball [32]. The softening point is reported as the mean of the temperatures at which the two disks soften enough to allow each ball, enveloped in bitumen, to fall a distance of 25 mm. The standard test method for softening point test is (AASHTO, 2000) and (ASTM,D36-95).

As per the specifications specimens are prepared exactly in precisely dimensioned brass rings and maintained at a temperature of not less than 10°C below the expected softening point for at least 30 minutes before the test. The rings, assembly and two ball bearings, are placed in a liquid bath filled to a depth of 105 ± 3 mm and the whole maintained at a temperature of 5 ± 1°C for 15 minutes. [Freshly boiled distilled water is used.

The temperature at which each bitumen specimen touches the base plate is recorded to the nearest 0.2°C. The mean temperature of the two specimens (which shall not differ by more than 1°C) is recorded as the softening point.

Figure 3.2 Major penetration testing.

Figure 3.3 Softening point testing apparatus.

3.2.1.3 Super-pave Binder Tests

The super-pave binder tests and the supporting test procedures are outcomes of the Strategic Highway Research Program (SHRP), a 5-year research effort from 1987 to 1992 which targeted $50 million for asphalt research. SHRP researchers designed the binder specification to address the asphalt's contribution to three failures seen in asphalt concrete pavement. These distresses are rutting, fatigue and thermal cracking or low-temperature cracking (AASHTO M320). Performance related tests were developed or adapted to address these distresses. The idea is that the distresses are related to the climate in which the roadway exists.

Grade Selection

To specify a performance graded asphalt binder, one needs to determine the temperature extremes under which the pavement must perform. A grade is determined by indicating the high and low temperatures for performance. As an example, we expect PG 64-22 to perform at a high temperature of 64°C and a low temperature of -22°C. The grading system uses increments of 6°C for the high and low temperature designation.

The high temperature designation represents the 7-day average high pavement temperature. The low temperature designation represents a single occurrence low pavement temperature.

3.2.1.3.1Rolling Thin Film Oven Test (RTFOT)

Asphalt binders age primarily due to two different mechanisms: loss of light oils present in the asphalt (volatilization) and reaction with oxygen from the environment (oxidation). During manufacturing of asphalt concrete in the hot mixing facility and during lay down and construction, binders age due to both mechanisms which are the high temperature and air flow involved in the process. The rolling thin film oven (RTFO) is used to simulate this form of aging.

The RTFO is used because it is repeatable and continually exposes fresh binder to heat and air flow. Its rolling action, in some cases, keeps modifiers (e.g., some polymers) dispersed in the asphalt. Another advantage of the RTFO is that it takes only 85 minutes to perform.

This test serves two purposes: one is to provide an aged asphalt product that can be used for further testing of physical properties. The second is to determine the mass quantity of volatiles lost from the asphalt during the test [32].

The RTFO procedure requires an electrically heated convection oven. The oven contains a vertical circular carriage that contains holes to accommodate sample bottles. The carriage is mechanically driven and rotates about its center. The oven also contains an air jet that is positioned to blow air into each sample bottle at its lowest travel position while being circulated in the carriage. The standard test method for the effect of Heat and Air on a Moving Film of Asphalt (Rolling Thin-Film Oven Test) is AASHTO T 240 and ASTM D 2872.

Figure 3.4 RTFO Apparatus.

According to the test specifications the sample is heated until it is fluid to pour. The sample is stirred to ensure homogeneity and remove air bubbles. If a determination of mass change is desired, two RTFO bottles are labeled and weighed them empty. These are designated as the “mass change” bottles. The weights are recorded (35 g) of asphalt binder are poured into each bottle. The bottles are cool down for 60 to 180 minutes. After cooling, the weights of the two mass change bottles are recorded again.

The bottles are placed in the RTFO oven carousel and rotates at 15 RPM for 85 minutes. During this time, the oven temperature is maintained at 325°F (163°C) and the airflow into the bottles at 244 in3/min (4000 ml/min). The RTFO residue should be tested within 72 hours of aging. After cooling the two mass change bottles for 60 – 180 minutes, they are weighted and the residues are discarded. The weights are again recorded.

Measured Parameters

- The mass change of a specimen as a percent of original mass.
- The RTFO is mainly used to simulate short term bitumen binder aging for use in other tests.

The RTFO Specification For The Performance Graded Asphalt

Materials	Value	Specification	Property of Concern
Un-aged binder	Mass loss[1]	≤ 1.0%	None

Normallythe mass loss value is in the range of 0.05 to 0.5 percent.

3.2.1.3.2 Dynamic Shear Rheometer Test (DSR)

The Dynamic Shear Rheometer (DSR) (Figure 3.3) characterizes the viscous and elastic manners of bitumen binders at average to high temperatures. The characterization is used in the Super-pave PG asphalt binder specification. The real temperatures expected in the area where the asphalt binder will be placed, determine the test temperatures used.

The basic DSR test uses a thin asphalt binder sample (Figure 3.4) sandwiched between two circular plates. The lower plate is fixed while the upper plate oscillates back and forth across the sample at 10 rad/sec (1.59 Hz) to create a shearing action (Figure 4). DSR tests are conducted on un-aged, RTFO aged and PAV aged asphalt binder samples. The test is largely software controlled. The standard for DSR test is AASHTO T 315.

The DSR measures a specimen's complex shear modulus (G*) and phase angle (δ). The complex shear modulus (G*) can be considered as the sample's total resistance to deformation when repeatedly sheared, while the phase angle (δ), is the lag between the applied shear stress and the resulting shear strain (Figure 5). The larger the phase angle (δ), the more viscous the material. Phase angle (δ) limiting values are:

- Purely elastic material: $\delta = 0$ degrees
- Purely viscous material: $\delta = 90$ degrees

In the Super-pave asphalt specification, permanent deformation (rutting) is controlled by requiring the $G^*/\sin\delta$ of the binder at the highest anticipated pavement temperature to be greater than 1.0 kpa before aging and 2.2 kpa after the RTFO process. Fatigue cracking is controlled by requiring that a binder after PAV should have a $G^*\sin\delta$ value of less than 5000 kpa at a specified intermediate pavement temperature [32].

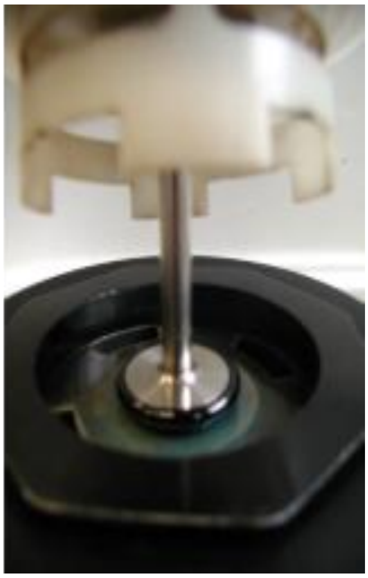

Figure 3.5 The Dynamic Shear Rheometer (DSR).

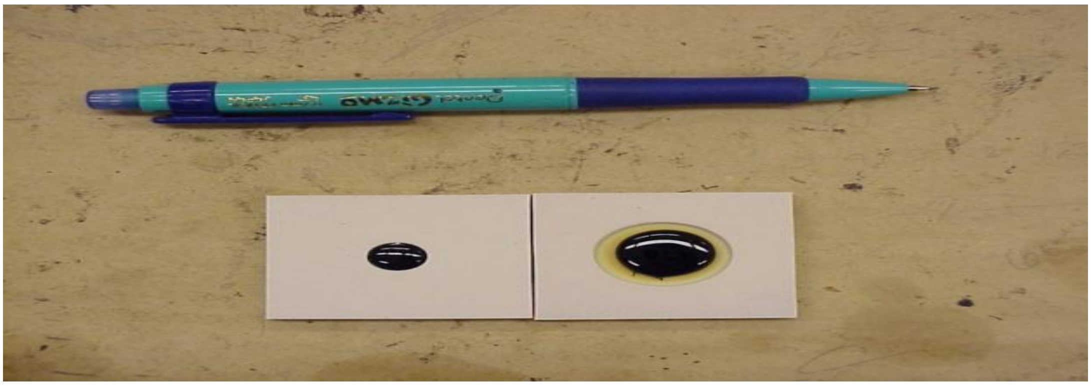

Figure 3.6 DSR sample for testing.

Measured Parameters:

1. Complex modulus (G*)

2. Phase angle (δ)

The DSR Specifications of Performance Graded Asphalt Binder

Material	Value	Specification	HMA Distress of Concern
Un-aged binder	G*/sinδ	≥ 1.0 kPa (0.145 psi)	Rutting
RTFO residue	G*/sinδ	≥ 2.2 kPa (0.319 psi)	Rutting
PAV residue	G*sinδ	≤ 5000 kPa (725 psi)	Fatigue cracking

The complex modulus (G*) can range from about 0.07 to 0.87 psi (500 to 6000 Pa), while the phase angle (δ) can ranges from about 50 to 90°. A δ of 90° is essentially complete viscous behavior. Polymer-modified asphalt binders generally exhibit a higher G* and a lower δ. This means they are, in general, a bit stiffer and more elastic than unmodified asphalt cements [32].

3.2.1.3.3 Bending Beam Rheometer Test (BBR)

Bending Beam Rheometer (BBR), test is used to measure the stiffness of asphalt binders at low surface temperature. For specification testing the test samples are fabricated from PAV-aged asphalt binders and tested at 10^{O}C above the expected minimum pavement temperature, (Tmin). The Super-pave binder specification requires the stiffness at the test temperature after 60 seconds to be less than 300 Mpa to control low temperature cracking and m-value at 60 seconds to be greater than or equal to 0.30.

Since one of the greatest factors that may affect the performance characteristics of asphalt mixtures is the asphalt binder's properties so asphalt binders that have excellent properties may control permanent deformation and fatigue cracking. In order to know the influences of carbon nano-tube addition on mass change with short term aging, and effect on long term aging, fatigue cracking and stiffness, four different percentages of carbon nano-tube (0.1%, 0.4%, 1.0% and 1.4%) by weight of base binder were added and the properties investigated.

3.2.1.4 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM), analysis or SEM microscopy, is used incredibly efficiently in microanalysis and failure analysis of solid materials. Scanning electron microscopy is achieved at higher enlargements, generates high-resolution images and accurately measures very small features and substances. In order to know such such micromechanical characteristics of CNT in asphalt, the microsturcture and morphology of fracture surfaces of CNT-modified samples were studied using the SEM.

3.2.2 Tests On Bituminous Binders

3.2.2.1 Mix Design

In The purpose of mix design is to create an economical optimum mixture of component materials for a given function. This includes thorough evaluations of aggregate, asphalt and cement as well as the determination of their optimum blending ratios. In the developing of this blend the designer needs to think of both the first cost and the life cycle cost of the project. Considering only the first cost may result in a higher life cycle cost. The HMA mixture which is placed on the highway should meet certain requirements.

- The mix must have enough asphalt to ensure a durable, compacted pavement by thoroughly coating, bonding and waterproofing the aggregate.

- Sufficient stability to satisfy the demands of traffic without dislocation or distortion (rutting).

- Adequate amount of voids to allow a minor amount of added compaction under traffic loading without bleeding and loss of stability. However, the volume of voids should be low enough to keep out harmful air and moisture. To achieve this the mixes are usually designed by 4% VTM in the lab and compacted to less than 7% VTM in the field.

- Enough workability to allow placement and proper compaction without segregation.

Asphalt mix design has been accomplished using the following four methods:

- Marshall method
- Hveem method
- Superpave method
- GTM method

Out of these four methods the most common and widely used one in Turkey and across the world is the Marshall mix design method. It has been used in about 75% mix design applications throughout the US. In 1995 the Superpave mix design procedure was introduced into use. It builds on the knowledge from Marshall and Hveem procedures. The primary differences between the three procedures is the machine used to compact the specimens and strength tests used to evaluate the mixes. Due to the reliability that is gained by adopting Marshall method, it is decided to use the Marshall Mix Design method.

3.2.2.1.1 Marshall Mix Design

This test procedure is used in designing and evaluating bituminous paving mixes and is extensively used in routine (usual or practical) test programs for the paving jobs. There are two major features of the Marshall method of designing mixes namely:

- Stability - Flow test and
- Density - Voids analysis

Strength is measured in terms of the 'Marshall's Stability' of the mix following the specification ASTM D 1559 (2004), which is defined as the maximum load carried by a compacted specimen at a standard test temperature of 60˚C. In this test compressive loading was applied on the specimen at the rate of 50.8 mm/min till it was broken. The temperature 60˚C represents the weakest condition for a bituminous pavement [33].

The flexibility is measured in terms of the 'flow value' which is measured by the change in diameter of the sample in the direction of load application between the start of loading and at the time of maximum load. During the loading, an attached dial gauge measures the specimen's plastic flow (deformation) due to the loading. The associated plastic flow of specimen at material failure is called flow value. The density- voids analysis is done using the volumetric properties of the mix, which will be described in the following sub sections.

3.2.2.1.2 Volumetric Properties

Fundamentally mix design is meant to determine the volume of bitumen binder and aggregate necessary to produce mixture with the desired properties (Roberts et al., 1996). Since weight measurements are typically much easier, weights are taken and then converted to volumes by using specific gravities. Below is the discussion of the important volumetric properties of bituminous mixtures:

1) The theoretical maximum specific gravity Gmm, Gt
2) The bulk specific gravity of the mix Gmb, Gm
3) Percentage air voids VA, Vv(%)
4) percentage volume of bitumen Vb(%)

5) percentage void in mineral aggregate VMA(%)
6) Percentage voids filled with bitumen VFB(%)

THEORITICAL SPECIFIC GRAVITY (Gt): The specific gravity without considering the air voids , expressed as :

$$Gt = \frac{w1 + w2 + w3 + wb}{\frac{w1}{G1} + \frac{w2}{G2} + \frac{w3}{G3} + \frac{wb}{Gb}}$$

Where:

w_1 = weight of coarse aggregate in the total mix
w_2 = weight of fine aggregate in total mix
w_3 = weight of filler in the total mix
w_b = weight of bitumen in total mix
G_1 = the apparent specific gravity of coarse aggregate
G_2 = the apparent specific gravity of fine aggregate
G_3 = the apparent specific gravity filler
G_b = the apparent specific gravity of bitumen

BULK SPECIFIC GRAVITY OF MIX (Gm): The specific gravity considering air voids is expressed:

$$G_m = \frac{Wm}{Wm - Ww}$$

W_m = the weight of mixture in air
W_w = the weight of mixture in water

AIR VOIDS PERCENTAGE Vv(%): It is the percentage of air voids by volume in the sample and is expressed:

$$Vv = \frac{(Gt - Gm) * 100}{Gt}$$

G_t = the theoretical specific gravity of the mix,
G_m = the bulk specific gravity of the mix.

VOLUME OF BITUMEN PERCENTAGE Vb(%): The percentage of volume of bitumen to the total volume of the sample and is expressed as:

$$Vb = \frac{\frac{Wb}{Gb}}{\frac{W1+W2+W3+Wb}{Gm}}$$

W1, W2, W3 and Wb are the weights of coarse aggregate, Fine aggregates, Filler and weight of bitumen in the total mix respectively.

Gb = apparent specific gravity of bitumen,

Gm = bulk specific gravity of mixture.

VOIDS IN MINRAL AGGREGATES PERCENTAGE VMA(%): voids in mineral aggregate percentage VMA % is the volume of voids in the aggregates, and it is the addition of air voids and volume of bitumen, and is equal to:

$$VMA(\%) = Vv(\%) + Vb(\%)$$

Vv(%) = is the air voids percentage in the mixture,

Vb = is the volume of bitumen percentage in the total mixture.

VOIDS FILLED WITH BITUMEN PERCENTAGE VFB (%): VFB % is the voids in mineral aggregate filled with bitumen and it represent effective bitumen content, it is inversely related to air voids and it is calculated as:

$$VFB = \frac{Vb * 100}{VMA}$$

Vb = is the percentage content in the mixture

VMA = is the percentage of voids in the mineral aggregate

The decrease of VFB indicates a decrease of effective bitumen film thickness between aggregates, which will result in higher low-temperature cracking and lower durability of bitumen mixture since bitumen perform the filling and healing effects to improve the flexibility of mixture.

3.2.2.1.3 Role of Volumetric Parameters of Mix

Bitumen holds the aggregates in position, and the load is taken by the aggregate mass through the contact points. If all the voids are filled with bitumen, the one to one contact of the aggregate particles may lose, and then the load is transmitted by hydrostatic pressure through bitumen, and hence the strength of the mix reduces. That is why stability of the mix starts reducing when bitumen content is increased further beyond a certain value.

During summer season, bitumen softens and occupies the void space between the aggregates and if void is unavailable, bleeding is caused. Thus, some amount of void is necessary in a bituminous mix, even after the final stage of compaction. However excess void will make the mix weak from its elastic modulus and fatigue life considerations.

Table 3.4 Design specification for wearing course used by KGM-Ankara-2012.

Design Parameter	Wearing Course Specifications	
	Min.	Max.
Blow No.	75	
Stability (Kg)	900	-
Flow (mm)	2	4
Air Void (%)	3	5
Voids Filled With Bitumen VFB(%)	65	75
Filler/Bitumen Content	-	1.5
Bitumen Content	4	7
Void In Mineral Aggregate VMA(%)	14	-

PREPARATION OF SAMPLES:

- 1150 gm of aggregate and filler are heated to a Temperature of 160-170˚C.
- At the same time Bitumen binder is heated to a temperature of 145-155˚C.

- Now a particular percentage of binder (3.5% by weight of mineral aggregate is mixed thoroughly (by Marshall Mixer) with aggregate mix at 135˚C.
- After mixing the mix is poured into a preheated-mould and then compacted by Marshall-compactor , which has a specific weight of 4.5 Kg, and falls-down from a height of 45.7 cm.
- Each side of the specimen is compacted with 75 blows at a temperature of 120˚C. (to prepare a laboratory specimen of 63.5±3mm and diameter of 101.5±1mm.
- After compaction the samples are extracted with special extractor and now in order to determine the Volumetric Properties ,each sample's height is measured(three heights are taken for each sample and then the AVG is counted as the real one).

Figure 3.7 Standard Marshall Samples Prepared For Testing.

Three samples of each % of the bitumen content is produced and then the AVG is taken as actual value. The bitumen binder content is changed and again the above procedure is repeated , Usually the binder is increased with an increment of 0.5%.

Figure 3.8 Marshall Mix Design Major Apparatus.

3.2.2.1.4 Marshall Stability and Flow Determination

When the samples are ejected from the moulds (after compaction and cooling), and once the specimen heights and weights (weights in air, water and saturated dried) are taken , then we place the specimen inside a particular Water-Bath having inside Temperature of 60˚C for about 30±5 mints. After the bath period is completed we take the sample and placed inside the Breaking-Head and then the load is applied at a constant rate of 50.8mm/min (Figure 3.9).

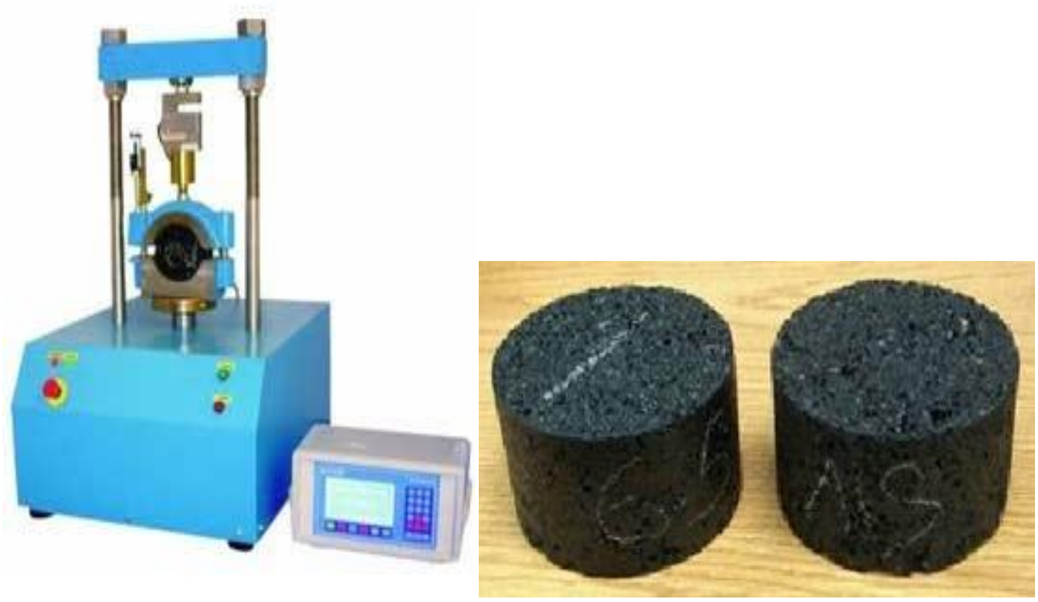

Figure 3.9 Marshall Stability Apparatus and Standard Marshall Samples For Testing.

When the stability test is in progress a dial gauge is used to measure the vertical - deformation of sample, which is actually the measurement of flow.

The deformation at failure point expressed in units of 0.25mm is called the Marshall-Flow value of the specimen. Stability value is that (maximum) load, which just produce failure or break the sample. After the Stability and Flow values are found-out, we check them with the Marshall Mix Design specification chart.

Mixes with high (very high) stability values and low flow values are not suitable, because the pavements constructed with such mixes are most likely to develop Cracks due to heavy moving loads.

Finding Out The Optimum Bitumen Content:

To determine the OBC six graphs are sketched-out , these graphs are

1) Bulk Specific Gravity vs Bitumen Content
2) Voids Content (%) vs Bitumen Content
3) Stability (KN) vs Bitumen Content
4) Flow (mm) vs Bitumen Content
5) Voids filled with Bitumen vs Bitumen Content
6) Voids filled with Aggregates vs Bitumen Content

After these graphs are plotted, the Bitumen Content that corresponds to MAXIMUM BULK SPECIFIC GRAVITY, MAXIMUM STABILITY and the bitumen content that corresponds to 4% AIR VOIDS are figure-out and then the average of these values are taken as out OPTIMUM BITUMEN CONTENT.

The average (which is the optimum binder %) is compared with the specifications of the remaining volumetric properties, if it is in the range of standard specifications (given in table attached) , then it is decided to be the OPTIMUM BITUMEN CONTENT, if so it is discarded and the test should be repeated.

The same procedure is followed as explained above for our experiments, the Optimum Bitumen Content was found for the proposed mix design gradation. Three samples were prepared for each % of bitumen content within the range of 3.5% - 6.0% and at 0.5% increments, according to (ASTM D 1559) using 75 blows of compaction per each side of the specimen.

Following is the average mix design data of each bitumen content percentage, OPTIMUM BITUMEN CONTENT.

Table 3.5 The Average mix design data of each bitumen content.

Sample No	Bit. %	Weight of Bitu. gm	Volume cm3	Bulk Sp.Gr (Gm)	Th.Sp. Gr (Gt)	Air Void Vv%	VAB %	VMA %	VFB %	Flow Corr. mm	St. Corr. Kn	Flow mm	St. Kn
1	3.5	40.3	526.2	2.40	2.73	12.62	7.55	20.59	38.68	2.470	7.81	2.90	8.03
2	4	46	513.2	2.44	2.71	10.41	9.21	19.64	46.94	3.142	9.13	3.87	8.88
3	4.5	51.75	503.6	2.45	2.64	8.97	10.34	19.30	53.64	3.508	8.59	3.81	8.36
4	5	57.5	494.8	2.52	2.61	6.61	11.78	18.36	64.08	3.550	8.23	3.57	7.82
5	5.5	63.25	489.9	2.55	2.60	4.20	13.03	17.24	75.83	5.409	8.20	3.57	7.52
6	6	69	487.2	2.51	2.59	5.18	13.94	19.12	73.01	8.110	9.10	8.10	8.35

from the graphs above it can be find:

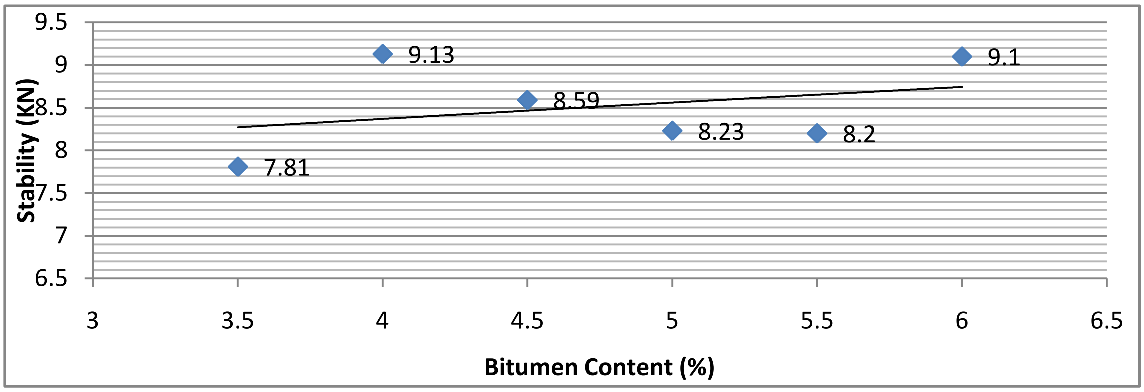

Figure 3.10 Stability vs Bitumen Content.

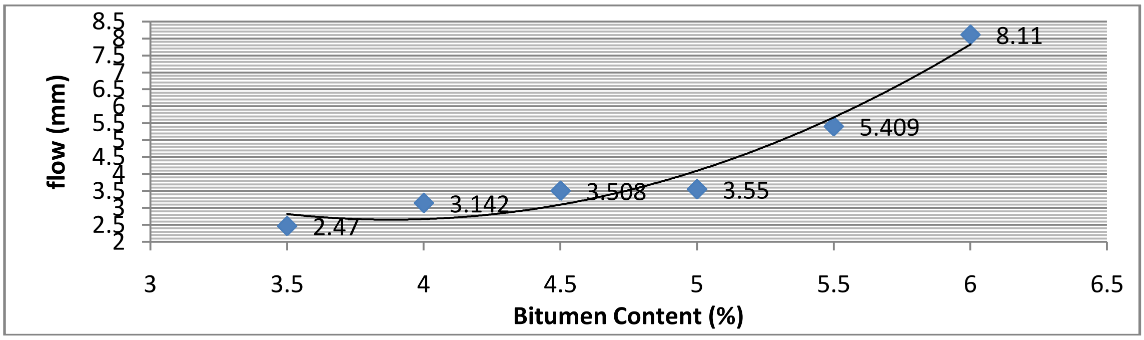

Figure 3.11 Flow vs Bitumen Content.

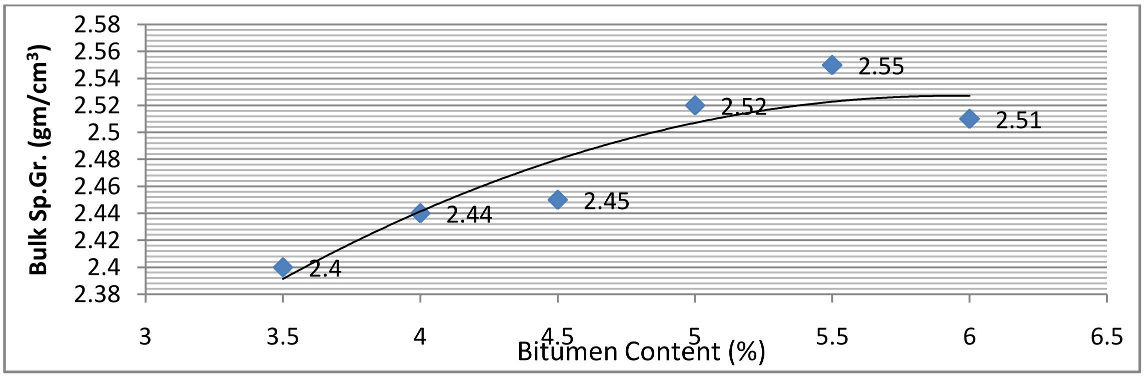

Figure 3.12 Bulk Specific Gravity vs Bitumen Content.

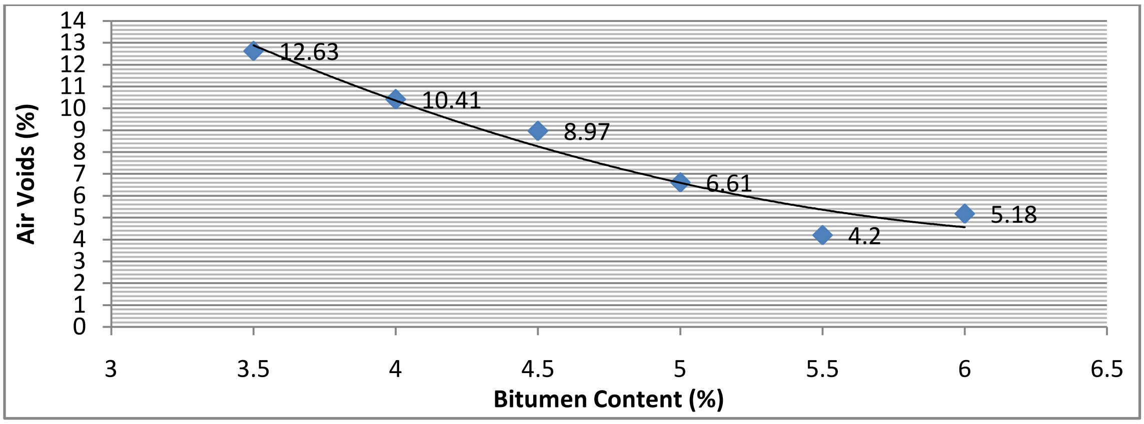

Figure 3.13 Void Content vs Bitumen Content.

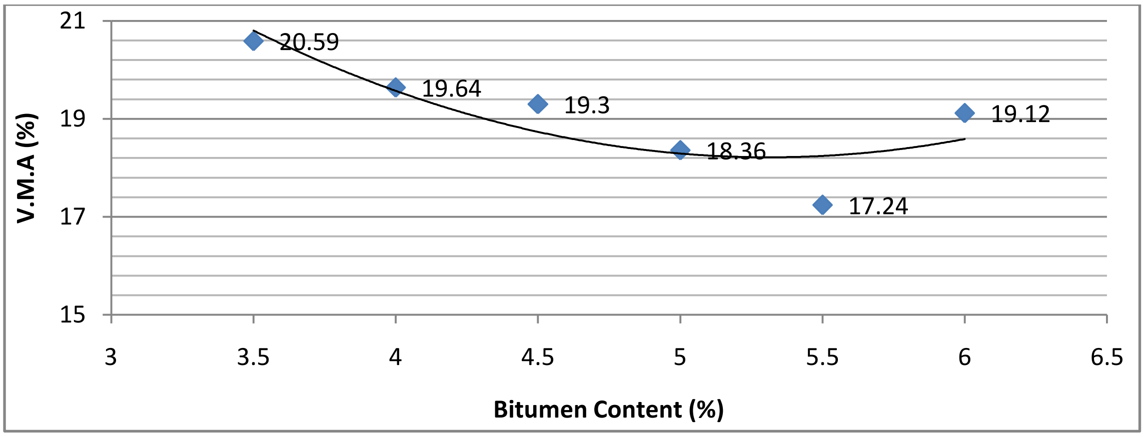

Figure 3.14 VMA% vs Bitumen Content.

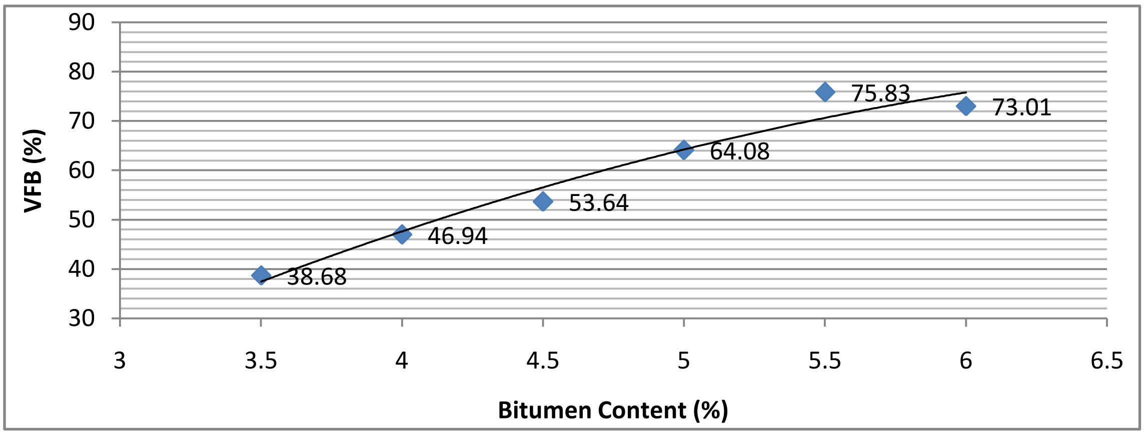

Figure 3.15 VFB% vs Bitumen Content.

1) Maximum stability which is 9.13kN corresponds to 4.05% of Bitumen Content.

2)Maximum bulk specific gravity at 2.55 corresponds to 5.5% of Bitumen Content.

3) 4(%) of Air voids corresponds to 5.55% of Bitumen Content. and also

And the average bitumen content =5.03%

Now we have to check for acceptance with specifications which are given in Table 3.4:

At 5.03% of B.C the flow=3.8 mm which falls within limits (2-4).

At 5.03% of B.C the VFB = 66% which falls within limits (65-75).

At 5.03% of B.C the VMA = 18.2%. which falls within limits (min 14%).

So the Optimum bitumen content is for this type of aggregates is = 5.03

After determining the optimum bitumen content (5.03%), the Marshall Stability and Flow test was carried out at optimum bitumen content modified with different percentages of carbon nanotubes (0%, 0.1%, 0.4%, 1.0% and 1.4% by weight of bitumen). The results of these tests can be seen from table (B.1), appendix B.

3.2.2.2 Determining The Compaction Effort At 7%±1% Air Voids For Indirect Tensile Strength Test

The standards suggest of caring out tests on bituminous samples at 7±1% air voids, in order to find out the effect of moisture susceptibility on hot bituminous mixtures. In this study different specimens were prepared at optimum bitumen content and with applying different compaction efforts changes in void contents were investigated.

For this aim mixtures were prepared and compacted with 30, 40, 50, 60 and 75 blows on each side. For each compaction effort 3 samples were prepared and void content investigated. Table (3.5) shows the details.

Table 3.6 Details of Samples prepared at 5.07% Bitumen Content.

Blow No	Sample No	Weight Of Bitumen (gm)	Weight Of Mixture (gm)	Weight In Air (gm)	Weight In Water(gm)	S.S.D. Weight In Air(gm)	Bulk. Sp. Gr (Gm)	Th. Sp. Gr (Gt)	Air Void %Vv
30	1	57.845	1207.845	1201.50	714.30	1210.80	2.42	2.601	10.51
	2	57.845	1207.845	1217.98	725.30	1222.17	2.45	2.601	9.35
	3	57.845	1207.845	1205.60	718.50	1214.20	2.43	2.601	10.05
	Average						2.43	2.601	9.97
40	1	57.845	1207.845	1212.34	726.85	1214.80	2.48	2.601	8.12
	2	57.845	1207.845	1202.36	717.80	1204.93	2.47	2.601	8.72
	3	57.845	1207.845	1204.80	719.90	1205.30	2.48	2.601	8.21
	Average						2.48	2.601	8.35
50	1	57.845	1207.845	1199.27	720.80	1200.58	2.50	2.601	7.56
	2	57.845	1207.845	1208.82	728.10	1210.17	2.51	2.601	7.26
	3	57.845	1207.845	1204.34	722.38	1206.44	2.49	2.601	7.99
	Average						2.50	2.601	7.60
60	1	57.845	1207.845	1203.88	726.10	1205.99	2.51	2.601	7.22
	2	57.845	1207.845	1205.95	723.70	1207.16	2.49	2.601	7.75
	3	57.845	1207.845	1205.58	725.30	1206.74	2.50	2.601	7.39
	Average						2.50	2.601	7.46
75	1	57.845	1207.845	1207.88	732.80	1208.82	2.54	2.601	6.16
	2	57.845	1207.845	1204.88	731.40	1206.06	2.54	2.601	6.13
	3	57.845	1207.845	1204.20	730.50	1205.30	2.54	2.601	6.20
	Average						2.54	2.601	6.16

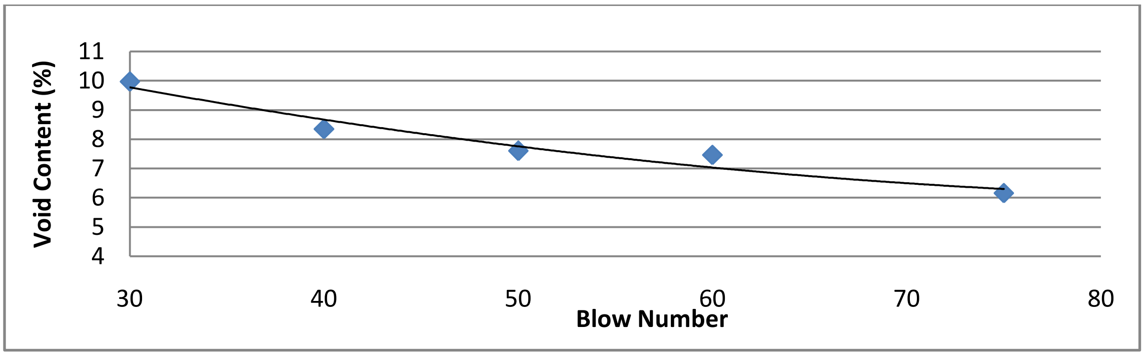

Figure 3.16 Relationships Between Void Content And Blow Number.

From Table 3.6 and Figure 3.12 the compaction effort (blow number) of 60 blows, which produces the void content of 7.4% is selected as the compaction effort.

3.2.2.3 Indirect Tensile Strength And Retained Stability Tests

The presence of moisture or water in hot mix asphalt mixtures, which is the combination of various materials is a considerable aspect, that can lead the flexible pavement to fail. As the Asphalt binder is the glue , which hold the aggregates together. If the asphalt could not stick to the aggregates firmly a rapid failure of the pavement can be expected. The adhesion loss of aggregates with asphalt binder is studied by utilizing retained stability and indirect tensile strength test. Stripping resistance is evaluated with these tests for bituminous mixtures. The test is specified in IRC: SP 53-2002, ASTM D 1075-1979 and also AASHTO T 283 specification [32].

3.2.2.3.1 Retained Marshall Stability Test

The retained stability test is conducted mostly in US as an alternative to determine the effect of moisture on the bituminous mixtures. The test consist of preparing the standard Marshall specimens at optimum bitumen content and after that testing them in dry (standard Marshall testing procedure) and wet condition (after 24 hours of saturation at 60 centigrade inside the water-bath) using the Marshall stability apparatus. The Stability and flow tests for retained stability is exactly the same as it is for the standard Marshall stability and flow tests. The retained stability is expressed as the stability after conditioning the samples in water-bath for 24 hours divided by stability under standard conditions and multiplied by 100 [34], [35].

$$\text{Retained stability} = \frac{\text{Soaked stability}}{\text{Standard stability}} * 100$$

THE ADDITION OF CARBON NANOTUBE TO BITUMEN

In this study four different percentages of Carbon Nano Tube (CNTs) were chosen to produce bitumen-CNT blends (0.1%, 0.4%, 1.0% and 1.4% by weight of the base binder). Table (B.2) to table (B.6) shows the details of retained stability index and also compared to control samples (the samples produced with 0% CNTs at the same condition of mixtures that also contains CNTs).

Figure 3.17 Carbon Nanotube Before Mixing With Bitumen.

Fig. 3.18 Carbon Nanotube After Mixed WithBitumen.

3.2.2.3.2 Indirect Tensile Strength Test

The values of IDT strength may be used to evaluate the relative quality of bituminous mixtures in conjunction with laboratory mix design testing and for estimating the potential for rutting or cracking. The results can also be used to determine the potential for field pavement moisture damage when results are obtained on both moisture-conditioned and unconditioned specimens.

The indirect tensile test involves loading a cylindrical specimen with compressive loads which act parallel to and along the vertical diametrical plane, as shown in (Figure 3.19). To distribute the load and maintain a constant loading area, the compressive load is applied through a ha1f-inch-wide stainless steel loading strip which is curved at the interface with the specimen and has a radius equal to that of the specimen. This loading configuration develops a relatively uniform tensile stress perpendicular to the direction of the applied load and along the vertical diametrical plane, which ultimately causes the specimen to fail by splitting or rupturing along the vertical diameter as shown in (Figure 3.20). The tensile stress in the center of the specimen can be calculated using the following equation [36].

$$\mathrm{St} = \frac{2P}{\pi \mathrm{dt}}$$

St: Indirect Tensile Stress;

d: Diameter of Specimen, (inches)

P: Vertical Load applied to Specimen, (Pounds)

h = height of specimen at beginning of test, (inches)

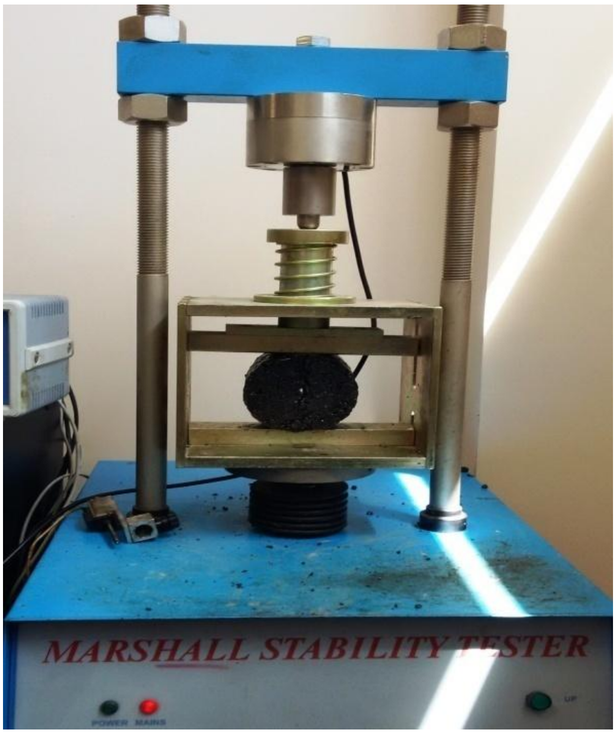

Figure 3.19 Indirect Tensile Strength Apparatus During Testing.

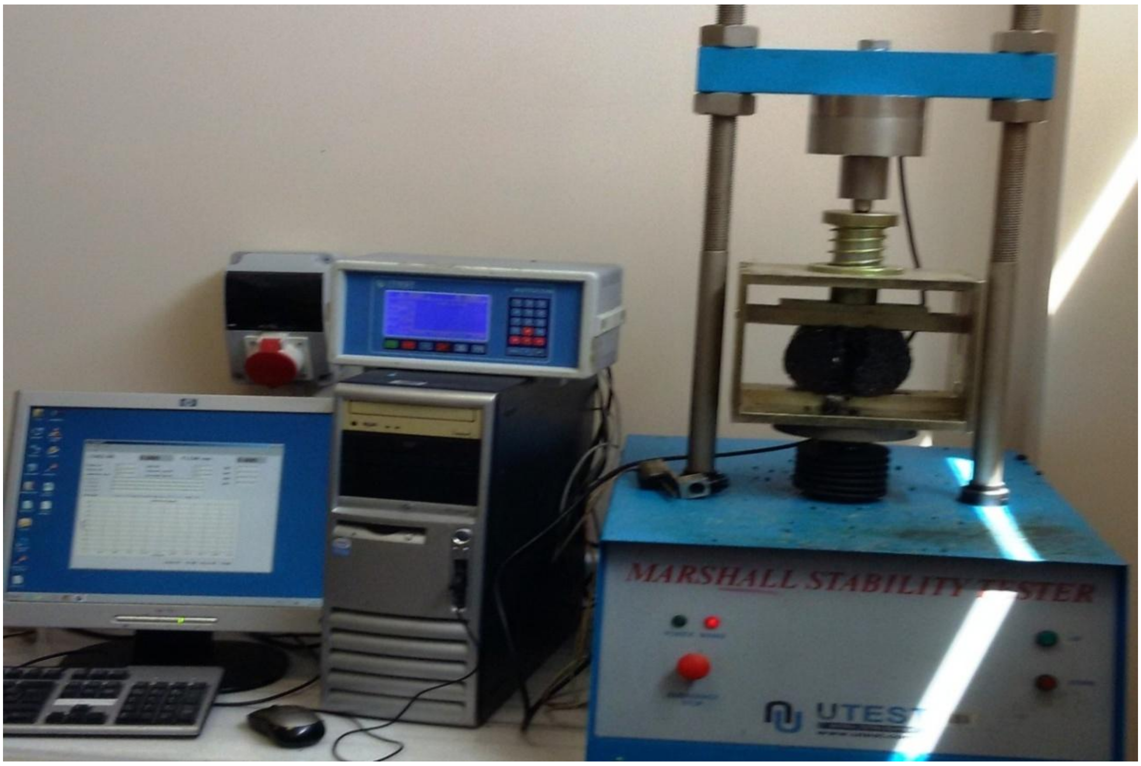

Figure 3.20 Indirect Tensile Strength Test After The Sample Has Broken.

The Indirect tensile strength test is conducted by making standard Marshall samples of (100 mm diameter and 63.5 mm height) at optimum bitumen content and compacting each side of the specimen with 60 blows, in order to get an air void content of 7%±1%.

THE ADDITION OF CARBON NANOTUBE TO BITUMEN

In this study four different percentages of Carbon Nano-tube (CNTs) were chosen to produce bitumen-CNT blends (0.1%, 0.4%, 1.0% and 1.4% by weight of the base binder). Table (B.7) to table (B.11) shows the details of indirect tensile strength and tensile strength ratios.

CHAPTER 4

DATA ANALYSIS AND RESULTS

4.1 OPTIMUM BITUMEN CONTENT, VOLUMETRIC PROPERTIES AND RETAINED STABILITY

The aggregates used in this study were brought from one of the prominent gravel and asphalt construction site of Turkey. The Turkish gradation of aggregates (KGM-Ankara-2008), was followed with the nominal designed size of 12.5mm and the minimum size of 0.075mm. The aggregate gradation specification selected can be seen from Table (3.2).

The next step to progress was the determination of optimum bitumen content for the asphalt concrete mixtures. Five different percentages of bitumen binder by weight of aggregate were selected (3.5%, 4%, 4.5%, 5% and 5.5%), after testing it was found that with the increase in amount of bitumen binder the void content decreased, stability increased and also the flow reduced, (figure 4.1) . The whole procedure that led in finding and selecting the amount of optimum bitumen content is well illustrated from figure (3.7) to figure (3.12), it was found that 5.03% has been the optimum bitumen content. The wearing course specification limits specified for design (stability, flow and void content) are tabulated in table (3.4).

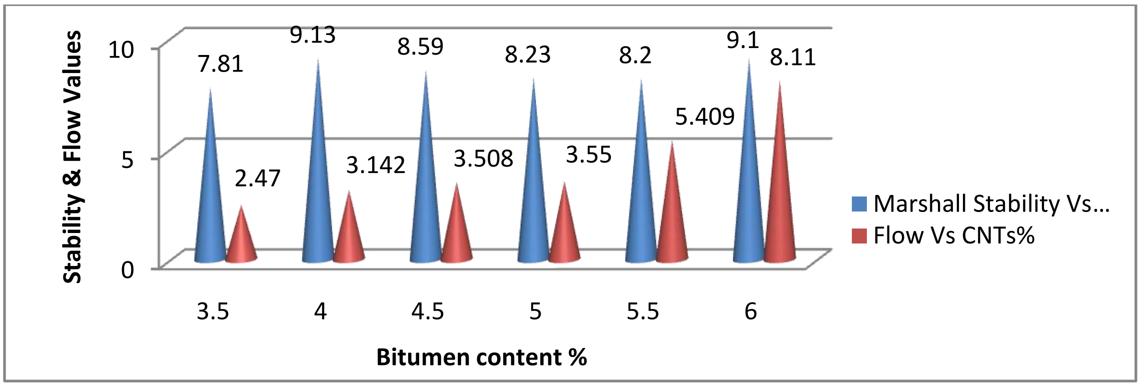

Figure 4.1 Stability & Flow Values For Determining The Optimum Bitumen Content.

Marshall stability, measures the maximum load sustained by the bituminous material at a loading rate of 50.8 mm/minute and at 60oC. It is related to the resistance of bituminous materials to distortion, displacement, rutting and shearing stresses. The stability is derived mainly from internal friction and cohesion. Cohesion is the binding force of binder material while internal friction is the interlocking and frictional resistance of aggregates. As bituminous pavement is subjected to severe traffic loads from time to time, it is necessary to adopt bituminous material with good stability and flow. Marshall flow is a measure of deformation (elastic plus plastic) of the asphalt mix determined during the stability test. Excessive amount of flow indicates that pavement will experience permanent deformation under traffic loading.

The amount of binder to be added to a bituminous mixture should not be too excessive or too little. The principle of designing the optimum amount at a void content of 4% is to include sufficient amount of binder so that the aggregates are fully coated with bitumen and the voids within the bituminous material are sealed up. As such, the durability of the bituminous pavement can be enhanced by the impermeability achieved. Moreover, a minimum amount of binder is essential to prevent the aggregates from being pulled out by the abrasive actions of moving vehicles on the carriageway. However, the binder content cannot be too high because it would result in the instability of the bituminous pavement. In essence, the resistance to deformation of bituminous pavement under traffic load is reduced by the inclusion of excessive binder content.

The effect of moisture on asphalt mixtures have been investigated for many years , using various additives, but on macro scale tests. The moisture effects in asphalt is mostly associated with asphalt chemistry and adhesion characteristics, which are below the micron scale phenomenon.

In this research study asphalt chemistry and adhesion values were evaluated in nano-scale in order to determine the moisture resistance of asphalt mixtures modified with carbon nanotube (CNTs). Carbon nanotube are molecular graphitic carbon tubes that possess brilliant properties and believed to be one of the strongest and stiffest materials ever tested with asphalt binders.

The standard Marshall samples of 100 mm diameter and 63.5mm height at optimum bitumen content were prepared with carbon nanotube modified asphalt binder. Four samples (two conditioned and two un-conditioned) from each CNTs percentages (0.1%, 0.4%, 1.0% and 1.4%) were prepared and the effects of CNTs were investigated through standard Marshall tests and retained Marshall tests. The details and results of these tests are presented in table (B.2) to table (B.6) in appendix (B).

The results display the differences that are made with the addition of CNTs in the values of Marshall stability & flow and as well as in the values of retained stability index (RSI). RSI for control samples was (58.12%), with the addition of 0.1% CNTs the RSI increased to (60.86%), with 0.4% of CNTs the RSI increased to (68.44%), with 1.0% CNTs the RSI amount increased to (74.57%) and with the addition of 1.4% CNTs the RSI goes back to (52.66%). The CNTs content versus the RSI% are clearly presented in Figure (4.1).

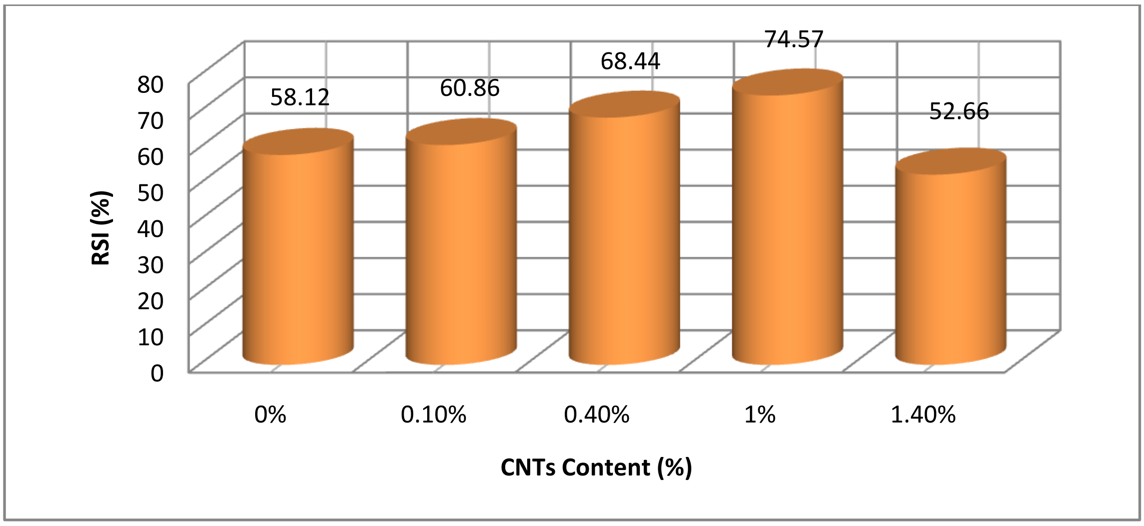

Figure 4.2 RSI Values For Various CNT Percentages Added To Bitumen.

4.2 INDIRECT TENSILE STRENGTH AND TENSILE STRENGTH RATIO

The tensile properties of bituminous mixtures are of interest to pavement engineers because of the problems associated with cracking. HMA is not as strong in tension as it is in compression. The indirect tensile strength test (IDT) is used to determine the tensile properties of the bituminous mixture which can further be related to the cracking properties of the pavement. Tensile strength depends on the cohesion of asphalt and the adhesion between asphalt binder and aggregates. A higher tensile strength means better resistance to fatigue and thermal cracking. At the same time, mixtures that are able to tolerate higher strain prior to failure are more likely to resist cracking than those unable to tolerate high strains (Tayfur et al., 2007).

Four different percentages of CNTs were added to the bitumen binder , which were further used in preparing the test samples for indirect tensile strength. The impacts of CNTs on indirect tensile strength and tensile strength ratios can be evaluated from the results of tests, the details of which are given in table (B.7) to table (B.11).

The TSR ratios for the asphalt concrete mixtures modified with carbon nanotube (0.1%, 0.4%. 1.0% and 1.4%) are well illustrated in (figure 4.2). It can be seen that the TSR% value was 85.84% for control samples but with the addition of 0.1% CNTs the TSR% raised to 90.44%, and increased further to 96.45 with the addition of 0.4% of

CNTs and similarly for 1.0% CNTs it went up to 105.10% , which gave the highest TSR% among the four percentages we used. Now again with the addition of 1.4% of CNTs the TSR% decreases to 103.41% as compared to 1.0% of CNTs.

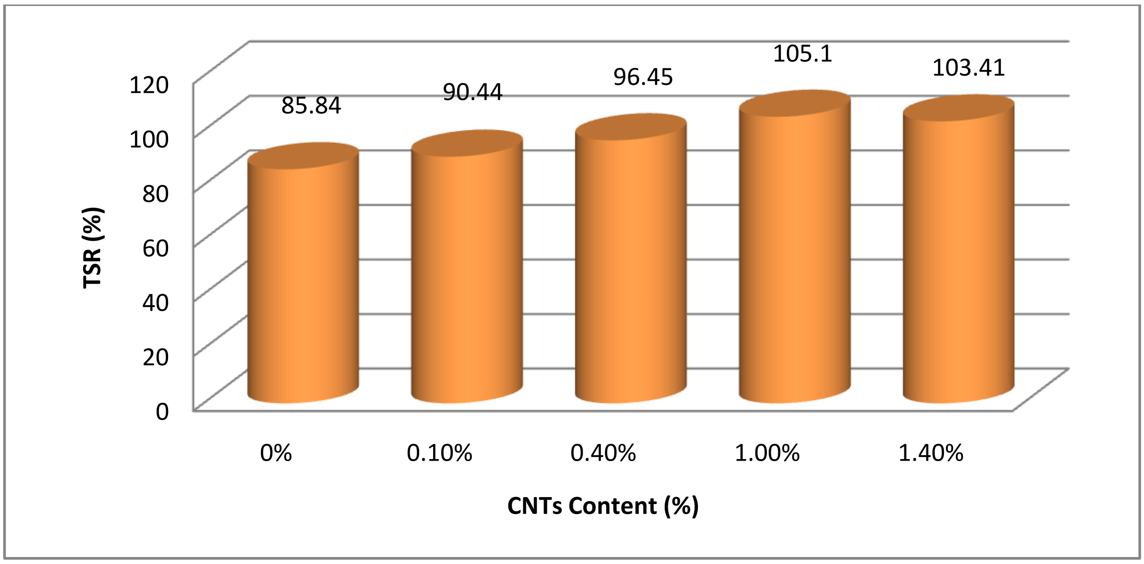

Figure 4.3 TSR values for various percentages of CNTs added to bitumen.

4.3 ASPHALT BINDER TESTS RESULTS

The bitumen binder used in this study was taken from Tupras-Izmit refinery. Only one grade of bitumen binder was used for investigation purpose, which was 50/70 penetration grade. The properties of these virgin binders are shown in table (3.3). A commercially available multi-wall carbon nano-tube was used as an additive. Four different percentages of (0.15, 0.4%, 1.0% and 1.45 by weight of asphalt) of CNTs were employed and were blended with asphalt binder.

In this research the Rolling Thin Film Oven test (RTFOT), Dynamic Shear Rheometer (DSR), Penetration and softening point tests were carried out on asphalt binders, considering both original asphalt samples (samples without the addition of carbon nanotubes) and modified asphalt samples (samples with the addition of carbon nanotubes). The results of penetration and softening point tests can be seen from the tables (A. 1) and (A. 2) appendix A.

From the results of penetration and softening point tests, it can be said, though there is a significant difference between the values of original and modified asphalt binder with carbon nanotubes at different percentages, but does not necessarily changed the grade of bitumen and the values mostly meet the requirements specified by (KGM-Ankara 2012). Figures 4.3 and 4.4 illustrates the results of penetration and softening point tests for CNTs percentages added to bitumen binder.

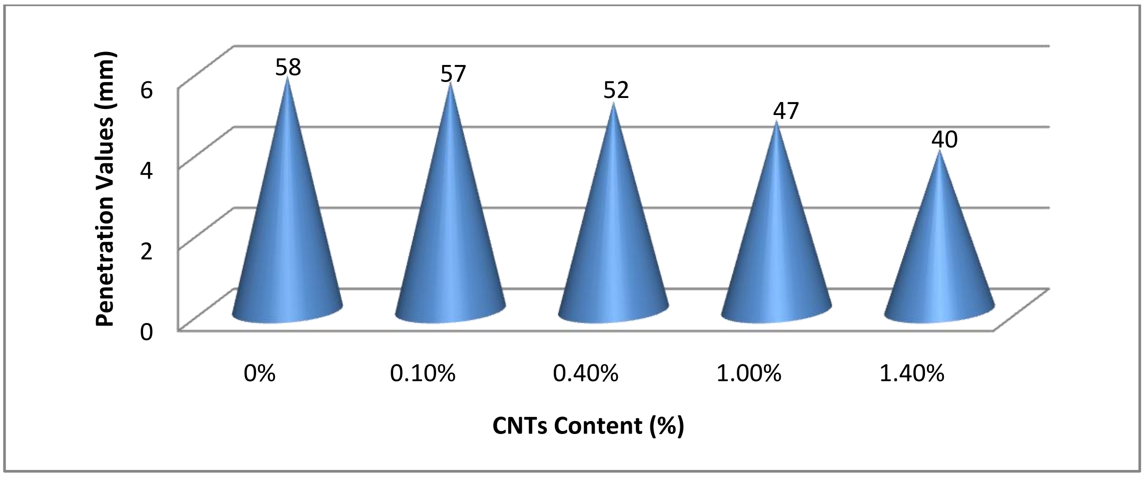

Figure 4.4 The Penetration Values For CNTs (Mixed With Bitumen Binder).

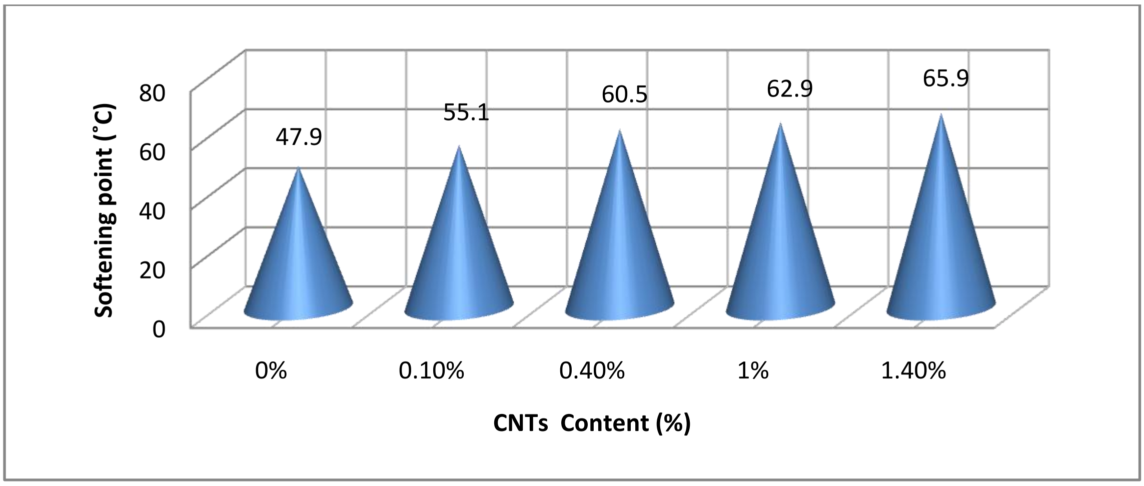

Figure 4.5 The Softening Point(˚C) vs CNT Content (%).

From the tests results of RTFO and DSR the increase in G*/sinδ' can be clearly seen, as soon as the percentages of CNTs in bitumen binder increases , which means an enhancement in permanent deformation (rutting). And with the addition of 1.4 % CNTs it gives the highest G*/sinδ' values. In addition to that we can see a significant change in performance grade with the increase of the amount of CNTs, as in case of original asphalt the PG is 64, but a binder containing 0.1% CNTs caused an increase of one level of PG (60^{O}C), and it remained the same for 0.4% and 1.0% of CNTs. However with further increase in the amount of CNTs to 1.4% the grade further changes to 88.

The complex modulus (G*), which is one of the main parameter describing the behavior of bitumen, has been significantly improved with the CNT's modified asphalt. Furthermore, the phase angle (δ) as an important parameter has also been influenced (improved) by the use of CNT's modified bitumen. From the results obtained from DSR, the values of G* and δ shows that the addition of CNTs has increased the complex modulus (G*) tremendously, while at the same time the phase angle (δ) has been decreased. The bitumen types with high complex modulus and small phase angle possess a better performance, specially at high temperatures.

On the other hand, the results of BBR tests which were carried out on RTFO aged asphalt samples, show that the m-value has increased gradually with an increase in the amount of carbon nano-tube and also but the stiffness values are quite variable with different amount of carbon nano-tube. In case of -6^{O}C the addition of 1.4% of CNT gives the highest m-value and the lowest stiffness value, which is an indication of improvement.

Similarly, for -12^{O}C and -18^{O}C the addition of 1.4% of CNT shows the highest m-values, however the stiffness values has decreased with the addition of CNT percentages for these cases, and it got the highest stiffness values for 1.4% of CNT. However the overall results of m-value and stiffness are within the limits.

M-value indicates the rate of change of stiffness(S) with loading time. the effect of (m-value and S) on thermal cracking is analogous to the effect of G* and Delta on rutting and fatigue cracking. As the stiffness increases the thermal stress developed in the pavement due to thermal shrinking also increases and thermal cracking become more likely to happen.

On the other hand, as the m-value decreases the rate of stress relaxation decreases. It means that the ability of the pavement to relieve the stress that occurs due to a rapid temperature drop will decrease which result in high possibility of thermal cracking.

Moreover, the images obtained from Scanning Electron Microscopy (SEM) regarding the microstructure and morphology of CNT surfaces in HMA samples are shown in the figures below:

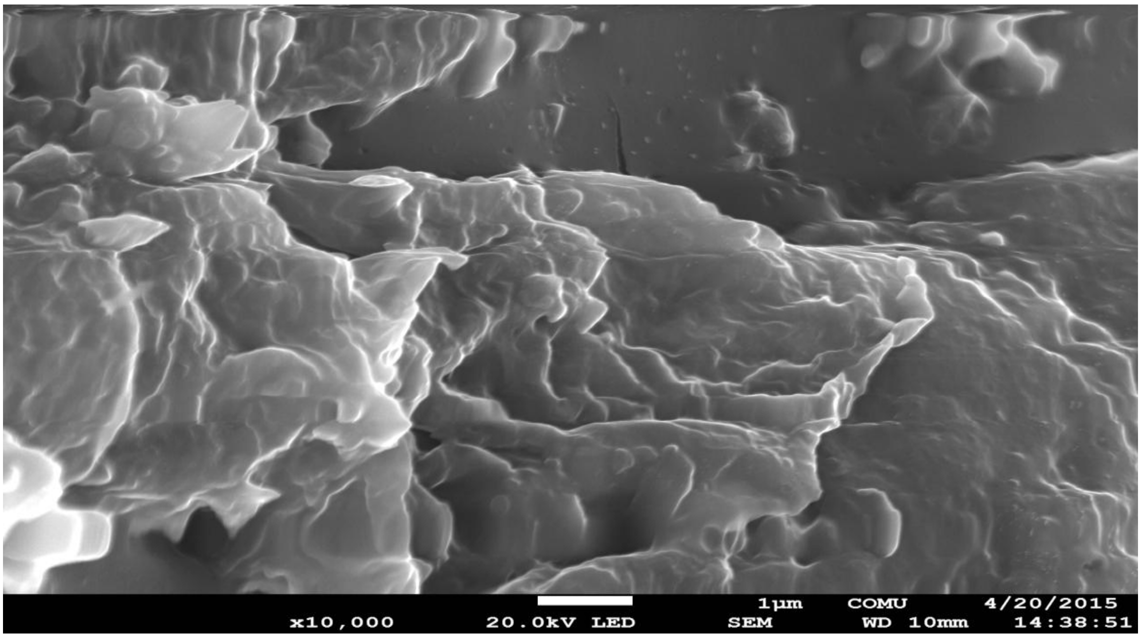

Figure 4.6 SEM Image of Modified Asphalt Binder with 0.0% of CNT.

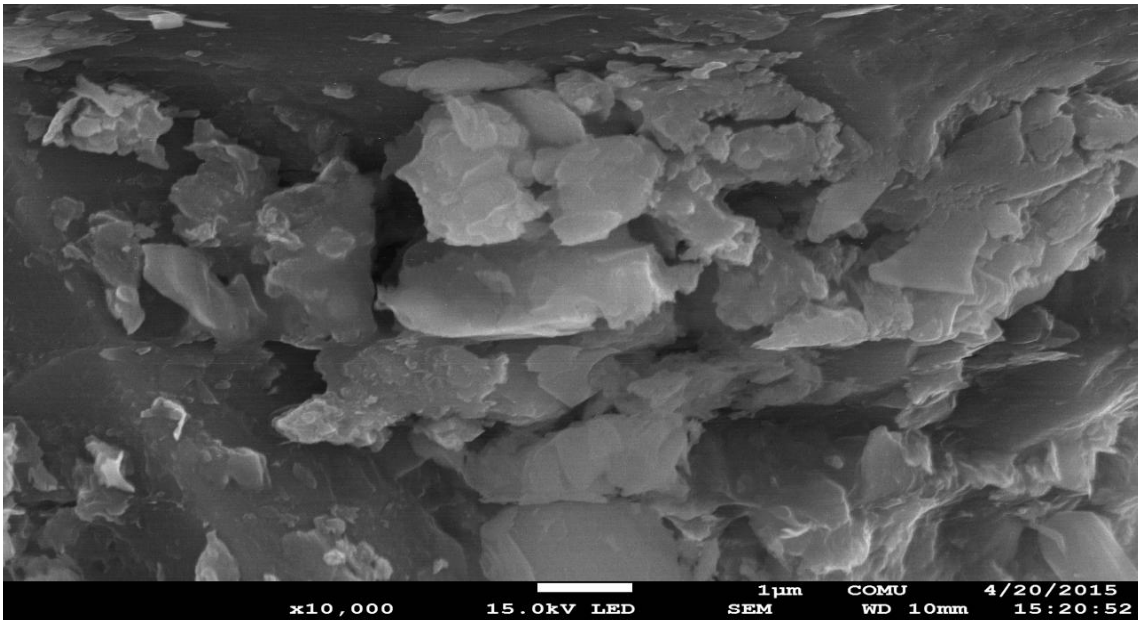

Figure 4.7 SEM Image of Modified Asphalt Binder with 0.1% of CNT.

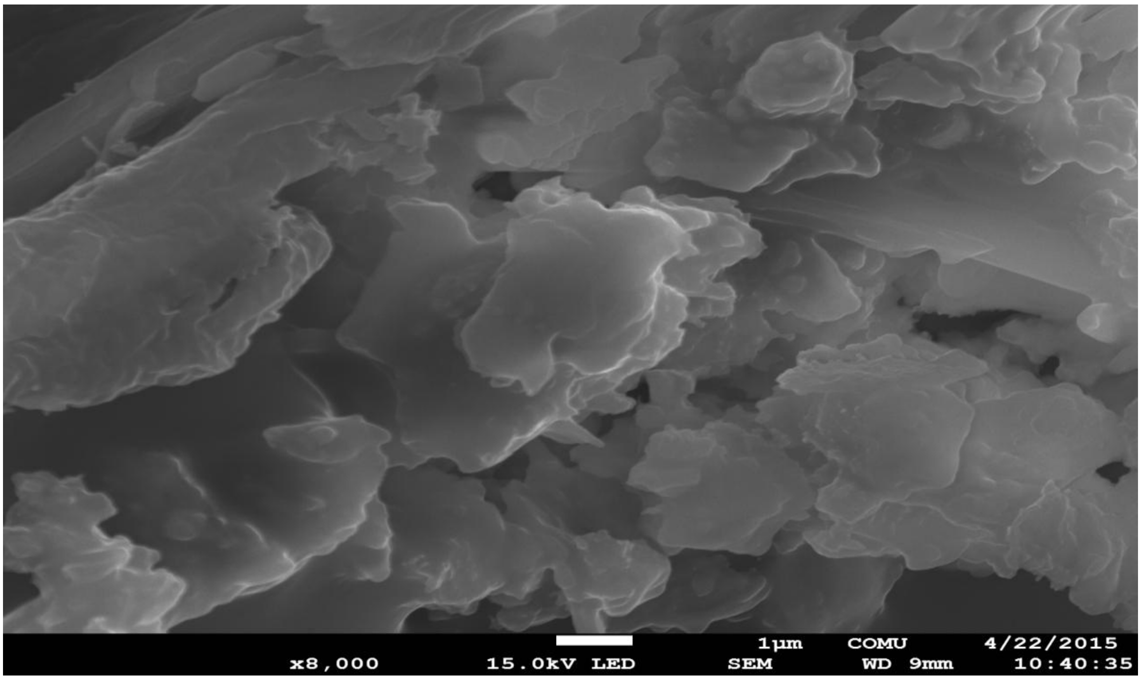

Figure 4.8 SEM Image of Modified Asphalt Binder with 0.4% of CNT.

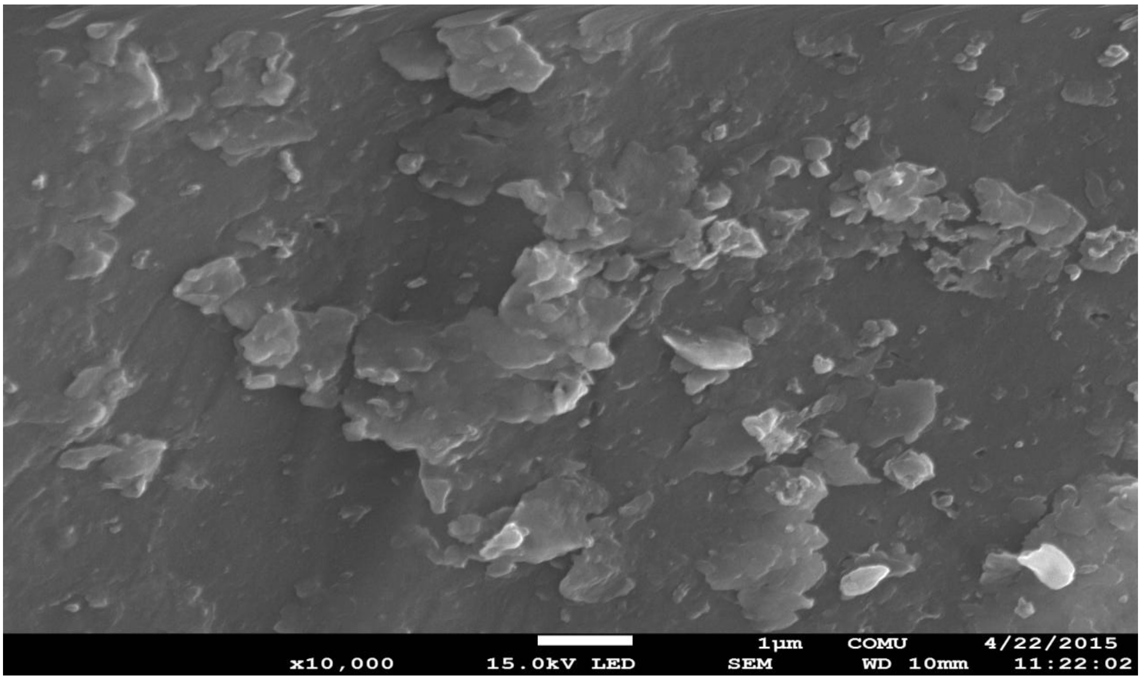

Figure 4.9 SEM Image of Modified Asphalt Binder with 1.0% of CNT.

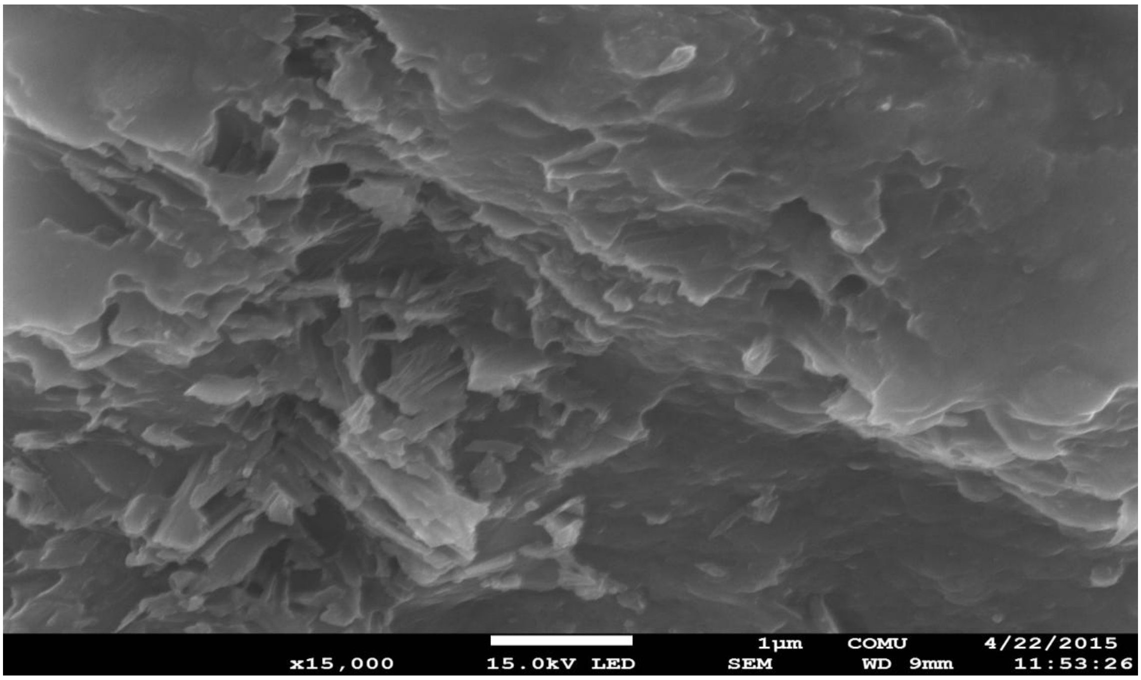

Figure 4.10 SEM Image of Modified Asphalt Binder with 1.4% of CNT.

Figures (4.6 - 4.10) shows the external morphology or texture of CNT-modified asphalt binder studied by Scanning Electron Microscopy' (SEM) imaging for four different dosages of CNT-modified samples including the control one (0.0% CNT). The figures are presented at different magnifications factor but close to each other. From these figures it can be seen that the interconnectivity and density of all the samples modified with CNT are higher and illustrates a good network . The CNT-networking, bridging in between the CNT particles and high density can be better observed from the figures specially in the case of o.4% and 1.4% of CNT, which illustrate a better network and higher number of CNT bridging among the other contents of CNTs. This kind of particles-networks are believed and most likely to resist micro cracks propagations and control crack development due to applied loading, which may result in enhancing the fatigue life and rutting characteristics of HMA (37).

The test results of RTFO, DSR and BBR can be seen from appendix (A), tables (A.3), (A.4), (A.5), (A.6), (A.7) (A.8), (A.9) and (A.10).

CHAPTER 5

CONCLUSION

5.1 CONCLUSION AND RECOMMENDATIONS

The tests performed in this study consisted of two categories, tests carried out on bitumen binders and those performed on asphalt mixtures. CNTs, which is one of the high-technology modifiers, has been blended with bitumen in different percentages by weight of base binder (0.1%, 0.4%, 1.0% and 1.4%), and its impacts on various properties of bitumen has been investigated through classical and functional experiments.

The results indicate that with the addition of this modifier the physical and functional properties of bitumen has been significantly improved. The penetration values has been gradually increased with the addition of the percentages of CNTs, as it can be seen from the results. The penetration value for the control samples was 58, however as soon as 0.1% CNTs is added the penetration value has decreased to 57 and with addition of other percentages it kept decreasing which finally for 1.4% of CNTs the penetration value is 40, which clearly shows how CNTs affected and strengthened the asphalt binder's consistency. And similarly if the softening point results are evaluated, the softening temperature for control samples was 48 but it went up to 55 with the addition of 0.1% of CNTs and kept going up for increasing the percentage of CNTs, which finally for 1.4% of CNTs it gave the highest value of 66. It can be seen that for both penetration and softening point tests 1.4% of CNTs gives the highest values.

Moreover, the results obtained from DSR tests , it can be seen that the complex modulus and phase angle values have been tremendously improved with CNTs modified bituminous samples as compared to the standard bitumen samples. It can be seen as the grade for control sample was 64, but with the addition of 0.1% of CNTs it changed to 70 and stayed the same for 0.4 % and 1.0% of CNTs, however it further improved to 88 in the case of 1.4% of CNTs, and not only that the phase angle decreases as the CNTs increases for the cases and at the same time the G* values increases with the increases of CNTs percentage, which is a great sign of improvement. In general it is desirable for asphalt to show higher complex modulus and lower phase angle, it is believed that a material with higher complex modulus and lower phase angle have high resistance to rutting or permanent deformation.

Furthermore, the results obtained from Marshall retained stability and indirect tensile strength tests, show that the moisture damage on asphalt concrete mixtures have been greatly improved with the addition of CNTs. As the retained stability index (RSI%) for control samples was 58.12 but the asphalt concrete samples modified with 0.1% of CNTs took RSI % to 60.86 and it kept improving with the increase in percentage of CNTs, with 1.0% of CNTs the RSI % was 74.6% but it decreased back to 52.7 in the case of 1.4% of CNTs. Similarly from the tensile strength ratios (TSR%) it can be seen that for control samples the TSR% was 85.8, but it improved to 90.44 with the addition of 0.1% of CNTs and kept increasing with the increase in percentages of CNTs. At 1.0% of CNTs it gave 105.10 , however it decreased to 103.41 in the case of 1.4% of CNTs. In both RSI and TSR cases at 1.0% of CNTs it gives the highest values. From the findings presented above it can be expressed that using CNTs as modifier of bitumen can greatly decrease the moisture susceptibility and improves the hot mix asphalt service life.

The results from SEM analysis indicates that CNT provides better nano and micro-crack bridging mechanism and improve adhesion characteristics.

There is no doubt that the results are impressive and encouraging, but some points are needed to be given the attention for using such nano-structured binders.

- The dispersion of carbon nano-tube in bitumen is a hard task and requires a suitable technique in order to get a homogenous mixture.
- Since carbon nano-tube's modified materials consist of higher viscosity as compared to the neat bitumen binder ,so the identification of mixing and compaction temperature may be critical.

Finally, the investigations and findings of this study indicate that modification of asphalt binders with carbon nanotubes (CNT) looks more reliable and promising approach toward improving the field performance of bituminous mixtures. However it also suggests that more comprehensive research is needed to simplify this research and come up with the most suitable framework through which the desired influences are obtained.

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Appendix A: Tables That Contains Details of Penetration, Softening Point, RTFO, DSR and BBR Tests Results.

Table A.1 Penetration Tests Results.

CNTs (%)	No. of Sample	Penetration Values			Average	Overall Average	
						cm	mm
(0.0) Control samples	1	5.93	5.73	5.67	5.78	5.81	58.1
	2	5.82	5.80	5.93	5.85		
0.1	1	5.75	5.50	5.63	5.62	5.68	56.8
	2	5.80	5.41	5.5o	5.77		
0.4	1	5.30	5.10	4.99	5.13	5.17	51.7
	2	5.09	5.00	5.04	5.04		
1.0	1	4.98	4.86	4.90	4.91	4.72	49
	2	4.89	4.95	4.92	4.92		
1.4	1	4.01	4.00	3.98	3.99	3.99	40
	2	3.99	4.02	3.98	3.99		

Table A.2 Softening Point Tests Results.

CNTs (%)	Softening Temperature Values (˚C)	Average
0.0	47.5	47.9
	48.3	
0.1	55	55.1
	55.2	
0.4	60.3	60.5
	60.7	
1.0	62.8	62.9
	63	
1.4	65.8	65.9
	66	

Table A.3 Details of RTFO and DSR Tests Results of 0.0% CNTs Added To Bitumen.

Sample Reference	Test Name	Test method	Results	**Specification**
Izmit B50/70	Mass Change (%)	(RTFOT), TS EN 12607-1	-0.09	≤ 1.00%
Dynamic Shear Rheometer (DSR) TS EN 14770, AASHTO T 315				
Test Temperature ^{o}C	G*, kpa	Phase Angle δ'	G*/sin δ', kpa	≥2.2 Kpa
64	3.9697	85.13	3.9841	
70	1.8555	86.51	1.8589	

Pass Fail Temp68.7^{o}C
Grade 64 Oscillation Mode: Grade
Frequency 10.0 rad/s Sample Mode: RTFOT

Table A.4 Details of (RTFO and DSR) tests results of 0.1% CNTs added to bitumen.

Sample Reference	Test Name	Test method	Results	Specification
Izmit B50/70	Mass Change (%)	(RTFOT), TS EN 12607-1	0.03	≤ 1.00%
Dynamic Shear Rheometer (DSR) TS EN 14770, AASHTO T 315				
Test Temperature ^{o}C	G*, kpa	Phase Angle δ'	G*/sin δ', kpa	≥2.2 kpa
70	3.4767	84.52	3.4927	
76	1.6813	85.94	1.6855	

Pass Fail Temp 73.8^{o}C
Grade 70
Frequency 10.0rad/s

Oscillation Mode : Grade
Sample Mode : RTFOT

Table A.5 Details of (RTFO and DSR) Tests Results Of 0.4% CNTs Added To Bitumen.

Sample Reference	Test Name	Test method	Results	**Specification**
Izmit B50/70	Mass Change (%)	(RTFOT), TS EN 12607-1	0.19	≤ 1.00%
Dynamic Shear Rheometer (DSR) TS EN 14770, AASHTO T 315				
Test Temperature ^{o}C	G*, kpa	Phase Angle δ'	G*/sin δ', kpa	≥2.2 kpa
70	3.6390	84.18	3.6579	
76	1.7807	85.44	1.7864	

Pass Fail Temp 74.3^{o}C
Grade 70
Frequency 10.0rad/s

Oscillation Mode : Grade
Sample Mode : RTFOT

Table A.6 Details of (RTFO and DSR) tests results Of 1.0% CNTs added to bitumen.

Sample Reference	Test Name	Test method	Results	Specification
Izmit B50/70	Mass Change (%)	(RTFOT), TS EN 12607-1	0.51	≤ 1.00%
Dynamic Shear Rheometer (DSR) TS EN 14770, AASHTO T 315				
Test Temperature ^{o}C	G*, kpa	Phase Angle δ'	G*/sin δ', kpa	≥2.2 kpa
70	4.2615	81.49	4.3089	
76	2.1086	82.48	2.1269	

Pass Fail Temp 75.7^{o}C
Grade 70 Oscillation Mode : Grade
Frequency 10.0rad/s Sample Mode : RTFOT

Table A.7 Details of (RTFO and DSR) tests results of 1.4% CNTs added to bitumen.

Sample Reference	Test Name	Test method	Results	Specification
Izmit B50/70	Mass Change (%)	(RTFOT), TS EN 12607-1	0.79	≤ 1.00%
Dynamic Shear Rheometer (DSR) TS EN 14770, AASHTO T 315				
Test Temperature ^{o}C	G*, kpa	Phase Angle δ'	G*/sin δ', kpa	≥2.2 kpa
76	7.7566	70.67	8.2198	
82	4.3462	70.52	4.6102	
88	2.5575	69.92	2.723	

Pass Fail Temp 90.4^{o}C
Grade 88 Oscillation Mode : Grade
Frequency 10.0rad/s Sample Mode : RTFOT

Table A.8 BBR-Tests Results at Max. Temperature of -6^{O}C.

CNT (%)	**Maximum Temperature (-6^{O}C)**			
	M-Value	Stiffness (MPa)	Specification	
			M-Value	Stiffness
0.0	0.418	87.1	**≥ 0.300**	**≤ 300 MPa**
0.1	0.379	114		
0.4	0.358	116		
1.0	0.391	101		
1.4	0.457	74.5		

Table A.9 BBR-Tests Results at Max. Temperature of -12^{O}C.

CNT (%)	**Maximum Temperature (-12^{O}C)**			
	M-Value	Stiffness (MPa)	Specification	
			M-Value	Stiffness
0.0	0.337	199	**≥ 0.300**	**≤ 300 MPa**
0.1	0.303	233		
0.4	0.308	224		
1.0	0.334	209		
1.4	0.343	208		

Table A.10 BBR-Tests Results at Max. Temperature of -18^{O}C.

CNT (%)	Maximum Temperature (-18^{O}C)			
	M-Value	Stiffness (MPa)	Specification	
			M-Value	Stiffness
0.0	0.246	439	**≥ 0.300**	**≤ 300 MPa**
0.1	0.218	449		
0.4	0.225	473		
1.0	0.242	474		
1.4	0.263	496		

APPENDIX B: TABLES THAT CONTAINS DETAILS OF MARSHALL STABILITY & FLOW, RSI AND TSR TEST VALUES.

Table (B.1) Marshall Stability and Flow Values of CNTs-Modified asphalt samples.

No. Of Sample	CNTs %	AVG HEIGHTS	WT. IN AIR	WT. IN WATER	SSD. IN AIR	GM	GT	Vv%	Vb%	VMA %	VFB%	FLOW mm	STABILITY KN	CORR. STABILITY KN
1	0.0%	61.10	1206.12	724.85	1207.10	2.500	2.601	3.973	11.73	15.72	74.62	5.041	10.701	11.06
2	0.0%	61.65	1207.34	725.98	1208.42	2.461	2.601	5.230	11.54	16.77	68.81	3.502	9.196	10.00
AVERAGE								4.60	11.63	16.24	71.71	4.21	9.95	10.53
3	0.1%	61.90	1203.78	720.99	1204.81	2.493	2.601	4.150	11.69	15.84	73.80	3.880	10.377	10.85
4	0.1%	60.85	1209.03	725.39	1210.45	2.511	2.601	3.460	11.78	15.24	77.38	4.001	9.961	10.05
AVERAGE								3.81	11.73	15.54	75.59	3.940	10.17	10.45
5	0.4%	60.84	1205.22	729.12	1206.14	2.501	2.601	3.845	11.57	15.57	75.34	3.640	8.920	9.530
6	0.4%	61.00	1204.76	718.50	1205.10	2.486	2.601	4.421	11.66	16.08	72.51	4.210	8.991	9.609
AVERAGE								4.13	11.61	15.82	73.92	3.925	8.95	9.57
7	1.0%	61.60	1202.65	716.99	1204.32	2.500	2.601	3.973	11.73	15.70	74.71	3.660	8.510	9.004
8	1.0%	61.43	1205.09	719.89	1206.95	2.499	2.601	3.921	11.72	15.64	74.94	3.610	8.903	9.421
AVERAGE								3.95	11.72	15.67	74.85	3.63	8.71	9.21
9	1.4%	61.84	1205.12	714.23	1207.04	2.454	2.601	5.620	11.51	17.13	67.19	3.810	7.520	8.210
10	1.4%	62.00	1206.10	713.99	1207.45	2.451	2.601	5.700	11.50	17.20	66.86	4.320	7.561	8.320
AVERAGE								5.65	11.50	17.16	67.02	4.07	7.54	8.26

Table B.2 Details of retained stability index (RSI) for control samples at optimum bitumen content (5.03%).

Blow No.	UNCONDITIONED SAMPLES	AVG HEIGHTS	WT. IN AIR	WT. IN WATER	SSD. IN AIR	GM	GT	Vv%	Vb%	VMA%	VFB%	FLOW mm	STABILITY KN	CORR. STABILITY KN	RETAINED STABILITY INDEX
75	1	61.10	1206.12	724.85	1207.10	2.500	2.601	3.973	11.73	15.72	74.62	5.041	10.701	11.06	58.12
	2	61.65	1207.34	725.98	1208.42	2.461	2.601	5.230	11.54	16.77	68.81	3.502	9.196	10.00	
	AVERAGE							4.60	11.63	16.24	71.71	4.21	9.95	10.53	
	CONDITIONED SAMPLES														
75	**3**	61.55	1207.80	723.61	1208.98	2.494	2.601	4.110	11.78	15.89	74.13	5.210	6.187	6.210	
	4	61.90	1203.45	726.34	1205.00	2.496	2.601	4.036	11.71	15.75	74.35	3.921	5.980	6.030	
	AVERAGE							4.07	11.74	15.82	74.24	4.56	6.10	6.12	

Table B.3 Details of retained stability index (RSI) for 0.1% CNTs added to bitumen.

Blow No.	**UNCONDITIONED SAMPLES**	**AVG HEIGHTS**	**WT. IN AIR**	**WT. IN WATER**	**SSD. IN AIR**	**GM**	**GT**	**Vv%**	**Vb%**	**VMA%**	**VFB%**	**FLOW mm**	**STABILITY KN**	**CORR.STABILITY KN**	**RETAINED STABILITY INDEX**
75	1	61.90	1203.78	720.99	1204.81	2.493	2.601	4.150	11.69	15.84	73.80	3.880	10.377	10.85	60.86
	2	60.85	1209.03	725.39	1210.45	2.511	2.601	3.460	11.78	15.24	77.38	4.001	9.961	10.05	
	AVERAGE							3.81	11.73	15.54	75.59	3.940	10.17	10.45	
	CONDITIONED SAMPLES														
75	**3**	61.10	1206.66	724.09	1208.23	2.500	2.601	3.970	11.73	15.70	74.71	4.030	6.560	6.610	
	4	61.31	1205.17	723.10	1206.94	2.499	2.601	3.921	11.72	15.64	74.94	4.209	6.051	6.104	
	AVERAGE							3.95	11.72	15.67	74.82	4.128	6.30	6.36	

Table b.4 Details of retained stability index (RSI) for 0.4% CNTs added to bitumen.

Blow No.	UNCONDITIONED SAMPLES	AVG HEIGHTS	WT. IN AIR	WT. IN WATER	SSD. IN AIR	GM	GT	Vv%	Vb%	VMA %	VFB%	FLOW mm	STABILITY KN	CORR.STABILITY KN	RETAINED STABILITY INDEX
75	1	60.84	1205.22	729.12	1206.14	2.501	2.601	3.845	11.57	15.57	75.34	3.640	8.920	9.530	68.44
	2	61.00	1204.76	718.50	1205.10	2.486	2.601	4.421	11.66	16.08	72.51	4.210	8.991	9.609	
	AVERAGE							4.13	11.61	15.82	73.92	3.925	8.95	9.57	
	CONDITIONED SAMPLES														
75	**3**	60.50	1207.00	722.10	1207.09	2.489	2.601	4.305	11.67	15.97	73.07	3.732	6.540	6.601	
	4	59.97	1203.13	723.76	1204.10	2.515	2.601	3.315	11.88	15.19	78.21	4.302	5.901	6.510	
	AVERAGE							3.81	11.77	15.58	75.64	4.02	6.22	6.55	

Table B.5 Details of retained stability index (RSI) for 1.0% CNTs added to bitumen.

Blow No.	UNCONDITIONED SAMPLES	AVG HEIGHTS	WT. IN AIR	WT. IN WATER	SSD. IN AIR	GM	GT	Vv%	Vb%	VMA %	VFB%	FLOW mm	STABILITY KN	CORR.STABILITY KN	RETAINED STABILITY INDEX
75	1	61.60	1202.65	716.99	1204.32	2.500	2.601	3.973	11.73	15.70	74.71	3.660	8.510	9.004	74.57
	2	61.43	1205.09	719.89	1206.95	2.499	2.601	3.921	11.72	15.64	74.94	3.610	8.903	9.421	
	AVERAGE							3.95	11.72	15.67	74.85	3.63	8.71	9.21	
	CONDITIONED SAMPLES														
75	**3**	62.00	1207.00	717.73	1209.03	2.498	2.601	3.960	11.72	15.68	74.74	3.901	6.210	6.788	
	4	61.96	1206.40	719.10	1208.06	2.501	2.601	3.845	11.73	15.57	75.34	3.900	6.172	6.754	
	AVERAGE							3.90	11.72	15.62	75.04	3.90	6.19	6.86	

Table B.6 Details of retained stability index (RSI) for 1.4% CNTs added to bitumen.

Blow No.	UNCONDITIONED SAMPLES	AVG HEIGHTS	WT. IN AIR	WT. IN WATER	SSD. IN AIR	GM	GT	Vv%	Vb%	VMA %	VFB%	FLOW mm	STABILITY KN	CORR.STABILITY KN	RETAINED STABILITY INDEX
75	1	61.84	1205.12	714.23	1207.04	2.454	2.601	5.620	11.51	17.13	67.19	3.810	7.520	8.210	52.66
	2	62.00	1206.10	713.99	1207.45	2.451	2.601	5.700	11.50	17.20	66.86	4.320	7.561	8.320	
	AVERAGE							5.65	11.50	17.16	67.02	4.07	7.54	8.26	
	UNCONDITIONED SAMPLES														
75	3	61.16	1208.09	717.09	1208.99	2.460	2.601	5.420	11.54	16.96	68.04	4.450	4.301	4.600	
	4	60.97	1203.56	715.87	1205.12	2.476	2.601	4.800	11.61	16.41	70.75	4.509	4.001	4.101	
	AVERAGE							5.11	11.57	16.68	69.39	4.56	4.15	4.35	

Table B.7 Details of indirect tensile strength and TSR% for control samples at optimum bitumen content (5.03%).

Blow No.	Conditioned samples	H1	H2	H3	AVG. Heights	WT.IN AIR	WT.IN WATER	SSD WT. In Air	saturation	GM	GT	Vv%	Load KN	Load N	St.tensile strength	TSR%
60 blows	1	60.80	60.80	60.70	60.77	1203.35	707.26	1205.07	Yes	2.421	2.601	6.960	9.850	9850	0.990	85.84
	2	60.20	60.40	60.30	60.30	1199.68	705.35	1200.96	Yes	2.432	2.601	7.101	9.991	9991	1.010	
	Average											7.03			1.000	
	Unconditioned samples															
60 blows	1	60.30	60.50	61.00	60.60	1200.27	705.28	1201.85	No	2.425	2.601	6.777	10.89	10890	1.140	
	2	60.40	60.60	60.30	60.43	1203.70	706.25	1204.95	No	2.411	2.601	7.310	11.15	11150	1.190	
	Average											7.04			1.165	

Table B.8 Details of indirect tensile strength and TSR% for 0.1% CNTs added to bitumen.

Blow No.	Conditioned samples	H1	H2	H3	AVG. Heights	WT.IN AIR	WT.IN WATER	SSD WT. In Air	saturation	GM	GT	Vv%	Load KN	Load N	St.tensile strength	TSR%
60 blows	1	60.60	60.30	60.50	60.51	1204.76	708.80	1205.90	Yes	2.420	2.604	6.96	8.630	8630	0.820	90.44
	2	60.00	60.20	60.10	60.10	1205.27	706.17	1206.61	Yes	2.410	2.604	7.34	8.221	8221	0.809	
	Average											7.15			0.814	
	Unconditioned samples															
60 blows	1	60.70	60.30	60.80	60.60	1205.65	705.60	1206.68	No	2.411	2.601	7.340	9.105	9105	0.901	
	2	60.50	60.60	60.40	60.50	1207.79	709.10	1209.37	No	2.421	2.601	6.920	9.021	9021	0.900	
	Average											7.13			0.90	

Table B.9 Details of indirect tensile strength and TSR% for 0.4% CNTs added to bitumen.

Blow No.	Conditioned samples	H1	H2	H3	AVG. Heights	WT.IN AIR	WT.IN WATER	SSD WT. In Air	saturation	GM	GT	Vv%	Load KN	Load N	St.tensile strength	TSR%
60 blows	1	62.00	62.30	61.90	62.07	1204.75	708.00	1203.10	Yes	2.420	2.601	6.97	8.034	8034	0.850	96.45
	2	61.70	61.70	61.50	61.63	1206.10	706.20	1202.89	Yes	2.412	2.601	7.27	7.625	7625	0.860	
	Average											7.12			0.855	
	Unconditioned samples															
60 blows	1	60.90	61.10	61.40		1205.18	705.12	1207.30	No	2.410	2.601	7.17	9.211	9211	0.910	
	2	61.00	61.00	61.20		1202.30	709.00	1203.90	No	2.421	2.601	6.921	8.610	8610	0.872	
	Average											7.04			0.891	

Table B.10 Details of indirect tensile strength and TSR% for 1.0% CNTs added to bitumen.

Blow No.	Conditioned samples	H1	H2	H3	AVG. Heights	WT.IN AIR	WT.IN WATER	SSD WT. In Air	saturation	GM	GT	Vv%	Load KN	Load N	St.tensile strength	TSR%
60 blows	1	62.00	62.00	62.00	62.00	1203.84	708.00	1205.16	Yes	2.427	2.601	6.771	8.350	8350	0.860	
	2	62.20	62.00	62.00	62.07	1202.80	704.27	1203.92	Yes	2.410	2.601	7.340	8.600	8600	0.871	
	Average											7.06			0.86	
	Unconditioned samples															105.10
60 blows	1	61.70	61.90	61.80	61.80	1204.20	707.17	1205.48	No	2.421	2.601	6.921	7.500	7500	0.810	
	2	62.00	62.10	61.90	62.00	1201.10	706.10	1203.17	No	2.430	2.601	6.664	7.861	7861	0.840	
	Average											6.88			0.82	

Table B.11 Details of indirect tensile strength and TSR% for 1.4% CNTs added to bitumen.

Blow No.	Conditioned samples	H1	H2	H3	AVG. Heights	WT.IN AIR	WT.IN WATER	SSD WT. In Air	saturation	GM	GT	Vv%	Load KN	Load N	St.tensile strength	TSR%
60 blows	1	62.10	62.00	62.00	62.03	1207.60	699.90	1209.10	Yes	2.378	2.601	8.520	9.210	9210	0.910	
	2	62.20	61.90	62.10	62.07	1200.10	700.85	1203.14	Yes	2.404	2.601	7.570	9.730	9730	0.934	
	Average											8.05			0.92	
	Unconditioned samples															**103.41**
60 blows	1	62.90	62.80	62.90	62.87	1202.03	701.09	1203.29	No	2.399	2.601	7.775	8.781	8781	0.891	
	2	63.10	63.00	63.00	63,03	1205.28	702.27	1207.10	No	2.396	2.601	7.882	9.012	9012	0.900	
	Average											7.837			0.89	