PERFORMANCE EVALUATION OF BITUMEN PARTIALLY REPLACED WITH WASTE COOKING OIL AND TIRE RUBBER POWDER IN THE PRESENCE OF BAGASSE ASH AS MODIFIER

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A thesis submitted in partial fulfilment of the requirement for

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Master of Science

In

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Islamabad, Pakistan

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THESIS ACCEPTANCE CERTIFICATE

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Dedication

I dedicate this research work to my beloved parents and teacher whose constant support and guidance make it possible to accomplish this difficult task.

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Abstract

Bitumen is a complex hydrocarbon, sourced from petroleum refineries as by product in frictional distillation. It is widely used as a binder in flexible pavement in road construction and rehabilitation. Petroleum is a natural resource, and its reserve are depleting rapidly with the passage of time. Production of bitumen directly depends on petroleum. Bitumen consumption has been increased by road construction industry for the past few years due to rapid expansion in road networks in developing countries, causing excessive environmental pollution due to hazardous fume produce during burning of bitumen in pavement paving process. The depleting reserve of petroleum and environmental concern led researcher to look for an alternative binder that could replace partially certain percentage of bitumen and environment friendly. Bio oil which include vegetable oil have proven as good alternative binder so far. Although they decrease viscosity of bitumen but increases resistance against fatigue and low temperature cracking of bitumen. Tire rubber powder and bagasse ash are waste materials obtained from tire and sugar industry respectively. Tire rubber powder and bagasse ash both contain silica in substantial percentage which impart them with good physical interaction properties, resulting in improving physical properties of bitumen, including penetration softening point and viscosity. This research work explicitly aims at replacing certain optimum percentage of bitumen with Waste cooking oil (WCO) Tire rubber powder (TR) in the presence of Bagasse ash (BA) used as additive. About 5 % of WCO and 15% TR were used as a replacement percentage in bitumen in the presence of 8 % bagasse ash used as additive. Alternative binder obtained were tested to carry out performance evaluations, the results obtained were promising. Results shows that modified bitumen shows more resistance to rutting, fatigue and low temperature cracking than virgin bitumen. Cost analysis were evaluated in cost of bitumen used per kilometer. Cost analysis shows that cost of modified bitumen incorporated with waste material is lower as compared to cost of Virgin bitumen. The research work directly contributes to the global initiative of making sustainable use of Bitumen (natural resourced material) and environmental protection by developing alternative binder for flexible pavement by incorporating waste material in to bitumen. The use of waste material in bitumen will solve the waste disposal problem and will minimize unhealthy environmental hazardous fume produced during bitumen burning, thus minimizing environmental degradation.

Keywords: Bitumen, Waste materials, Alternative Binder

LIST OF ABBREVIATIONS

ASTM	American Society of Testing Materials
WCO	Waste Cooking Oil
TR	Tire rubber powder
BA	Bagasse Ash
G*	Shear Modulus
δ	Phase Angle
G*/Sin δ	Complex Modulus
W5	5% replacement of bitumen with waste cooking
TR15	15% replacement of bitumen with rubber powder
B8	8 % Bagasse ash
W5TR15B8	5% Waste cooking oil, 15% Tire rubber powder and 8% Bagasse ash
60-70	Virgin Bitumen (PN-Grade 60-70) without modification
PARCO	Pak Arab Refinery Ltd
PAV	Pressure Aging Vessel
DSR	Dynamic Shear Rheometer
BBR	Bending Beam Rheometer
RTFO	Rolling Thin Film Oven
M	Creep Rate (Slope)
S	Creep stiffness

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CHAPTER 1

INTRODUCTION

1.1 Background

Bitumen is a complex hydrocarbon obtained as by product in frictional distillation. The origin of Asphalt binder is dated back to ancient Sumerian in 6000 B.C. world is familiar with different uses of bitumen many times ago. French and Americans used bitumen in solid rock form for footpath and water proofing. In early 1800 first bitumen road was paved in Washington D.C. This was the beginning of bitumen use in road construction. Since then, bitumen is using extensively in road construction and rehabilitation.

Bitumen is sourced from petroleum refineries as by-product which is widely used as binder for flexible pavement. Petroleum is a natural resource and its reservoir are depleting with the passage of time. Such depleting reservoir of petroleum will directly affect the production of bitumen. In addition, bitumen during road construction burns at high temperature, producing hazardous fume which are injurious to health and causes environmental degradation. These issues led researcher to look for alternative binder that could minimize the use of bitumen. Different researcher had conducted research in that effort and conclude that material mainly obtained from bio oil such as waste cooking oil, coconut oil, polymer can be use affectively as alternative binder. This research work explicitly aims at replacing certain percentage of bitumen with waste cooking oil, tire rubber powder and bagasse ash. Pakistan import about 95% of its edible oil to meet its consumer demand. Waste cooking are produced in local restaurants in high quantity in Pakistan. Tire rubber powder and bagasse ash are waste material produce in tire and sugarcane industry respectively. Disposal of these waste poses serious threat to clean environment as well as leading to reduction in land fill area. Waste cooking oil when use alone without modifier, as a replacement in bitumen, may lead to excessive softening of bitumen which is not desirable. Tire rubber powder and bagasse ash, both improve bitumen hardness and other properties of bitumen. Waste cooking oil and tire rubber powder were used as a replacement of certain bitumen percentage while bagasse ash is used as additive in it. The purpose of adding tire rubber powder and bagasse ash is to increase the percentage replacement of bitumen without affecting properties of bitumen as well as incorporating waste materials into useful product.

1.2 Research Objectives.

The main objectives of this research work were set to obtain the following:

- Modification of bitumen with waste cooking oil and other supplementary materials in Bitumen.
- Determination of optimum percentage of bitumen that can be replaced with Waste Cooking oil, Tire rubber powder and bagasse ash.
- Investigation of physical, and rheological property of the modified bitumen and its Comparison with the neat bitumen binder.

1.3 Research Methodology

In order to achieve the above-mentioned objectives, a research plan was prepared as shown in Figure The main research assignments are break down, and listed below

- Literature review and collection of information about 60-70 bitumen, WCO and Tire rubber powder the effect of WEO and Tire rubber powder on rheology at high, intermediate and low temperature.
- Collection of Grade 60-70 virgin asphalt binder from Parco oil refinery regional office Rawalpindi, tire rubber powder from local market Rawalpindi, and bagasse ash from sugar-cane waste obtained from Mardan sugar mill.
- Characterization of available virgin asphalt binders (Grade 60-70) and their blends involving WCO Tire rubber powder and Bagasse ash (In different proportion), using the state-of-the-art equipment i.e. Rolling Thin Film Oven (RTFO), DSR and BBR.

1.4 Scope of the Research

The research work is directly related with the sustainable use of natural resources. The research work incorporates waste materials to minimize the use of bitumen (Natural Resourced) and environmental degradation. This research work focuses mainly on replacing optimum percentage of bitumen with waste material and comparison of modified bitumen with virgin bitumen. Detail test Metrix comprising of different percentage of waste materials in bitumen were prepared as shown in the following table

Table 1-1 Test Metrix

Type of sample	Virgin Bitumen replaced (%)	WCO (%)	TRP (%)	BA (%)	Penetration Test (mm)	Softening TEST	PAV Test	RTFO TEST	DSR TEST	Viscosity TEST	Total Sample
Virgin Bitumen	_	_	_	_	3	3	3	3	3	3	18
Virgin bitumen + WCO	5	5	_	_	3	3	_	_	_	_	6
	10	10	_	_	3	3	_	_	_	_	6
	15	15	_	_	3	3	_	_	_	_	6
Virgin bitumen + WCO + Rubber powder	10	5	5	_	3	3	_	_	_	_	6
	15	10	5	_	3	3	_	_	_	_	6
	20	12.5	7.5	_	3	3	_	_	_	_	6
	25	15	10	_	3	3	_	_	_	_	6
Virgin + WCO + Rubber Powder + bagasse ash	10	5	2.5	2.5	3	3	3	3	3	3	18
	15	5	6.5	3.5	3	3	3	3	3	3	18
	20	5	10	5	3	3	3	3	3	3	18
	25	5	12.5	7.5	3	3	3	3	3	3	18
Total Samples					36	36	15	15	15	15	132

CHAPTER 2

LITERATURE REVIEW

2.1 General

Transportation by roads play a vital role in the country economy. Road network provide access across different part of the country which ultimately increase the potential of providing access to different market across the country. Bitumen is a hydrocarbon which is widely use in road construction as a binder across the world. Bitumen is petroleum by-product. About 83 thousand. Metric ton of bitumen is consumed every year. Out of this amount about 70 % of bitumen is use for road construction. Analysis shows that through 2019, global demand for asphalt is projected to expand 2.8 percent per year to 122.5 million metric tons (742.5 million barrels). Such an increasing demand of bitumen let researcher to explore alternative ways to reduce the usage of bitumen in road industry.

Bitumen is a very complex, viscoelastic, rheological and non-crystalline material (black or dark brown in color), which is substantially soluble in carbon disulfide (CS_2) and exhibits adhesive and water-proofing characteristics. Beside its physical and chemical properties, it possesses a unique and fundamental microwave property (permittivity). It is a low loss material as loss tangent, tan δ ($\varepsilon''/\varepsilon'$) <0.5 and its microwave permittivity (dielectric constant, ε') value ranges from 2 to 7 depending on the grade of bitumen, asphaltenes content, temperature and microwave frequency. It is composed of aromatic hydrocarbons and particularly includes 80% of carbons (C) approximately, 15% of hydrogen (H) and remnants such as oxygen, sulfur, nitrogen, metals etc.

Bitumen causes severe impact to health as it is burn at high temperature which produces toxic fumes, extremely dangerous to health and have detrimental effect on environment. Due to the above reasons there is a need to look for bitumen alternatives or replace certain percentage of bitumen with another material.

2.2 Level of Research Already Carried Out

Finding alternative binder which can replace bitumen up to certain extent is the leading research trend in today’s transportation industry. Due to rapid depletion in petroleum reserve a world will comes once to a point where petroleum reserve will diminish or consumed. The demand of bitumen had already exceeded than its production. Many researchers have contributed somehow in finding bitumen alternative. So far now researchers can find bio oil and waste lubricating oil as best bitumen alternative because of the chemical and physical properties these wastes material possess. Finding alternative binder has become leading recent trend these days. (H Wang et al, 2012) conducted research on the recent advances production and utilization trend of bio oil and conclude that bio oil is environment friendly, and economical and can also be used as petroleum substitute. [1]. Sources of bio oil can be divided into three broad categories. (1) agricultural or forest production wastes (like maize straw), wood wastes (like sawdust and bark), and urban organic wastes (like microalgae and coffee grounds), (2) animal wastes that include but are not limited to swine manure, bovine feces, (3) waste oil wastes that include but are not limited to cooking oil/residues, waste auto engine oil/residues, cottonseed and soybean oil residues. [2] [3] In these sources of bio oil waste cooking oil obtained from frying activity and waste engine oil are the two bio oil on which extensive research had been consider as the potential substitute of bitumen. Previous study evaluated that 5-10% of bio-oil obtained from agricultural or forest production waste , and less than 10% of bio-oil obtained from an animal waste can be used with control asphalt to maintain its general characteristics. [4] [5]. Waste cooking oil can be used as rejuvenating agent as well as partial replacement of bitumen. Ravi Datt Sharma [6] Conducted research on use of waste cooking oil as rejuvenating agent. Aged bitumen or RAP is waste product which is recycled and reuse for flexible pavement construction. Waste cooking oil can act as a rejuvenating agent in the recycling process of aged asphalt. The research study concludes that 3 - 4% of WCO can rejuvenate 30/40 penetration grade of bitumen to meet the property of bitumen having penetration grade 60/70. Another study shows that waste cooking oil can be used as an additive in asphalt cement use for the construction of flexible pavement. Aslam A. Al-Omari. [7] Conducted research on the characterization of asphalt binder modified with waste vegetable oil/waste cooking oil. In this research waste vegetable oil was mixes in different percentages such as 1%,2%, 4%,6%, 8% by volume of asphalt was mixed with asphalt and extensive laboratory test were performed. The result shows an improvement in the ductility, fire point, flash point and fluidity of asphalt cement modified with waste vegetable oil, also it shows that Asphalt cement modified with waste vegetable oil shows less viscosity, which is important to have good workability and decreased creep stiffness. In another study bitumen was modified with bio oil such as waste vegetable oil and properties of modified bitumen were assess. The results were further extended to conclude that bitumen modified with waste cooking oil when use in flexible road construction it minimizes several types of distresses such as bleeding binder drain down, which ultimately causes sticking of surface dressing with tire. In this study surface micro mechanical properties using AFM were conducted to investigate the adhesion of bitumen with aggregate which shows that bitumen modified with waste cooking oil show improved adhesion and ultimately minimizing binder drain down A. Guarin et al. [8]

2.3 Asphalt Binder

Asphalt binder also known as bitumen is a highly viscous hydrocarbon obtained from frictional distillation of petroleum as residue. It has a boiling point of 160C. it releases toxic fume into the air when it burns at high temperature. Asphalt is a viscoelastic material. It imparts flexibility to pavement causes pavement to deform when subjected to load. the basic principal of load transfer mechanism of flexible pavement is that load is transfer from the asphaltic course also known as HMA course to aggregate base which is further transfer to earth sub-grade.

Flexible pavement is subjected to two main type of deformation i.e. rutting and fatigue. When the stiffness of bitumen is decreases at high temperature a channel in path of wheel can be observed this type of deformation in pavement is called rutting. Cracking can be attributed to fatigue and thermal cracking. fatigue cracking occurs due to repeated loaded and unloaded of pavement occur and thermal cracking is related to surrounding temperature. Extremely variation in surrounding temperature will causes pavement contraction and expansion at different intervals which will ultimately develop stresses in pavement. Bitumen replacement with Waste cooking oil tire rubber powder and in the presence of Bagasse ash as modifier should yield bitumen which will stiff enough to resist damage from rutting, but soft enough to dissipate stresses build up to absorb stresses without fracture. This way we will be able to obtain our objective which include replacing bitumen with alternative material so that modified bitumen gives properties nearly equal or good than the virgin bitumen.

2.4 Superpave Grading System

The Super pave is a complete package which includes tests methodology, specifications and best engineering practice that enable engineers to select suitable material and guideline for mixing the material properly to meet the heavy traffic and extreme climatic condition for highway project.

In 1984 transportation research board (TRB) published a report title as "Transportation special report 202" with emphasize on six research areas with long term pavement performance program as one of key research area. Accelerating the search for innovation, the need for publishing such report was to find a solution premature failure of flexible pavement which was matter of concern for the US government as large amount of tax payer money was spent on it. This report paved the way for US congress to sanction 50 million dollar for the Federal Highway Administration (FHWA) (transportation, 2017-2018).It started an agenda which is known as (SHRP).This report provided certain recommendation to (AASHTO) which made a start for inclusion of new testing methodology, new and improved tools and improved method of mix design of asphalt in a new edition of AASHTO. The strategic highway program which was started in 1987 ended in 1993 which primary focus on pavement deteriorations like rutting phenomenon, cracks due to fatigue and low temperature cracking which were the major causes for the premature failure of asphalt pavement. In order to curb pavement distresses new testing methods and tools were developed which simulate field condition in lab better three main area of research of strategic highway research program is mentioned as below:

- Specifications of Performance Graded (PG) Asphalt Binder
- Mix Design (Volumetric)
- Prediction of Mix Analysis Performance

The important breakthrough of SHRP was to make a technique to classify the binder which was termed as performance graded asphalt binder specification. This approach helps engineers to classify the bitumen based on maximum average seven days pavement temperature. For example, 76-16 means that this bitumen can be used in a locality where the maximum seven days temperature is 76°c while the lowest seven days pavement temperature is -16°c. It is important to mention that maximum seven days pavement temperature can be calculated my measuring Seven-day hottest day of the year and taking average of it similarly average lowest temperature can be measured and taking average of it this temperature data can obtained from metrological department of Pakistan. There are different approaches to convert maximum air temperature to maximum seven-day pavement temperature, the most prominent is model developed by strategic highway research program

T (pav) = (T (air), - 0.00618 Lat2+0.2289 Lat +42.4) 0.9545 - 17.78 (Mampearachchi, Mihirani, Binduhewa, & Lalithya, 2012)

Where: Tpav= High pavement temperature at 20 mm which is lower than atmospheric temperature (Celsius)

Tair= air temperature (high)

Lat=Latitude of the section, degrees

AASHTO Specification for minimum values of complex modulus (Fini, 2016; Institute, 2003)

Material	**Value**	**Specification**	**HMA Distress Concern**
Un aged binder	$G*/sin\ \delta$	≥ 1.0 KPa	Prone to Rutting
RTFO aged	$G*/sin\ \delta$	≥ 2.2 KPa	Prone to Rutting
PAV aged	$G*.sin\ \delta$	≤ 5000 KPa	Prone to Fatigue

2.5 Waste Cooking Oil.

Waste cooking oil is produced in huge quantity every day in a country. It possesses properties slightly similar to bitumen. Due to depletion of petroleum reserve bitumen production goes consistently on decreasing. In order to address this issue, various research studies have been carried out in an effort of finding alternative binder. Researcher have proved that, bio oil obtained from WCO can be used as a potential alternative binder. Waste cooking oil use in bitumen can improve bitumen properties like rutting resistance. Wen et al. evaluated the performance of waste cooking use in bitumen and conclude that, WCO modified bitumen shows good resistance against thermal cracking as the percentage of WCO increases. Now a days Reclaimed asphalt pavement (RAP) is extensively uses in road construction in which RAP is rejuvenated by adding rejuvenating agent. Waste cooking oil can be used as a potential rejuvenating agent in RAP. Hallizza Asli [9] conducted research on the physical properties of binder, rejuvenated with bitumen. Physical properties of original bitumen, aged bitumen and bitumen rejuvenated with waste cooking were compared. Results shows that bitumen rejuvenated with waste cooking oil shows the same physical properties as the original bitumen. Previous studies show that waste cooking oil can improve rheological properties also. A research studies were conducted in which the rheological properties of bitumen modified with Waste cooking oil, penetration grade 70 bitumen and SBS modified bitumen were evaluated. The result shows that bitumen modified with waste cooking oil shows better resistance against rutting. This can be attribute to the fact that bitumen interacts chemically with the waste cooking oil through van der wall forces. Another research shows that, performance of WCO is dependent on quality of Waste cooking oil. WCO having low acidity or low PH tends to show better performance than WCO which is more acidic.

2.6 Bagasse Ash

Bagasse ash is by product of sugar industry produced when sugar is extracted from cane and the left off pulpy is allowed to dry in sun and burn after that to form ash. Pakistan produced about 70 million tons of every year. Bagasse obtained from sugarcane is used partially in sugar industry as fuel, but a huge amount of waste sugar cane is disposed of every year causing environmental problems. Recently different research work had been conducted on agricultural wastes such as rice husk ash, fly ash and bagasse ash. Research work has shown that these agricultural wastes possess good pozzolanic properties and can be used as supplementary material in soil, cement and bitumen. So far research had been conducted on Bagasse ash usage in cement only and it had never been used in bitumen before. Rice husk ash has been previously used in bitumen for improving its physical and Rheological properties. (Raissa Romastarika, 2016) conducted research in which he mixes different percentages of rice husk ash with bitumen. Result shows that, the addition of rice husk in greatly improve both physical and Rheological properties of bitumen [10] It improve resistance against rutting by decreasing viscosity. Bagasse ash possess similar chemical characteristics as that of rice hush ash. It had been used previously for soil stabilization purpose. Bagasse ash will help to improve bitumen rheological and physical properties. In this research work Bagasse ash will be used as modifier in Bitumen, which is partially replaced with WCO and Tire rubber powder.

2.7 Tire Rubber Powder

Bitumen is a complex hydrocarbon obtained from petroleum industry as a residue. Bitumen consist of maltenes and asphaltenes. Bitumen is widely use in road construction. The main problem concerning bitumen is its visco-elastic behavior. Bitumen become brittle at low temperature and become very flowable at high temperature. To improve visco-elastic behavior of bitumen different modifier were used. Tire rubber powder has been determined as a potential and cheap available modifier to improve visoc-elastic behavior of bitumen Tire rubber powder can improve both physical and Rheological properties of bitumen. Marek Pszczoła [11] conducted research in which physical and Rheological properties of bitumen modified with crumb rubber were evaluated. The result shows that with the addition of rubber bitumen obtained will have properties similar to polymer modified bitumen. His research work concluded that use of crumb rubber in bitumen enhance the bitumen resistance against rutting and low temperature cracking. Another research work shows that bitumen modified with rubber shows better performance at both Low and high temperature. Nuha Salim Mashaa [12] conducted research and conclude that properties of bitumen modified with crumb rubber is greatly influenced with the blending temperature and does not affected significantly with the blending time. Tire rubber powder interact physically with bitumen. Different studies were carried out on the interaction of tire rubber with bitumen and it was concluded that the only reason of interaction of tire rubber powder with bitumen is that tire rubber when comes in contact with bitumen absorb some of the oil from bitumen and swell up and impart stiffness to bitumen.

2.8 Summary of the Chapter

Road Transportation is an important tool of connecting different cities with in any country. It comprises of facilities such as bridges, highways, tunnels to ensure safe and efficient movement of people and goods within inside or between countries. A good and efficient Highways network directly contribute in country's economy by increasing GDP Growth. Paved Roads are of either flexible or rigid type. During construction of flexible pavement bitumen is use as the binder. Bitumen is a complex hydrocarbon obtained as residue during the process of fractional distillation of petroleum. Bitumen is non-renewable resource and its production depend on petroleum reserve which are depleting with the passage of time. In addition to it bitumen when burn at high temperature emit hazardous fume that are unhealthy to the environment. These issues had forced numeral government highway agencies to look for alternative binder that can substitute certain percentage of bitumen and is environment friendly. Researcher have concluded bio oil such as waste cooking oil as the best alternative binder and can be used in bitumen as bitumen replacement. Thousands of tons waste cooking oil are produced in almost every country. So, it can be incorporate in bitumen industry along with other material such as tire rubber powder and bagasse ash which are also produce in world in large amount. These are waste material and poses many environmental degradations. Until now about 5 percent of waste cooking has been used in bitumen as replacement in the presence of different modifier. The reluctance for higher WCO Percentage is due to too much softening of mix which can be attributed to chemistry of bitumen. Bitumen is chemically composed of three compounds: aromatics, resins and asphaltenes, the first two compounds are collectively known as maltenes. Increased in asphaltenes can cause stiffening of bitumen. Waste cooking oil consist of aromatics, resin and saturates. Waste cooking oil has lower asphaltene and higher resin components. Waste cooking oil help in improving resistance against fatigue and low temperature cracking. Tire rubber powder improve the stiffness as well as elastic recovery potential of bitumen. (F. J. Navarro) conducted research on rubber addition in bitumen and conclude that, rubber modified bitumen possesses high resistance against rutting, fatigue and low temperature cracking. Bagasse ash is an Agro waste and obtained from sugar cane industry. It is used as modifier to improve stiffness of bitumen. Bagasse ash has never used before in bitumen. Pakistan import about 75% of total edible to meet its consumer demand. A high amount has been paid for it each year. Incorporating waste cooking oil into bitumen will have impact on reducing cost of bitumen usage in road construction and rehabilitation: thus, will equalize economy.

CHAPTER 3

METHODOLOGY

3.1 General

The main objectives of this research studies to partially replace bitumen with waste cooking oil and tire rubber powder and bagasse ash and carry out performance evaluation to compare modified bitumen with neat bitumen. While conducting this research work following methodology has been followed

- Virgin bitumen 60/70 was blinded with different percentages of waste cooking oil tire rubber powder and bagasse ash
- Penetration Softening point and viscosity test were performed to determine the optimum percentage of bitumen that can be replaced with WCO, TRP and BA.
- After determining the optimum percentage of WCO, TRP, and BA that can be replaced successfully in bitumen Rheological properties of modified bitumen were determined and compare with the neat bitumen either 60/70 or 80/100.
- Chemical Analysis of Bitumen Partially replaced and modified with WCO TRP and BA were conducted using different chemical analysis test such as FTIR, XRD and SEM.

3.2 Material Selection:

The objective of this research is to partially replace bitumen with other material so for this purpose selection of modified alternative binder material is very crucial step. Material are selected as per the availability and cost efficiency of the material. Following mixture of material were used for preparation of samples for different experimentation.

- Bitumen 60/70 PN-Grade
- Tire rubber powder
- Bagasse Ash
- Waste cooking oil.

3.2.1 Bitumen:

Bitumen use were 60/70 Penetration Grade which were obtained from Parco sale office Rawalpindi. Bitumen without any modification in this research is termed as neat or virgin bitumen.

3.2.2 Waste Cooking oil & PH-Test

Waste cooking oil were obtained from three different reputable restaurants in Islamabad. Literature review shows that the quality of waste cooking oil greatly affects the properties of bitumen modified with waste cooking oil. Literature review also shows that as the acidic content of waste cooking oil increases its quality increases and it has bad effect on bitumen physical as well as rheological properties.

PH test were performed on all three samples of waste cooking oil where were obtained from three different restaurants. WCO having lowest value were selected for further experimentation.

3.2.2.1 WCO Filtration

Waste cooking oil obtained from different restaurants contain suspended particles of different sizes. Literature review shows that waste cooking oil free from any impurities shows better performance than WCO having suspended particle and impurities. For this purpose, WCO were filtered by using centrifugation and filter paper to remove all impurities and suspended particles.

3.2.2.1.1 Centrifugation method

Universal centrifuge was used for centrifugation of waste cooking oil. The basic principal of this centrifuge is that a material from which impurities has to be removed is placed in glass container which are four in number. Capacity of each glass bottle is 500ml. after placing samples in glass tubes machine is started which is rotated at different speed(rpm), based on size of impurities to be removed. Due to high speed, impurities are separated based on difference in densities. Material having high density settles at the bottom.

- Waste cooking oil were poured into 4 glass tube and placed in centrifuge
- The speed of centrifuge was set to 4000 rpm
- Temperature inside centrifuge were set to $27^{o}C$
- The time for which centrifugation has to be carried out were set as 30 minutes

Waste cooking oil filtered in centrifuge were passed through filter paper to further remove any suspended impurities.

Figure 3-1 Universal Centrifuge

Figure 3-2 Universal Centrifuge (Bottles)

Figure 3-3 Waste Cooking Oil Filtrations Through Filter paper

3.2.3 Tire rubber powder

In this research work tire rubber powder use were obtained from local shop in Rawalpindi, Pakistan, where tire rubber powder is produced in recycling process at ambient temperature. Local shop uses different grinding tools for scraping tires of different size. During this process rubber powder is produced. Such type of tire rubber powder has a large surface area and spongy like surface. Tire rubber powder obtained were passes through different sieves, tire powder passes #200 sieve were used for further experimentation. The average size of the particle of tire rubber powder were about 0.0002mm.

3.2.4 Bagasse Ash

Bagasse ash is by product of sugar industry produced when sugar is extracted from cane and the left off pulpy is allowed to dry in sun and burn after that to form ash. The disposed BA from Mardan Sugar Mills located in the city of Mardan and province of Khyber Pukhtun kwah was procured for further processing. After removing the initial moisture by keeping the raw BA in oven at 120° C for 72 hours, processing of BA was done in two steps. First step was to sieve the raw bagasse ash through 300 µm (No.50) sieve to separate out the unburnt fibrous carbonic material and then to carry out the controlled burning of the sieved ash at 650° C for 120 minutes in an electric furnace to reduce partially burnt fine carbon particles. The second step was to enhance the pozzolanic activity by increasing the surface area of BA particles through grinding. Processing methodology used in first step was motivated from the study of (Bahurudeen et al. 2015). Target fineness for grinding operation was the time until the particles retained on 45µm were less than 5% by weight of the sample and this target fineness was adopted from the studies (Chusilp et al. 2009; Rerkpiboon et al. 2015). Same ball mill and grinding procedure was employed for grinding of BA which was adopted for grinding of RHA. Grinding time of 120 minutes was found to be sufficient for achieving the target fineness. Silica content of BA was found to be over 76%. To study the phases of silica, XRD analysis was carried out. XRD pattern of BA is given in figure 3.8. There is a clear gradually dispersed peak around Bragg's angle of 22° affirming the presence of considerable proportion of silica in amorphous form. There are also sharp peaks at some angles indicating the presence of two crystalline phases of silica i.e. Quartz and Cristobalite. It can be observed that quartz phase is relatively higher in proportion than cristobalite phase. Resembling XRD patterns were found by (Bahurudeen and Santhanam 2015; Cordeiro et al. 2008). The presence of Quartz in higher proportions in BA is due to the sand which remains adhered to sugarcane even after washing the harvested sugarcane (Cordeiro et al. 2008). LOI of the raw BA was over 19% but after processing, the LOI of final BA sample was 4.13%. So, an appreciable amount of undesirable carbon particles was removed. Color of raw BA was black, after processing steps involving initial sieving, control burning and grinding, and color of final BA was dark grey. Same can also be observed visually in figure 3.9. Raw ash in fig 3.4 (a) is black and contains coarse unburnt fibrous materials and after initial sieving through No.50 sieve, fibrous unburnt material was removed as shown in fig 3.4 (b). BA was still black after initial sieving due to the presence of fine partially-burnt carbon particles. But keeping the BA at 650° C for two hours burned considerable amount of fine partially burnt carbon particles as the color of BA turned grayish. Finally, BA was ground to target fineness and fig 3.4 (d) shows the final BA used in this study.

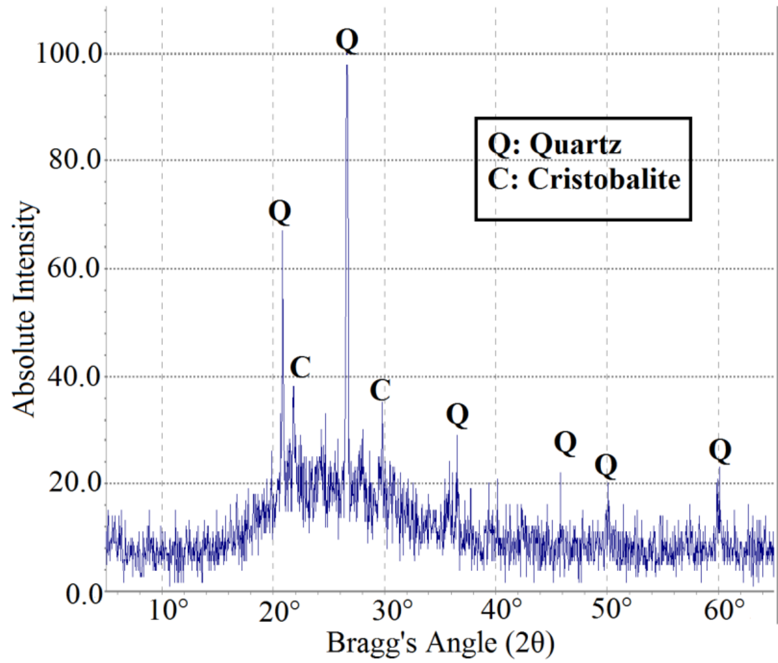


Figure 3-4 XRD pattern of BA

(a)

(b)

(c)

(d)

Fig. 3.4. (a) Sample of raw BA (unprocessed) (b) BA sample after initial sieving through No. 50 sieve (c) Controlled burning of sieved sample of BA in an electric furnace (d) Final processed sample of BA

3.3 Mixing of WCO TRP and BA with Bitumen

Manual mixing method were adopted in which bitumen were heated to a temperature of 135C and were stirred continuously for over 30 minutes. The stirring was done with glass rod gently so that TRP WCO and BA can be completely dissolved in bitumen.

3.4 Material Testing

Physical and Performance testing were performed on virgin bitumen PG 60-70 and modified bitumen. Modified bitumen was obtained through extensive physical testing by determining an optimum percentage of WCO, tire rubber powder and Bagasse ash that can replace bitumen. Results of the Physical and performance testing, which includes Low temperature cracking, rutting and Fatigue resistance of virgin and modified bitumen were compared.

3.4.1 Physical Test:

Physical test includes Penetration Test, Softening point test and viscosity Test. These tests were conducted to determine optimum percentage of bitumen that can be successfully replaced with WCO, TRP and BA.

3.4.1.1 Penetration (AASHTO T-49 ASTM D-94)

This test is important for determining Grade of bitumen, which is further used for determining suitability of bitumen use under different climatic conditions. It measures the hardness or consistency of bituminous material.

Following are the apparatus that is use in penetration test.

- Penetrometer
- Digital timer
- Water bath
- Penetration cup

3.4.1.1.1 Procedure

- Turn on the oven, set it at temperature 100ºC to 110ºC. Place the bitumen sample in it and allow it to change from solid to liquid state
- When bitumen attains liquid state pour it to a cup shaped container and allow it to cool at room temperature 25ºC for about 60 min.
- Place the cup shaped container in water bath and for about 90 minutes. Maintain the temperature of w-water bath at 25ºC
- Properly Set the penetrometer on level surface and set the water bubble. The water bubble will indicate whether the surface on which penetrometer is leveled is level or not.
- Place the sample in penetrometer and allow the needle to fall on surface of sample and touch the sample surface very slowly
- Set the timer at 5 sec.
- Press the start button and start the timer. Record the readings after 5 second.
- Take average value of the readings by repeating the test three time so that any error occurred may be omitted or minimized.
- Maximum difference between reading should not exceed 4

3.4.1.2 Softening point (T 53-06. ASTM designation: D 36-95

Softening point of bitumen is a mean temperature at which bitumen is soft enough to allow ball to cover a distance of 25 mm

Following are the apparatus used in softening point test.

- Heating and magnetic stirrer unit
- Glass beaker
- Ring and ball
- Holding assembly
- Thermometer

Figure 3-5 Softening Point Test

Figure 3-6 Softening Point Test Samples

3.4.1.3 Rotational viscosity (AASHTO T-316: ASTM D 4402)

The basic principle upon which rotational viscometer function is that it senses the torque which is required to move spindle immersed in fluid with constant speed the torque measured is proportional to dynamic viscosity. This test measures the viscosity at high temperature which provide basis for the selection of mixing and compaction temperature.

Following Apparatus are used in Finding Rotational Viscosities of samples.

- Rotational Viscometer
- Temperature Controller
- Spindles

- Griping Pliers
- Sample Chambers
- Sample Chamber Rack
- Thermosdal

3.4.1.3.1 Procedure

- Pre heat the thermosdal and sample temperature to a temperature at which test has to be done. Usually the temperature is 135ºC and 165ºC.
- Heat the sample in oven at temperature 110° C such that it is sufficiently fluid.
- Pour the sample in sample chamber
- Place the sample chamber in thermosel and lower spindle in it
- Run the test for 30 minutes and read last three reading at interval of minute and take an average value.

Figure 3-7 Rotational Viscometer

3.4.2 Performance Testing

3.4.2.1 Dynamic shear rheometer (AASHTO T 315: ASTM D 4402)

DSR is used to determine the viscoelastic behavior of asphalt. It measures the complex shear modulus and the phase angle of bitumen. DSR is also use calculate performance grade of bitumen. Apparatus for DSR test is as below

- Apparatus include main unit which consist of DSR which oscillate continuously and apply shear force which produce shearing action. The oscillation frequency range is between 0HZ to 100HZ

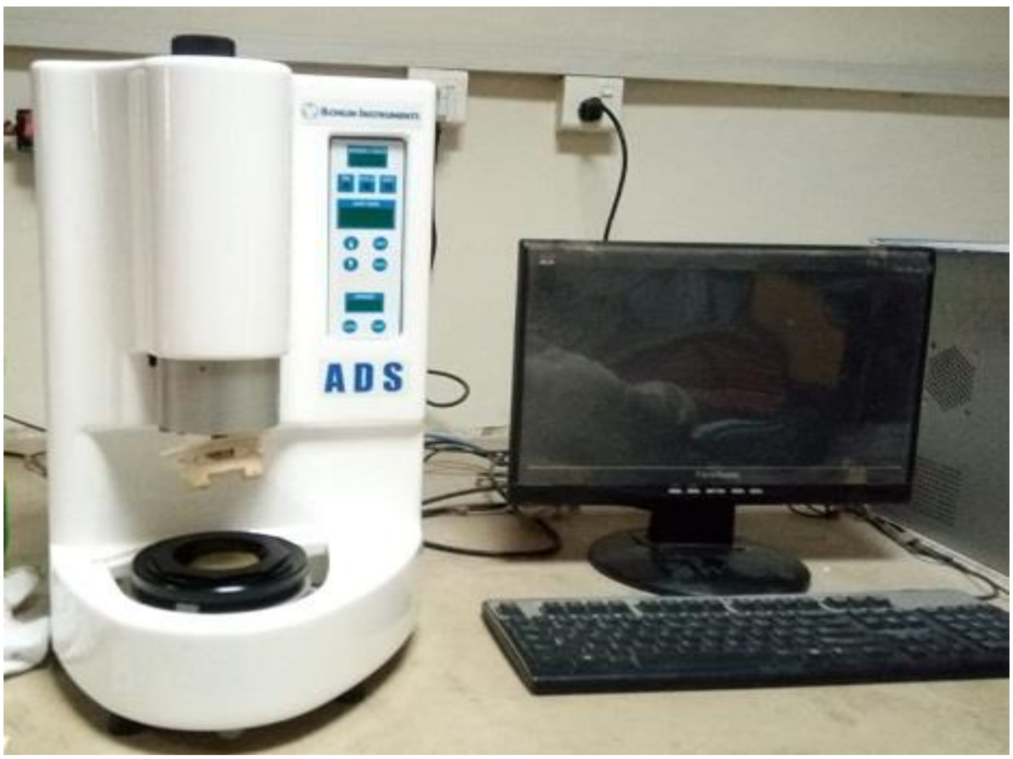


Figure 3-8 Dynamic Shear Rheometer

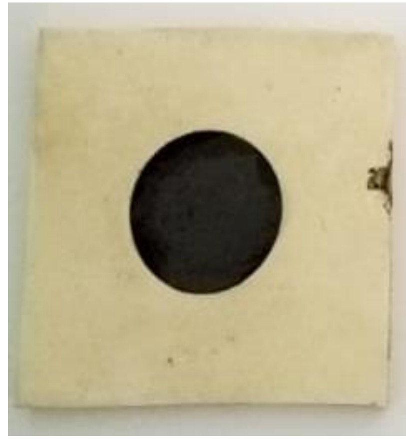


Figure 3-9 DSR Test under Progress **Figure 3-10 DSR TEST Sample**

3.4.2.2 Rolling thin film oven (AASHTO T 240: ASTM D 2872)

Evaluation of rheological properties after short term aging of bitumen is very important to assess quality of bitumen. This test is used to stimulate short term aging in lab. Asphalt in RTFO are aged by heating and blowing of hot air at 163ºC for 80 minutes. Short term aging is loss of volatile in an asphalt binder when it is heated at asphalt plant and subsequently laid at site.

Apparatus include the following

- Glass bottles
- Balance
- Back for bottles.
- Oven equipped with carousal which exhibit circular motion and a nozzle for hot air.

Figure 3-11 Rotational Thin fil Oven (RTFO)

Figure 3-12 RTFO Test Sample

3.4.2.3 Pressure ageing vessel (AASHTO R 28: ASTM D6521)

This test is used to simulate long term ageing of asphalt i.e. 7 to 10 years by applying heat and pressure for 20 hours.

Apparatus includes the following.

- Sample Pan/trays
- PAV
- Vacuum oven

Figure 3-13 Pressure Aging vessel (PAV)

Figure 3-14 PAV Test Samples

3.4.2.4 Bending beam rheometer (AASHTO T 313: ASTM D 6648)

Bending beam Rheometer is a device used to measure cracking in asphalt that occur in low temperature. This device is also used to determine Low temperature grade of bitumen.

Apparatus includes the following

- BBR molds
- Deflection calibration beams
- LVDT calibration disc
- Ethanol, methanol or glycol-methanol mixture
- M-value and s-value is than selected at 60 sec.

Figure 3-15 Bending Beam Rheometer (BBR)

3.5 Summary

In this chapter methodology adopted to obtain the desire objectives is discussed. Virgin bitumen 60/70 was selected based on the climatic condition of Pakistan. in Pakistan in most of the region bitumen penetration Grade 60/70 is used. Waste cooking oil were selected based on the acidic which were determined by Ph test. Waste cooking oil having Less Ph value were obtained from well-known restaurant in Islamabad. Baggas ash was obtained from sugar cane industry as it is the waste product produce size were kept approximately equal to the size of soil particle. Tire rubber powder were selected based on the size. Tire shop in Rawalpindi which carry out the grinding of tire provided us tire rubber powder. Bitumen is blended with different percentages of waste cooking oil tire rubber powder and bagasse ash. The percentage of waste cooking oil and tire rubber powder blended with bitumen in different trial were 5, 10, 15 18(WCO) and 5 7.5 10 12 15 (TRP) respectively. The size of the tire rubber powder was of the size 2 micron. Bitumen is mixed first with tire rubber powder and mix it at temperature 160. After that waste cooking oil and bagasse ash were added to the heated mixture of bitumen and rubber powder. The overall mixture was again heated at temperature 110 for 15 minutes. Baggas ash was added as modifier. Optimum percentage of WCO TRP and BA replacing bitumen were selected on the basis of physical test which include penetration softening test and viscosity test. The selected optimum blend with WCO, TRP and BA is tested for rutting, fatigue and low temperature cracking. The aim is to determine that with replacement of bitumen with WCO TRP and BA whether it will act like a normal bitumen or not.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

Pakistan is a developing country. It had a vast network of road span all across the country. The network of road is extending every year and month. A heavy quantity of bitumen is use in construction and rehabilitation of these roads. Bitumen is petroleum by product and price of petroleum goes high with time. Pakistan meet its bitumen demand by exporting it from neighboring countries such as Iran Saudi Arab. Pakistan pay a heavy price for the bitumen exports. World bank carry out a study in 2005 and conclude that asphalt production in Pakistan is 299,000 metric ton while demand is estimated to be 395,000 and the demand is expected to increase to 438,000 metric ton by the end of 2010. To meet the future demand of bitumen based on environment, economic and natural resources depletion concern there is a hug need of research to look for alternate binder. WCO is used as alternate binder.

This study focuses on the use of WCO TRP and BA as bitumen replacement. Bitumen is blended with different percentages of WCO TRP and BA. An optimum blend was selected, and different performance test were performed on that optimum blend. The aim is to determine the quantity of Bitumen that can be replaced with WCO, TRP and BA without adversely affecting virgin bitumen properties.

4.2 Physical Test Results

Physical test was performed on virgin bitumen and bitumen blend with different percentages of WCO, TRP and BA. Physical test include penetration softening and viscosity test. Physical test was conducted to determine the optimum blended bitumen percentage.

4.2.1 Penetration Test Result

Penetration test was used to determine the optimum percentage of bitumen that can be replaced with different percentages of WCO TRP and BA. The optimum percentages of WCO, TRP and BA were determined by carry out a series of penetration test on 75 samples. Result indicate that with the addition of 5 percent of waste cooking oil increases the value of penetration of 60/70 Grade bitumen to about 160/ 180 penetration value. The addition of tire rubber powder makes the sample stiffer as the percentage of tire rubber powder increases the penetration value decreases. With further addition of Bagasse ash as modifier the value of penetration decreases and comes in the range of 80/100 Grade bitumen. The final optimum amount at which the bitumen Grade become 80-100 were selected for performance testing.

Following are the results of all trail performed to obtain penetration Grade in range of 80-100.

Table 4-1 Penetration result of virgin bitumen replaced with WCO

Type of sample (Total sample weight=200gm)	**Sample Code**	**Bitumen Replaced (%)**	**Penetration**			
			Sample 1	**Sample 2**	**Sample 3**	**Average**
60-70 -5%WCO	W5	5	210	205	190	201
60-70 -10%WCO	W10	10	288	295	300	294
60-70 -15%WCO	W15	15	325	348	332	334
60-70 -20%WCO	W20	20	356	362	366	361

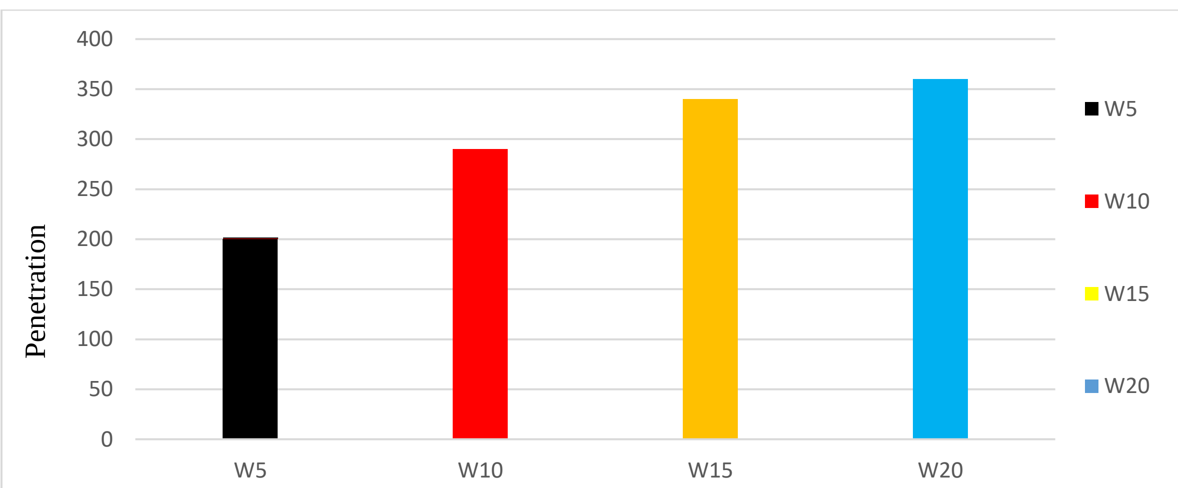


Figure 4-1 Variation of bitumen penetration with varying percentage replacement of WCO

As the percentage of waste cooking oil increases the penetration value of the bitumen increases. It is due to the fact that, with the addition of waste cooking oil asphalt consistency changes asphalt become soft because the viscosity of waste cooking oil is lower than that of bitumen.

Table 4-2 Penetration result of bitumen replaced with WCO and TR

Type of sample (Total Sample weight=200gm)	Sample Code	Bitumen Replaced (%)	Penetration			
			Sample 1	Sample 2	Sample 3	Average
60-70 -5%WCO-5%TR	W5TR5	10	131	137	141	136
60-70 -5%WCO-8%TR	W5TR8	13	117	126	121	122
60-7, -5%WCO-12%TR	W5TR12	17	109	118	114	114
60-7, -5%WCO-15%TR	W5TR15	20	106	113	108	109
60-7, -5%WCO-18%TR	W5TR18	23	103	107	104	105

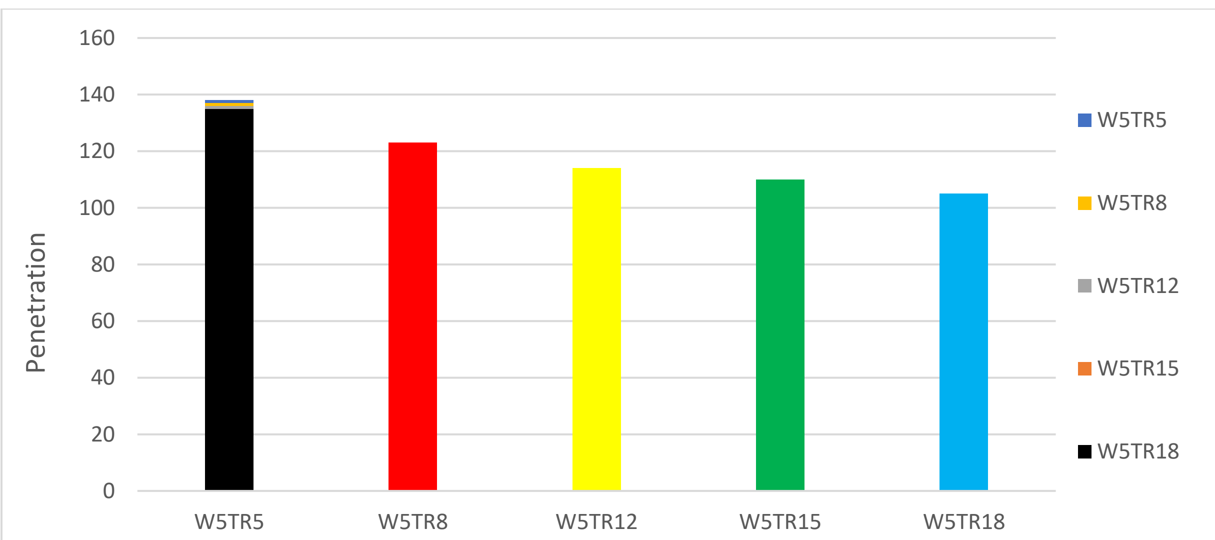


Figure 4-2 Comparison of Penetration test result of virgin bitumen partially replaced with WCO and TR

As we know now that the increase in WCO percentage can causes an increase in bitumen penetration grade value. This increase is due the fact that waste cooking oil, which is obtained from bio oil, mainly contain resin, aromatics and saturates which are responsible for increasing softness' of bitumen. With the addition of tire rubber powder the penetration value will be decreases. Because in Pakistan we mostly use bitumen penetration Grade 60/70 or 80/100. So, in order to replace 60/70 bitumen with WCO we tend to have modified bitumen of Grade 60/70 or 80/100. Tire rubber powder is a stiff material which can decrease the bitumen softness and can provide extra stiffness to bitumen High percentage of tire rubber in bitumen is not recommended as it absorb oily from bitumen which will leads to excessive decrease ductility. For this purpose, in order to decrease the penetration value to 80/100 Grade bitumen we need to use a modifier that decreases the penetration value without absorbing oily part from bitumen. So, bagasse ash is introduced into the mi. Bagasse ash were added in different percentages of sample to the mixture and penetration test were conducted the result are shown below

Table 4-3 Penetration result of virgin bitumen replaced with WCO and TR and in the presence of Bagasse ash as modifier

Type of sample (Total sample weight=200gm+%BA)	Sample Code	Bitumen Replaced (%)	Penetration			
			Sample 1	Sample 2	Sample 3	Average
60-70 -5%WCO-5%TR+5%BA	W5TR5B5	10	138	129	133	133
60-70 -5%WCO-8%TR+7%BA	W5TR8B7	13	121	126	128	125
60-7, -5%WCO-12%TR+7.5%BA	W5TR12B7.5	17	109	118	113	113
60-7, -5%WCO-15%TR+8%BA	W5TR15B8	20	104	113	107	108
60-7, -5%WCO-18%TR+10%BA	W5TR18B10	23	108	102	106	105

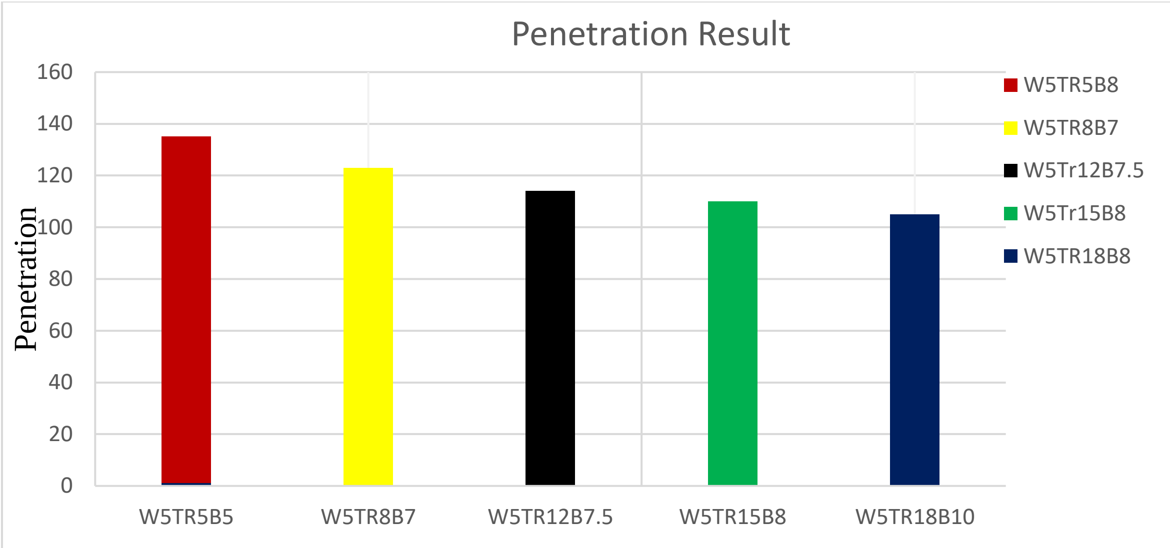


Figure 4-3 comparison of penetration test result values when bagasse ash is used as modifier

4.2.2 Softening point Test Result

It is measure of temperature susceptibility of the bitumen. Usually greater the penetration grade of bitumen less will be the softening point. Similarly, lesser the penetration grade value of bitumen more will be the softening point. Softening point is a temperature at which two ring ball of 9.5mm diameter touches the bottom of apparatus. Softening point test were conducted on 84 samples. Same as penetration test softening point test were also conducted on different trial of blending of waste cooking oil tire rubber powder and bagasse ash in an effort to find the best suitable mixing ratios of these waste materials used in bitumen. Table 4-4 indicate that with the addition of Waste cooking oil the softening point decreases. With the addition of tire rubber powder and bagasse ash in different trials, modified bitumen become stiff and softening point decreases.

Figure 4-4 softening point test

Figure 4-5 Softening point test samples

Table 4-4 showing Softening point result of virgin bitumen replaced with WCO

Type of sample (Total sample weight=200gm)	Sample Code	Bitumen Replaced (%)	Softening Point (^oC)		
			Left Sample	Right Sample	Average
Virgin Bitumen	60-70	0	47	45	46
60-70 -5%WCO	W5	5	24	22	23
60-70 -10%WCO	W10	10	22	19	20.5
60-70, -15%WCO	W15	15	17	20	19
60-70-20%WCO	W20	20	17	19	18

As it can be clearly seen that with the increase in percentage of waste cooking oil the softening point value decreases. It indicates that the addition of waste cooking increases the bitumen softness and decreases the bitumen softening point. Table 5 shows the softening point of final trail which introduce addition of tire rubber powder and bagasse ash into bitumen containing waste cooking oil.

Table 4-5 Softening point Test result of virgin bitumen replaced with WCO and TR and addition of Bagasse ash

Type of sample (Total sample weight=200gm+%BA)	Sample Code	Bitumen Replaced (%)	Softening Point (º C)		
			Left Sample	Right Sample	Average
60-70 -5%WCO-5%TR+5%BA	W5TR5B5	10	36	39	37
60-70 -5%WCO-8%TR+7%BA	W5TR8B7	13	38	40	39
60-7, -5%WCO-12%TR+7.5%BA	W5TR12B7.5	17	42	46	44
60-7, -5%WCO-15%TR+8%BA	W5TR15B8	20	47	49	48
60-7, -5%WCO-18%TR+10%BA	W5TR18B10	23	52	55	53

4.2.3 Rotational Viscosity

Viscosity of bitumen is measure of its flow at particular temperature. With the addition of Waste cooking oil, viscosity of bitumen decreases substantially. Because Waste cooking oil contain acid which are lighter than compound in bitumen. With the increase in percentage of waste cooking in bitumen, viscosity tend to be decreases. With the increase in temperature the viscosity of waste cooking oil tends to increase. With the introduction of Tire rubber and Baggers ash into the bitumen, viscosity value of the mix decreases substantially. It is due the fact that tire rubber powder and bagasse ash impart stiffness to bitumen which poses resistance towards the flow, in addition, rubber powder and bagasse ash act as pozzolanic material which increases stiffness and increases viscosity. The following table show the viscosity values of final trail, containing different blends of bitumen with WCO, TR and BA.

Table 4-6 Rotational Viscosity Test result of virgin and modified bitumen

Type of sample (Total sample weight=200gm+%BA)	Sample Code	Bitumen Replaced (%)	Viscosity (C.P)		Viscosity Ratio (135°C /160°C)
			135°C	160°C	
Virgin Bitumen	60-70	0	450	140	3.214
60-70 -5%WCO-5%TR+5%BA	W5TR5B5	10	493	290	1.7
60-70 -5%WCO-8%TR+7%BA	W5TR8B7	13	740	390	1.89
60-7, -5%WCO-12%TR+7.5%BA	W5TR12B7.5	17	900	470	1.91
60-7, -5%WCO-15%TR+8%BA	W5TR15B8	20	1300	490	2.653
60-7, -5%WCO-18%TR+10%BA	W5TR18B10	23	1730	501	3.45

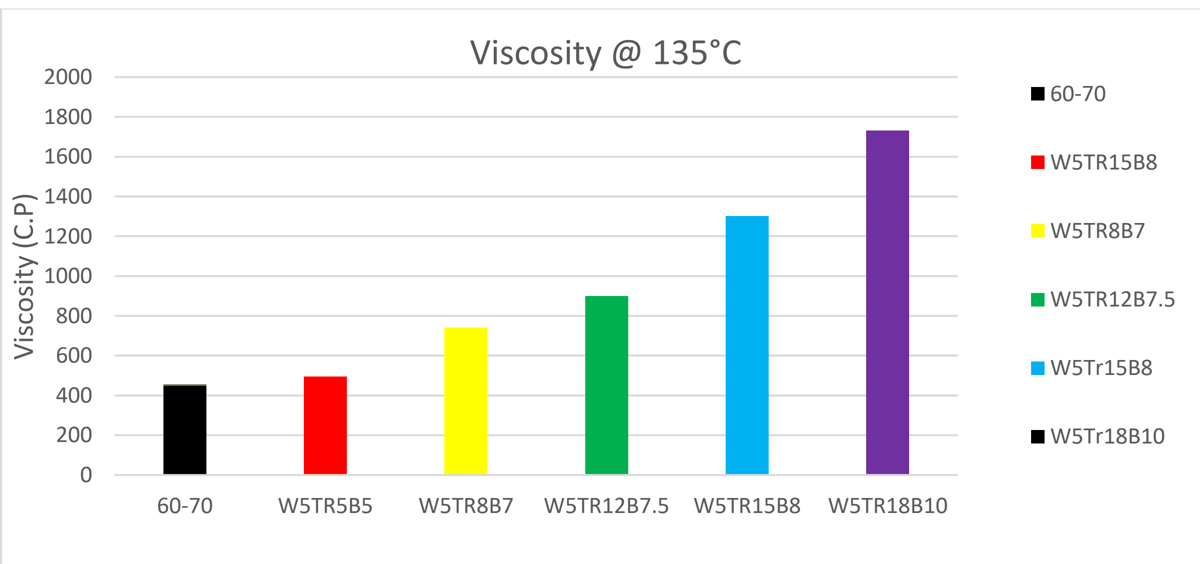


Figure 4-6 Comparison of viscosity at (135ºC) of virgin modified bitumen

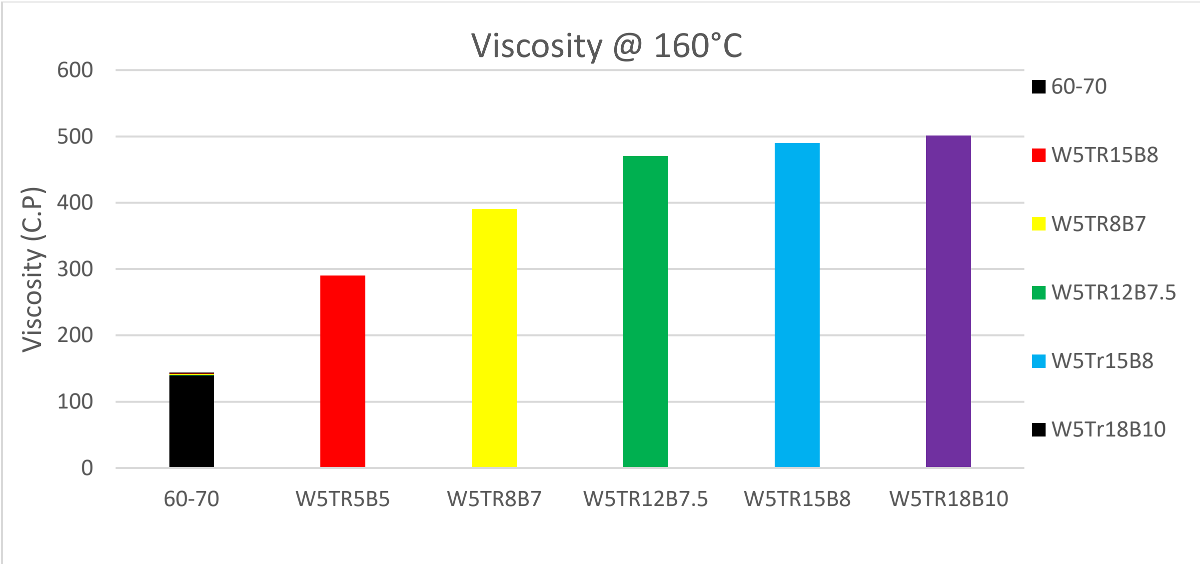


Figure 4-7 Comparison of Viscosity of virgin bitumen and modified bitumen at 160°C

4.3 Performance Testing

Performance test were performed to assess the performance of bitumen, modified and replaced with WCO BA and Tire Rubber Powder. An optimum percentage of WCO Tire rubber powder and Bagasse ash were determined through physical testing. Performance Test include rutting resistance Test, Fatigue resistance test and low temperature cracking resistance test.

4.3.1 Dynamic Shear Rheometer Test

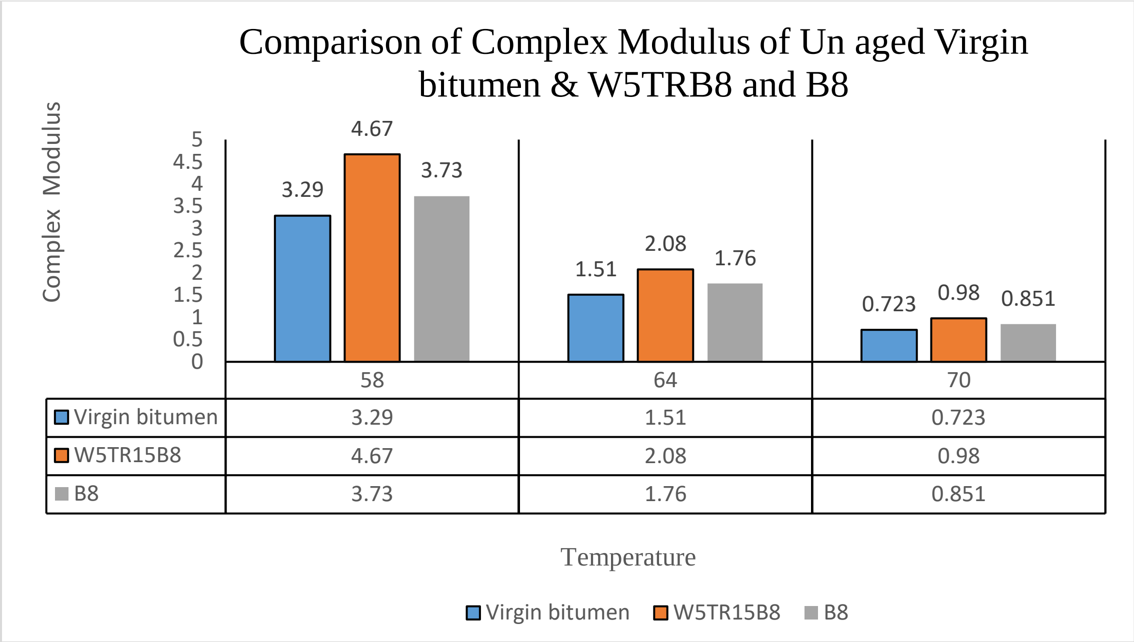
DSR test were conducted on Un aged, and Aged samples. Asphalt binder Performance Grade determination is very important for assessing the properties of asphalt under field condition. In order to evaluate performance grade asphalt binder Performance Grade asphalt binder were tested on DSR Un aged, RTFO aged and PAV aged.

4.3.2 DSR (High temperature Penetration Grade)

DSR testing is capable of evaluating the viscoelastic behavior of asphalt. It determines the value of phase angle and complex modulus G*. The rutting potential of asphalt can be assessed by factor G* / sin δ. Un aged sample of virgin bitumen, bitumen replaced and modified with WCO TRP and BA having sample code W5TR15B8 and bagasse ash added bitumen B8 were tested in DSR apparatus. Result indicate that with the increase in temperature complex modulus G* decreases which shows decreases in rutting susceptibility. It also shows that temperature has negative impact on rutting potential of asphalt binder. Result shows that virgin bitumen can resist rutting up to 64^{o}C, followed by bagasse ash B8 showing the same rutting resistance as that of virgin bitumen. Sample W5TR15B8 contain high content of rubber powder and bagasse ash, in Un aged condition fail at 70^{o}C showing same resistance against rutting as that of virgin bitumen and B8. It may be point to the fact that, with the addition of waste cooking oil in bitumen, complex modulus decreases, while phase angle increases at medium and high temperature (Zhaojie Sun 2016). Thus, it can be concluded that, adding waste cooking oil decreases deformation resistance and elastic recovery. However Even after replacement with Waste cooking and tire rubber powder sample W5TR15B8 behave like the virgin one which is good from the economic point of view. Table shows the result obtained from DSR apparatus for Un aged sample.

Table 4-7 Showing DSR result of Un aged virgin, W5TR15B8, and B8

Sample type	**Sample code**	**Temperature**	**Complex modulus**	**PG Grade**
Virgin bitumen 60-70	60-70	58	3.29	Criteria G*/sin(delta)>1 PG 64
		64	1.51	
		70	0.723	
Virgin bitumen + 8%BA	B8	58	3.73	PG-64
		64	1.76	
		70	0.851	
60-70-5%WCO-15%RP+8%BA	W5TR15B8	58	4.67	Criteria G*/sin(delta)>1 PG 64
		64	2.08	
		70	0.98	



	58	64	70
Virgin bitumen	3.29	1.51	0.723
W5TR15B8	4.67	2.08	0.98
B8	3.73	1.76	0.851

Figure 4-8 Comparison of Complex Modulus of Un aged Virgin bitumen & W5TRB8 and B8

4.3.3 DSR High performance Grade (RTFO aged)

Sample of virgin sample, bitumen replaced and modified with Waste cooking oil tire rubber powder and Bagasse ash (W5TR15B8) and Bagasse ash added bitumen B8 were aged for 82 minutes in rotatory thing film oven in order to determine the performance of bitumen after short term aging. The ages sample were tested on DSR. Criteria for higher performance Grade is G*/sin δ is 2.2 KP. Result shows that virgin bitumen has the same performance Grade as that of Un aged bitumen which is 64^{o}C. Sample W5TR15B8 and B8 fails at temperature 75^{o}C and hence having high temperature performance Grade as 70^{o}. Sample W5TR15B8 and B8 shows improvement in rutting resistance. Improved rutting resistance of sample W5TR15B8 can be attributed to the high content of rubber powder and bagasse ash which both improve rutting resistance. Sample B8 containing only bagasse ash also shows improvement in rutting resistance due to the presence of bagasse ash.

Table 4-8 Complex modulus of RTFO aged virgin W5TR15B8 and B8

Sample type	Sample code	Temperature	Complex modulus	PG Grade
Virgin bitumen (60-70)	60-70	58	3.29	Criteria G*/sin(delta)>2 PG 64
		64	1.51	
		70	0.723	
Virgin bitumen +8%BA	B8	64	4.86	PG-70
		70	2.43	
		75	1.24	
60-70-5%WCO-15%TRP+8%BA	W5TR15B8	58	7.81	Criteria G*/sin(delta)>2 PG 70
		64	4.3	
		70	2.48	
		75	1.3	

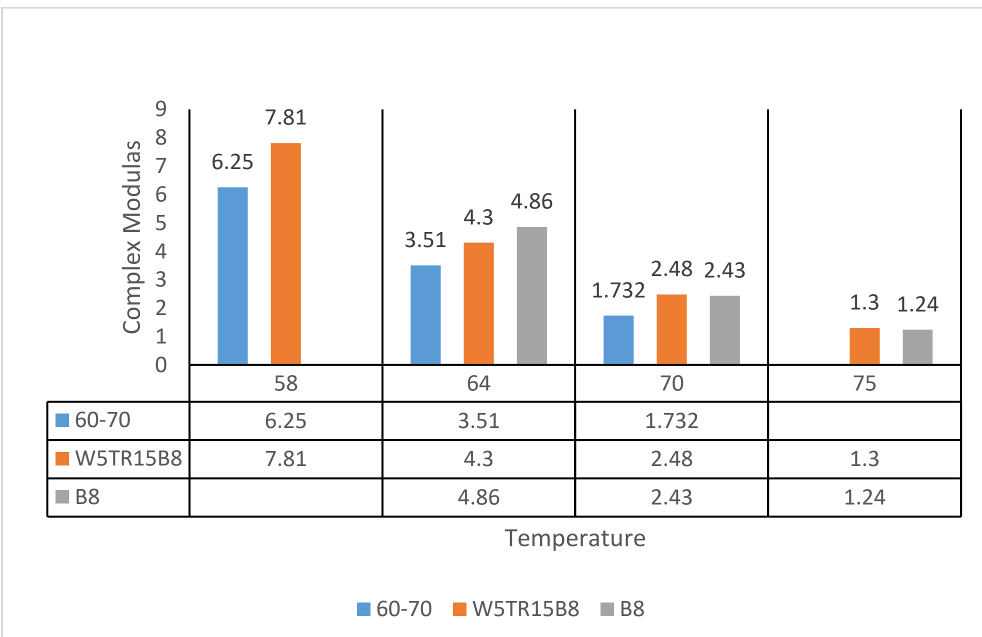


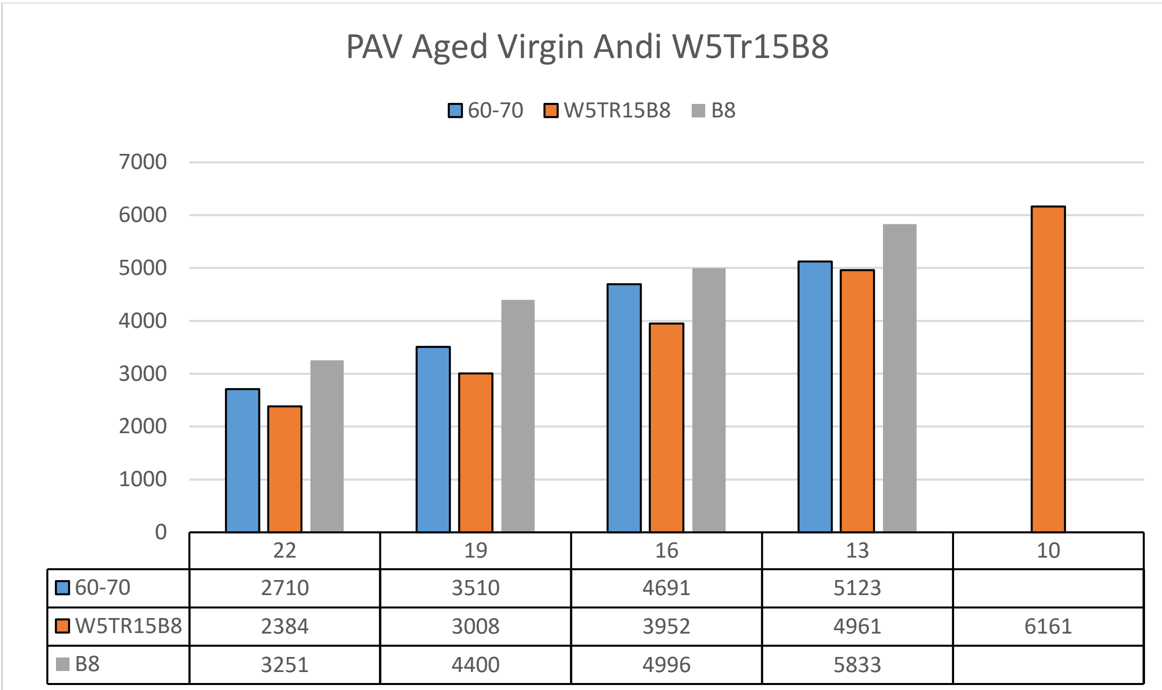
Figure 4-9 Comparison of complex modulus RTFO aged, virgin, W5TR15B8 and B8

4.3.4 DSR Intermediate Temperature Performance Grade PAV Aged/Fatigue Resistance

This test is conducted on bitumen to determine its fatigue resistance. Fatigue resistance usually occur at intermediate temperature. To resist maximum fatigue cracking asphalt must be viscous in intermediate temperature range. According to super pave specification (G*sin δ) can be used to assess fatigue resistance. Lower the value of (G*sin δ) h) higher will be the resistance against fatigue. The higher value of (G*sin δ) indicate that asphalt binder rapidly dissipates the shearing strain energy under load. (G*sin δ) factor can be assess using Dynamic shear Rheometer. RTFO aged sample is placed in PAV equipment for 20 hours. The temperature and pressure inside the PAV equipment were maintained 100C and 2.2 pascal. After that sample is tested in DSR equipment to determine temperature till which it will behave positively against fatigue. Both Virgin bitumen and Bagasse added sample B8 fails the criteria for fatigue cracking resistance at 13^{o}C, showing both have the same fatigue resistance, while sample W5TR15B8 fails the criteria for fatigue cracking at 10^{o}C. It shows that sample W5TR15B8 poses greater resistance towards fatigue than virgin sample and B8. Improved fatigue resistance of sample W5TR15B8 may be attributed to the fact that, tire rubber and waste cooking oil improve the elastic recovery potential of bitumen.

Table 4-9 Complex modulus of PAV aged virgin and W5TR15B8

Type of sample	Sample Code	Temperature	Complex modulus	Result
Virgin	60-70	22	2760	G*.sin(delta)<5000
		19	3500	
		16	4530	
		13	5123	
Virgin-5%WCO-15%TRP+8%BA	W5TR15B8	22	2384	G*.sin(delta)<5000
		19	3008	
		16	3952	
		13	4961	
		10	6161	
Virgin +8BA	B8	22	3251	G*.sin(delta)<5000
		19	4400	
		16	4996	
		13	5833	



	22	19	16	13	10
60-70	2710	3510	4691	5123	
W5TR15B8	2384	3008	3952	4961	6161
B8	3251	4400	4996	5833	

Figure 4-10 Comparison of G*.sin (delta) value of PAV aged 60-70, W5Tr15B8 and B8 sample

4.3.5 BBR low temperature results (D6648 – 08)

Low temperature cracking is an important pavement distress that is found in colder region. Tensile stresses in generated in pavement due to low temperature causes cracking. Performance Evaluation of bitumen at low temperature must be assessed to determine the suitability of bitumen use in colder region. Bending Beam Rheometer is use for determining low temperature performance Grade of bitumen. It measures two parameters represented by m and S, where m is the creep rate and S is the Creep stiffness. These parameters determine bitumen ability to resist low temperature cracking. BBR test were conducted on PAV Aged Virgin bitumen and W5TR15B8 and the result were compared. The objective of the test is to determine low temperature resistance capability of the bitumen (Partially replaced with WCO, TRP and modified with BA) and compare it with the virgin bitumen. BBR can take measurement in temperature range 36C to 0C. The minimum value of creep stiffness S is 300 and creep rate 0.300.

Virgin sample, W5TR15B8 and B8 sample were tested in BBR apparatus to at different temperature to determine low temperature cracking resistance of these sample. Result shows that virgin sample and bagasse ash added sample B8 can resist cracking up to -12 and -10 respectively, while sample W5TR15B8 containing waste cooking oil tire rubber powder and bagasse ash can resist cracking up to -18^{o}C which shows that this sample has highest potential resistance toward low temperature cracking. the highest low temperature resistance of sample W5TR15B8 can be attributed to the presence of waste cooking oil and tire rubber powder in the sample which both are responsible for improving low temperature cracking resistance. The presence of rubber can enhance low temperature resistance of bitumen. Waste cooking oil improve the stress relaxing capability (indicated by m) of the bitumen. Bagasse ash show less resistance to cracking at low temperature and can resist low temperature cracking up to -10. It may be attributed to the fact that bagasse ash makes the sample stiffer and decreases its elastic recovery potential. The use of tire rubber powder in bitumen can enhance its resistance against skidding, durability and low temperature resistance as already determined in BBR experiment Marek Pszczoła, 2016. [1]. Hainian Wang [3]. Conducted research on the performance evaluation of bitumen modified with tire scrap rubber and conclude that rubber uses in bitumen can significantly improve both the viscosity of bitumen at higher temperature and creep stiffness at lower temperature.

Table 4-10 Creep Stiffness and Slope value of 60-70, W5TR15B8 and B8 sample at varying temperature

Sample type	Sample Code	Temperature	Creep Stiffness	M (Slope)	Remarks
Virgin	60-70	-6	59.68	0.50	Stiffness<300 m>=0.3
		-12	133.5414	0.32	
		-18	205.688	0.2257	
Virgin +8BA	B8	-6	170	0.322	
		-12	208.2372	0.2344	
		-18	304.3278	0.1349	
Virgin-5%WCO-15%TRP+8%BA	W5TR15B8	-6	56.3390	0.554	
		-12	74.043	0.38	
		-18	111.339	0.301	
		-22	208.46	0.24	

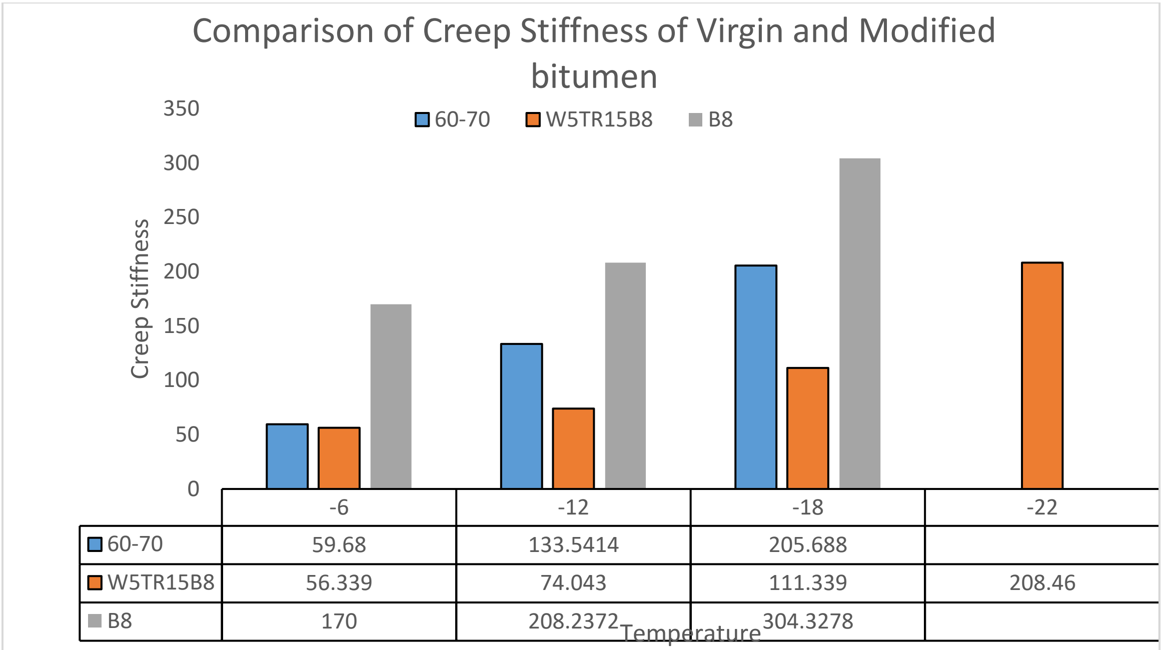


Figure 4-11 Comparison of Creep Stiffness of Virgin, W5TR15B8 and B8 sample

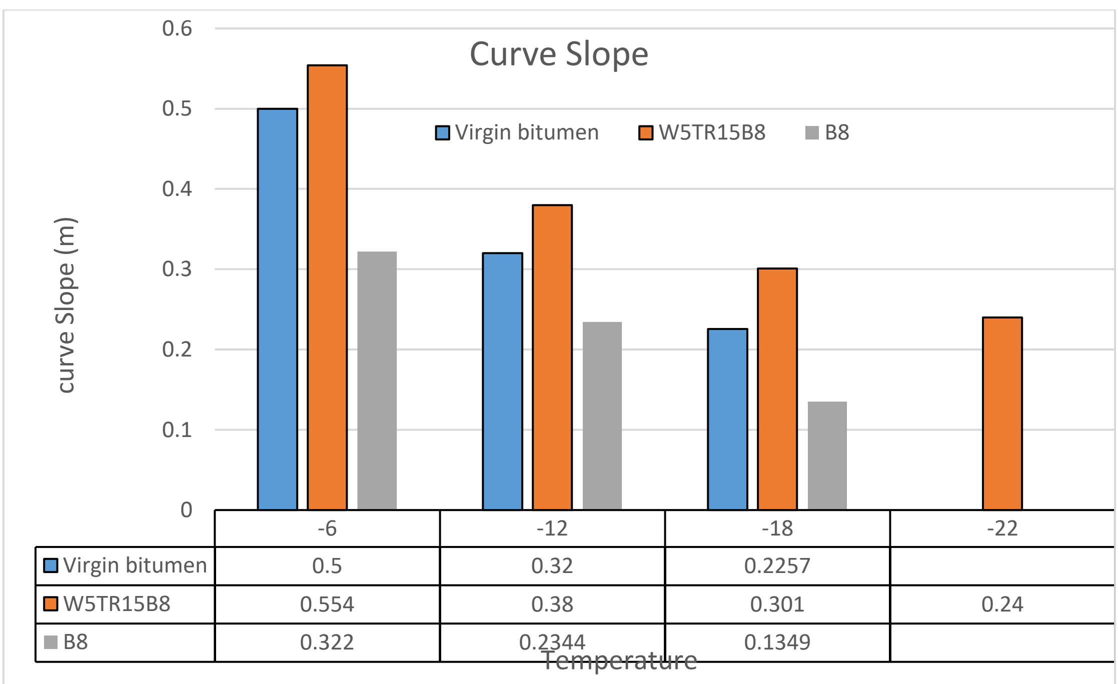


Figure 4-12 Comparison of Slope value of Virgin, W5TR15B8 and B8 at different temperature

4.4 Chemical Analysis.

Chemical analysis is important to understand interaction of various compound in bitumen modified with WCO, TRP and Bagasse Ash. The main objective of chemical testing conducted were to determine the interaction of Waste cooking oil tire rubber powder and Bagasse ash with bitumen and to see the effect of these material on chemical stability of bitumen. For this very purpose functional group analysis were conducted with the help of Fourier Transform infrared spectroscopy

FTIR test is performed to determine functional Group of organic material in order to quantify the functional group of structural indices of S=O and C=O. (Lamontagne et al. 2001) computed structural index by numerical integrating peak of target functional group and then dividing it by entire area between 600 cm-1 to 2000 cm-1. The principal of its working is that a beam of variable light frequency is shine on a sample. Light is absorbed by sample at different wavelength. Computer takes all this data and calculate what the absorption is at each wavelength.

Table 4-13 shows structural indices of Virgin bitumen, W5, B8, TR15 and W5TR15B8. The comparison of structural indices of control, W5TR15B8 and B8 are shown graphically in a figure 4.17. Results indicate that S=O and C=O are two functional group responsible for hardening of bitumen of asphalt binder. Sample TR15 shows higher number of structural indices of S=O and C=O as compare to virgin sample which shows that with the addition of tire rubber powder S=O and C=O increase cause the binder's hardening. Addition of WCO to binder in sample W5, shows decrease in a structural index of S=O and C=O which point to the fact that waste cooking oil had reacted chemically that lead to decrease in functional group of S=O and C=O. Sample B8 shows increase in structure indices which point to the fact that with the addition of bagasse ash bitumen become hard. Increase in structure indices of S=O and C=O in case of Sample TR5 and B8 shows that reaction of tire rubber powder with bitumen and bagasse ash is physical.

Table 4-11 Structure indices of Virgin, Tr15 WCO5, BA and W5TR15B8

Type of Sample	Sample Code	Functional Group	Wave Number	Structural Indices
60-70	Virgin	S=O	1050	0.01191
		C=O	1750	0.00287
60-70-15%TR	TR15	S=O	1050	0.01491
		C=O	1750	0.01300
60-70-5%WCO	WCO5	S=O	1050	0.00012
		C=O	1750	0.00016
60-70+ 8%BA	8BA	S=O	1050	0.00191
		C=O	1750	0.00175
60-70-W5-TR15+B8	W5TR15B8	S=O	1050	0.01092
		C=O	1750	0.00291

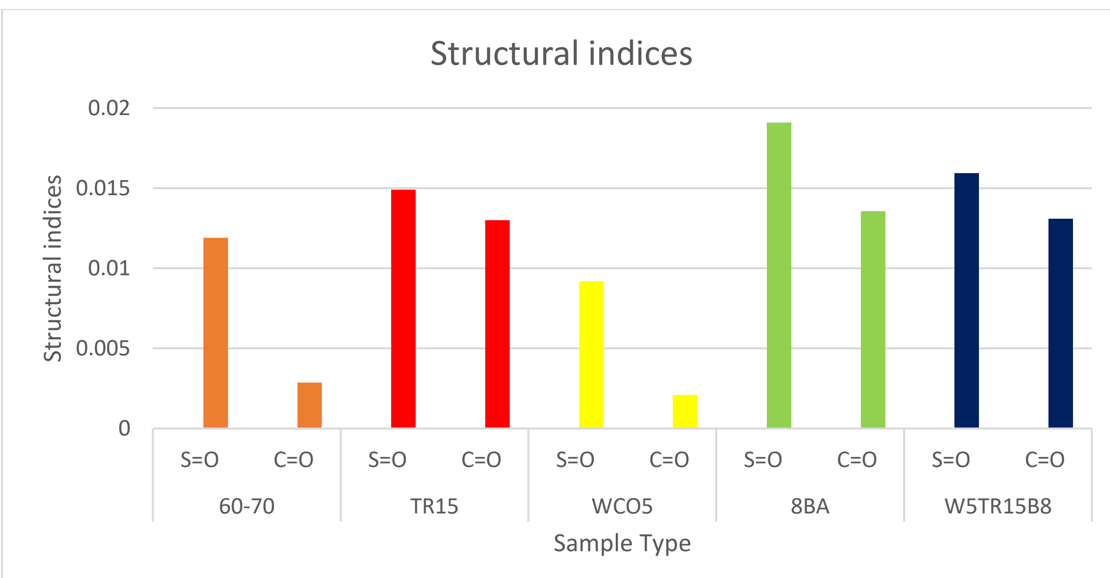


Figure 4-13 comparison of structure indices of virgin, TR15, WCO5, W5TR15B8

4.5 Cost Analysis

Cost analysis were carried out in term of price of bitumen use in one kilometer of road. Price of waste material such as tire rubber powder, waste cooking oil and bagasse ash have been incorporated while carrying out cost analysis.

Pakistan import about 75% of its edible oil to meet its consumer demand. According to survey around 1300 thousand metric tons cooking oil is being used every year in Pakistan. Scrap tire has been used in metric tons in Pakistan.

Comparison of cost of bitumen use in one kilometer of road were carried out between virgin Bitumen (60-70) and modified bitumen (W5TR15B8). Result indicate that cost of bitumen/km of road decreases by 25.7 percent with the use of modified of bitumen.

Table 4-12 Cost per/km of virgin and modified bitumen

Type of Sample	Sample Code	Cost (PKR)/Km	Percentage Decrease in cost
60-70	60-70	4072152	0
60-70-15%TR-5%WCO+8%BA	W5TR15B8	3425752	15.9

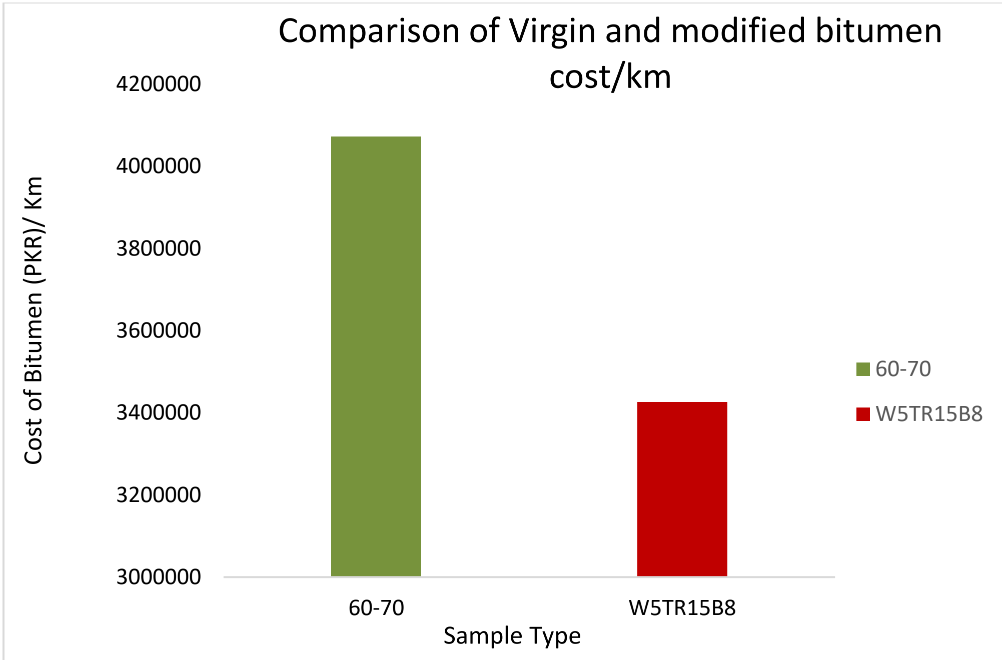


Figure 4-14 comparison of Cost of bitumen/km of Virgin and modified bitumen

4.6 Summary of the Chapter

4.6.1 Penetration

- Penetration of virgin bitumen decreases with addition of waste cooking oil, decreasing hardness of sample
- up to 15% tire rubber replacement in bitumen and addition of 8% bagasse ash (W5TR15B8) can increases penetration of sample. Further increase in percentage replacement of tire rubber powder will decrease penetration value because of loss of binding property of bitumen.

4.6.2 Softening point

- Softening point of virgin bitumen decreases with addition (as percentage replacement of bitumen) of waste cooking oil, can be seen in sample W5, W10.
- Softening point of bitumen is increased with addition of tire rubber powder and bagasse ash.
- Blend of 5 percent waste cooking oil, 15 percent tire rubber powder and 8 percent bagasse ash show higher softening point than control sample.

4.6.3 Rotational viscosity

- Rotational Viscosity decreases with the addition of waste cooking oil.

- Rotational viscosity increases when tire rubber powder and bagasse ash incorporated into bitumen containing waste cooking oil.
- Sample W5TR15B8 shows higher viscosity than virgin bitumen.

4.6.4 Performance Evaluation of Modified binder

- Modified bitumen shows better resistance against rutting. Complex modulus value of modified bitumen (W5TR15B8) is higher as compared to virgin bitumen 60-70.
- Modified bitumen shows better resistance against fatigue as compare to virgin bitumen.
- modified bitumen W5TR15B8 shows improved resistance against low temperature cracking as compare to virgin bitumen

4.6.5 Chemical Analysis

- Chemical analysis shows that waste cooking oil increases softness of bitumen.
- Interaction of waste cooking oil with bitumen is chemical and structure indices of S=O and C=O, which are indicator of hardness of bitumen decreases with the addition of waste cooking oil as can be seen in sample W5.
- Tire rubber powder and bagasse add react physically with bitumen. Sample B8 shows higher structure indices of S=O, C=O, which indicate that it increases hardness of bitumen because penetration increases with the addition of bagasse ash into bitumen.
- The induction of tire rubber powder and bagasse ash into bitumen increases the structure indices of C=O and S=O.
- XRD analysis of Bagasse ash shows that it contains silica in access

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

After successful completion of extensive lab testing, carried out in an effort to obtain the targeted objectives of this research work, following conclusions can be drawn based on lab test result and logical observations.

- All physical and performance test result shows that about 20% of bitumen can be replaced with waste cooking oil, tire rubber powder in the presence of bagasse ash use as modifier. The incorporation of these waste into bitumen will solve the disposal problem and will also help in protecting environment, by minimizing, hazardous fume releases from bitumen and disposal of waste which also causes environmental problems.
- As compare to virgin bitumen, modified bitumen W5TR15B8 shows remarkable resistance against rutting which can be attributed to the presence of tire rubber and bagasse ash.
- Modified bitumen shows good resistance to fatigue cracking than virgin bitumen. It is attributed to the presence of waste cooking oil and tire rubber powder which both increases the elastic recovery potential of bitumen.
- Modified bitumen shows remarkably good resistance to low temperature cracking as compare to virgin bitumen. Modified bitumen W5TR15B8 can resist low temperature as low as up to -18^{o}C. Such high resistance against low temperature cracking is due to the presence of waste cooking oil and tire rubber powder.
- Cost analysis of virgin and modified bitumen shows that use of modified bitumen is both economical and environmentally friendly. It can decrease bitumen cost used per kilometer by 25%

5.2 Recommendation

- This research study aims to obtain modified bitumen by replacing certain percentage of bitumen with waste cooking oil tire rubber powder and bagasse ash thus helping in protecting environment by incorporating waste material into useful product.
- In this research work, modified bitumen has been assessed through performance and physical testing.

- The focus of new research studies should be to use this modified bitumen (W5TR15B8) in asphaltic pavement and carrying out performance testing such as Rutting, Fatigue, Marshall Stability, flow test.

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APPENDICES

APPENDICES I DSR HIGH TEMPERATURE REPORTS

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1008961
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	Aged virgin(60-70).gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\RTFO
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PG64 TruGrade Temperature (°C) : 69.81

Ancillary Information :

Sample :	Vergin 60-70
User Id :	
Test Number :	0
Terminal Location :	17/8/2017
Grade :	60-70
Tank Number :	1
Lot Number :	1
Project :	Lab Test

Parameters :

Measurement Type :	High Temperature Range
Initial Temperature (°C) :	58.00
Strain Amplitude (%) :	12.00
Measuring System :	PP 25 DSR
Plate Diameter (mm) :	25
Plate Gap (mm) :	1.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Original Binder (High)

Grade Run 1 Results : 1/1/2004 12:16:21 AM

Complex Modulus (kPa) :	2.08
Phase Angle (°) :	81.8
G*/Sin(Delta) (kPa) :	2.1
Strain (%) :	11.90
Shear Stress (Pa) :	246.757
Temperature (°C) :	64.00
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 12:46:22 AM Malvern Instruments Page 1 of 2

TruGrade Software

Grade Run 2 Results :		1/1/2004 12:16:21 AM
	Complex Modulus (kPa) :	0.969
	Phase Angle (°) :	83.9
	G*/Sin(Delta) (kPa) :	0.975
	Strain (%) :	12.03
	Shear Stress (Pa) :	116.144
	Temperature (°C) :	70.01
	Frequency (rad/s) :	10.03
	Result :	Fail

Operator Notes :

60-70

Performance Grade Asphalt Analysis

TruGrade Software

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1008981
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	w5tr15b8.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\RTFO
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PG70 TruGrade Temperature (°C) : 71.12

Ancillary Information :	
Sample :	W5TR15B8
User Id :	
Test Number :	1
Terminal Location :	TN LAB
Grade :	80/100
Tank Number :	1
Lot Number :	
Project :	
Parameters :	
Measurement Type :	High Temperature Range
Initial Temperature (°C) :	58.00
Strain Amplitude (%) :	10.00
Measuring System :	PP 25 DSR
Plate Diameter (mm) :	25
Plate Gap (mm) :	1.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Rolling Thin Film (High)
Grade Run 1 Results :	1/1/2004 12:20:01 AM
Complex Modulus (kPa) :	2.32
Phase Angle (°) :	69.6
G*/Sin(Delta) (kPa) :	2.48
Strain (%) :	10.08
Shear Stress (Pa) :	232.869
Temperature (°C) :	70.01
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 12:47:05 AM   Malvern Instruments   Page 1 of 2

TruGrade Software

Grade Run 2 Results :	1/1/2004 12:20:01 AM
Complex Modulus (kPa) :	1.23
Phase Angle (°) :	70.9
G*/Sin(Delta) (kPa) :	1.3
Strain (%) :	10.12
Shear Stress (Pa) :	122.827
Temperature (°C) :	75.98
Frequency (rad/s) :	10.03
Result :	Fail

Operator Notes :

Characterizing the existing asphalt grades in Pakistan according to PG specification

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1006981
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	unaged ba.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\Baggas
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PG64
	TruGrade Temperature (°C) : 68.66

Ancillary Information :	
Sample :	UNAGED BA
User Id :	
Test Number :	1
Terminal Location :	TN LAB
Grade :	
Tank Number :	1
Lot Number :	
Project :	
Parameters :	
Measurement Type :	High Temperature Range
Initial Temperature (°C) :	64.00
Strain Amplitude (%) :	12.00
Measuring System :	PP 25 DSR
Plate Diameter (mm) :	25
Plate Gap (mm) :	1.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Original Binder (High)
Grade Run 1 Results :	8/15/2018 1:39:03 PM
Complex Modulus (kPa) :	1.75
Phase Angle (°) :	85.0
G*/Sin(Delta) (kPa) :	1.76
Strain (%) :	12.14
Shear Stress (Pa) :	212.03
Temperature (°C) :	63.99
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 11:32:41 PM | Malvern Instruments | Page 1 of 2

TruGrade Software

Grade Run 2 Results :	8/15/2018 1:39:03 PM
Complex Modulus (kPa) :	0.849
Phase Angle (°) :	86.5
G*/Sin(Delta) (kPa) :	0.851
Strain (%) :	11.94
Shear Stress (Pa) :	101.214
Temperature (°C) :	70.00
Frequency (rad/s) :	10.03
Result :	Fail

Operator Notes :

Characterizing the existing asphalt grades in Pakistan according to PG specification

MALVERN
TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1008961
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	rtfo aged BA.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\Baggas
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PG70 TruGrade Temperature (°C) : 71.07

Ancillary Information :	
Sample :	RTFO aged BA
User Id :	
Test Number :	1
Terminal Location :	TN LAB
Grade :	
Tank Number :	1
Lot Number :	
Project :	
Parameters :	
Measurement Type :	High Temperature Range
Initial Temperature (°C) :	64.00
Strain Amplitude (%) :	10.00
Measuring System :	PP 25 DSR
Plate Diameter (mm) :	25
Plate Gap (mm) :	1.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Rolling Thin Film (High)
Grade Run 1 Results :	8/15/2018 12:45:59 PM
Complex Modulus (kPa) :	2.43
Phase Angle (°) :	78.8
G*/Sin(Delta) (kPa) :	2.48
Strain (%) :	9.93
Shear Stress (Pa) :	240.984
Temperature (°C) :	70.01
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 11:32:07 PM Malvern Instruments Page 1 of 2

TruGrade Software

Grade Run 2 Results :	8/15/2018 12:45:59 PM
Complex Modulus (kPa) :	1.24
Phase Angle (°) :	80.5
G*/Sin(Delta) (kPa) :	1.26
Strain (%) :	10.14
Shear Stress (Pa) :	125.605
Temperature (°C) :	75.98
Frequency (rad/s) :	10.03
Result :	Fail

Operator Notes :

Characterizing the existing asphalt grades in Pakistan according to PG specification

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1008981
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	Virgin bitumen (60-70).gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\unaged
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PG64 TruGrade Temperature (°C) : 67.38

Ancillary Information :	
Sample :	Virgin bitumen 60-70
User Id :	
Test Number :	1
Terminal Location :	17/8/2017
Grade :	60/70
Tank Number :	1
Lot Number :	1
Project :	Lab Test
Parameters :	
Measurement Type :	High Temperature Range
Initial Temperature (°C) :	58.00
Strain Amplitude (%) :	12.00
Measuring System :	PP 25 DSR
Plate Diameter (mm) :	25
Plate Gap (mm) :	1.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Original Binder (High)
Grade Run 1 Results :	1/1/2004 1:41:43 AM
Complex Modulus (kPa) :	1.51
Phase Angle (°) :	84.5
G*/Sin(Delta) (kPa) :	1.51
Strain (%) :	11.92
Shear Stress (Pa) :	178.947
Temperature (°C) :	64.01
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 12:48:00 AM Malvern Instruments Page 1 of 2

MALVERN
TruGrade Software

Grade Run 2 Results :	1/1/2004 1:41:43 AM
Complex Modulus (kPa) :	0.723
Phase Angle (°) :	85.7
G*/Sin(Delta) (kPa) :	0.725
Strain (%) :	12.09
Shear Stress (Pa) :	87.1216
Temperature (°C) :	70.01
Frequency (rad/s) :	10.03
Result :	Fail

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1006981
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	Unaged W5TR15B8.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\unaged
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PG64 TruGrade Temperature (°C) : 69.78

Ancillary Information :	
Sample :	W5TR15B8
User Id :	
Test Number :	1
Terminal Location :	NIT LAB
Grade :	60/70
Tank Number :	1
Lot Number :	1
Project :	
Parameters :	
Measurement Type :	High Temperature Range
Initial Temperature (°C) :	58.00
Strain Amplitude (%) :	12.00
Measuring System :	PP 25 DSR
Plate Diameter (mm) :	25
Plate Gap (mm) :	1.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Original Binder (High)
Grade Run 1 Results :	1/1/2004 12:18:26 AM
Complex Modulus (kPa) :	2.05
Phase Angle (°) :	82.9
G*/Sin(Delta) (kPa) :	2.07
Strain (%) :	12.01
Shear Stress (Pa) :	246.188
Temperature (°C) :	64.00
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 12:48:41 AM Malvern Instruments Page 1 of 2

TruGrade Software

Grade Run 2 Results :	1/1/2004 12:18:26 AM
Complex Modulus (kPa) :	0.973
Phase Angle (°) :	84.7
G*/Sin(Delta) (kPa) :	0.978
Strain (%) :	12.04
Shear Stress (Pa) :	116.765
Temperature (°C) :	69.96
Frequency (rad/s) :	10.03
Result :	Fail

APPENDICES II DSR INTERMEDIATE TEMPERATURE REPORTS

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1008981
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	60-70 PAV.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\PAV
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PAV
	TruGrade Temperature (°C) : 15.28

Ancillary Information :	
Sample :	01
User Id :	
Test Number :	0
Terminal Location :	NIT LAB
Grade :	60-70 pav
Tank Number :	1
Lot Number :	1
Project :	MS Thesis
Parameters :	
Measurement Type :	Intermediate Temperature Range
Initial Temperature (°C) :	40.00
Strain Amplitude (%) :	1.00
Measuring System :	PP 8 DSR
Plate Diameter (mm) :	8
Plate Gap (mm) :	2.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Pressure Aging Vessel (Int)
Grade Run 1 Results :	1/1/2004 12:33:04 AM
Complex Modulus (kPa) :	8.54E3
Phase Angle (°) :	33.3
G*.Sin(Delta) (kPa) :	4.69E3
Strain (%) :	1.01
Shear Stress (Pa) :	86238.4
Temperature (°C) :	16.00
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 11:27:01 PM Malvern Instruments Page 1 of 2

TruGrade Software

Grade Run 2 Results :	1/1/2004 12:33:04 AM
Complex Modulus (kPa) :	1.17E4
Phase Angle (°) :	31.8
G*.Sin(Delta) (kPa) :	6.13E3
Strain (%) :	1.01
Shear Stress (Pa) :	117769
Temperature (°C) :	12.99
Frequency (rad/s) :	10.03
Result :	Fail

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1008961
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	PAV BA.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\Baggas
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PAV TruGrade Temperature (°C) : 17.63

Ancillary Information :

Sample :	PAV BA
User Id :	
Test Number :	1
Terminal Location :	
Grade :	
Tank Number :	1
Lot Number :	
Project :	

Parameters :

Measurement Type :	Intermediate Temperature Range
Initial Temperature (°C) :	28.00
Strain Amplitude (%) :	1.00
Measuring System :	PP 8 DSR
Plate Diameter (mm) :	8
Plate Gap (mm) :	2.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Pressure Aging Vessel (Int)

Grade Run 1 Results : 1/1/2004 1:01:55 AM

Complex Modulus (kPa) :	7.78E3
Phase Angle (°) :	34.4
G*.Sin(Delta) (kPa) :	4.4E3
Strain (%) :	1.01
Shear Stress (Pa) :	78488.6
Temperature (°C) :	18.99
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 1:09:55 AM Malvern Instruments Page 1 of 2

TruGrade Software

Grade Run 2 Results :	1/1/2004 1:01:55 AM
Complex Modulus (kPa) :	1.08E4
Phase Angle (°) :	32.6
G*.Sin(Delta) (kPa) :	5.83E3
Strain (%) :	1.01
Shear Stress (Pa) :	109147
Temperature (°C) :	16.01
Frequency (rad/s) :	10.03
Result :	Fail

8/30/2018 1:09:55 AM Malvern Instruments Page 2 of 2

TruGrade Software

Performance Grade Asphalt Analysis

Test Mode :	Grade Determination (Auto-stress)
Instrument Serial Number :	MAL1006961
Temperature Control :	Mk2 TCU
Software Version :	TruGrade 1.1.0.0
File Name :	Vergin bitumen PAV.gdp
File Location :	E:\waste cooking oil\Results compilation\DSR Lab Testing\PAV
Temperature Calibration Date :	
Temperature Verification Date :	
Result :	Final Grade : PAV TruGrade Temperature (°C) : 12.91

Ancillary Information :	
Sample :	Virgin Bitumen
User Id :	
Test Number :	1
Terminal Location :	25/8/2017
Grade :	60/70 pav
Tank Number :	1
Lot Number :	1
Project :	Lab Test
Parameters :	
Measurement Type :	Intermediate Temperature Range
Initial Temperature (°C) :	34.00
Strain Amplitude (%) :	1.00
Measuring System :	PP 8 DSR
Plate Diameter (mm) :	8
Plate Gap (mm) :	2.000
Thermal Equilibrium Time (min) :	10
Sample Type :	Pressure Aging Vessel (Int)
Grade Run 1 Results :	1/1/2004 1:31:10 AM
Complex Modulus (kPa) :	1.08E4
Phase Angle (°) :	27.5
G*.Sin(Delta) (kPa) :	4.97E3
Strain (%) :	0.95
Shear Stress (Pa) :	102426
Temperature (°C) :	13.00
Frequency (rad/s) :	10.03
Result :	Pass

8/30/2018 11:29:13 PM Malvern Instruments Page 1 of 2

TruGrade Software

Grade Run 2 Results :	1/1/2004 1:31:10 AM
Complex Modulus (kPa) :	1.39E4
Phase Angle (°) :	26.2
G*.Sin(Delta) (kPa) :	6.16E3
Strain (%) :	1.01
Shear Stress (Pa) :	140940
Temperature (°C) :	10.00
Frequency (rad/s) :	10.03
Result :	Fail

APPENDICES III BBR TEST REPORTS

Test Identification

```
         Operator: Junaid
       Start Time: 15-Aug-2018 at 12:08:35
        Test Type: BBR Specimen Test
       Project ID: Virgin
  Specimen Number: 00
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
        File Name: Virgin.S00
```

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	20.5	0.0000	--	--	--	--
0.5	1227.0	0.0039	--	--	--	--
8.0	1005.9	0.5106	160.7273	160.8060	0.048914	0.446776
15.0	986.6	0.6699	120.1564	121.0172	0.711295	0.467653
30.0	986.9	0.9204	87.4807	86.4858	-1.150332	0.501699
60.0	986.3	1.3414	59.6883	60.3661	0.626043	0.505746
120.0	985.1	1.9153	41.9623	41.1523	-1.968366	0.569792
240.0	982.4	2.9160	27.4863	27.3996	-0.316390	0.603838
250.0	8.0	1.9676	--	--	--	--

Correlation Coefficient R^2 = 0.999758
Regression Coefficients: A = 5.554631, B = -0.33464, C = -0.05655

Minimum and Maximum Load from 0.5 to 240 s was 950.2 and 1227.0 mN.
Average Load from 0.5 to 240 s was 989.8 mN.
Maximum Load Deviation from 0.5 to 5 s was 237.1 mN.
Maximum Load Deviation from 5.0 to 240 s was 150.0 mN.
Minimum and Maximum Temperature from 0.5 to 240 s was -6.0 and -6.0 C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software Version 4.03W.

Elapsed Time In Bath: 60 minutes

The most recent machine calibrations were:

Type	Date	Result
Deflection (LVDT)	15-Aug-2018	1.898E+00 um/(ADC Count)
Load (Load Cell)	15-Aug-2018	6.191E-01 mN/(ADC Count)
Compliance	15-Aug-2018	4.157E+00 um/N
Confidence Check	15-Aug-2018	2.191E+02 GPa
Temperature (RTD)	11-Dec-2013	

NOTES:
Default specimen file

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Test Identification

Operator: Junad
Start Time: 17-Aug-2018 at 01:45:10
Test Type: BBR Specimen Test
Project ID: W5TR15B8
Specimen Number:
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: W5TR15B8.S

Test Results

t Time (sec)	P Load (mN)	D Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	17.8	0.4398	--	--	--	--
0.5	985.0	0.0812	--	--	--	--
8.0	973.2	0.5343	150.1317	150.1229	-0.005791	0.457866
15.0	975.3	0.7211	111.4665	111.6236	0.140734	0.484920
30.0	978.9	1.0153	78.9423	78.9388	-0.004435	0.514752
60.0	977.6	1.4753	56.3390	56.6819	-0.627116	0.554584 *
120.0	982.0	2.1651	37.1209	37.1037	-0.046146	0.574416
240.0	978.8	3.2511	24.6532	24.6610	0.031560	0.604249
250.0	14.8	1.5565	--	--	--	--

Correlation Coefficient R^2 = 0.999898
Regression Coefficients: A = 5.549534, B = -0.368374, C = -0.04955

Minimum and Maximum Load from 0.5 to 240 s was 969.6 and 985.0 mN.
Average Load from 0.5 to 240 s was 979.9 mN.
Maximum Load Deviation from 0.5 to 5 s was 7.9 mN.
Maximum Load Deviation from 5.0 to 240 s was 10.2 mN.
Minimum and Maximum Temperature from 0.5 to 240 s was -5.7 and -5.1 C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

```
 Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type               Date        Result
   Deflection (LVDT)  17-Aug-2018 1.899E+00 um/(ADC Count)
   Load (Load Cell)   17-Aug-2018 6.195E-01 mN/(ADC Count)
   Compliance         17-Aug-2018 7.098E+00 um/N
   Confidence Check   17-Aug-2018 2.139E+02 GPa
   Temperature (RTD)  11-Dec-2012

NOTES:
Default specimen file

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```
========================================================================
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                           Test Identification
========================================================================
=
           Operator: Junad
         Start Time: 15-Aug-2018 at 12:15:28
          Test Type: BBR Specimen Test
         Project ID: Virgin
    Specimen Number: 1
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
          File Name: Virgin.S1

========================================================================
=
                              Test Results
========================================================================
=
 t        P        d         Measured    Estimated
Time     Load     Defl       Stiffness   Stiffness  Difference   m-value
(sec)    (mN)     (mm)        (MPa)       (MPa)        (%)
------------------------------------------------------------------------
-
  0.0    21.8    0.0098        --          --          --          --
  0.5   969.1    0.1537        --          --          --          --
  8.0   935.3    0.3048     250.3508    252.6644    0.924109    0.276690
 15.0   976.6    0.3723     214.0249    215.0712   -1.380078    0.294580
 30.0   972.1    0.4631     171.2494    172.7322   -0.301996    0.306409
 60.0   970.5    0.5842     133.5414    134.1231    0.429187    0.327238
*
120.0   954.9    0.7363     105.8080    106.9739    1.101927    0.358067
240.0   988.3    0.9658      83.4871     82.8617   -0.749072    0.378897
250.0    46.4    0.6647        --          --          --          --
------------------------------------------------------------------------
-

Correlation Coefficient R^2 = 0.9997266
Regression Coefficients:  A = 5.624204,  B = -0.214202,  C = -0.034597

Minimum and Maximum Load from 0.5 to 240 s was 931.4 and 995.8 mN.
Average Load             from 0.5 to 240 s was 966.5 mN.
Maximum Load Deviation   from 0.5 to   5 s was 26.7 mN.
Maximum Load Deviation   from 5.0 to 240 s was 35.1 mN.
Minimum and Maximum Temperature from 0.5 to 240 s was -11.2 and -11.1 C.

========================================================================
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                            Test Conditions
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=
This test was performed using an Applied Test Systems Bending Beam
Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software
version 4.03W.
```

```
Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type                Date          Result
   Deflection (LVDT)  15-Aug-2018  1.901E+00 um/(ADC Count)
   Load (Load Cell)   15-Aug-2018  6.207E-01 mN/(ADC Count)
   Compliance         15-Aug-2018  4.143E+00 um/N
   Confidence Check   15-Aug-2018  2.225E+02 GPa
   Temperature (RTD)  11-Aug-2012

NOTES:
60/70

========================================================================
=
```

Test Identification

Operator: Junad
Start Time: 16-Aug-2018 at 12:26:33
Test Type: BBR Specimen Test
Project ID: W5TR15B8
Specimen Number: 02
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: W5TR15B8.S02

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	19.5	0.0381	--	--	--	--
0.5	1079.4	0.2827	--	--	--	--
8.0	1038.2	0.5913	143.2472	142.6680	-0.404354	0.281473
15.0	980.5	0.6812	117.4430	118.3882	0.804784	0.312063
30.0	976.8	0.8457	94.2271	94.2521	0.026595	0.345793
60.0	996.1	1.0976	74.0434	73.3027	-1.000383	0.379524
*						
120.0	973.9	1.4369	55.2966	55.6923	0.715507	0.413254
240.0	967.7	1.9076	41.3888	41.3348	-0.130434	0.446985
250.0	20.1	1.4043	--	--	--	--

Correlation Coefficient R^2 = 0.9998925
Regression Coefficients: A = 5.36283, B = -0.180282, C = -0.056025

Minimum and Maximum Load from 0.5 to 240 s was 944.9 and 1080.0 mN.
Average Load from 0.5 to 240 s was 980.3 mN.
Maximum Load Deviation from 0.5 to 5 s was 99.7 mN.
Maximum Load Deviation from 5.0 to 240 s was 99.3 mN.
Minimum and Maximum Temperature from 0.5 to 240 s was -11.1 and -11.1 C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

```
 Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type                Date        Result
   Deflection (LVDT)   16-Aug-2018 1.901E+00 um/(ADC Count)
   Load (Load Cell)    16-Aug-2018 6.207E-01 mN/(ADC Count)
   Compliance          16-Aug-2018 4.143E+00 um/N
   Confidence Check    16-Aug-2018 2.225E+02 GPa
   Temperature (RTD)   11-Dec-2012

NOTES:
Default

========================================================================
=
```

Test Identification

Operator: Junaid
Start Time: 28-Aug-2018 at 12:08:35
Test Type: BBR Specimen Test
Project ID: Virgin
Specimen Number: 00
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: Virgin.S00

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	20.5	0.0000	--	--	--	--
0.5	1227.0	0.0039	--	--	--	--
8.0	1005.9	0.5106	260.7273	260.8060	0.048914	0.126776
15.0	986.6	0.6699	238.1564	238.0172	0.711295	0.167653
30.0	986.9	0.9204	217.4807	217.4858	-1.150332	0.191699
60.0	986.3	1.3414	205.6883	206.3661	0.676043	0.225746
120.0	985.1	1.9153	141.9623	141.1523	-1.968366	0.269792
240.0	982.4	2.9160	127.4863	127.3996	-0.316390	0.363838
250.0	8.0	1.9676	--	--	--	--

Correlation Coefficient R^2 = 0.999758
Regression Coefficients: A = 5.554631, B = -0.33464, C = -0.05655

Minimum and Maximum Load from 0.5 to 240 s was 950.2 and 1227.0 mN.
Average Load from 0.5 to 240 s was 989.8 mN.
Maximum Load Deviation from 0.5 to 5 s was 237.1 mN.
Maximum Load Deviation from 5.0 to 240 s was 150.0 mN.
Minimum and Maximum Temperature from 0.5 to 240 s was -17.1 and -17.1 C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software Version 4.03W.

```
Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type               Date         Result
   Deflection (LVDT)  28-Aug-2018  1.898E+00 um/(ADC Count)
   Load (Load Cell)   28-Aug-2018  6.191E-01 mN/(ADC Count)
   Compliance         28-Aug-2018  4.157E+00 um/N
   Confidence Check   28-Aug-2018  2.191E+02 GPa
   Temperature (RTD)  28-Dec-2013

NOTES:
Default specimen file

========================================================================
=
```

Test Identification

Operator: Junaid
Start Time: 28-Aug-2018 at 01:45:10
Test Type: BBR Specimen Test
Project ID: W5TR15B8
Specimen Number:
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: W5TR15B8.S

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	17.8	0.4398	--	--	--	--
0.5	985.0	0.0812	--	--	--	--
8.0	973.2	0.5343	174.1317	174.1229	-0.005791	0.197866
15.0	975.3	0.7211	139.4665	139.6236	0.140734	0.224920
30.0	978.9	1.0153	123.9423	123.9388	-0.004435	0.264752
60.0	977.6	1.4753	111.3390	111.6819	-0.627116	0.304584
*						
120.0	982.0	2.1651	87.1209	87.1037	-0.046146	0.334416
240.0	978.8	3.2511	54.6532	54.6610	0.031560	0.364249
250.0	14.8	1.5565	--	--	--	--

Correlation Coefficient R^2 = 0.999898
Regression Coefficients: A = 5.549534, B = -0.368374, C = -0.04955

Minimum and Maximum Load from 0.5 to 240 s was 969.6 and 985.0 mN.
Average Load from 0.5 to 240 s was 979.9 mN.
Maximum Load Deviation from 0.5 to 5 s was 7.9 mN.
Maximum Load Deviation from 5.0 to 240 s was 10.2 mN.
Minimum and Maximum Temperature from 0.5 to 240s was -17.1 and -17.1C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

```
 Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type               Date        Result
   Deflection (LVDT)  28-Aug-2018 1.899E+00 um/(ADC Count)
   Load (Load Cell)   28-Aug-2018 6.195E-01 mN/(ADC Count)
   Compliance         28-Aug-2018 7.098E+00 um/N
   Confidence Check   28-Aug-2018 2.139E+02 GPa
   Temperature (RTD)  11-Dec-2012

NOTES:
Default specimen file

========================================================================
=
```

Test Identification

Operator: Factory
Start Time: 28-Aug-2018 at 14:38:06
Test Type: BBR Specimen Test
Project ID: BA 8
Specimen Number: 00
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: BA 8.S00

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	18.6	0.0031	--	--	--	--
0.5	989.7	0.1486	--	--	--	--
8.0	992.2	0.2782	356.3898	356.0430	-0.465959	0.09991
15.0	991.8	0.3190	341.4025	341.1207	-0.508425	0.11834
30.0	989.4	0.3832	323.6503	323.7986	0.070339	0.12665
60.0	990.2	0.4743	304.3278	305.0934	0.765552	0.13495
*						
120.0	991.1	0.5962	245.6254	245.8050	-0.608591	0.165325
240.0	991.1	0.7774	226.0132	226.1063	-0.879565	0.190816
250.0	21.0	0.3571	--	--	--	--

Correlation Coefficient R^2 = 0.9990861
Regression Coefficients: A = 5.581747, B = -0.069507, C = -0.0711735

Minimum and Maximum Load from 0.5 to 240 s was 985.4 and 992.2 mN.
Average Load from 0.5 to 240 s was 989.8 mN.
Maximum Load Deviation from 0.5 to 5 s was 4.4 mN.
Maximum Load Deviation from 5.0 to 240 s was 4.3 mN.
Minimum and Maximum Temperature from 0.5 to 240 s was -17.1 and-17.1 C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

Elapsed Time In Bath: 60 minutes

The most recent machine calibrations were:

Type	Date	Result
Deflection (LVDT)	28-Aug-2017	1.784E+00 um/(ADC Count)
Load (Load Cell)	28-Aug-2017	6.201E-01 mN/(ADC Count)
Compliance	28-Aug-2017	6.605E+00 um/N
Confidence Check	28-Aug-2017	2.532E+02 GPa
Temperature (RTD)	11-Dec-2012	

NOTES:
Default specimen file

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Test Identification

Operator: Junaid
Start Time: 28-Aug-2018 at 01:45:10
Test Type: BBR Specimen Test
Project ID: WCO
Specimen Number:
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: WCO.S

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	17.8	0.4398	--	--	--	--
0.5	985.0	0.0812	--	--	--	--
8.0	974.2	0.5343	198.1317	198.1229	-0.005791	0.297866
15.0	977.3	0.7211	179.4665	179.6236	0.140734	0.334920
30.0	978.9	1.0153	163.9423	163.9388	-0.004435	0.374752
60.0	977.6	1.4753	151.3390	151.6819	-0.627116	0.414584
120.0	982.0	2.1651	87.1209	87.1037	-0.046146	0.464416
240.0	978.8	3.2511	44.6532	44.6610	0.031560	0.494249
250.0	14.8	1.5565	--	--	--	--

Correlation Coefficient R^2 = 0.999898
Regression Coefficients: A = 5.549534, B = -0.368374, C = -0.04955

Minimum and Maximum Load from 0.5 to 240 s was 969.6 and 985.0 mN.
Average Load from 0.5 to 240 s was 979.9 mN.
Maximum Load Deviation from 0.5 to 5 s was 7.9 mN.
Maximum Load Deviation from 5.0 to 240 s was 10.2 mN.
Minimum and Maximum Temperature from 0.5 to 240s was -17.1 and -17.1C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

```
 Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type               Date        Result
   Deflection (LVDT)  28-Aug-2018 1.899E+00 um/(ADC Count)
   Load (Load Cell)   28-Aug-2018 6.195E-01 mN/(ADC Count)
   Compliance         28-Aug-2018 7.098E+00 um/N
   Confidence Check   28-Aug-2018 2.139E+02 GPa
   Temperature (RTD)  11-Dec-2012

NOTES:
Default specimen file

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Test Identification

Operator: Junaid
Start Time: 28-Aug-2018 at 01:45:10
Test Type: BBR Specimen Test
Project ID: W5TR15B8
Specimen Number:
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: W5TR15B8.S

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	17.8	0.4398	--	--	--	--
0.5	995.0	0.0812	--	--	--	--
8.0	992.2	0.5343	294.1317	294.1229	-0.005791	0.117866
15.0	988.3	0.7211	239.4665	240.6236	0.140734	0.134920
30.0	987.9	1.0153	223.9423	223.9388	-0.004435	0.174752
60.0	981.6	1.4753	208.3390	208.6819	-0.627116	0.244584
*						
120.0	982.0	2.1651	174.1209	174.1037	-0.046146	0.274416
240.0	978.8	3.2511	133.6532	133.6610	0.031560	0.314249
250.0	14.8	1.5565	--	--	--	--

Correlation Coefficient R^2 = 0.999898
Regression Coefficients: A = 5.549534, B = -0.368374, C = -0.04955

Minimum and Maximum Load from 0.5 to 240 s was 969.6 and 985.0 mN.
Average Load from 0.5 to 240 s was 979.9 mN.
Maximum Load Deviation from 0.5 to 5 s was 7.9 mN.
Maximum Load Deviation from 5.0 to 240 s was 10.2 mN.
Minimum and Maximum Temperature from 0.5 to 240s was -21.1 and -21.1C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

```
 Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type                Date        Result
   Deflection (LVDT)   28-Aug-2018 1.899E+00 um/(ADC Count)
   Load (Load Cell)    28-Aug-2018 6.195E-01 mN/(ADC Count)
   Compliance          28-Aug-2018 7.098E+00 um/N
   Confidence Check    28-Aug-2018 2.139E+02 GPa
   Temperature (RTD)   11-Dec-2012

NOTES:
Default specimen file

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```

Test Identification

Operator: Junaid
Start Time: 28-Aug-2018 at 01:45:10
Test Type: BBR Specimen Test
Project ID: WCO
Specimen Number:
Specimen Dimensions: 102.00 mm x 12.70 mm x 6.35 mm
File Name: WCO.S

Test Results

t Time (sec)	P Load (mN)	d Defl (mm)	Measured Stiffness (MPa)	Estimated Stiffness (MPa)	Difference (%)	m-value
0.0	17.8	0.4398	--	--	--	--
0.5	1005.0	0.0812	--	--	--	--
8.0	996.2	0.5343	407.1317	407.1229	-0.005791	0.217866
15.0	992.3	0.7211	365.4665	365.6236	0.140734	0.234920
30.0	988.9	1.0153	343.9423	343.9388	-0.004435	0.264752
60.0	985.6	1.4753	321.3390	321.6819	-0.627116	0.294584
120.0	982.0	2.1651	274.1209	274.1037	-0.046146	0.334416
240.0	978.8	3.2511	207.6532	207.6610	0.031560	0.394249
250.0	14.8	1.5565	--	--	--	--

Correlation Coefficient R^2 = 0.999898
Regression Coefficients: A = 5.549534, B = -0.368374, C = -0.04955

Minimum and Maximum Load from 0.5 to 240 s was 969.6 and 985.0 mN.
Average Load from 0.5 to 240 s was 979.9 mN.
Maximum Load Deviation from 0.5 to 5 s was 7.9 mN.
Maximum Load Deviation from 5.0 to 240 s was 10.2 mN.
Minimum and Maximum Temperature from 0.5 to 240s was -21.1 and -21.1C.

Test Conditions

This test was performed using an Applied Test Systems Bending Beam Rheometer, serial number yy-zzzz, device ID ATS BBR 01, using software version 4.03W.

```
 Elapsed Time In Bath:  60 minutes

The most recent machine calibrations were:

   Type               Date        Result
   Deflection (LVDT)  28-Aug-2018 1.899E+00 um/(ADC Count)
   Load (Load Cell)   28-Aug-2018 6.195E-01 mN/(ADC Count)
   Compliance         28-Aug-2018 7.098E+00 um/N
   Confidence Check   28-Aug-2018 2.139E+02 GPa
   Temperature (RTD)  11-Dec-2012

NOTES:
Default specimen file

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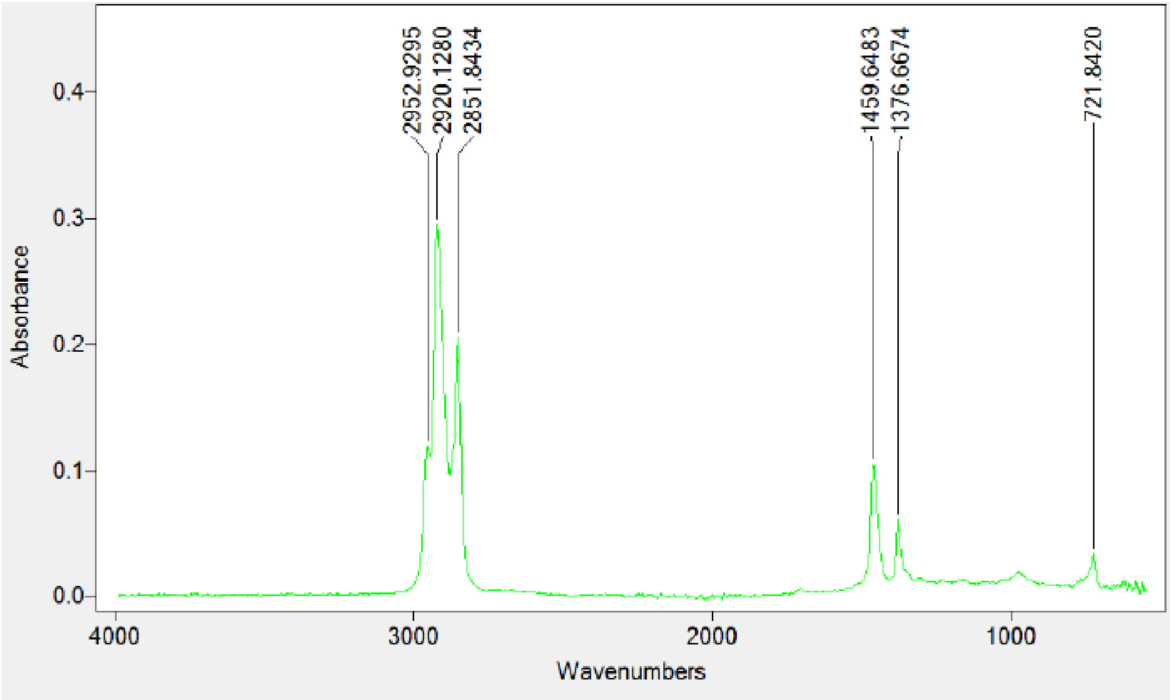
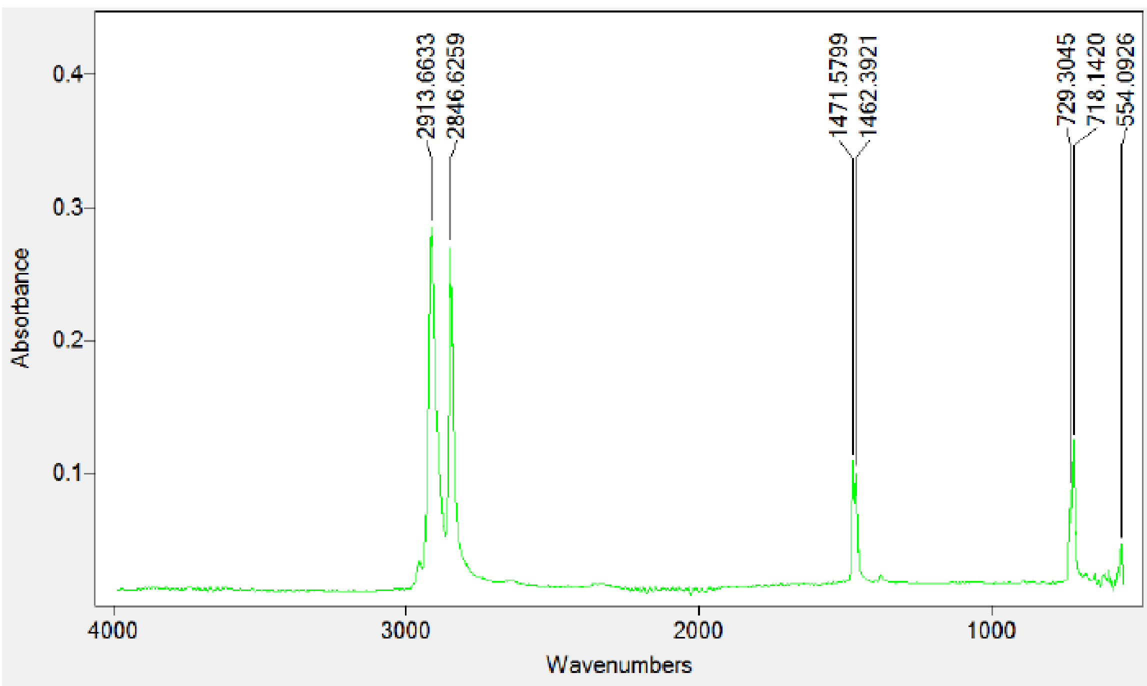
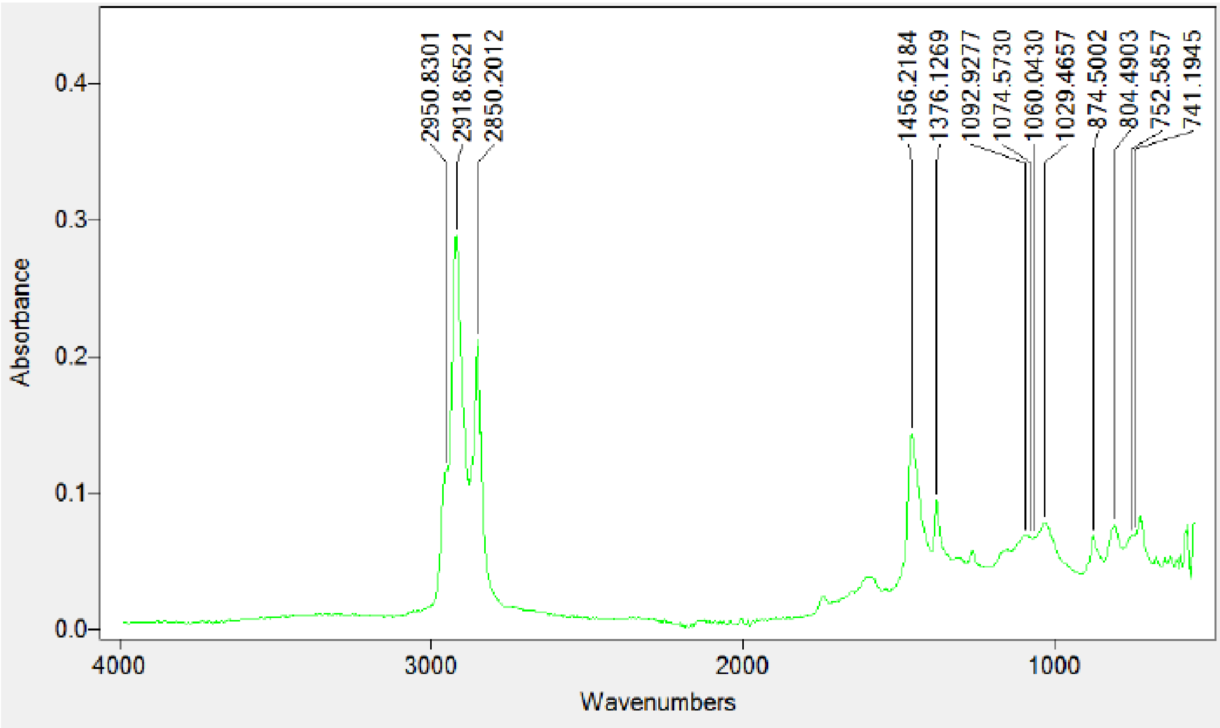
APPENDICES IV FTIR REPORTS



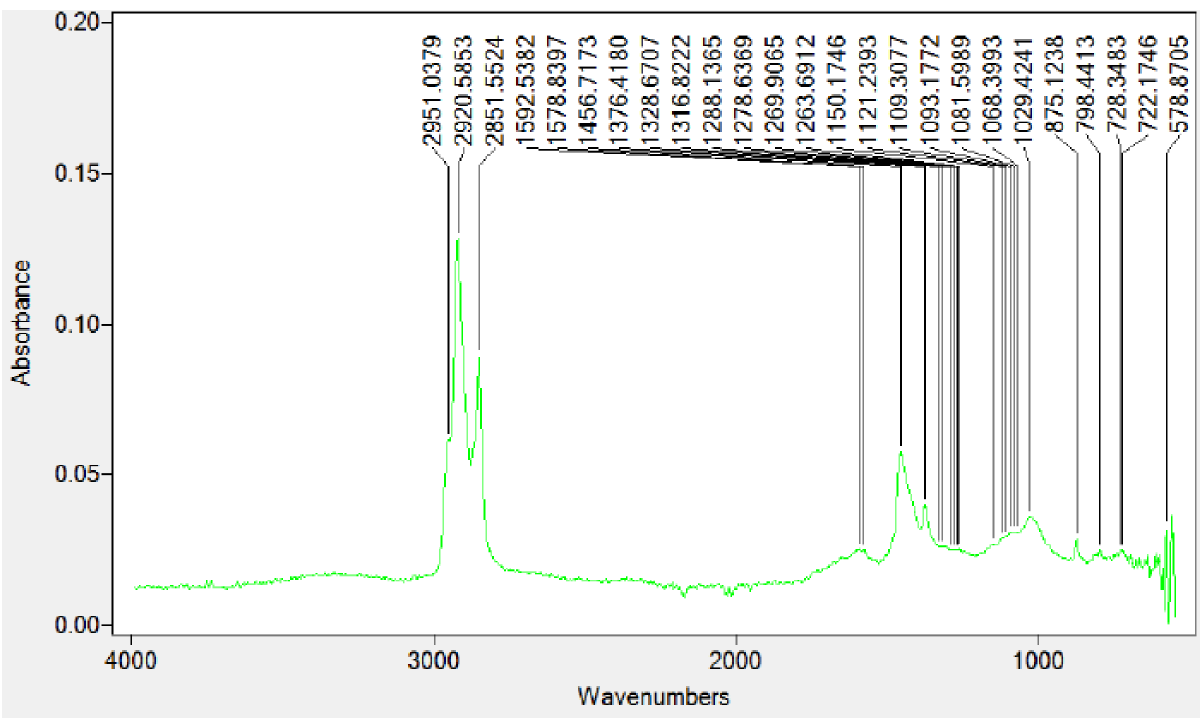
Waste Cooking Oil



60-70-15%TR



60-70-5%WCO



60-70-W5-TR15+B8