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INVESTIGATING NANO MATERIALS EFFECT ON HOT MIX ASPHALT CONCRETE (USING ZYCOTHERM)

By

ILHAM IBRAHIM MOHAMMED

May 2014

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By

Ilham Ibrahim Mohammed

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APPROVAL PAGE

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Ilham Ibrahim Mohammed

M.S. Thesis - Civil Structures and Infrastructures Engineering
May 2014

Thesis Supervisor: Assist. Prof. Dr. Hatice Nur Aras Mehan

ABSTRACT

Nano-sized particles can be used in various applications in order to improve the properties of various materials. Increasing the use of nanotechnology in civil engineering and as an effective alternative for asphalt binder modification has become a great and an important issue. Nanotechnology has the potential to create many new materials and devices. Nano composite materials that discovered in recent years, are the most exciting and promising materials. In order to get more efficiency for flexible pavement, bituminous binders and asphalt aggregate are modified with various polymers and Nano materials, and this has opened new horizons on transportation engineering. Especially with the starting modification of bituminous binders and polymer-based additives and , as well as asphalt aggregate mixtures, we can see that the properties such as rutting , low heat cracks, fatigue cracks, peeling are extremely reduced and high temperature resistant and service life increased and getting longer with the comparison to normal flexible coating .Therefore good road construction requires that the asphalt maintain its integrity without stripping (deboning of asphalt binder from aggregate surface especially when exposed to moisture or water) and provide resistance to rutting with adequate strength, fatigue cracking and low temperature cracking .stripping of asphalt pavements has become a major problem in recent years .stripping can lead to premature rutting, fatigue cracking or disintegration (potholing) of asphalt pavements. In this study, the effects of nanomaterial zycotherm on the performance of asphalt mixtures will be studied. The project will be performed take in two stages. The first stage of this process will be carried out by mixing asphalt with Zycotherm in various proportions. The asphalt concrete samples are prepared by Marshall Method. In the second stage Zycotherm diluted inside water at 1:400 and the solution will be sprayed on the aggregates and asphalt concretes will be fabricated with these aggregates. Then nanomaterial's effect on asphalt concrete will be examined with the help of these tests: Marshall Stability and flow test, Indirect tensile strength test, retained stability test, boiling test, Dynamic shear rheometer test, Bending beam rheometer test, Rolling thin film oven test.

Keywords: Nanomaterial, Zycotherm, Moisture Susceptibility, Stripping, Rutting, Fatigue, Tensile strength, Retained Stability Index, Dynamic Shear Rheometer, Bending Beam Rheometer.

INVESTIGATING NANO MATERIALS EFFECT ON HOT MIX ASPHALT CONCRETE (USING ZYCOTHERM)

Ilham Ibrahim Mohammed

Yüksek Lisans Tezi – İNŞAAT YAPI VE ALTYAPI MÜHENDİSLİĞİ
Mayis 2014

Tez Danışmanı: Yrd. Doç. Dr. Hatice Nur Aras Mehan

ÖZ

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This work is dedicated to my Family and my teachers

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LIST OF SYMBOLS AND ABBREVIATION

SYMBOL/ABBREVIATION

ASTM American Society of Testing and Materials

AASHTO American association of highway and transportation officials

CHAPTER ONE 1

INTRODUCTION

1.1 INTRODUCTION

Highway flexible pavements constitute more than 90% of the national pavements of the country, produced from hot mix asphalt (which is a mixture of well graded aggregate combined with a bituminous binder to provide a stable structure for passing vehicles) continually face different failure problems. These problems may source from hot mix asphalt or it may source from underlying layers either base, sub base or subgrade failures. Hot mix asphalt concrete will have a long life even under heavy traffic, severe conditions like moisture; high and low temperature, when designed and laid properly.

During past years different researchers studied causes of failure of flexible pavements and offered variety of alternatives for solving these failure problems. Some researchers focused on failures that source from hot mix asphalt and it is believed that resistance of an asphalt concrete mixture to fatigue, rutting, ravelling and low temperature cracking will determine the performance of an asphalt concrete mixture.

It is believed that moisture or water is one of the greatest factors which will have an inverse effect on the performance of hot mix asphalt, it manifests itself as a reduction in overall strength and it will increase rutting potential, decreasing fatigue life and accelerating stripping potential of the mixture.

Cohesion within aggregate particles and adhesion between aggregate particles and bitumen determine with great extent strength and durability of the mixture. And controlling two parameters which are moisture sensitivity and temperature susceptibility will guarantee long life of the pavement. It is known that temperature and moisture are ever exist, means that it is impossible to make them constant in a range, so the solution is provided by using materials that are neither moisture sensitive nor temperature susceptible or different techniques to reduce their effect on the pavement. If this was impossible also we have to face different techniques such as using mineral filler, Nano materials, polymers and different additives to reduce sensitivity and susceptibility of moisture and temperature of the materials.

Working on solving these problems related to flexible pavement up to now at macro and micro level didn’t gave satisfaction to engineers and road administers and also didn’t gave the idea of solving the problems from root, so the need of solving problems at Nano levels became necessary. since bitumen itself has a great influence on the characteristics of mixture and because bitumen in most refineries produced in the secondary process, or it can be said it is a byproduct and it will not lead the bitumen to compete with fuel and other products, and from here it is obtained that better performing asphalt in petroleum refining is not a common strategy. When the product does not meet pavement structure requirements, modification has been used as one of the best and most attractive alternatives for improving asphalt and asphalt concrete properties.

In this study Zycotherm Nano material will be used with both asphalt binder and aggregate and hot mix asphalt property at Nano level will be investigated.

1.2 AIM AND OBJECTIVE OF THE STUDY

1.2.1 AIM OF THE STUDY

Aim of the study is to investigate the effects of zycotherm nano material after using in two different modification method with two type of aggregate (limestone and basalt) after asphalt concrete(asphalt mixtures) produced with them.

1.2.2 OBJECTIVE OF THE STUDY

The above aim achieved through the following objectives:

Using two type of aggregate (basalt and limestone) and comparing the effectiveness of using zycotherm Nano material on:

- Volumetric properties of hot mix asphalt such as Marshal Stability, flow, marshal quotient, air void, void in mineral aggregate and voids filled with asphalt in a mixture.
- Resistance of compacted asphalt mixture to moisture induced damage.
- Mixing zycotherm with bitumen binder to investigate (DSR) dynamic shear rheometer test parameter and (BBR) bending beam rheometer test parameter to estimate rutting and fatigue life.
- Stripping value of aggregate when aggregate mixed with zycotherm treated bitumen

CHAPTER TWO 2

LITERATURE REVIEW

2.1 PERFORMANCE CHARACTERISTICS OF HOT MIX ASPHALT

Raw material for hot mix asphalt concrete production is aggregate and bitumen with different additive and modifiers which gives various performance ability to the mixture. different methods available for designing and finding optimum bitumen content of the mixture ,most common are (marshal method , Superpave gyratory method and Hveem method) performance characteristics of hot mix asphalt depend on proper selection of aggregate and bitumen .

Asphalt mixture or pavement performance can be classified in two categories: structural failure and functional failure. Structural failures are associated with the pavement structure not being able to carry the design load. Functional failures are concerned with vehicle stability, comfort of the ride and safety. Often, functional failures trigger the rehabilitation of pavement structures although the pavement structure may still be structurally sound. [4]

There are many factors that affect durability and performance characteristics of hot mix asphalt: mixture properties, construction methods, traffic volume, environmental factors such as temperature and water and to a great extent adhesion between asphalt binder and aggregate.

Prior to deciding which type of aggregate and bitumen to be used for asphalt concrete production; aggregates physical properties such as: mineralogy, durability, particle shape, voids in mineral aggregate, specific gravity and gradation and also Bitumen binder properties such as viscosity, penetration, ductility, aging and temperature susceptibility could be looked at to decide best mixture property and performance characteristic of the mix.

Selecting most suitable time for paving the mixture followed by proper compaction procedure within the limit of the designed temperature will guarantee durable and high performance mixture.

Increasing volume of traffic will adversely affect performance of the mixture and causes asphalt pavement overstress which will fail the pavement. This point can be solved during design stage by proper estimating the total amount of vehicles and axle loads that will pass over during pavements life span. Environmental factors such as temperature play a vital role in the degradation of asphalt concrete pavement. Asphalt binder play an important role in the reaction of the asphalt mixture to temperature. Asphalt binders that have high temperature susceptibility are not desirable.

Stroup-Gardiner et al. (1995) reported that temperature susceptibility became more project-specific as the coarseness of the aggregate gradation increased. Temperature effects the elastic and viscoelastic properties of an asphalt concrete mixture. The modulus of the asphalt is dependent up on the pavement temperature; with an increase in temperature there is a decrease in asphalt modulus. The susceptibility to temperature is a property of the asphalt binder and can be evaluated by evaluating the change in stiffness with temperature [4].

Asphalt concretes are damaged due to water and moisture that penetrate inside the mixture and separate the asphalt film over the aggregate in which a phenomenon is produced which is called stripping. Stripping is the loss of bond between aggregates and asphalt binder, which typically begins at the bottom of the hot mix asphalt layer and progresses upward [5].

Since it will take a long time in order to see the stripping trace on the surface of the mixture so determining stripping is difficult. On the other side surface stripping that is available may come from wheel trace, detachments and also cracks. In order to determine actual stripping potentials sections from the mixture samples should be investigated. There are different ways from which moisture and water can enter the mixture. Under ground water raises by capillary action, rain and seepage from surrounding ground are the main gates for water entry. If there is no enough drainage voids inside the mixture will be saturated with moisture and water and the adhesion between aggregate and asphalt will be vanished and causes failure of the pavement. Aggregates that are not clean or dusty prohibits complete coverage of the aggregate with asphalt binder. Dust and dirty materials on the surface of the aggregate decrease the asphalt film thickness around aggregate and allow the water or moisture to penetrate easily and break the bond between both. [30]

There are numerous mechanisms that can explain the associated phenomenon relating to HMA stripping:

Detachment, Displacement, Spontaneous emulsification, Film rupture, Pore pressure, Hydraulic scouring, PH instability.

2.2 ADHESION OF AGGREGATE AND ASPHALT

Adhesion can be defined as the molecular force that holds the two unlike bodies, Terrell and Shute hypothesized 3 theories to explain the adhesion bond between asphalt cement and aggregate. These are:

I. Chemical reaction
II. Molecular orientation and surface energy
III. Mechanical interlocking

Physical and chemical properties of both asphalt and the aggregate to a great extent affect the adhesion between both, and this adhesion is reduced in the presence of water [5].

According to Terrell and Shute one of these theories cannot explain adhesion alone. Adhesion can be explained with the combination of three of them [30].

Seven factors are identified that affect adhesion of asphalt to aggregate:

Viscosity of asphalt,

Surface texture of the aggregate,

Porosity of the aggregate,

Cleanliness of the aggregate,

Chemical composition of the asphalt and aggregate

Surface tension of the asphalt and aggregate,

Moisture content and temperature of aggregate during mixing with asphalt cement.

(Terrell and al-swailmil, 1990).

2.3 COHESION

Cohesion is defined as the intermolecular force that holds molecules in a solid or liquid together at the macro level of a compacted bituminous mixture. Capability of asphalt molecular structure that resists deformation is termed as cohesive strength of asphalt. Rheological indicators such as viscosity and mechanical indexes like tensile strength are commonly used for evaluating cohesion of asphalt, some researchers used dynamic shear rheometer and bending beam rheometer (DSR) and bending beam rheometer (BBR) in order to determine cohesion strength of asphalt and effect of cohesion on water stability of asphalt mixtures, like kanitpong et al [40]. Viscosity is one of the greatest responsible parameter for cohesive force development in asphalt binder, and it is known that viscosity of asphalt binder depend on temperature, and cohesive forces developed is inversely proportional to temperature [2].

2.4 NANO TECHNOLOGY IN ASPHALT MIXTURES

Nano technology is the creation of new materials, devices, and systems at the molecular level associated with atomic and molecular interactions strongly influence macroscopic material properties [24]. In recent years nanotechnology has gradually been incorporated into the field of modified asphalt [25]. In the last decade or so, nanotechnology emerged as the potential solution to greatly enhance the performance and durability of construction materials. Nano materials are defined as materials with at least one dimension that falls in the length scale 1-100 nm. Due to the small size and high surface area the property of Nano materials is much different from normal size materials [15]. To demonstrate many of the prospective applications, researchers have conducted a series of positive and effective efforts dealing with the preparation of modified asphalt to demonstrate the mechanism of modification and the resultant improvement in performance [25]. Some research showed that fatigue cracking, rutting and moisture resistance of asphalt binders and mixtures are improved with the addition of nanomaterials [15]. Ghafarpur 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 bitumen such as stiffness and aging resistance and decrease phase angle in compare to unmodified asphalt and asphalt mixtures properties such as stability, resilient modulus, and indirect tensile strength [32], [34]. And also F. Moghadas Nejad performed a test to investigate the performance characteristic of asphalt mixture by using zycosoil as an anti-strip agent with limestone and granite aggregate and it was seen that; fatigue life increased because of formation of a hydrophobic nano layer on aggregate, and aggregate coverage with zycosoil increased the amount of filler and decreased void content in asphalt mixture, and also zycosoil modified aggregate surface caused a better compaction of asphalt mixture [35]. Rohith N. et al investigated stability and Marshall property of hot mix asphalt specimens produced at 155‘C, 130’C and 115’C and compared with warm mix asphalt specimens contain zycotherm nanomaterial produced at 130’C and 115’C and it was concluded that stability and marshall property were improved with the addition of 0.1% of zycotherm nanomaterial [18]. Another study conducted on asphalt and asphalt mixtures by using nanosilica at 4% and 6% by weight of bitumen to estimate characteristic of nanomodified asphalt binder and mixture. Different tests such as (Rotational Viscosity RV, Dynamic Shear Rheometer DSR, Bending Beam Rhometer BBR, Fourier Transform Infrared Spectroscopy FTIR, Scanning Electron Microscopy SEM, Asphalt Pavement Analyzer APA, Dynamic Modulus DM and flow number FN were performed to analyze the change in chemical bonding and rheological properties of modified asphalt binder and also the performance characteristic of asphalt mixture after modification. From the results in general, the findings from the study show that the anti-aging property and rutting and fatigue cracking performance of nanosilica modified asphalt binders are enhanced and the addition of nanosilica in the control asphalt mixture significantly improves the dynamic modulus, flow number and rutting resistance of asphalt mixtures [41].

In particular to asphalt and asphalt mixture properties, Nanotechnology has the following known benefits:

- ❖ 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 [33]

CHAPTER THREE 3

RESEARCH PROGRAM

3.1 MATERIAL PREPARATION

3.1.1 AGGREGATE PREPARATION

Two types of aggregate materials (basalt and limestone) selected since they are mostly used for asphalt production because of their reliable physical properties such as strength, hardness, toughness and specific gravity and also their chemical composition which behave different in response to water and temperature are used in this study for asphalt mixture production to investigate zycotherm Nano material effect on their behavior and to compare them with respect to their usage with this material. Aggregates brought from two different sources in turkey …???…… and …???…Physical properties of both basalt and limestone are given in table (1) and (2).

Table (1) shows limestone aggregate test results

tests	units	test method	results	specification wear type 1
abrasion loss (loss angeles) max.	%	TS EN 1097-2	22	30
abrasion max.	%	TS EN 1097-6	0,61	2,0
Magnesium sulphate freezing loss max.	%	TS EN 1367-2	4,2	16
flatness index	%	TS 9582 EN 933-3	14	-
Peeling Strength, min.	%	KTŞ 2006	65-75	50
bulk specific gravity of coarse aggregate	g/cm^3	TS EN 1097-6	2,699	-
apparent specific gravity of coarse aggregate	g/cm^3	TS EN 1097-6	2,744	-
bulk specific gravity of fine aggregate	g/cm^3	TS EN 1097-6	2,650	-
apparent specific gravity of fine aggregate	g/cm^3	TS EN 1097-6	2,725	-
apparent specific gravity of filler	g/cm^3	TS EN 1097-7	2,779	-

Table (2) shows basalt aggregate test results

tests	units	test method	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.	%	KTŞ 2006	30-40	50
bulk specific gravity of coarse aggregate	g/cm^3	TS EN 1097-6	2,891	-
apparent specific gravity of coarse aggregate	g/cm^3	TS EN 1097-6	2,974	-
bulk specific gravity of fine aggregate	g/cm^3	TS EN 1097-6	2,812	-
apparent specific gravity of fine aggregate	g/cm^3	TS EN 1097-6	2,918	-
apparent specific gravity of filler	g/cm^3	TS EN 1097-7	2,940	-

3.1.2 AGGREGATE GRADATION

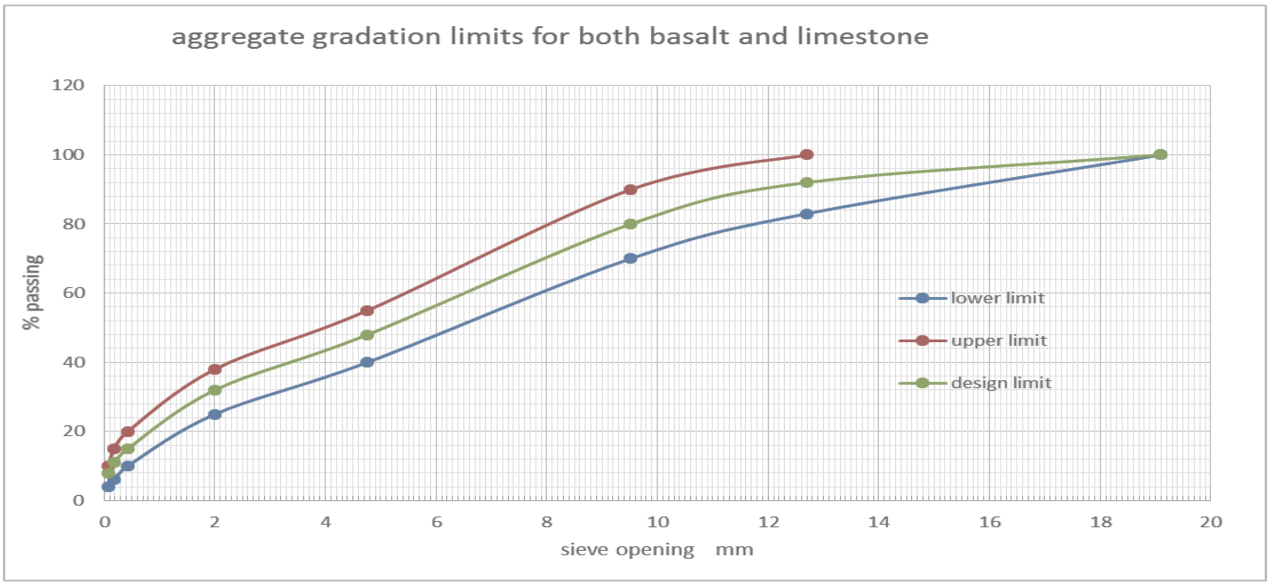
Aggregate gradation is one of the most important factors for design of bituminous mixtures, it affects the HMA performance in many respects including stiffness, durability, stability, permeability, workability, resistance to rutting and fatigue cracking and frictional resistance. Gradation that satisfy the requirement of general directorate of highways (KGM-

Ankara 2012) for wearing course was selected. Specification for wearing course selected and detail tabulated down in table (3).

Table (3) show aggregate gradation and specification limit.

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 (1) shows aggregate gradation limit for both limestone and basalt.



3.1.3 BITUMEN BINDER

Bitumen used for this study taken from tupras-izmit refinery. One grade of bitumen used for investigation purpose and it was 50/70 penetration grade bitumen. Physical properties such as penetration, softening point, specific gravity and other properties are given below in table (4).

Table (4) shows 50/70 grade bitumen test results.

tests	units	test method	50/70 Bitumen grade	specification
penetration	0,1 mm	TS EN 1426	61,0	**50 - 70**
softenning point	℃	TS EN 1427	50,4	**46 - 54**
Fraass max.	℃	TS EN 12593	-12	**-8**
flash point test min	℃	TS EN ISO 2592	332	**230**
specific gravity($d_{25/25}$)	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 (Thin Film Oven Test on the Move) Max.	%	TS EN 12607-1	0,5	**-**

3.1.4 ZYCOTHERM ADDITIVE

Zycotherm silane is an additive used for modifying hot mix asphalt concrete and offers a wide spectrum of benefits, due to the unique properties of Alkyl Siloxane Nano layer produced;

It resist Water and Frost

Extend service life of bitumen layers

Allows an extended construction season

Environment friendly

No Capital Expenditure on the bitumen mixing plant

Significant Benefits at low cost

Used for modifying bitumen and bituminous concrete mixtures in two way:

I. Zycotherm added directly to bitumen by weight of bitumen
II. Zycotherm diluted in water in (1:400) range and aggregate marinated with this dilution by weight of aggregate

3.1.5 TYPICAL SPECIFICATION OF ZYCOTHERM

Active ingredient : organosilane compound

Solid content : 65%±2%

Appearance : clear pale yellow liquid

Flash point : >80’C

Solubility in water : soluble

Figure (1) shows zycotherm dilution in water with 1:400

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

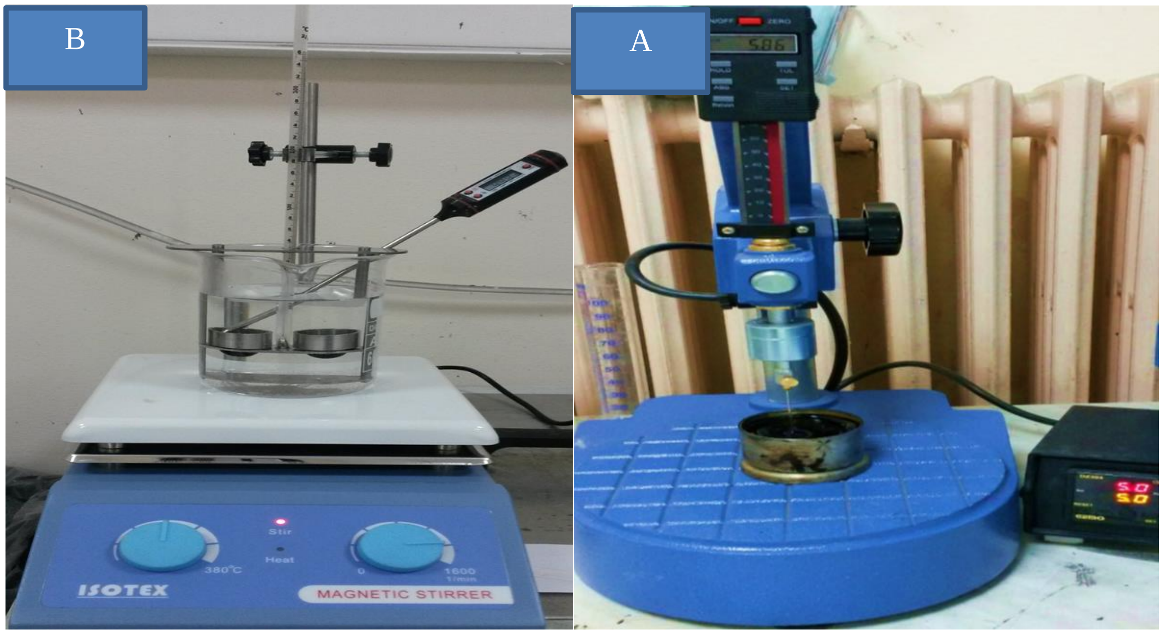
3.2.1 TESTS ON BITUMEN BINDER

3.2.1.1 PENETRATION TEST

Penetration test is one of the oldest bitumen tests, basic principle of this test is to determine the depth of penetration to which a sewing needle penetrate an asphalt sample under specified conditions of load, time and temperature.

As per AASHTO T 49 and ASTM D 5 standards the depth of penetration is measured in units of 0.1mm and reported in penetration unit under these conditions: load of 100 gm, time 5 seconds and temperature of 25’C (77’F) [42].

Figure (2) shows (A) penetration test equipment and (B) softening point test apparatus.



3.2.1.2 SOFTENING POINT TEST

Standard test method for softening point test is AASHTO T 53 and ASTM D 36: (Ring-and-Ball Apparatus) The softening point is defined as the temperature at which a bitumen sample can no longer support the weight of a 3.5-g steel ball. Basically, 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. 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 (1.0 inch) (AASHTO, 2000) [42].

3.2.1.3 SUPERPAVE BINDER TESTS

SHRP strategic highway research program spent nearly 50$ million which took approximately 5 year research effort (1987 to 1992) to establish Superpave binder specification, it is designed to address contribution of an asphalt binder to rutting, low temperature cracking, and fatigue cracking (AASHTO M320).weather and climate condition that the pavement will experience has the greatest influence on choosing or selecting asphalt binder for a particular mix design. Based on the climatic variation, the asphalt binders are tested and classified according to performance. For example, an asphalt binder that is expected to perform adequately in a climate with an average 7-day maximum temperature of 64’C and a minimum pavement design temperature of -22’C would be classified PG 64-22. (TxDOT) The brief descriptions of the Superpave binder tests are as follows:

3.2.1.3.1 ROLLING THIN FILM OVEN TEST (RTFOT)

Basically Rolling Thin Film Oven Test (RTFOT) designed for serving two purposes. The first purpose is to age the asphalt binder to represent the aging (oxidation and volatilization) associated with the production of asphalt mixtures. The second is to determine the mass quantity of volatiles lost from the asphalt during the process. The RTFOT continually exposes fresh films of binder to heat and air flow. A binder sample is heated until fluid, not exceeding 150°C. RTFOT bottles are loaded with 35 grams of binder. Eight sample bottles are required for Superpave binder testing. After aging, the two bottles containing the mass loss samples are weighed to the nearest 0.001 grams. The other bottles are poured into a single container to be used for Dynamic Shear Rheometer (DSR) testing and to be transferred into Pressure Aging Vessel (PAV) pans for additional aging [8].

Figure (3) shows (A) rolling thin film test and (B) pressure aging vessel test apparatus.

3.2.1.3.2 PRESSURE AGING VESSEL (PAV)

The Superpave Pressure Aging Vessel (PAV) procedure is used to simulate long-term aging of asphalt binders over time in the pavement. Pans containing 50 grams of RTFOT residue are placed in the PAV, which is pressurized with air at 2070 kPa, and aged for 20 hours. The range of PAV temperature used is between 90 and 110°C [8].

3.2.1.3.3 DYNAMIC SHEAR RHEOMETER TEST (DSR)

The Dynamic Shear Rheometer (DSR) test measures the viscoelastic shear properties of asphalt binders by testing them in an oscillatory mode. A sample of asphalt binder is placed between two parallel steel plates. The DSR test is used for measuring the asphalt properties at high and intermediate service temperatures for specification purposes. The two values to be measured from each test are the complex shear modulus, G*, and the phase angle, δ. These two test values are then used to compute G*/sinδ and G*sinδ. In the Superpave asphalt specification, permanent deformation is controlled by requiring the G*/sinδ 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 aging should have a G*sinδ value of less than 5000 kPa at a specified intermediate pavement temperature [8].

3.2.1.3.4 BENDING BEAM RHEOMETER TEST (BBR)

The Bending Beam Rheometer (BBR) test is used to measure the stiffness of asphalt binders at low surface temperatures. For specification testing, the test samples are fabricated from PAV-aged asphalt binders and tested at 10°C above the expected minimum pavement temperature, Tmin. The Superpave binder specification requires the stiffness at the test temperature after 60 seconds to be less than 300 MPa to control low-temperature cracking and the m-value at 60 seconds to be greater than or equal to 0.30 [8].

Figure (4) shows(A) bending beam rheometer test apparatus and (B) DSR test apparatus.

. Since one of the greatest factors that may affect the performance characteristics of asphalt mixture is the asphalt binder's properties so asphalt binders that have excellent properties may control permanent deformation and fatigue cracking and also provide a good stiffness in order to resist low temperature cracking. In order to know the effect of zycotherm addition on mass change with short term aging, effect on long term aging, fatigue cracking and stiffness three percentage (0.1%, 0.3% and 0.5%) by weight of binder added and these properties investigated.

3.2.2 TESTS ON BITUMINOUS MIXTURES

3.2.2.1 MIX DESIGN WITH MARSHAL METHOD

The aim and purpose of bituminous mix design is to determine an economical and efficient blend through several trial mixes. Properly designed bituminous mix will carry heavy traffic loads even under bad weather and climatic conditions. And also the requirement of structural and pavement surface characteristics will be maintained. The optimum bitumen content and aggregate gradation should provide these:

- ❖ Stable structure to resist deformation under sustained or repeated load. Aggregate interlocking and cohesion generally provide this resistance.
- ❖ Flexibility to withstand bending and deflection without cracking. Proper amount and grade of bitumen is essential for obtaining flexibility.
- ❖ An efficient construction operation in laying the mixture by providing sufficient workability.
- ❖ Durable pavement with sufficient binder content by providing a coating on the aggregate to produce a waterproofing layer.
- ❖ Space for additional compaction under traffic loading by providing sufficient air voids in the total mix.

Four principal bituminous mix design methods are in use, which vary in the size of the test specimen, compaction, and other test specification they are marshal method, hveem method, Superpave method and GTM methods. Marshal method is the widely used method throughout turkey. That is why we decided to design our samples with marshal method.

3.2.2.1.1 MARSHAL MIX DESIGN

For designing and evaluating hot mix asphalt this test procedure is used in routine test programs for the paving jobs. With this test procedure two main features assessed, density and voids analysed and also stability and flow can be tested.

Strength is measured in terms of 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 50.8mm/min till it was broken. The temperature 60'C represents the worst condition for a bituminous pavement that will experience [37].

Flow value which is an indicator of mixtures flexibility can be read with the change in samples diameter in the direction of load application between load application start and the time of maximum load by attaching a dial gauge to read plastic deformation due to loading, and this is called flow value.

VOLUMETRIC PROPERTIES

Fundamentally mix design is meant to determine the volume of asphalt binder and aggregate necessary to produce a mixture with the desired properties (Robert et al., 1996).

Because of weight measurement was much easier so weights were taken and converted to volume by using specific gravities.

Important volumetric properties are;

Theoretical maximum specific gravity (Gt)

Bulk specific gravity (Gm)

Air void percentage (Vv %)

Volume of bitumen percentage (Vb %)

Voids in mineral aggregate percentage (VMA %)

Voids filled with bitumen percentage (VFB %)

Theoretical specific gravity is the specific gravity without considering air voids, it's expressed as:

$$G_t = \frac{W_1 + W_2 + W_3 + W_b}{\frac{W_1}{G_1} + \frac{W_2}{G_2} + \frac{W_3}{G_3} + \frac{W_b}{G_b}}$$

Where w1= weight of coarse aggregate in the total mix,

W2= weight of fine aggregate in the total mix,

W3= weight of filler in the total mix,

Wb= is the weight of bitumen in the total mix,

G1 is the apparent specific gravity of coarse aggregate,

G2 is the apparent specific gravity of fine aggregate,

G3 is the apparent specific gravity of filler,

Gb is the apparent specific gravity of bitumen.

Bulk specific gravity of mix Gm

The bulk specific gravity of the mixture Gm is the specific gravity considering air voids and is expressed as:

$$G_m = \frac{W_m}{W_m - W_w}$$

Wm =is the weight of mixture in air,

Ww =is the weight of mixture in water.

Air void percentage (Vv %)

Air void percentage Vv% is the percentage of air voids by volume in the sample and is expressed as:

$$V_v = \frac{(G_t - G_m)100}{G_t}$$

Gt = is the theoretical specific gravity of the mix,

Gm = is the bulk specific gravity of the mix.

Volume of bitumen percentage (Vb %)

The volume of bitumen percentage Vb% is the percentage of volume of bitumen to the total volume of the sample and expressed as:

$$V_b = \frac{\frac{W_b}{G_b}}{\frac{W_1+W_2+W_3+W_b}{G_m}}$$

W1 =is the weight of coarse aggregate in the total mixture,

W2 =is the weight of fine aggregate in the total mixture,

W3 =is the weight of filler in the total mixture,

Wb =is the weight of bitumen in the total mixture,

Gb is the apparent specific gravity of bitumen,

Gm is the bulk specific gravity of mixture.

Voids in mineral aggregate 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 void percentage in the mixture,

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

Voids filled with bitumen percentage (VFB %)

V FB% 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:

$$VFB = \frac{V_b \times 100}{VMA}$$

Vb = is the percent of bitumen content in the mixture

VMA = is the percentage of voids in the mineral aggregate.

3.2.2.1.2 MIX VOLUMETRIC PARAMETERS IMPORTANCE

Aggregates are bind together with asphalt binder 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 [37].

Hot weather condition of the summer causes bleeding if there is not enough void space between aggregate particles, though a specific amount of void is necessary even after the last phase of compaction. However a point should be taken into consideration that having too much void will weaken the mixture from its elastic modulus and fatigue life point of view.

Table (5) Design specification for wearing course that is used by (KGM-ankara-2012)

design parameter	wearing course specification	
	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**	**-**

Laboratory works of mixture designs were done with Marshall Method and the test procedure was as follow:

Preparation of test specimen

1150 gm of aggregate and filler is heated to a temperature of 160-170’C and bitumen binder is heated to 145-155 ‘C. a trial percentage of binder such as 3.5% by weight of mineral aggregate was selected as the first trial and mixed thoroughly with the heated aggregate at 135’C. The mix was poured in a preheated mold and compacted by a hammer having a weight of 4.5 kg and failed from height of 45.7 cm, each side compacted with 75 blow at 120’C to prepare laboratory specimens of 63.5±3mm and diameter of 101.5±1 mm. three 3 samples of each trial produced and the average taken as the actual value. The bitumen binder content is changed and the above procedure is repeated for producing next samples, usually bitumen content is increased with 0.5% increments.

After producing required amount of samples, the samples are extracted with special extractor and then in order to determine volumetric properties each specimen's height measured three 3 times in order to determine volume of the specimen.

After finding the bulk specific gravity of each specimen by weighing them in air, in water and saturated surface dry weight and measuring each of the volumetric properties like theoretical specific gravity, air void percentage, volume of bitumen percentage, voids filled with bitumen and voids in mineral aggregate. In order to determine stability and flow the specimens are conditioned by putting them inside water bath at 60'C and remain inside it for 30±5 min before testing. After conditioning the samples are taken out and surface dried with a cloth and then tested.

Figure (5) agroup standard marshal specimen of hot mix asphalt produced for testing

Determining Marshall Stability and flow

Stability of a test sample is the maximum load needed to produce failure or break when the sample is conditioned as mentioned above and placed in a special test head (shown in figure (6)) and the load is applied at a constant strain (5 cm/min). While the stability test is in progress a dial gauge is used to measure the vertical deformation of the sample. The deformation at the failure point expressed in units of 0.25 mm is called Marshall Flow value of the specimen.

The stability value and Marshall flow value is then checked with Marshall mix design specification chart given in table (5). Mixes with very high stability value and low flow value are not desirable as the pavements constructed with such mixes are likely to develop cracks due to heavy moving loads.

Figure (6) shows the standard marshal test device.

Optimum bitumen content determination

In order to determine optimum bitumen content 6 six graphs are sketched, these graphs are:

Bulk specific gravity versus bitumen content,

Void content % percentage versus bitumen content,

Stability (KN) versus bitumen content,

Flow (mm) versus bitumen content,

Voids filled with bitumen % percentage versus bitumen content,

Voids in mineral aggregate % percentage versus bitumen content.

And then bitumen content that corresponds to: Maximum bulk specific gravity, Maximum stability, 4% air void

Is determined and the average of these 3 design specification is taken.

The average bitumen content is compared with each: Flow, Voids filled with bitumen % percentage, Voids in mineral aggregate % percentage.

If it’s in the range that is given in table (5) then it is decided to be the optimum bitumen content, if so it is discarded and the test should be repeated.

The above procedure followed for our test and optimum bitumen content was found for two types of aggregate basalt and limestone, for the proposed mix design gradation three 3 samples prepared for each bitumen content within the range of 3.5% --6.0% at 0.5% increments.in accordance to ASTM D 1559 using 75 blows per each side . All the bitumen content were in percentage by weight of the aggregate mix.

Optimum Bitumen content for limestone aggregate

Volumetric properties of each mix is given in table (6) and also the graphs illustrating the relation between bitumen content and (bulk specific gravity, stability, flow, void content, voids in mineral aggregate and voids filled with aggregate) respectively are presented down:

Maximum stability at 10.45 kn corresponds to 4.05 % bitumen content

Maximum bulk specific gravity at 2.47 corresponds to 5.05 % bitumen content

4% void content corresponds to 4.9 % bitumen content

And average bitumen content = **4.05+5.05+4.9 / 3 =4.67 %**

At 4.67% bitumen content flow =3.2 mm which falls within the limit (2-4)

At 4.67% bitumen content voids filled with bitumen=68% which falls within the limit (65-75)%

At 4.67% bitumen content voids in mineral aggregate = 15.85 % which falls within the limit (minimum 14%).

Optimum Bitumen content for limestone aggregate = 4.67%

Table (7) shows average mix design data of each bitumen content percentage.

SPECIMEN	BITUMEN	WHEIGHT OF	WHEIGHT OF	VOLUME	BULK SP.GR.	TH.SP.GR.	AIR VOID	WHEIGHT OF	V.M.A	V.F.B.	FLOW	STABILITY	FLOW	STABILITY
NO	%	MIXTURE (gm)	BITUMEN (gm)	cm3	Gm	Gt	% Vv	BITUMEN Vb%	VMA %	% Vfb	COR mm	COR. KN	mm	kn
1.00	3.50	1,190.25	40.25	527.34	2.33	2.61	10.54	7.72	18.26	42.33	2.60	7.62	3.00	7.93
2.00	4.00	1,196.00	46.00	532.32	2.39	2.59	7.59	9.00	16.59	54.41	3.21	10.38	3.34	10.66
3.00	4.50	1,201.75	51.75	524.11	2.42	2.57	5.83	10.19	16.03	63.65	2.94	9.43	2.98	9.43
4.00	5.00	1,207.50	57.50	504.80	2.45	2.55	3.74	11.44	15.18	76.10	3.94	9.44	4.17	9.02
5.00	5.50	1,213.25	63.25	501.56	2.43	2.53	3.79	12.43	16.22	76.65	5.26	9.24	5.28	8.89

Figure (7) shows stability versus bitumen content.

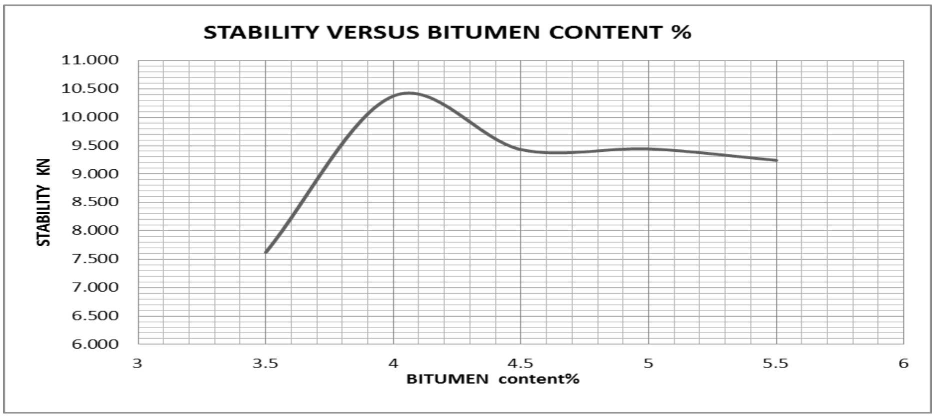


Figure (8) shows flow versus bitumen content.

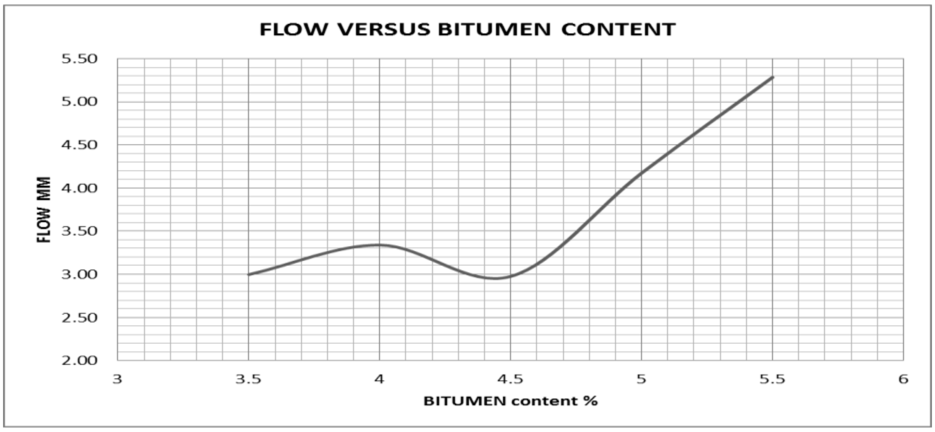


Figure (10) shows bulk specific gravity versus bitumen content.

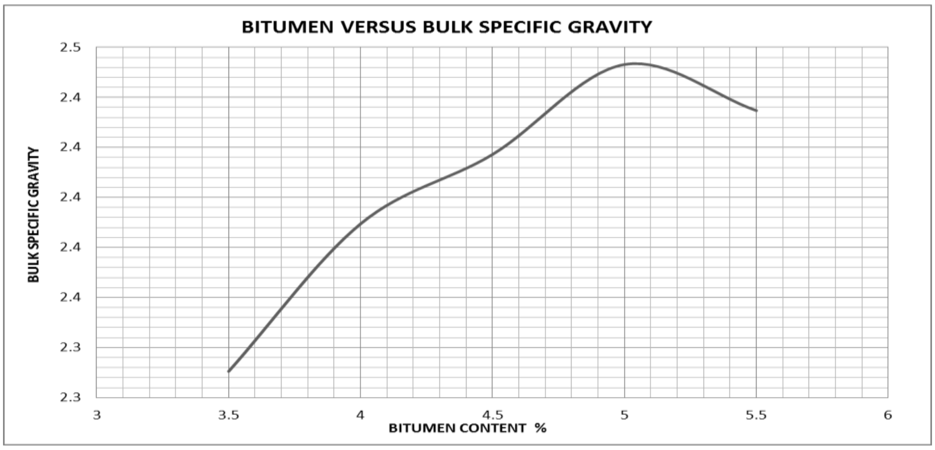


Figure (9) shows void content versus bitumen content.

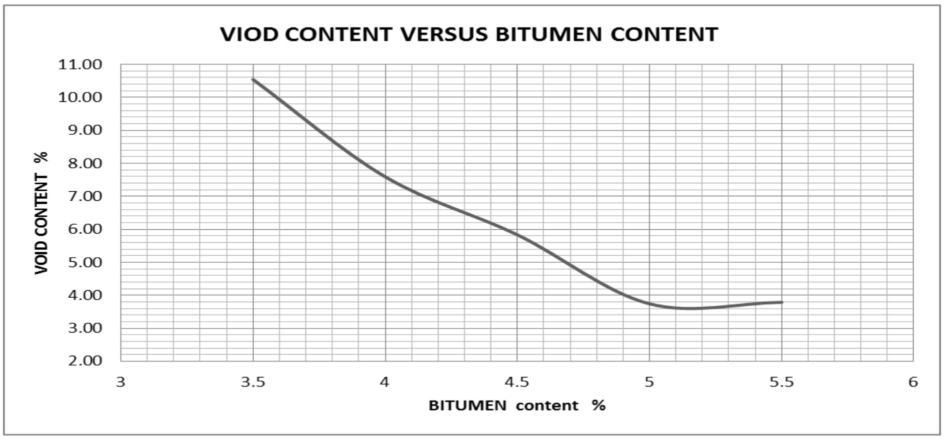


Figure (11) shows VMA content versus bitumen content.

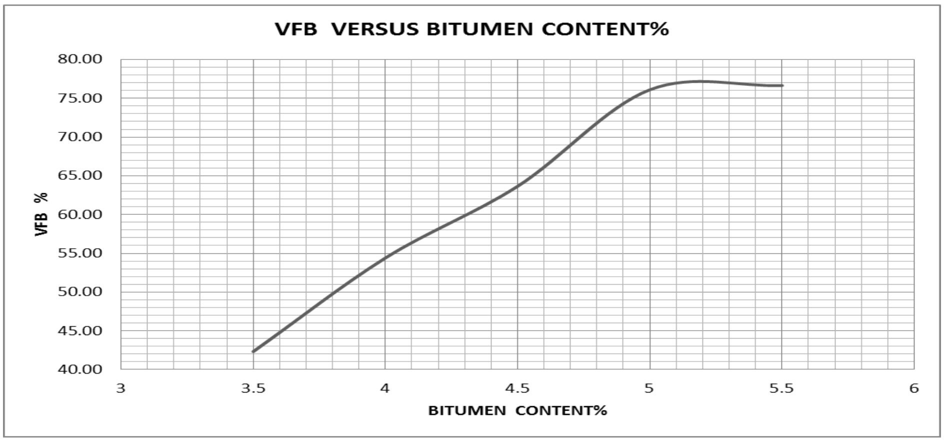
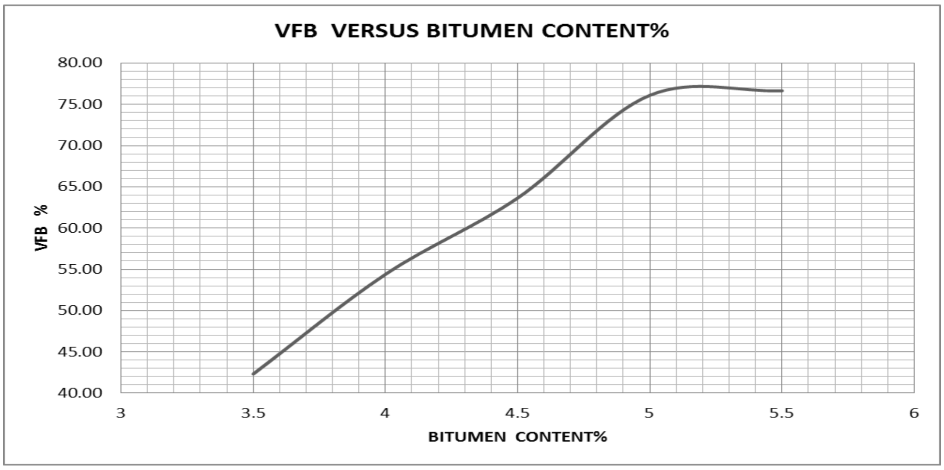


Figure (12) shows VFB content versus bitumen content.



Optimum Bitumen content for basalt aggregate

Volumetric properties of each mix is given in table (9) and also the graphs illustrating the relation between bitumen content and (bulk specific gravity, stability, flow, void content, voids in mineral aggregate and voids filled with aggregate) are presented down:

Maximum stability at 9.13 KN corresponds to 4.05% bitumen content

Maximum bulk specific gravity at 2.55 corresponds to 5.5% bitumen content

4% void content corresponds to 5.55% bitumen content

And average bitumen content = 5.55+5.5+4.05/3=5.03%

At 5.03% bitumen content flow =3.6 mm which falls within the limit (2-4)

At 5.03% bitumen content voids filled with bitumen=65% which falls within the limit (65-75) %.

At 5.03% bitumen content voids in mineral aggregate = 18.5 % which falls within the limit (minimum 14%).

Optimum Bitumen content for basalt aggregate = 5.03%

Table (10) shows average mix design data of each bitumen content percentage.

SPECIMEN NO	BITUMEN %	WHEIGHT OF MIXTURE (gm)	WHEIGHT OF BITUMEN (gm)	VOLUME cm3	BULK SP.GR. Gm	TH.SP.GR. Gt	AIR VOID % Vv	VOLUME OF BITUMEN Vb%	V.M.A VMA %	V.F.B. % Vfb	FLOW CORRECTED mm	STABILITY CORRECTED kn	FLOW mm	STABILITY kn
1	3.5	1190.3	40.3	526.2	2.40	2.75	12.63	7.55	20.59	38.68	2.470	7.81	2.90	8.03
2	4	1196	46	513.2	2.44	2.72	10.41	9.21	19.64	46.94	3.142	9.13	3.87	8.88
3	4.5	1201.75	51.75	503.6	2.45	2.69	8.97	10.34	19.30	53.64	3.508	8.59	3.81	8.36
4	5	1207.5	57.5	494.8	2.52	2.70	6.61	11.78	18.36	64.08	3.550	8.23	3.57	7.82
5	5.5	1213.25	63.25	489.9	2.55	2.67	4.20	13.02	17.24	75.83	5.409	8.20	5.57	7.52
6	6	1219	69		2.51	2.66	5.18	13.94	19.12	73.01	8.11	9.10	8.10	8.35

Figure (13) shows stability versus bitumen content.

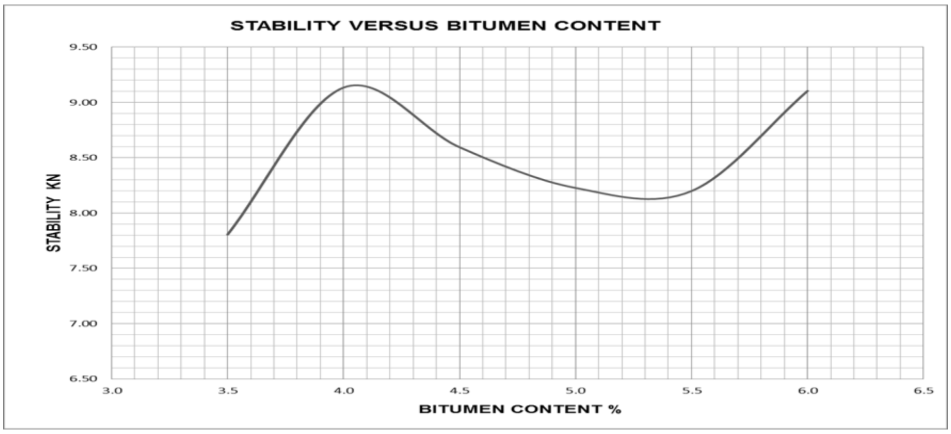


Figure (14) shows flow versus bitumen content.

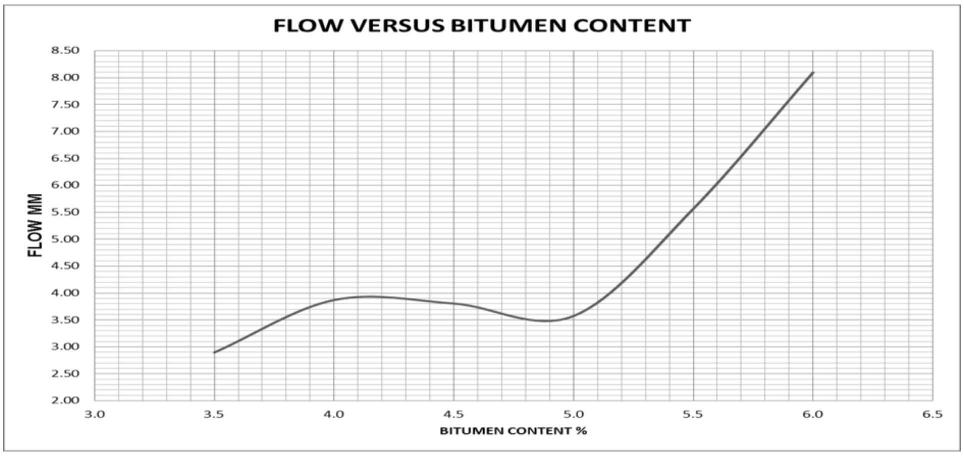


Figure (15) shows bulk specific gravity versus bitumen content.

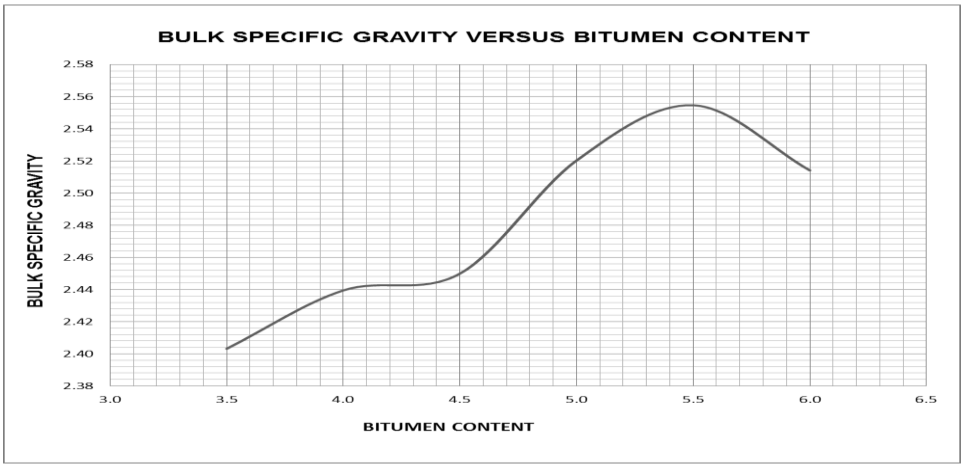


Figure (16) shows void content versus bitumen content.

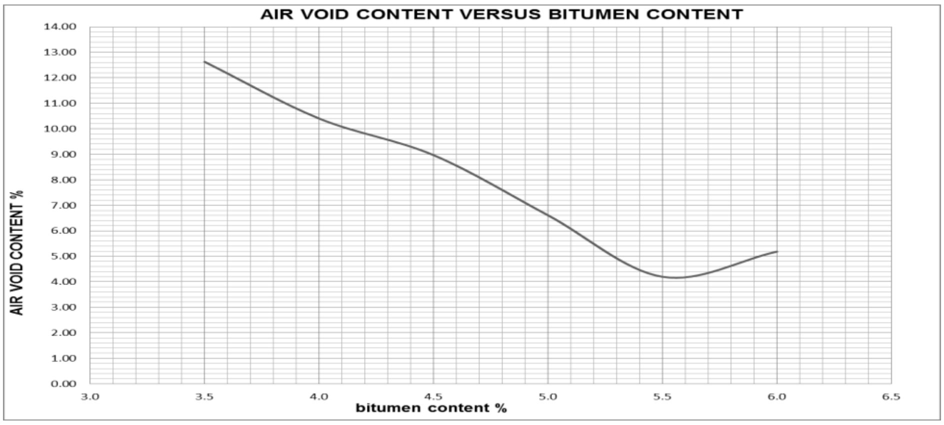


Figure (17) shows VMA versus bitumen content.

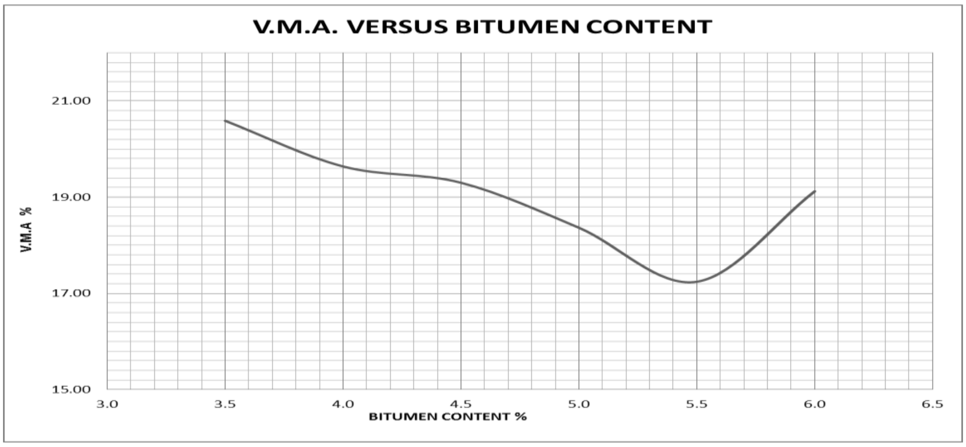
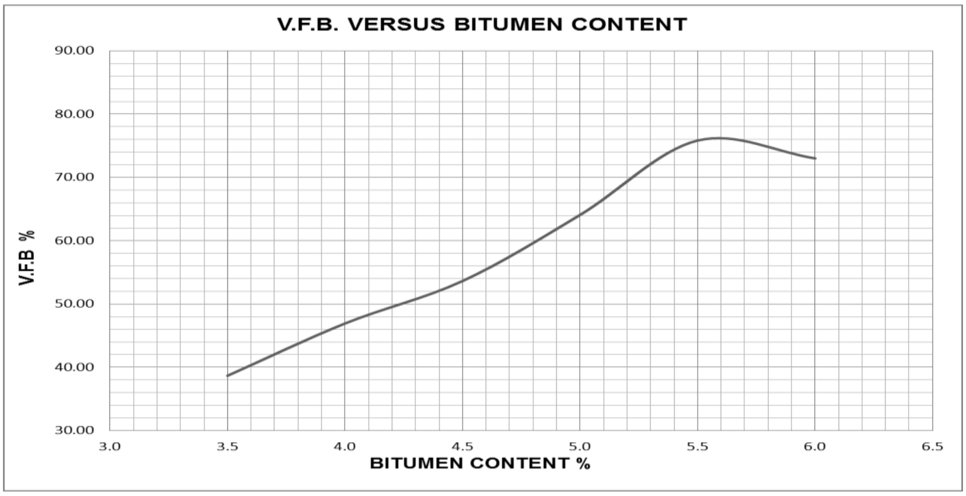


Figure (18) shows VFB versus bitumen content.



3.2.2.2 DETERMINING COMPACTION EFFORT AT 7%±1% AIR VOID FOR INDIRECT TENSILE TEST

In order to determine the effect of moisture susceptibility on hot bituminous mixtures standards prefers to perform the test on bituminous samples at 7±1% air voids. In this study different specimens prepared at optimum bitumen content and with applying different compaction effort changes in void contents were investigated. For this purpose mixtures 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 (11) and (12) shows the detail of the samples prepared at optimum bitumen content for both limestone and basalt and compacted with different compaction effort and void content change investigated.

Table (11) shows the detail of basalt samples prepared at 5.03% bitumen content.

BLOW NO	SPECIMEN NO	WHEIGHT OF BITMEN (gm)	WHEIGHT OF MIXTURE (gm)	WHEIGHT IN AIR (gm)	WHEIGHT IN WATER (gm)	S.S.D.WHEIGHT IN AIR (gm)	BULK SP.GR. Gm	TH.SP.GR. Gt	AIR VOID % Vv
30	1	57.845	1207.845	1,201.50	714.30	1,210.80	2.42	2.704	10.51
	2	57.845	1207.845	1,217.98	725.30	1,222.17	2.45	2.704	9.35
	3	57.845	1207.845	1,205.60	718.50	1,214.20	2.43	2.704	10.05
	AVERAGE						2.43	2.704	9.97
40	1	57.845	1207.845	1,212.34	726.85	1,214.80	2.48	2.704	8.12
	2	57.845	1207.845	1,202.36	717.80	1,204.93	2.47	2.704	8.72
	3	57.845	1207.845	1,204.80	719.90	1,205.30	2.48	2.704	8.21
	AVERAGE						2.48	2.704	8.35
50	1	57.845	1207.845	1,199.27	720.80	1,200.58	2.50	2.704	7.56
	2	57.845	1207.845	1,208.82	728.10	1,210.17	2.51	2.704	7.26
	3	57.845	1207.845	1,204.34	722.38	1,206.44	2.49	2.704	7.99
	AVERAGE						2.50	2.704	7.60
60	1	57.845	1207.845	1,203.88	726.10	1,205.99	2.51	2.704	7.22
	2	57.845	1207.845	1,205.95	723.70	1,207.16	2.49	2.704	7.75
	3	57.845	1207.845	1,205.58	725.30	1,206.74	2.50	2.704	7.39
	AVERAGE						2.50	2.704	7.46
75	1	57.845	1207.845	1,207.88	732.80	1,208.82	2.54	2.704	6.16
	2	57.845	1207.845	1,204.82	731.40	1,206.06	2.54	2.704	6.13
	3	57.845	1207.845	1,204.20	730.50	1,205.30	2.54	2.704	6.20
	AVERAGE						2.54	2.704	6.16

Figure (16) shows the relation between void content and blow number for basalt aggregate

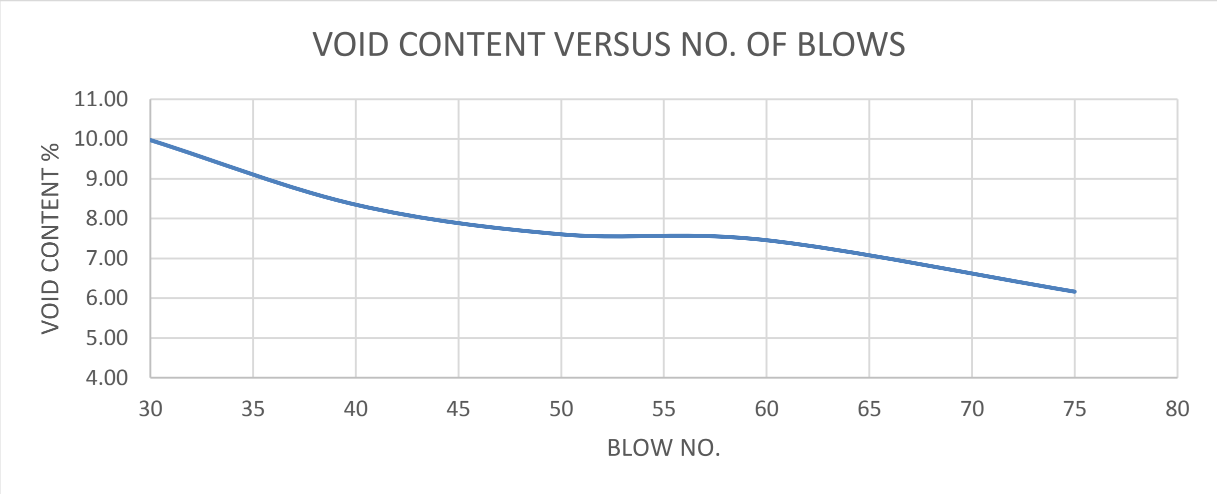


Table (12) shows the detail of limestone samples prepared at 4.67% bitumen content.

BLOW NO.	SPECIMEN NO	WHEIGHT OF BITUMEN (gm)	WHEIGHT OF MIXTURE (gm)	WHEIGHT IN AIR (gm)	WHEIGHT IN WATER (gm)	S.S.D.WHEIGHT IN AIR (gm)	BULK SP.GR. Gm	TH.SP.GR. Gt	AIR VOID % Vv
30	1	53.705	1203.705	1,197.04	698.01	1,208.50	2.345	2.560	8.40
	2	53.705	1203.705	1,201.20	700.60	1,210.30	2.357	2.560	7.94
	3	53.705	1203.705	1,202.30	699.30	1,209.80	2.355	2.560	8.00
	AVERAGE						2.352	2.560	8.12
40	1	53.705	1203.705	1,200.65	699.22	1,211.48	2.344	2.560	8.44
	2	53.705	1203.705	1,203.10	700.90	1,208.08	2.372	2.560	7.34
	3	53.705	1203.705	1,201.70	700.10	1,209.80	2.358	2.560	7.90
	AVERAGE						2.358	2.560	7.90
50	1	53.705	1203.705	1,202.08	701.32	1,206.86	2.378	2.560	7.12
	2	53.705	1203.705	1,201.35	704.20	1,206.73	2.391	2.560	6.62
	3	53.705	1203.705	1,200.50	701.32	1,204.35	2.387	2.560	6.78
	AVERAGE						2.385	2.560	6.84
60	1	53.705	1203.705	1,203.40	703.70	1,207.40	2.389	2.560	6.67
	2	53.705	1203.705	1,200.59	702.60	1,205.70	2.386	2.560	6.78
	3	53.705	1203.705	1,202.30	702.25	1,205.63	2.388	2.560	6.70
	AVERAGE						2.388	2.560	6.72
75	1	53.705	1203.705	1,201.61	710.20	1,203.53	2.44	2.561	4.88
	2	53.705	1203.705	1,197.94	706.14	1,200.02	2.43	2.561	5.27
	3	53.705	1203.705	1,200.30	708.00	1,202.05	2.430	2.560	5.10
	AVERAGE						2.430	2.560	5.07

Figure (17) shows the relation between void content and blow number for limestone aggregate.

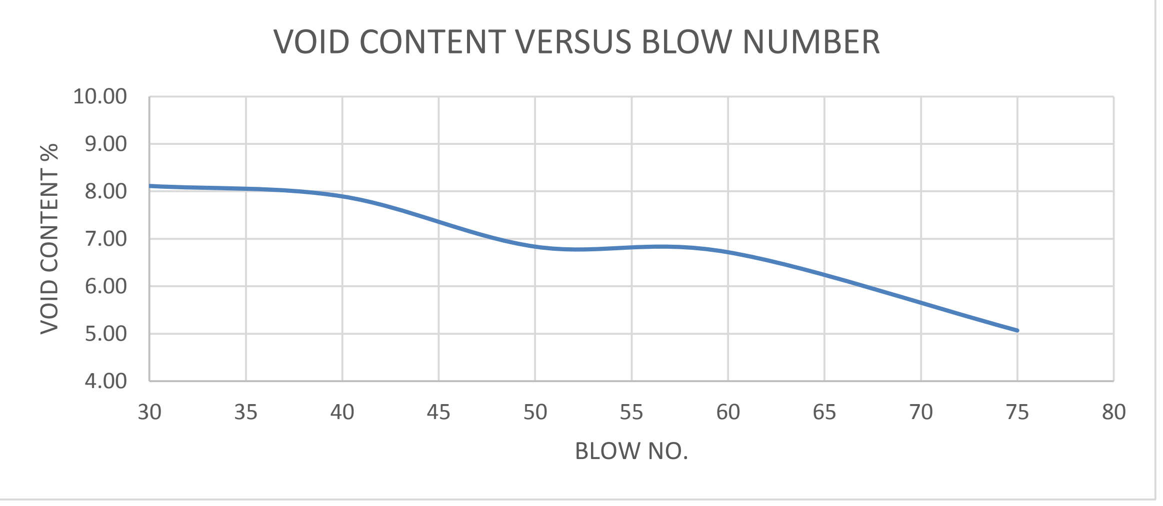


Figure (16) and (17) shows the relation between void content and compaction effort (or blow number) for mixtures produced with both limestone and basalt aggregate.

From figure (16) compaction effort (or blow number) of 50 selected as the compaction effort which produces a void content of 6.8% for limestone.

From figure (17) compaction effort (or blow number) of 60 selected as the compaction effort which produces a void content of 7.4% for basalt.

3.2.2.3 INDIRECT TENSILE STRENGTH, RETAINED STABILITY AND STRIPPING VALUE OF AGGREGATE TESTS

Asphalt mixtures made from certain combination of materials maybe sensitive to water in the finished pavement or it can be said that Presence of moisture or water in a mixture of hot mix asphalt is a critical factor, and it may cause the flexible pavement to fail. Since the asphalt binder is the glue that holds the aggregate together so rapid failure of the pavement can be expected if the asphalt cannot stick to the aggregate. Adhesion loss of aggregate with asphalt binder is studied by utilizing retained stability and indirect tensile strength test to examine the effect of zycotherm Nano material additive on resistance to moisture induced damage. 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.

3.2.2.3.1 RETAINED STABILITY TEST

The test is performed by preparing standard marshal samples (of 100 mm diameter and 63.5mm height) at optimum bitumen content by applying 75 blows on each side for both limestone and basalt aggregate. Three specimens are selected as a control and tested without moisture conditioning and the three more are selected to be conditioned by keeping them in water bath maintained at 60 ‘C for 24 hrs. prior to testing. Marshal stability and flow of compacted specimens is determined after conditioning them with standard marshal testing machine. Retained stability expressed as the stability after conditioning samples in water maintained at 60‘C for 24 hrs. prior to testing divided by stability under standard conditions multiplied by 100 [37] [38].

Retained stability= (stability of conditioned samples/stability unconditioned samples)*100

Zycotherm addition to bitumen

In this study zycotherm Nano material added to the bitumen binder at 3 three different percentages (0.1%, 0.3% and 0.5%) by weight of bitumen and bituminous mixtures produced with both aggregate types, basalt and limestone. Table (13) to table (20) shows the detail. From these mixtures retained stability index was measured and compared with control samples (sample produced with 0% zycotherm at the same condition of mixtures that contain zycotherm).

Determining amount of zycotherm at each percentage used for one specimen:

Limestone aggregate (OBC= 4.67%)

1150*0.0467=53.705 gm

0.1% →

53.705*0.1/100=0.053705 gm

It means **0.054** gm pure zycotherm used for preparation of a specimen at 0.1% zycotherm addition.

0.3% →

53.705*0.3/100=0.16 gm pure zycotherm used for preparation of a specimen at 0.3% zycotherm addition.

0.5% →

53.705*0.5/100=0.27 gm pure zycotherm used for preparation of a specimen at 0.5% zycotherm addition.

Basalt aggregate (OBC=5.03%)

1150*0.0503=57.845 gm

0.1% →

57.845*0.1/100=0.057845 gm

It means **0.058** gm pure zycotherm used for preparation of a specimen at 0.1% zycotherm addition.

0.3% →

57.845*0.3/100=0.17 gm pure zycotherm used for preparation of a specimen at 0.3% zycotherm addition.

0.5% →

57.845*0.5/100=0.29 gm pure zycotherm used for preparation of a specimen at 0.5% zycotherm addition.

Aggregate marinating with zycotherm dilution

Zycotherm diluted in water at 1:400 means one part of zycotherm diluted in 400 parts of water. Both aggregate type limestone and basalt marinated with this dilution at 3 different percentages (1%, 3%, and 5%) by weight of aggregate. After this marinated aggregates heated up to 170'C and bituminous mixtures produced with the marinated aggregate, retained stability was measured and compared with control samples (sample produced with 0% zycotherm dilution at the same condition of mixtures that contain zycotherm dilution). Table (21) to table (26) shows the detail of both type of mixtures produced with this system.

Determining amount of zycotherm at each percentage used for one specimen.

Zycotherm diluted in water at 1:400 it means each percentage used (1%,3% and 5%) must be divided by 400 to get the amount of pure zycotherm used.

1% ⟶

1150*1/100=11.5 gm zycotherm dilution used at 1%

11.5/400=0.02875 gm pure zycotherm used for preparation of a specimen at 1% zycotherm dilution addition.

3% ⟶

1150*3/100=34.5 gm zycotherm dilution used at 3%

34.5/400=0.08625 gm pure zycotherm used for preparation of a specimen at 3% zycotherm dilution addition.

5% ⟶

1150*5/100=57.5 gm zycotherm dilution used at 5%

57.5/400=0.14375 gm pure zycotherm used for preparation of a specimen at 5% zycotherm dilution addition.

3.2.2.3.2 INDIRECT TENSILE STRENGTH TEST

Indirect tensile test is used to determine the structural properties of a hot bituminous mixture through laboratory testing. It is used also for evaluating moistures susceptibility of bituminous mixtures, and this can be shown with the reduction in the loss of indirect tensile strength after conditioning samples in water at 60’C. Tensile strength is produced with the cohesion inside asphalt binder and the adhesion between binder and aggregate.

Therefore, any additives that can generate a higher tensile strength in the HMA mix in the dry and moisture-conditioned stages will improve the long-term performance of an HMA pavement. [35].It is conducted by applying a diametral compressive load along a vertical plane of a cylindrical specimen. The indirect tensile strength St is the maximum horizontal stress that develops at the center of the specimen due to the loading. At failure, the horizontal (tensile) and vertical (compressive) stresses at the center reach the maximum values [39].

Indirect tensile strength is calculated with the following formula:

$$\mathbf{St = 2P/\pi dt}$$

St: indirect tensile strength

P: maximum applied load

d: specimen diameter

t: specimen height

The test is performed by preparing standard marshal samples of (100 mm diameter and 63.5 mm height) at optimum bitumen content and compacting each side of the specimen with (50 blow for limestone and 60 blow for basalt) to get an air void content of 7%±1%.

Figure (18) shows indirect tensile test apparatus

Zycotherm addition to bitumen

In this study zycotherm Nano material added to the bitumen binder at 3 three different percentages (0.1%, 0.3% and 0.5%) by weight of bitumen and bituminous mixtures produced with both aggregate types, basalt and limestone. Table (27) to table (34) shows the detail. From these mixtures indirect tensile strength and tensile strength ratio was measured and compared with control samples (sample produced with 0% zycotherm at the same condition of mixtures that contain zycotherm).

Aggregate marinating with zycotherm dilution

Zycotherm diluted in water at 1:400 means one part of zycotherm diluted in 400 parts of water. Both aggregate type limestone and basalt marinated with this dilution at 3 different percentages (1%, 3%, and 5%) by weight of aggregate. After this marinated aggregate samples heated up to 170’C inside oven and Bituminous mixtures produced with the marinated aggregate with zycotherm dilution, indirect tensile strength and tensile strength ratio was measured and compared with control samples (samples produced with 0% zycotherm dilution at the same condition of mixtures that contain zycotherm dilution). Table (35) to table (40) shows the detail of both type of mixtures produced with this system.

After determining the physical properties of the specimens like; bulk specific gravity, volume, theoretical specific gravity, void content, voids in mineral aggregate and voids filled with asphalt, according to the standards all the specimens were left in oven at 60‘C for 18 hours.

After this period they were taken out and divided in two groups, each group approximately have the same void content. And after that dry samples were left inside 25’C water bath for 1 hour and after that the samples were taken out and subjected to an axial load with the marshal test equipment and the maximum load that produces failure was determined and indirect tensile strength was calculated.

For the case of Conditioned samples they were placed inside water bath at 25’C and left for 8 hour till they were saturated approximately 80% and after this period they were put inside 60’C water bath and left for 24 hours. Just like dry samples, before testing them, conditioned samples after 24 hour taken out from the bath and they were left inside water bath at 25’C for extra 1 hour and after that they were subjected to an axial load with the marshal test equipment and the maximum load that produces failure was determined and indirect tensile strength was calculated for each specimen. By dividing indirect tensile strength of conditioned samples by indirect tensile strength of unconditioned (dry) samples a measure of moisture or water susceptibility which is tensile strength ratio (TSR) can be determined.

Tables (27) to table (40) shows the conditioned, unconditioned and tensile strength ratio of both basalt and limestone which were produced with zycotherm either by marinating aggregate or by adding directly to the bitumen.

3.2.2.3.3 STRIPPING VALUE OF AGGREGATE

IS: 6241-1971 states that stripping value of aggregate is the ratio of uncovered area observed visually to the total area of aggregates, it is expressed as percent.

The test carried out as this; 200 gm of clean aggregate which passed through 20 mm and retained on 12.5 mm sieves heat up to 170’C. Five percent of bitumen binder heated up to 150’C. The aggregate and the binder mixed thoroughly till completely coated and the mixture transferred to a 500 ml beaker and allowed to cool at room temperature for 2 hour. Distilled water added till the aggregate immersed in water. The beaker is kept in a water bath maintained at 40’C, the water in the bath should be at least half of the height of the beaker. After 24 hour the beaker taken out form the bath and stripping value estimated.

Control sample and samples with 0.1% zycotherm added to bitumen prepared with both type of aggregate limestone and basalt. It was observed that addition of zycotherm greatly influence the stripping resistance of both aggregate type in comparison to control samples.

CHAPTER FOUR 4

DATA ANALYSIS AND RESULTS

Tests performed within this research were two categories: tests on bitumen binder and tests on hot mix asphalt concrete. In order to determine effect of zycotherm addition with both addition system and on both aggregate type so each test result will be analysed and further influence on flexible pavement will be evaluated.

4.1 OPTIMUM BITUMEN CONTENT, VOLUMETRIC PROPERTIES AND RETAINED STABILITY

Aggregate Materials basalt and limestone required for hot mix asphalt concrete production were brought from two different sources in turkey and tests required to characterize and assure the reliability of using these materials performed as shown in table

(1) and table (2) and gradation with nominal size of 12.5mm were designed for testing and evaluation, and also gradation of these aggregates were done according to (KGM-ankara-2008) specification for wearing course given in table (3) for both aggregate types.

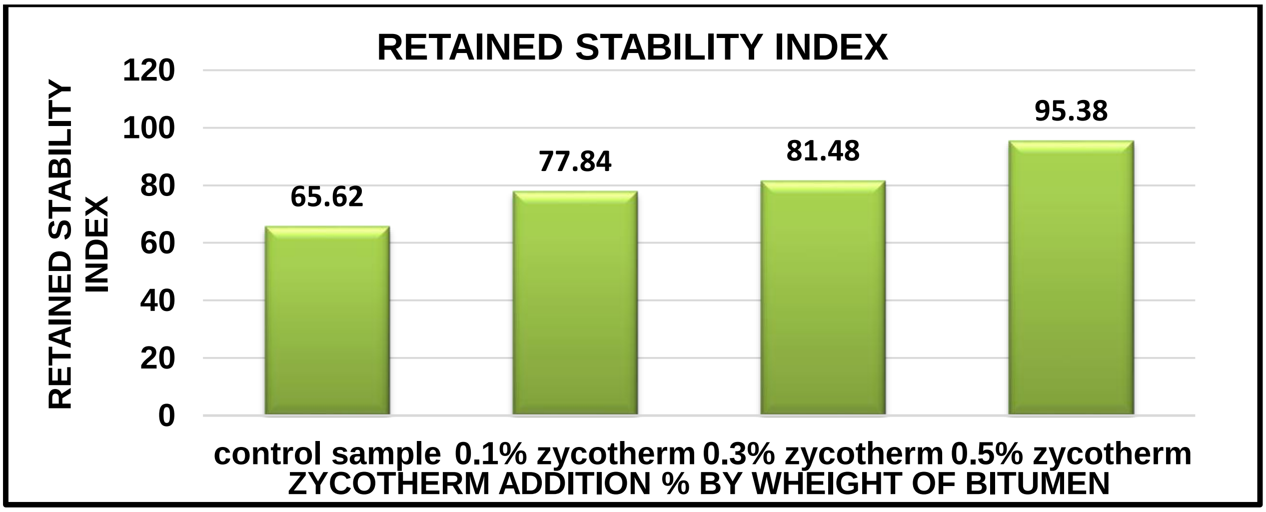
After this marshal specimens with 5 different bitumen content (3.5%, 4%, 4.5%, 5%, and 5.5%) for both aggregate types prepared to determine optimum bitumen content. It was noticed that with increasing bitumen content void content decreased, stability increased and flow decreased. Table (5) specifies limits for design parameters like void content, stability and flow. Form the figures (7) to figure (18) it was concluded that optimum bitumen content for limestone was 4.67% and for basalt was 5.03% by weight of aggregate.

Stability is a parameter which is affected by angle of internal friction and viscosity at 60’C, it means angularity of aggregates and viscous property of bitumen are the two most important parameters which affect stability of asphalt mixtures. Also flow can be defined as deformation of asphalt mixture, too much flow indicates that mixture will experience permanent deformation under traffic. Optimum bitumen content is determined at specific void content (4%), in order to allow further compaction of pavement under heavy traffic load without deformation.

In order to determine marshal properties and retained stability index property of samples produced with zycotherm and zycotherm dilution, four (4) samples from each zycotherm percentage with control samples for both type of aggregates prepared and the average results of the samples can be seen from conditioned and unconditioned parts of table (13) to table (26). since zycotherm chemically change the surface properties of aggregate, for which it will change chemically the water loving aggregate surface (hydrophilic property) to a water repellent (hydrophobic property), so just by looking at the ratio of conditioned stability to unconditioned stability (retained stability index) of the samples it can be seen that all the samples that contain zycotherm with both addition system for both limestone and basalt in comparison to control samples increased.

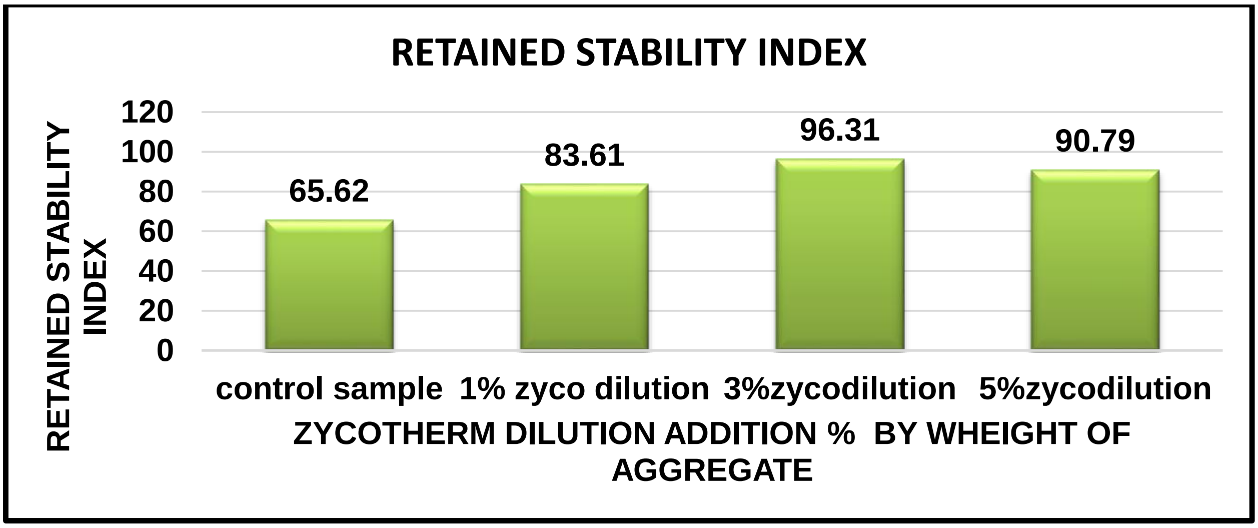
Limestone retained stability index for control samples was (65.62%), with the addition of 0.1% zycotherm it increased to 77.84%, further increase of zycotherm to 0.3% it increased to 81.48%, further increase to 0.5% retained stability increased to 95.38%. which means with the addition of 0.5% zycotherm retained stability which is a measure of moisture resistance of the mixture, increased approximately 35%. it can be seen clearly from the graph below (figure (19)).

Figure (19) shows RSI of mixtures made with limestone aggregate after zycotherm added to bitumen.



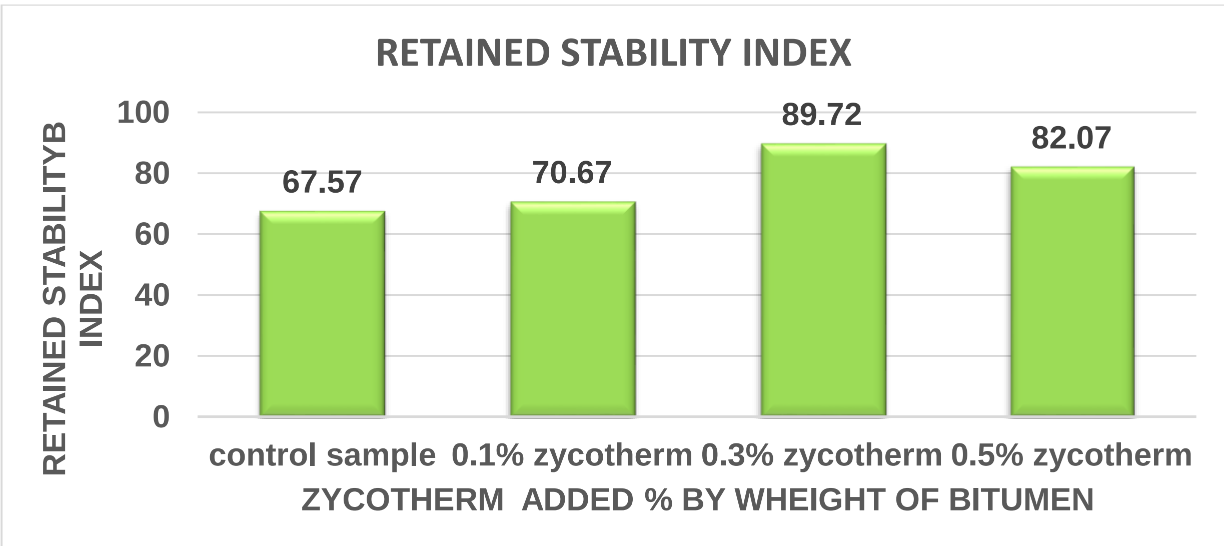
However when the modification system changed and limestone aggregate marinated with zycotherm dilution, retained stability increase changes as for 1% zycotherm dilution becomes 83.61% and for 3% further increase becomes 96.31%, however further increasing here up to 5% retained stability decrease to 90.79%. As shown in figure (20). When we look at the results it is noticed that for limestone aggregate an agreement over the increase in retained stability index exist with both modification system. it means moisture resistance of asphalt mixtures produced with limestone aggregate modified with zycotherm with both systems increased.

Figure (20) shows RSI of mixtures made with limestone aggregate after zycotherm dilution added to aggregate.



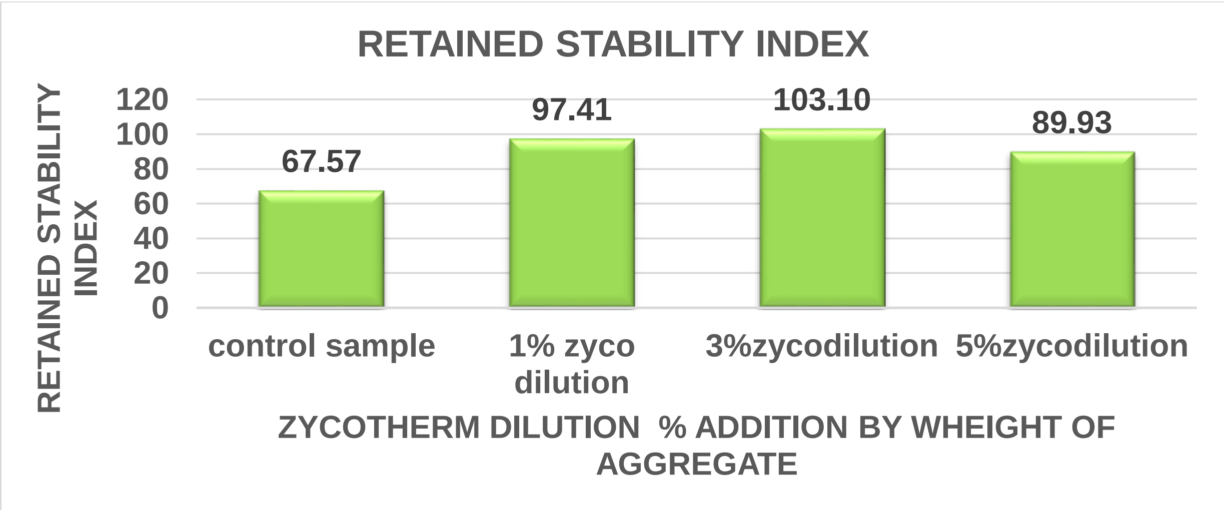
For samples made with basalt aggregate, retained stability index for control samples was (67.57%), with the addition of 0.1% zycotherm it increased to 70.67%, further increase of zycotherm to 0.3% it increased to 89.72%, further increase to 0.5% retained stability decreased to 82.07%. it can be seen clearly from the graph below (figure(21)).

Figure (21) shows RSI of mixtures made with basalt aggregate after zycotherm added to bitumen.



However when the modification system changed and basalt aggregate marinated with zycotherm dilution, retained stability increase changes as for 1% zycotherm dilution becomes 97.41% and for 3% further increase becomes 103.10%, however further increasing here up to 5% retained stability decrease to 89.93%. As shown in figure (22). When we look at the results it is noticed that for basalt aggregate an agreement over the increase in retained stability index exist with both modification system. it means moisture resistance of asphalt mixtures produced with basalt aggregate modified with zycotherm with both systems increased.

Figure (22) shows RSI of mixtures made with limestone aggregate after zycotherm dilution added to aggregate.



4.2 INDIRECT TENSILE STRENGTH AND TENSILE STRENGTH RATIO (TSR)

Since tensile strength depends on cohesion of the asphalt film and adhesion between asphalt binder and aggregate, and also an increase in tensile strength means better resistance to fatigue and thermal cracking, so it can be said long term performance of asphalt mixtures can be evaluated from indirect tensile strength.

In order to increase cohesion property of asphalt film and also adhesion between asphalt binder and aggregate zycotherm nano material used. To evaluate the effectiveness of using this material different percentage of this material in two different system (either by adding directly to bitumen or diluting and applying directly to aggregate) with two type of aggregates that vary in chemical composition (basalt and limestone) used and indirect tensile strength and tensile strength ratio results were investigated.

As the first trial control samples were prepared for both aggregate types and tested, after that samples contain 0.1%, 0.3% and 0.5% by weight of bitumen and 1%, 3% and 5% by weight of aggregate prepared for both aggregate type and tested. Detail of these samples and their results can be seen from table (27) to table (40).

If we have a look at the figures (23), (24), (25) and (26) we can see that there is an agreement over the increase in tensile strength ratio (TSR) after modification of mixtures produced with both aggregate types and with both modification systems (either directly applying to aggregate or adding to bitumen), but also it can be seen that behaviour of different aggregate is different with both systems.

Tensile strength ratio (TSR) for mixtures made with limestone aggregate with the addition of (0.1%, 0.3%, 0.5%, 1%, 3%and 5%) zycotherm and zycotherm dilution is (84.32%, 68.81%, 79.41%, 65.0%, 84.41% and 83.27%) respectively.

And also (TSR) for mixtures made with basalt aggregate with the addition of (0.1%, 0.3%, 0.5%, 1%, 3%and 5%) zycotherm and zycotherm dilution is (104.89%, 100.85%, 91.26%, 96.21%86% and 88.9%) respectively.

Figure (23) shows TSR of mixtures made with limestone aggregate after zycotherm added to bitumen.

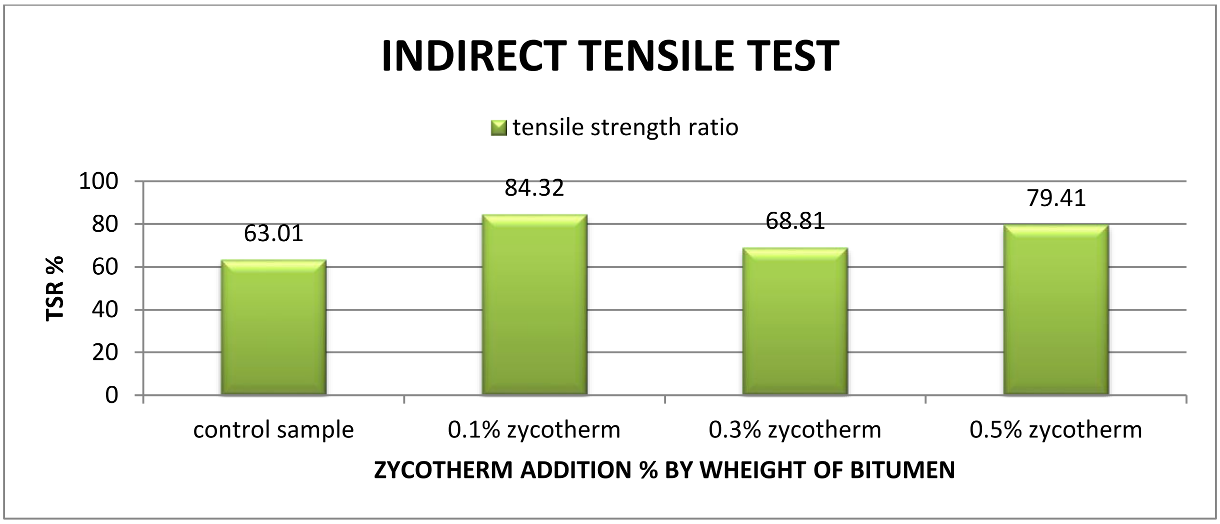


Figure (24) shows TSR of mixtures made with limestone aggregate after zycotherm dilution added to aggregate.

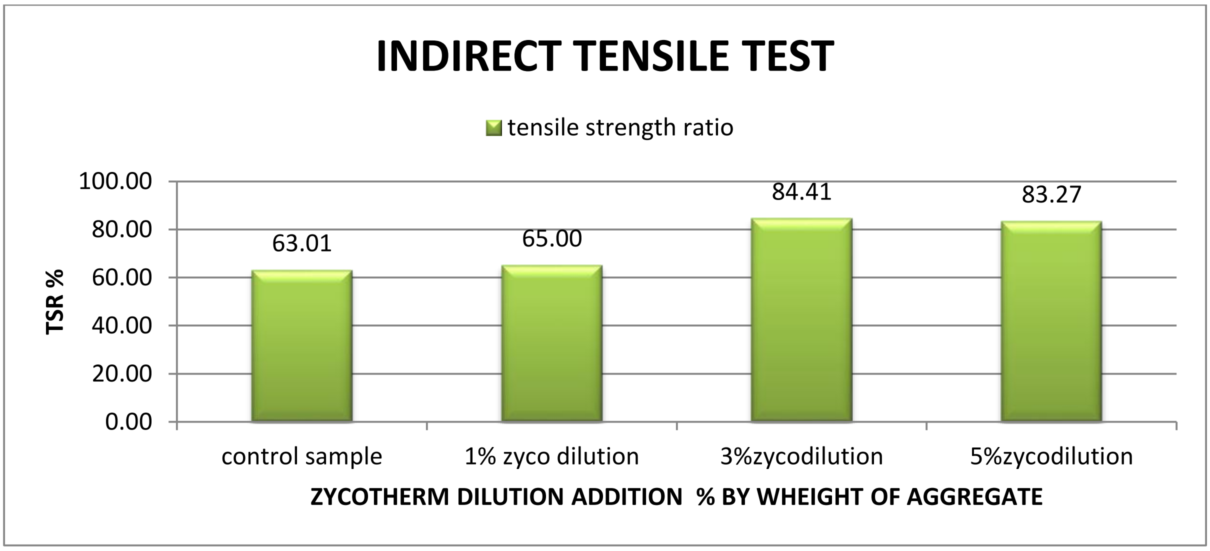


Figure (25) shows TSR of mixtures made with basalt aggregate after zycotherm added to bitumen.

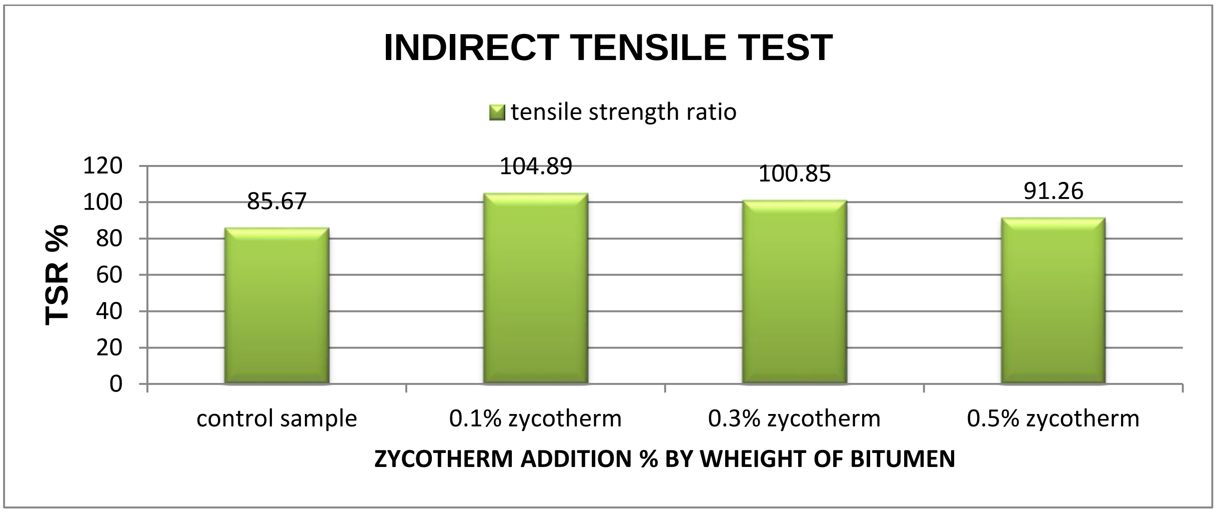
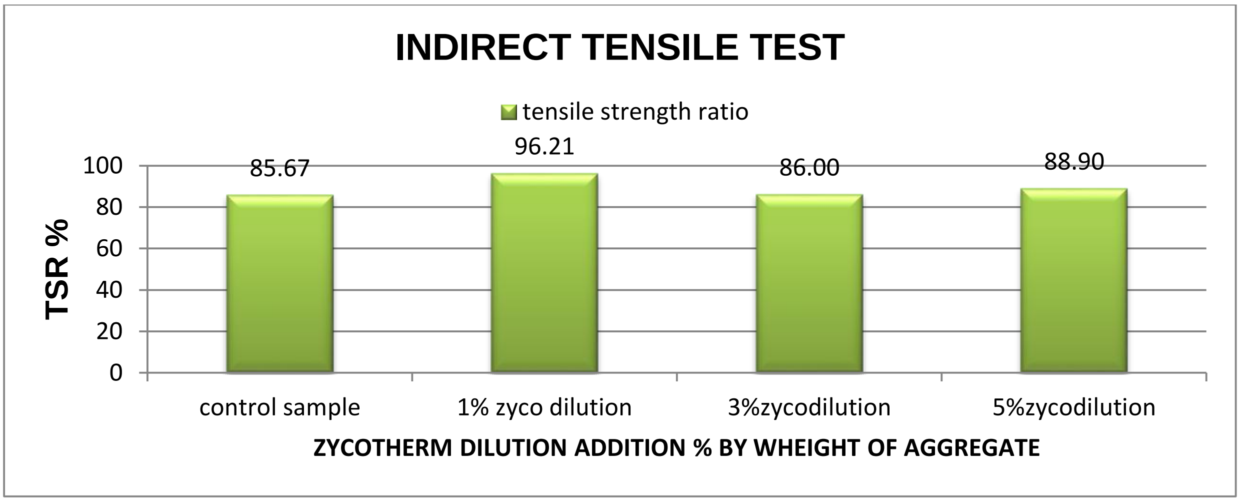


Figure (26) shows TSR of mixtures made with basalt aggregate after zycotherm dilution added to aggregate.



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