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Crumb Rubber in Performance-Graded Asphalt Binder

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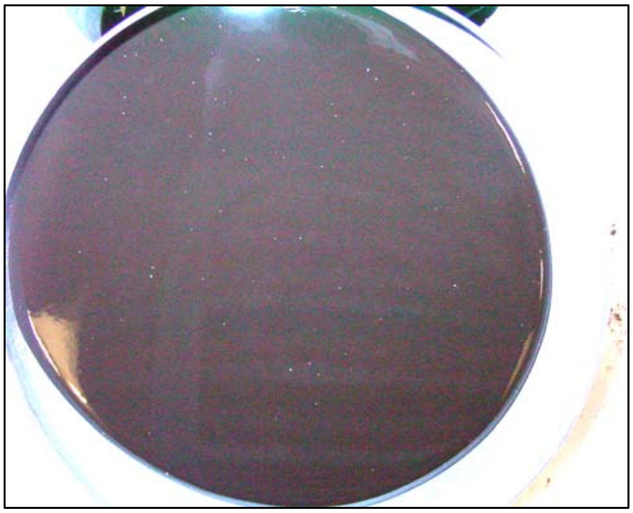

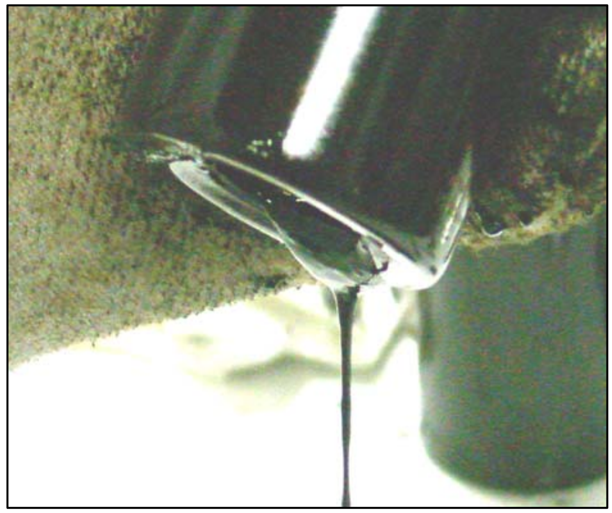

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16. Abstract

About four billion scrap tires currently reside in landfills and stockpiles across the nation. Most markets which use recycled tires as a raw material cannot support additional expansion. One of the available expanding markets for scrap tires is crumb rubber modified (CRM) asphalt for use as pavement. During production of CRM binders, there are two concerns relative to property measurement. The first is testing the appropriate properties to ensure quality control during binder production. The second is testing of binder properties to determine performance characteristics of the pavement. This study investigates the effects of various crumb rubber materials on binder performance-related parameters evaluated using performance graded testing procedures. The study also examines the interaction process variables of time and temperature to determine the appropriate parameters for producing CRM binders with specific performance related properties. Two types of interactions were evaluated. The first was short term interactions, which models actual binder production. The second was intermediate term interactions extending for up to several hours, which models a storage period. This study summarizes the effects of polymer modifiers as supplemental additives to improve the performance, stability and workability of CRM binders. The study includes recommendations for production processes and conditions, as well as suggestions for performance-related specifications pertaining to CRM asphalt production.

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Table of Contents

List of Tables

List of Figures

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CHAPTER ONE

INTRODUCTION

1.1 Background

The Environmental Protection Agency (EPA) estimates about four billion scrap tires currently reside in landfills and stockpiles across the nation (1). This number is growing by approximately 300 million tires each year, which makes the waste rubber tire issue one that will not disappear (1, 2). Scrap tires are offensive, health and fire hazards, and constitute a significant component of the solid waste management problem (1, 2, 3).

Most markets which use recycled tires as raw material cannot support additional expansion. The supply of retreated, reused, and reprocessed rubber products meets or exceeds current demand in most areas. Of the available expanding markets for scrap tires, only two have the potential to use a significantly larger number of scrap tires: fuel for combustion and crumb rubber modified (CRM) asphalt for pavement (1, 2, 3). Combustion has the potential to consume up to ten million scrap tires per licensed facility per year, or about thirty million tires annually (2). The second potential new market, CRM asphalt, presently consumes about two million tires per year (2, 4) but has the potential to recycle up to ten million scrap tires annually.

The environmental risks of health and fire hazard associated with scrap tires prompted legislative action in the early 1990's, when many states enacted laws to regulate the disposal of automobile and truck tires. At the beginning of 1991, forty-four states had drafted, introduced, regulated or enacted laws to manage the scrap tire problem (2, 4). The U.S. government passed the Intermodal Surface Transportation Efficiency Act (ISTEA) legislation in 1991. Section 1083 of this act was drafted to encourage states to use scrap tire rubber as a component of their asphalt pavements. However, due to problems with performance of pavements containing scrap rubber, ISTEA section 1083 has not been enforced since 1993 (5).

Increasing traffic volumes, heavier loads, higher tire pressures, and performance problems with pavement have goaded state highway agencies toward improving asphalt pavement quality. The Strategic Highway Research Program (SHRP) documented three types of pavement failure resulting from problems with asphalt mix design, including rutting, fatigue cracking and thermal cracking (4, 6). Evaluating the potential of an asphaltic concrete mix to develop these distresses requires testing of the aggregate, the binder and the mix design. SHRP's Superpave program specifically sought to improve testing procedures for asphalt binders. Superpave research programs also investigated the inadequacy of traditional and/or empirical binder tests to characterize asphalt pavement performance. While only partially responsible for some pavement problems, binders represent one area that has the

potential to significantly improve the overall asphalt mix and thus pavement performance. One method developed by SHRP to improve mix performance was the use of binders modified by polymers or other additives. Ground tire rubber is a binder modifier that has significant potential to improve mix performance. Addition of crumb rubber to asphalt binder produces a CRM binder that exhibits qualities similar to polymer-modified binders.

The effects of crumb rubber added to asphalt binders have historically been evaluated using traditional testing methods. Traditional testing methods measure non-fundamental binder properties and fail to test binder parameters related to pavement performance. Superpave research identified several fundamental rheological properties of asphalt binders and proposed newer testing methods that are directly related to pavement failure modes. Superpave research also established limits on binder performance-related properties to minimize failure potential and evaluate aging characteristics related to service conditions. Superpave testing methods provide a means of comparing different binders and determining the probable performance characteristics of pavements constructed using those binders.

One method of producing a CRM modified binder is the wet process. In the wet process, crumb rubber is mixed with and allowed to interact with asphalt cement at elevated temperatures prior to mixing with aggregate. As interaction of the crumb rubber and binder progresses, binder properties change. Viscosity has traditionally been used as a quality control criterion to measure the progression of the interaction between asphalt and rubber (1). SHRP research discovered that specific interaction properties, interaction time and temperature, are more important for modified asphalt binder performance than viscosity (4, 6). Interaction time and temperature need to be evaluated to determine the influence of these binder processing and production variables on pavement quality.

In the production of CRM binder, there are two concerns relative to property measurement. The first is measurement of properties necessary for quality control during production. The second is testing of binder properties during production to determine performance characteristics of the pavement. Ideally, quality control should be based upon the same measurements and data as performance testing to allow performance comparisons to be made during binder production. Use of Superpave testing procedures to monitor the production of CRM binders facilitates optimization of pavement performance characteristics.

1.2 Asphalt-Rubber Interaction

When crumb rubber is mixed and heated with asphalt cement, a modified binder is produced which has significantly different properties than the original. Property modification is due to physical and/or compositional changes during the interaction process. During the interaction process, the rubber particles swell due to absorption

of oils or volatile fluids and form a viscous gel. Rubber swells in a time and temperature dependent manner, which results in a reduction in the interparticle distance, thereby increasing viscosity. Once the rubber has swelled, if temperature is maintained too high or for too long a period, the rubber begins to disintegrate into the asphalt by partially depolymerizing. This depolymerization causes a reduction in viscosity. Change in the viscosity of the CRM asphalt binder has traditionally been used to indicate the progress of the interaction between asphalt and rubber.

Literature reports three stages of interaction that have been evaluated with regard to CRM asphalt (1, 3, 7):

1. An early stage that occurs immediately after mixing crumb rubber with asphalt.

2. An intermediate storage stage during which the binder is held at elevated temperatures for up to a few hours before mixing with aggregate.

3. An extended (storage) stage when CRM binders are stored for extended periods before mixing with aggregate. This stage was not evaluated in this study as it involves time and temperature changes well beyond those associated with the interaction process.

1.3 Relevant Properties

Traditional testing on crumb rubber modified binders has relied upon measuring non-fundamental properties such as viscosity. Using traditional testing methods, parameters of the interaction process could not be related to binder performance. Performance related properties of asphalt pavement can be extrapolated from fundamental properties of the materials and processes used to create the final product. Fundamental binder properties have been categorized into material variables and interaction variables. Material variables include asphalt cement properties and crumb rubber properties. Asphalt cement properties include stiffness and chemical composition while crumb rubber properties include source and method of processing. One other material factor affecting the binder properties is crumb rubber concentration. Interaction process variables include time and temperature. A material variable, compatibility between asphalt and CRM, must also be considered.

1.4 Problem Statement

There exist deficiencies in CRM binder research with regard to hot mix asphalt (HMA) used in pavement applications. Previous research studied properties that need to be considered primarily for producing CRM binders used in surface treatment applications. There has not been an in-depth effort to characterize the interaction processes, material variables, and their impact on binder properties for pavement

applications, especially when products must meet performance (Superpave) specifications.

Adding rubber particles to asphalt binder alters the performance of HMA for paving applications by modifying binder properties. Property modification depends on both material variables and interaction variables. Crumb rubber is produced with different physical properties from different sources, introducing the potential for material differences. The interaction variables of time and temperature are known to have effects on binder properties, but the nature of the interaction process has not been well documented. There is a need to characterize the interaction variables and to examine the differences in performance-related properties that result from changes in these variables.

CRM binders have been tested for their physical properties using a variety of standard as well as non-standard test procedures. However, tests which rely on empirical parameters indicate modifications to binder properties but do not address specific distress modes or measure fundamental properties that affect long-term pavement performance. CRM binders need to be tested against performance-based specifications to determine the effects that the addition of crumb rubber and subsequent asphalt-rubber interactions will have on pavement performance.

1.5 Study Objectives and Scope

The objective of this research was to develop performance specifications for CRM binders that will comply with current Superpave specifications. This study will document the specific steps required to produce terminal blended Superpave binders modified by addition of crumb rubber. A secondary goal of this research is to improve binder performance-related properties and to increase mix workability through the addition of virgin polymers or through further processing of the asphalt binder. The study will investigate asphalt-rubber interaction properties using fundamental material properties and Superpave performance-related testing during binder production steps. The study will:

- Investigate the effectiveness of different materials and interaction variables on the binder performance-related parameters when tested using Superpave procedures.
- Examine the interaction process to determine the possibility of producing CRM binders with specific performance-related properties. This will be done through monitoring two types of interactions:
    - Short-term reactivity that develops immediately after mixing asphalt with rubber which models on-line binder production.
    - Intermediate-term interactions that may extend for several hours, similar to an

intermediate storage period.

Characterization of the binder production process will provide a more fundamental understanding of the interaction process as well as optimize the performance characteristics of CRM binders.

This research will also develop suggestions for the Nebraska Department of Roads (NDOR) on adopting Superpave specifications for CRM binders. The study will evaluate physical property progressions during the interaction of asphalt and crumb rubber and will quantify the material variables commonly used in the production of CRM binders produced for HMA pavement applications. Property progression will be evaluated using the Superpave (performance based) testing methods. The interaction process variables considered are time and temperature. The main properties of crumb rubber (CR) to be considered are: rubber sources, polymer source and content, CR concentration, particle size and surface area. The effect of asphalt cement properties on the developed properties of the mixture will be demonstrated, and variations in the developed properties resulting from different binder sources and grade changes will be illustrated. Chemical composition of crumb rubber will not be considered. The effect of asphalt cement properties on the developed binder properties are beyond the scope of this study since obtaining relevant information would involve researching the chemical properties of both asphalt and rubber.

1.6 Expected Benefits

The binders developed through this study should be capable of being used for both surface applications and for hot-mix-asphalt (HMA) pavement. These binders will be characterized using performance grade specifications, eliminating the engineering concerns regarding the use of CRM asphalt as a Superpave mix. No special testing equipment (other than the Superpave equipment already being used) will be required when performing QA/QC tests on CRM HMA mixtures.

This research will promote recycling tire rubber for HMA applications in a manner that is environmentally approved. Research findings will improve current binder specifications and the material selection process for CRM asphalt applications. Findings will provide the information necessary to construct pavement sections that perform better and last longer while disposing of scrap tires as part of the process. The proposed research will evaluate locally available rubber sources for use as raw material within the newly developed specifications. This research will extend the service life of, improve the durability of and reduce damage to CRM asphalt pavement across the State of Nebraska.

The new specifications will allow local suppliers and contractors to produce terminal blended CRM binders for Superpave applications, which should significantly lower the cost of specifying CRM binders for NDOR projects. This study will develop and

document procedures for production of one or more terminal blend CRM binders that will enhance the performance and extend the service life of many asphalt pavements.

1.7 Report Outline

Chapter 2 reviews literature on the production, control, testing, and specifications of CRM binders. This review includes some historical aspects on the use of CRM in HMA pavement applications, factors affecting binder production, testing CRM binders using traditional testing methods and performance-based testing of CRM binders.

Chapter 3 presents the research considerations and procedures used in this study. It outlines material selection, procedures for the interaction experiments plus testing techniques and equipment.

Chapter 4 presents the results of this study and discusses the effects of interaction factors and different material factors on the performance-related properties of CRM asphalt.

Chapter 5 focuses on conclusions and recommendations and includes some suggestions for performance-related specifications pertaining to CRM asphalt production.

CHAPTER TWO

PRODUCTION, TESTING AND CONTROL OF CRM BINDERS FOR ASPHALT APPLICATIONS

2.1 Introduction

This chapter outlines some of the more important aspects of CRM binder testing and control procedures for HMA pavement applications including Superpave (4, 6). The information presented covers different aspects and considerations for laboratory characterization of binder properties to obtain improved HMA pavement performance, with a focus on the advantages of using Superpave tests and procedures. Reports on the field performance of CRM binders are included only when they relate to the engineering properties being studied in the lab.

2.2 The Dry Process

The Federal Highway Administration (FHWA) describes crumb rubber modifiers (CRM) as "a scrap tire rubber which has been processed by ambient grinding or granulating methods, reducing the rubber to particles which generally pass the 4.75-millimeter (No. 4) sieve (7, 8). The CRM may be obtained from any combination of tire sources." The FHWA defines asphalt rubber (AR) as "asphalt cement modified with crumb rubber modifier." The production of AR can be through a wet process, defined as "any method that blends crumb rubber modifier with the asphalt cement prior to incorporating the binder in the asphalt paving project." The result is a modified binder having significantly different properties than the original asphalt cement.

Another method for incorporating crumb rubber (CR) into asphalt pavements is the "dry process." The dry process was originally developed in Sweden under the trade name Rubit and subsequently registered in the U.S. under the trade name Plusride (9, 10). It differs from the wet process in that the crumb rubber is mixed with the aggregate before the asphalt binder is added. The dry process requires special aggregate gradation in order to avoid interaction of rubber crumb with the aggregate, which can lead to premature stripping (1). In addition, the dry process typically calls for 1.5 to 3% more liquid asphalt than conventional hot mix (1, 11, 12). The increased quantity of asphalt is needed to reach a void content below 3% to prevent premature raveling of the pavement. The dry process uses two to four times the quantity of binder the wet process would use to produce a similar HMA (2).

The dry process has thus far been primarily limited to use in HMA applications. Only recently has tire rubber been considered for hot mix asphalt as a binder modifier. A survey (2) found that currently thirty-eight states are using scrap tire rubber in their

pavements. About ten percent of these states use the dry process to incorporate tire rubber into HMA pavement (2).

Much of the previous research on crumb rubber in HMA applications has been directed toward mix design considerations. The approach followed was to modify the mix design procedures to account for the differing physical properties of AR. Design procedures for AR mixtures have normally included gradation limitations based on the nature of the binder, particularly with respect to the aggregate gradations most suitable for AR mixtures (11, 12, 13).

Since initial research on the wet process in the late 1960s, many variations have been developed. These variations result from differences in materials used and/or changes to interaction variables designed to modify binders for specific uses. Since the bulk of the early work on rubberized binder applications was for surface treatments and interlayer applications, most of the research on CRM binders has been focused there. Until the late 1980s, few states had considered using CRM binders in HMA mixtures. Arizona and California have been the primary users during the past fifteen years. The bulk of lab research and field evaluation of CRM material has been done in these two states, where research has been conducted on both the wet and the dry processes.

2.3 The Wet Process

C.H. McDonald, a materials engineer in the roads department for the city of Phoenix, Arizona, pioneered the development of rubber modified asphalt binders that evolved into what is known as the “wet process” (7). Swollen crumb rubber particles occupy more volume than dry particles. Expanded rubber particles fill the voids between aggregate and tend to minimize potential aggregate contact (1), so the wet process requires adjustment of the binder content. The wet process requires higher binder content than unmodified asphalt cement because the swelling rubber particles must be recoated with binder as they increase in size.

When using the "wet process," 14% - 20% (by weight) ground tire rubber (# 8 - # 20 mesh) is allowed to interact with asphalt at elevated temperature. The reaction, according to Heitzman, is not chemical in nature (1). The reaction involves absorption of aromatic oils from the asphalt cement into the polymer chains, which are the key components of the CRM asphalt. Heitzman reported that the reaction does not result from melting of the crumb rubber into the asphalt cement. Rather, rubber particles are swollen by absorption of the asphalt's oily phase at high temperatures, 160 °C to 200 °C, to form a gel-like material. The change in rubber particle sizes and formation of gel structures results in a reduction in the inter-particle distance and produces a modified gel which produces a viscosity increase of up to a factor of ten (1, 14, 15).

Rubber swells in a time and temperature dependent manner. When rubber swells after being mixed with asphalt, if the temperature is too high or for maintained for too long, the swelling will continue to the point where the rubber disperses into the asphalt as the rubber depolymerizes (1, 16). This causes a gradual reduction in viscosity, an undesirable occurrence. This gradual change in the viscosity of the AR has been used to indicate the progress of the interaction between asphalt and rubber. Chehovits reported that higher interaction temperature might result in greater swelling and a greater increase in viscosity. An aromatic kerosene fraction is often added to increase the fluidity of the gel-like structure. This "asphalt-rubber" is then mixed with aggregate to form HMA. The process requires the use of at least twenty percent more liquid asphalt than is used in a conventional hot-mix pavement (12). In some cases, forty to sixty percent more asphalt is used in the mixture, which imparts a significant increase in cost. Because of the potentially higher initial cost of HMA produced using the wet process, the wet process has been primarily used for control of reflective cracking. Asphalt-rubber has found applications in stress-absorbing membranes, as inter-layers, as crack and joint sealers, and, to a lesser degree, as a binder in thin asphalt overlays.

The use of asphalt-rubber raises engineering concerns as well as environmental concerns with regard to material suitability for hot mix paving applications. In addition to the performance/cost issues, other engineering concerns exist because of the adjustments necessitated by the gel nature of the CRM binders. CRM binders have shown higher variability when tested for conventional material properties (17, 18). This higher variability could limit the benefits gained by property modification. Environmental concerns result from the higher heating temperatures, which may produce harmful gases and the need to add aromatic oils to decrease viscosity (19). The cost of rubber modified hot-mix pavements is currently 60-150% above the cost of a conventional asphalt pavement. This coupled with uncertainty about the performance of CRM mixtures has deterred many transportation agencies from adopting this technology (20).

2.4 Traditional Tests for Properties of Asphalt Binders

The use of traditional test methods in monitoring the production of asphalt binders was found by SHRP (Superpave) to be inappropriate, as these tests lack sensitivity and do not measure properties related to pavement performance. The Superpave research program developed tests to measure the performance of binders using properties that are more fundamental in nature. Relationships developed by Superpave could be applied to CRM binders. The next section consists of a brief literature review on Superpave testing and parameters. It identifies performance-related parameters of binders that need to be monitored during binder production to identify and control the variables that affect binder properties from a pavement performance standpoint.

2.4.1 Performance-Related Properties

Research studies (6, 21) indicate that the fundamental rheological properties of asphalt binders can be correlated with their behavior in asphalt concrete mixtures. Literature (21, 22, 23, 24) lists the properties of special importance for paving applications, along with the fundamental response parameters, as:

- Rigidity: Total resistance to deformation. Higher rigidity is favored at higher temperature to resist rutting. Lower rigidity is favored at intermediate and low temperatures to resist fatigue and thermal cracking.

- Elasticity: Recovery of deformation using stored energy. Higher elasticity is favored to resist both rutting and fatigue. Less elasticity and greater stress relaxation is favored to resist thermal cracking.

- Brittleness: Failure at low strains. Enhanced strain tolerance, or ductility, is favored to improve fatigue and thermal cracking.

2.4.2 **Rheological Tests**

The significance of Superpave testing in characterizing binder performance is that rational rheological procedures are used in testing binder properties. Response to loading is expressed in terms of fundamental properties such as stress, strain, etc. This allows examination of relationships between pavement performance and binder properties. Superpave testing provides the ability to evaluate time-temperature dependency with regard to measured rheological properties (21, 22), because these procedures measure binder behavior at different temperatures.

2.4.3 **Fundamental Binder Properties Indicative of Material Structure**

Superpave research defines complex shear modulus, G^*, as the ratio of total shear stress to total shear strain. The time between applied stress and resulting strain is related to the phase angle, δ. As loading time or temperature changes, the values of both G^* and δ change. The rate of change is dependent upon material composition. The value of both G^* and δ are considered indications of the development of a network within the binder structure. Saylak et al. (25) indicate that the values of the elastic component have a direct relation to the degree of cross-linking of the material, which in turn gives the material its elastic characteristics. If the material ages or experiences changes in its cross-link density, this will result in a change in the magnitude of the elastic component. The values of the viscous component reflect changes in the material chain structure (25). Since both types of activity can be

occurring simultaneously during processing, the change in magnitude of the phase angle during material processing can be an indication of the primary mechanism involved. Hence, changes in δ represent a convenient parameter for monitoring the binder polymeric structure. The phase angle is used in this study to monitor progression of the asphalt-rubber interaction.

2.5 **CRM Binder Production**

One concern with using crumb rubber in HMA is the method and degree of control required when producing CRM binders. As both materials and process variables affect binder properties, a higher degree of quality control is necessary to produce CRM binders. Evaluating CRM binders using traditional testing methods has pointed out some factors affecting binder production, including quality control testing (11, 12, 13, 14). As noted earlier, it is desirable that quality control testing use the same procedures as performance testing. CRM binders can be produced using many different combinations of material and interaction variables. Each combination provides a different set of performance-related properties that may or may not be measured by traditional testing methods.

2.5.1 **Material Variables**

Material variables include rubber and asphalt cement chemical compositions, rubber morphology and gradations, asphalt grades and chemical composition of additives.

2.5.1.1 Asphalt Cements

Research on the effects of asphalt cement properties on CRM binder property modification is limited. Literature shows specific asphalt cement properties can significantly affect CRM binder conventional properties. While it has been reported that specific asphalts and rubbers may not be compatible, no research efforts have yet identified the key chemical components that affect compatibility (1, 14, 16). Limited literature was found about the effect of asphalt grade on the interaction process. Aromatic content is considered to be a factor that affects asphalt-rubber interaction. Bouldin, et al. (26) indicated that softer asphalts would be more compatible with rubber polymers and crumb rubber modification would be more effective than with stiffer asphalts. Green and Tolonen (27) listed asphalt viscosity as a factor that affects the time required for rubber particles to swell.

Hansen et al. (28) concluded that asphalt cement sources had little or no effect on the way the rubber reacted with asphalt. However, in another study by Western Research Institute (WRI), when crumb rubber reacted with different Superpave core asphalts, the opposite conclusion was reached (29). WRI found that asphalt source controlled the CRM binder properties and had significant effect on the way asphalt-rubber interacted at different temperatures (29).

In a study completed at the Texas Transportation Institute (TTI), asphalt cement properties did affect the binder improvement (30). Properties measured were creep stiffness, S(t) and logarithmic creep rate, m(t) using the Bending Beam Rheometer (BBR). The TTI study showed the binder creep stiffness decreased with time and the logarithmic creep rate increased with time, both property improvements. For asphalt cements with poor low temperature properties, increased interaction time caused further improvement at a constant rate. For asphalts with good low temperature properties, after improvement during the first hour, the interaction time had a negligible effect on creep stiffness.

2.5.1.2 Crumb Rubber Properties

Crumb rubber (CR) is usually created from generic tires from different sources with different compositions (31). In addition, CR has been produced with a variety of physical properties. Both the chemical and physical properties of CR affect binder properties (32). Each method of producing CR generates a unique particle with specific characteristics (33). Each production process affects the particle surface properties differently (32, 33, 34). From a specific grinding process, several different types of ground rubber are often produced based on particle size distribution and tire source. Three main processing methods are currently available for mass production of CR. These include (33):

- Ambient Grinding Process: All grinding takes place at ambient temperature, resulting in material gradation of 1/4 inch to # 40 mesh. The actual particle reduction is accomplished by tearing or ripping action. This creates a particle with a rough porous surface, often described as spongy.

- Cryogenic Grinding Process: The scrap tire rubber is cooled to its embrittlement point using liquid nitrogen. This causes the rubber to be very brittle and is easily fractured in a hammer mill. The resulting surface is very "clean faced or glass like" resulting in less particle surface area than ambient ground materials. The resulting gradation is 1/4 inch to # 60 mesh.

- Wet Grinding Process: The rubber granules are made into wet slurry and passed between grinding stones producing a finer particle (# 20 mesh to # 325 mesh) with relatively higher surface area.

Different rubber sources have significantly different chemical contents. The following categories are the most common types of rubber sources:

- Automotive tire rubber originates from two main sources, whole tire rubber, from passenger and truck tires, and tread tire rubber from passenger, truck or bus treads. Tread rubber is stiffer than sidewall rubber (33). Tread rubber is a more uniform product than whole tire rubber (11, 33)

- Non-automotive tire rubber originates from off-road tire rubber (heavy equipment and airplane tires)

CR properties have been reported to affect conventional binder properties. Oliver (32) found that natural rubber tends to be superior to synthetic rubber for elastic properties and that synthetic rubber is more stable than natural rubber with regard to the interaction conditions of time and temperature. Earlier studies reported that truck tires are considered rich in natural rubber, while passenger tires are rich in synthetic rubber (32). Recent studies and reports show the difference between truck tire rubber and passenger tires has been reduced (1). In the study by Western Research Institute (29) it was concluded that CR source has only a minor effect on the binder properties when tested using Superpave procedures.

CR physical properties, including characteristics such as size, gradation and morphology, influence the interaction with asphalt. Oliver found rubber surface morphology to be the most important factor affecting elastic properties (32). The rougher the rubber particle surface, the higher the surface area and the higher the elastic recovery of the modified binder. Data show that CR produced by the cryogenic process with a smooth particle surface is not as reactive (with asphalt cement) as rough particles produced by the ambient process (32). Rubber sources affect CR surface properties even when produced using the same production process. Oliver (32) showed that the same grinding process produces different surface properties when applied to rubber from different sources.

Literature shows contradiction in defining the effect of CR particle size on the binder properties. Oliver (32) concluded that elastic recovery of the asphalt-rubber binders tends to increase as the rubber particle size decreases. Chehovits et al. (18) showed that coarser particles create a mix that is more sensitive to rubber concentration or asphalt grade. Frobel et al. concluded that finer rubber particles result in higher ductility than larger particles, and that toughness increases as particle size decreases (35). Lalwani reported that toughness increased as particle size decreased and that particle size had no effect on elastic recovery (36).

Although not a material property, CR concentration has a significant effect on binder behavior. Hanson et al. (28) characterized the effect of CR concentration and gradations on the binder properties using the Superpave technology. Rubber concentration was found to have the largest effect on the final properties

of the binder. This conclusion regarding the effect of rubber concentration was confirmed by other studies (16, 18).

CR variables such as particle size gradation were apparently less significant. Common CR gradations range from #16 to #120 mesh. There were no clear trends in the data regarding differences in reaction rates for different gradations of rubber. CR gradations used in this study were not uniform between sources. Each CR source had a gradation composed of different particle sizes, which makes it difficult to draw conclusions about the effects of a gradation on binder properties. In a study by Texas Transportation Institute (TTI), finer rubber was more effective than coarser rubber with regard to modifying the low temperature properties (30). TTI report theorized that the increased surface area per volume or weight of finer particles enhances the ability of the particles to be swollen by, and thus bond with, the binder. Hui et al. (37) tested CRM binders at -20 °C, measuring the fracture toughness of unaged binder. CRM binders created from finer rubber (#80 mesh size) yielded performance superior to those created using coarser rubber (#20 mesh size).

Particle size controls the swelling mechanism over time and affects the binder matrix. Buckly and Berger (38) showed that the time required for swelling increases with the particle radius squared. Larger particle sizes require much greater times to swell. Finer particle sizes may require almost no time to react; an # 80 mesh mean particle size requires about one minute to react with an AC-30 grade asphalt at 163 °C (19). Experiments on binders made with finely ground rubber indicate faster property modification than coarser rubber. For the same specific interaction conditions, less fine rubber, as compared to coarser rubber, is required to achieve the same degree of property modification (37).

2.5.2 Interaction Mechanism

During interaction with asphalt, rubber particles swell to two to three times their original volume, causing an increase in viscosity compared to asphalt without rubber or asphalt with unswollen rubber early in the reaction process. The nature of this interaction is not fully understood. Recently, Bahia (39) interacted asphalt with rubber and stated that "It is clear from this study and many previous studies that the research community does not fully understand the mechanism by which the interaction between these two materials takes place." Two main types of activities that affect binder properties have been reported in the literature: particle swelling and detachment or depolymerization. These activities occur as the binder is subjected to different combinations of time and temperature.

Heitzman reported the interaction is a non-chemical reaction that does not result from melting of the CR into the asphalt cement (1). Rather, rubber particles swell by absorbing some of the asphalt's aromatic oils to form a gel-like material. The swelling of the rubber particles results in less free space between the swollen rubber particles and so the binder viscosity increases. Green and Tolonen (27)

emphasize the importance of controlling the swelling process through controlling the interaction time and temperature. They concluded that rubber particles absorb the lighter fractions of the “maltene phase” of the asphalt, so the viscosity of the “continuous phase” of the binder increases. They also concluded that the swelling process may continue at a reduced rate even at ambient temperature. The absorption of the light fractions of asphalt not only increases the effective volume of rubber particles, but also changes the nature of the asphalt liquid phase.

Bahia and Davies showed that the interaction between asphalt and rubber is not what they call inert (15). They claim the increase in binder viscosity cannot be accounted for only by the existence of the rubber swelling particles. They examined theories commonly used for particulate-filled composite materials to calculate the increase in viscosity of CRM binders. These theories underestimate the increase in viscosity by a large margin. They concluded that there has to be some type of interaction phenomenon that not only increases the effective volume of the rubber particles but also changes the nature of the liquid phase.

Chehovits et al. (18) postulated the interaction mechanism was a result of both chemical and physical interactions. Stroup-Gardener and Shuler (14, 40) indicated that component exchange occurs during the asphalt-rubber interaction. Not fully substantiated, Huffman (41) hypothesized that chemically, under the effect of temperature or time or both, the asphalt and rubber particles participate in an exchange of components. Under this concept, both time and temperature must be carefully controlled to minimize exchange of components from the rubber to the asphalt. Time and temperature control are critical to keep the rubber near its maximum possible volume so that the binder will be stiffer.

A key element in understanding the interaction process is the effect of temperature on the swelling activity. Green and Tolonen (27) considered a concept for the change in free energy and concluded that temperature has two effects on the interaction process. The first effect is on the rate of swelling of rubber particles. As the temperature increases, from 160 °C to 200 °C, the rate of swelling increases. The second effect is on the extent of swelling. As the temperature increases, the extent of swelling decreases as the rubber network becomes stiffer to achieve equivalent change in entropy. They explained that should experimental data show an increase in swelling with temperature, it would indicate that some other reaction is taking place, which they call detachment. The extent of swelling as discussed in the reference is defined as the maximum possible swelling.

When verifying their theory, swelling was larger at 191° C than that at 135° C (27). Green and Tolonen explained that the material network is loosening up as rubber detaches from carbon black particles. The effect of interaction conditions on rubber networking was also reported by Crane and Kay (42), who have shown

that aromatics can cause depolymerization of SBR polymers in twelve to twenty-four hours at 250° C to 275° C.

The binder network alters as the swelling extent changes. As rubber particles swell, a composite matrix of asphalt cement and swollen rubber particles is developed. Because crumb rubber particles are cross-linked and do not totally dissolve in asphalt, different particle sizes and shapes form different matrix structures. This leads to different rheological properties. However, there is no indication in the literature of how rheological properties change during this swelling stage compared to other stages.

Post-vulcanization (27) is an interesting phenomenon that sometimes occurs when mixing asphalt cement with rubber. During rubber processing there are sulfur and other agents that have not been entirely chemically bonded during vulcanization of the rubber. When mixing rubber with hot asphalt, the process of vulcanization will be reactivated and continue for some time, depending on the interaction temperature. This extra time is longer for lower interaction temperatures than for higher interaction temperatures. This extra time was reported as thirty minutes at 150 °C (27). Vulcanization delays the development of modified binder properties. This phenomenon should be suspected when CRM interacts with asphalt for short interaction periods and targeted properties are not achieved.

2.6 Effect of Interaction Conditions on Binder Properties

Oliver (32) examined the general trend of the progression of elastic strain recovery as a function of both time and temperature. A combined effect of both time and temperature was noted, with minimum elastic recovery value developed at maximum time and maximum temperature, two hours and 240° C, respectively. Oliver also found an effect of rubber morphology and sources with both time and temperature. Morphology, as determined by the bulk density test, was the main factor affecting the asphalt-rubber interaction. Bulk density relates to particle surface area and surface area affects the rate of swelling. Lower bulk density results in greater surface area, which yields faster propagation. The interaction conditions of time and temperature were more important with natural rubber sources than with the more stable synthetic rubber sources.

Lalwani (36) controlled asphalt-rubber binder elastic properties using non-standard testing through interaction temperature. In general, binders produced at relatively lower temperatures were far less variable than binders produced at higher temperatures. Binder elasticity was drastically reduced (by as much as three times) when temperature was increased from 200° C to 300° C, while no significant differences occurred due to changing temperature from 150° C to 200° C. In all cases, rubber concentration was the main controlling factor, particularly in reducing temperature sensitivity. Jimenez (43) reported that AR ductility was not affected by interaction temperature.

Chehovits (16) conducted standard and modified tests on CRM binders interacted at 350° F and reported that CRM binders generally maintain their physical properties for at least twenty-four hours at 350° F (177° C). At higher temperatures, CRM binders begin to depolymerize within three to six hours to such an extent that physical properties were affected. Time effects on binder properties vary depending on the rubber source. Synthetic rubber sources produce more stable CRM binders. Viscosity testing shows consistent increase in values with time, up to a maximum of six hours. Values at twenty-four hours were generally lower than at six hours. As interaction temperature decreased to 300° F (149° C), viscosities at twenty-four hours were higher than those at six hours, indicating continuing interaction at lower temperature. Data shows that twenty-four hours of heating at 300° F did not achieve equivalent properties to ninety minutes of interaction at 350° F. Higher viscosities, penetration, resilience, softening points and ductility testing were measured as temperature increased from 300° F to 350° F.

Hanson et al. (28) reported that the development of the complex modulus, G*, would be similar to changes in binder viscosity when interacted at 177° C. In this study, the complex modulus, at higher interaction temperatures, rose for three to five hours, then stabilized or dropped slightly. The development of creep stiffness and logarithmic creep rate was different in the study completed at TTI (30), as property development continued for significantly longer time. Binder modifications continued for forty-eight hours under a nitrogen blanket. Although the TTI study did not cover a full scale of interaction process variables, it pointed out that CRM binder low temperature properties, creep stiffness and logarithmic creep rate, could be controlled by controlling the material and/or the process variables.

2.7 Traditional Testing Methods for CRM Binders

Testing used to characterize CRM binders has traditionally relied upon both standard and non-standard procedures (14, 16, 18, 27, 32, 39, 44). In some reports where standard tests were used, the data do not show trends resulting from the effects of the interaction variables (time and temperature) on binder properties (14, 16, 18). Results were not consistent when different tests were used, and sometimes contradictory results were obtained. In most cases, standard testing procedures including penetration, ductility, etc. have limited application and produce unreliable conclusions (16, 18). Ductility testing was not able to detect a significant change in the binder property at a 300° F interaction even after twenty-four hours. Penetration test results do not indicate significant changes in the binder properties over time. Traditional testing methods have other limitations, even when applied to conventional binders. These limitations include incorrect testing temperatures, non-applicable testing parameters and questionable data extrapolation (21, 22). The unique behavior of asphalt binders as viscoelastic materials is dependent on their response to loading time and

testing temperature. For any combination of time and temperature, viscoelastic behavior must be characterized by at least two properties, the total resistance to deformation and the relative distribution of that resistance between an elastic and a viscous component.

Penetration, softening point and ductility measured at various temperatures and critical limits have been used to indicate the level at which distresses in the pavement are expected. These are empirical parameters which cannot be expressed in engineering units. These properties are not directly related to a rheological property of the binder. These values do not give an indication of the relative distribution of binder response between elastic or viscous components, nor do they consider the loading rate dependency of a binder. These measurements are independent in the sense that they use different loading modes, different loading rates, and different temperatures; they cannot be combined to estimate fundamental rheological properties.

As the use of modified binders became more widespread, altered test procedures were developed to better characterize their properties. Elastic recovery and force ductility tests were developed specifically for modified binders. These tests are better able to indicate the effects of modifications on binder properties, but they still measure empirical properties. These modified testing procedures do not consider fundamental material properties in characterizing binders. They cannot be used to reliably relate the test results to pavement performance (22). In a Superpave report on characterizing modified binders (23), the conclusion was drawn that there are so many problems associated with the traditional methods of characterizing binders that it is difficult to establish relationships and trends associated with the use of modified testing procedures.

2.8 Superpave Testing Procedures for CRM Binders

The nature of CRM binders with swollen rubber particles limits the direct applicability of Superpave testing procedures on these binders. Superpave specifications set the gap opening on the dynamic shear rheometer test to 1.0 mm for high temperature range testing and to 2.0 mm for intermediate range temperature testing. The gap setting with regard to particle size must be set so that testing parameters are not affected by the non-homogenous nature of binder containing rubber particles. This has been a concern among researchers (39, 45). In an official response from the Federal Highway Administration, CRM binders with particle sizes passing sieve # 60 (250 mm) sieve would be eligible for all Superpave specification testing (46). The gap setting could be changed to allow for testing and characterizing binders with larger particle sizes. The gap setting, in mm, must be at least four times the maximum particle size.

The suitability of Superpave aging processes on CRM binders with high rubber concentration or coarse rubber particles is another issue. Both the Rolling Thin-

Film Oven Test (RTFOT) and the Pressure Aging Vessel (PAV) test include elevated temperature for a specified time. Temperature is a main variable in the asphalt-rubber interaction process. The effect of the Superpave aging processes on modified binder properties has not been well documented (47, 48). In a study on asphalt polymer modifiers, it was reported that the modifications produced by a polymer modifier became less significant after the RTFOT and the PAV aging processes (49). Limited literature was found on the applicability of the RTFOT or the PAV on CRM binders. One study by McGennis (50) tested the applicability of Superpave testing on CRM binders. The behavior of CRM binders during the RTFOT aging was unlike that of a polymer-modified binder. The CRM binders tended to veil across the RTFOT bottle during aging, which rendered the results invalid.

2.9 Crumb Rubber Compared to Other Binder Modifiers

The mechanism by which CR changes binder properties is different from polymer modifiers. Polymer modifiers completely disperse in the asphalt and cause changes in its molecular structure. CR keeps its physical shape and behaves as flexible particulate filler between binder particles. Hence, CR binders are non-homogeneous in nature. Polymer modification results in a more homogeneous binder.

Oliver (32) performed a limited experiment to compare the effectiveness of CRM for modification of both high temperature and low temperature properties and concluded that there was no significant change in the phase angle or the complex shear modulus at low temperature. Bahia (22) reported that Superpave test parameters are sensitive to the effect of binder modifiers, and that certain modifiers can alter certain binder properties. For commonly used polymers such as Styrene-Butadiene (SB), Bahia (22) investigated the modification effects on Superpave parameters and reported that the main change is in rigidity, at both high and low temperature, while only secondary effects are produced in elasticity, expressed by δ. Thus, SB modifications will have a minor effect on the rate of stress relaxation and energy dissipation, as they are mainly functions of δ. Bahia (22) reported similar changes in G^* and δ when using crumb rubber to those reported with SB polymers and stated that "the relative changes of either parameter are of the same order of magnitude as for the SB polymer modification." G^* increased at low frequency (high temperature) and decreased at high frequency (low temperature). The δ values were lower at low frequencies but higher at high frequencies. The effects of SB and CRM modification on the binder can be described as mainly changes in rigidity.

2.10 Pavement Performance Using CRM Binders

There have been contradictions regarding the performance of asphalt concrete made with CRM binders, both in lab tests and in actual field performance. CRM mixes have performed very well in warmer climates. Studies from Arizona and

California show CRM mixes outperforming more conventional asphalt mixes in those states. Kurtz and Stroup-Gardener showed that mixes made with CRM binders are more resistant to rutting when compared to control mixes (51). Hoyt et al. (52) conducted laboratory studies on mixes made with CRM binders and compared these to more conventional mixes at the Texas Transportation Institute. Testing included resilient modulus, fatigue and fracture properties, creep compliance, and rutting. In all of these tests, asphalt-rubber mixes were superior to more conventional asphalt mixes, and in most cases were more cost effective. This was not the case in a study by Maupin, who concluded, based on limited testing, that mixtures containing asphalt-rubber binders are less resistant to permanent deformation than regular mixtures (53). Identical CRM binders were not used in all of these tests. Differences in materials, gradation and/or interaction conditions almost certainly existed and caused some of the discrepancies.

In Michigan, mixes made with both the wet and the dry processes were evaluated against control mixes (54). The control sections had higher tensile strength, higher resilient modulus and lower tensile strain at failure than both the dry and the wet process sections. Low rubber content mixes showed higher tensile strength and modulus than higher rubber content mixes. It was also concluded that rubber sections experienced less rutting than control sections and that the dry process sections disintegrated sooner and required more frequent patching.

In Washington State, Lundy et al. reported that lab test results on mixes made with CRM binders indicate the expected fatigue life of CRM mixes would exceed that of the neat asphalt mixes at any strain level (55). In the same study by Lundy, CRM mixes showed unacceptable low temperature stability, but no rutting was noticed after three year of service. This observation questions the stability test, along with other standard tests, as valid indicators of the field performance of CRM mixes.

Cost added to the construction process through use of CRM binders has not been documented in the literature. While studies from Arizona and California provide good performance/cost benefit data for warm climate performance, other field trials (56, 57) have been disappointing. Due to lack of published data, little information is available about the effects of CRM binders on pavement field performance in colder regions.

2.11 CRM Binder Specifications

CRM binder specifications have traditionally been based on viscosity measurements. Texas, Arizona and California are leading the effort to establish performance-based specifications for CRM applications. The Rubberized Asphalt Technology Center was formed as a cooperative effort by the County of Los Angeles, County of Sacramento and the California Integrated Waste

Management Board. The center promotes the use of crumb rubber from scrap tires in roadway rehabilitation projects by providing education, training, and consultation services to local agencies. The program is funded by the California Integrated Waste Management Board in an effort to reduce the state's stockpile of scrap tires and help conserve the state's landfills. The three most common applications for rubberized asphalt within California are:

1. As an asphalt rubber hot mix (ARHM) resurfacing over existing asphalt or concrete pavement.
2. As an asphalt rubber aggregate membrane (ARAM).
3. As a rubberized slurry seal.

Most current asphalt-rubber specifications require distinct gradations of the crumb rubber materials. In some cases, heating temperatures are also specified. The California Department of Transportation (CalTrans) has established the asphalt-rubber specifications shown in Table 2.1.

Arizona also uses two different types of CRM binder specifications, traditional specifications with minimum crumb rubber content of 20% and Superpave-based specifications that include minimum requirements (Table 2.2) (27). Florida accepts CRM binders within their Superpave binder specifications for modified asphalt. Texas has developed several different versions of CRM binder specifications depending on application (Table 2.3). Current CRM binder materials specifications in Texas include Superpave testing procedures (Table 2.4).

Table 2.1 California Traditional Asphalt-Rubber Specifications.

ASPHALT-RUBBER BINDER

Test Parameter	ASTM Test Method	Requirement	
		Min.	Max.
Cone Penetration @ 25°C, 1/10 mm	D 217	25	70
Resilience @ 25°C, Percent rebound	D 3407	18	—
Field Softening Point, °C	D 36	52	74
Viscosity @ 190°C, Pa • s (x10^{-3})	See Note	1500	4000

Note: The viscosity test shall be conducted using a hand held Haake Viscometer Model VT-02 with Rotor 1, 24 mm in depth x 53 mm in height, or equivalent, as determined by the Engineer. The accuracy of the viscometer shall be verified by comparing the viscosity results obtained with the hand held viscometer to 3 separate calibration fluids of known viscosities ranging from 1000 to 5000 Pa • s (x10^{-3}). The viscometer will be considered accurate if the values obtained are within 300 Pa • s (x10^{-3}) of the known viscosity. The known viscosity value shall be based on the fluid manufacturers standard test temperature or the test temperature versus viscosity correlation table provided by the fluid manufacturer. Viscometers used on the project shall be verified to be accurate. The test method for determining the viscosity of asphalt-rubber binder using a hand held viscometer is available at the Transportation Laboratory, Pavement Branch, Telephone 916-227-7300. The accuracy verification results shall be provided to the Engineer and shall be certified by a Certificate of Compliance. The Certificate of Compliance shall be furnished to the Engineer in conformance with the provisions in Section 6-1.07, "Certificates of Compliance," of the Standard Specifications.

Table 2.2 Arizona Superpave-Based Asphalt-Rubber Specifications.

Property	Requirement		
	Type 1	**Type 2**	**Type 3**
Grade of base asphalt cement	PG 64-16	PG 58-22	PG 52-28
Rotational Viscosity*: 350 ^{o}F; pascal seconds	1.5 - 4.0	1.5 - 4.0	1.5 - 4.0
Penetration: 39.2 ^{o}F, 200 g, 60 sec. (ASTM D 5); minimum	10	15	25
Softening Point: (ASTM D 36); ^{o}F, minimum	135	130	125
Resilience: 77 ^{o}F (ASTM D 5329); %, minimum	30	25	15

* The viscotester used must be correlated to a Rion (formerly Haake) Model VT-04 viscotester using the No. 1 Rotor. The Rion viscotester rotor, while in the off position, shall be completely immersed in the binder at a temperature from 350 to 355 degrees F for a minimum heat equilibrium period of 60 seconds, and the average viscosity determined from three separate constant readings (± 0.5 pascal seconds) taken within a 30 second time frame with the viscotester level during testing and turned off between readings. Continuous rotation of the rotor may cause thinning of the material immediately in contact with the rotor, resulting in erroneous results.

Table 2.3 Texas DOT Modified Asphalt Cement Specifications.

Polymer-Modified Asphalt Cement

Property	Test Procedure	AC-5 w/2% SBR Min	AC-5 w/2% SBR Max	AC-10 w/2% SBR Min	AC-10 w/2% SBR Max	AC-15P Min	AC-15P Max	AC-20-5TR Min	AC-20-5TR Max
Polymer		SBR		SBR		SBS		TR	
Polymer content, % (solids basis)	Tex-533-C	2.0	–	2.0	–	3.0	–	5.0	–
Dynamic shear, G*/sin δ, 64°C, 10 rad/s, kPa	T 315	–	–	–	–	–	–	1.0	–
Viscosity									
140°F, poise	T 202	700	–	1,300	–	1,500	–	2,000	–
275°F, poise	T 202	–	7.0	–	8.0	–	8.0	–	10.0
Penetration, 77°F, 100 g, 5 sec.	T 49	120	–	80	–	100	150	75	115
Ductility, 5cm/min., 39.2°F, cm	T 51	70	–	60	–	–	–	–	–
Elastic recovery, 50°F, %	Tex-539-C	–	–	–	–	55	–	55	–
Softening point, °F	T 53	–	–	–	–	–	–	120	–
Polymer separation, 48 hr.	Tex-540-C	None		None		None		None	
Flash point, C.O.C., °F	T 48	425	–	425	–	425	–	425	–
Tests on residue from Thin-Film Oven Test:	T 179								
Retained penetration ratio, 77°F	T 49	–	–	–	–	0.60	1.00	0.60	1.00
Tests on residue from RTFOT aging and pressure aging:	Tex-541-C and R 28								
Creep stiffness	T 313								
S, -18°C, MPa		–	–	–	–	–	–	–	300
m-value, -18°C		–	–	–	–	–	–	0.300	–

Table 2.4 Texas DOT Specifications for CRM Materials.

G. Crumb Rubber Modifier. Crumb rubber modifier (CRM) consists of automobile and truck tires processed by ambient temperature grinding.

CRM must be:

- free from contaminants including fabric, metal, and mineral and other nonrubber substances;
- free-flowing; and
- nonfoaming when added to hot asphalt binder.

When tested in accordance with Tex-200-F, Part I, using a 50-g sample, the rubber gradation must meet the requirements of the grades in Table 13.

CRM Gradations

Sieve Size (% Passing)	Grade A		Grade B		Grade C		Grade D	Grade E
	Min	Max	Min	Max	Min	Max		
#8	100	–	–	–	–	–	As shown on the plans	As approved
#10	95	100	100	–	–	–		
#16	–	–	70	100	100	–		
#30	–	–	25	60	90	100		
#40	–	–	–	–	45	100		
#50	0	10	–	–	–	–		
#200	–	–	0	5	–	–		

2.12 Summary

The current stockpile of waste tires is growing by approximately 200 million tires each year, which makes waste tires an issue that will continue to be a problem. One market with a high potential for using more scrap tires is CRM asphalt for pavement. One method of incorporating crumb rubber in asphalt pavement is through mixing crumb rubber with binder before adding aggregate (the wet process). Performance of asphalt pavement constructed with CRM binders is a critical issue that needs to be researched if crumb rubber is to assume a more prominent role in the nation's road system.

Most of the previous research effort has been focused on properties measured by traditional testing procedures. These tests lack the sensitivity required to differentiate the better performing binders and do not detect changes in the binder process variables that affect pavement performance. Most research findings indicate that traditional tests are not suitable for use with CRM binders.

There are many variables affecting CRM binder properties, including material property variables and interaction variables. In this study, the interaction variables of time and temperature were evaluated to determine their effects on binder properties. Changes that occur during the interaction process are very complex. Rubber particle swelling is one reaction that occurs during mixing crumb rubber with asphalt binder. Depending on the interaction time and temperature, other reactions can occur as well. These extent and duration of these reactions influence the binder's performance-related properties.

Testing of rheological properties provides better indicators of changes in the performance-related properties of binders. Superpave has already developed specifications and testing methods for polymer modified asphalt binders, based upon rheological parameters that are performance-related. Rheological properties can be adjusted during binder production to control performance-related properties of the final product. Discovering the magnitude and extent of changes necessary to produce CRM binders with specific performance-related properties is the subject of this research.

Many states are currently developing separate specifications for CRM pavement and surface treatment applications. Not all of these specifications are based on performance testing, but the concept of using performance testing to control binder production is becoming more accepted since many state transportation agencies have adopted Superpave binder specifications for asphalt pavement.

CHAPTER THREE

RESEARCH CONSIDERATIONS AND PROCEDURES

3.1 Introduction

In Chapter 2, a literature search documented some of the variables that can be modified during processing to improve asphalt pavement performance. Investigation of these variables requires a detailed examination of different materials and their properties when processed under precisely controlled conditions representative of binder production. Two main categories of factors will be considered, material properties and interaction conditions. Material properties include asphalt type, rubber source, rubber particle surface properties, and rubber particle sizes. Interaction conditions include interaction time and temperature. This chapter will present three main topics: (1) equipment used in the interaction process, an example is shown in Figure 3.1, (2) materials used to modify the developed binder, an example is shown in Figure 3.2 and (3) the properties of the developed binder, an example is presented in Figure 3.3.

3.2 Equipment

This study required equipment to maintain constant temperature, provide high shear, provide precise control and obtain exact measurements. The equipment used in this research included:

- High Shear Mixer (ROSS Model No. HSM -100LM -2), shown in Figure 3.1
- Dynamic Shear Rheometer (Bohlin Instruments CVO)
- Bending Beam Rheometer (ATS)
- Rolling Thin Film Oven (CS – 325B)
- Pressure Aging Vessel (Model No. PAV 9300)
- Vacuum Oven (Model No. 9900)
- Oven maintained at 325^0F for the cigar tube test
- Oven maintained at 180^0C (356^0F) for stage one of the experimental design
- Oven for general purpose heating
- Heating mantles of different sizes corresponding to the container size (Omega 110B – TM618 and Omega 100B – TM614), shown in Figure 3.1
- Bench-type temperature controller (MCS 2110J – R)
- Temperature Probes (TJC – 36 - 6in, 9 in, 12 in), shown in Figure 3.1
- Fan to aid temperature control
- Freezer

- Lab accessories (metal cans (2 oz., 4 oz. 16 oz.), spoon, spatula, rags, etc……)

Figure 3-1 Ross mixer, Temperature Controller, Mantel and Cans used in Experiments.

Figure 3-2 Firestone polymer Stereon 841A and Entire Recycling CRM.

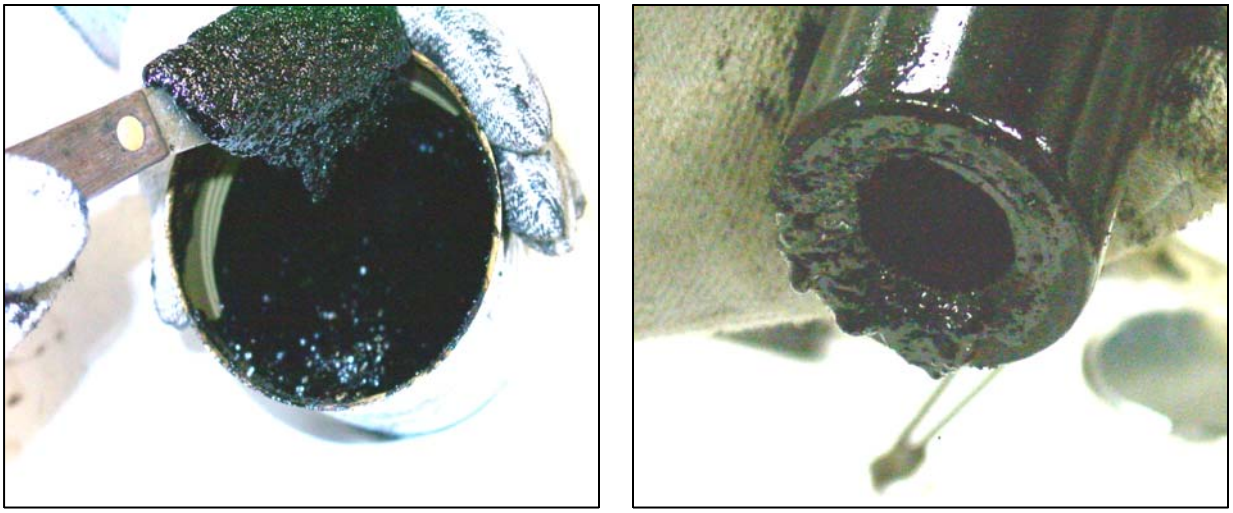


Figure 3-3-a Binder Produced Through Traditional Asphalt-Rubber Interaction.

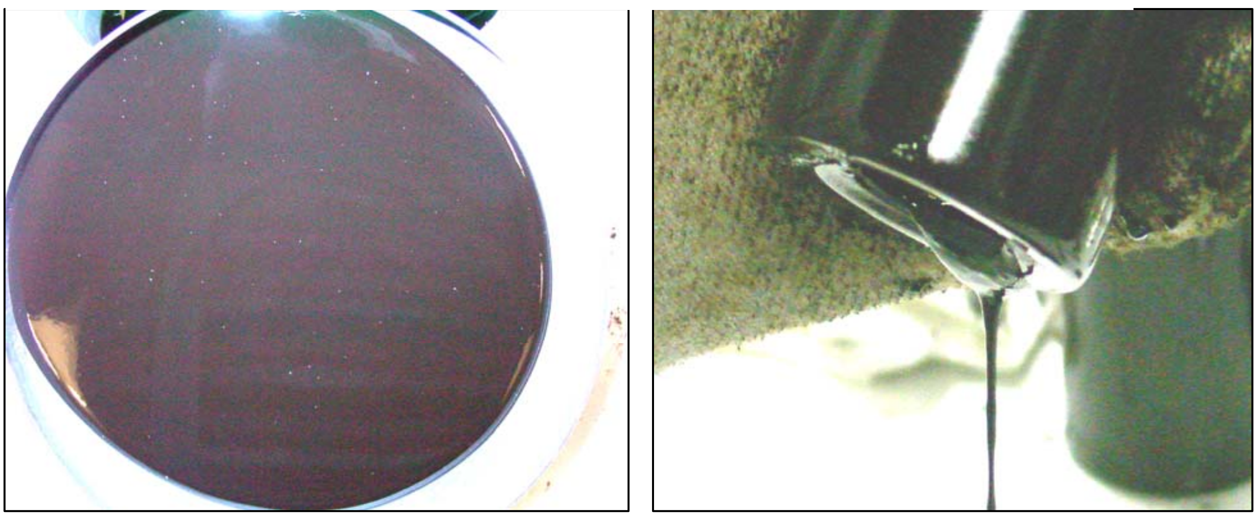


Figure 3-3-b Binder Produced Through the Interactions in This Study.

3.3 Materials Used

Three different performance graded binders were investigated in combination with two different polymers and two local sources of crumbed rubber. Flint Hills (PG 58 – 28) was obtained from Flint Hills Resources of Wichita, KS, Jebro (PG 52 – 34) was obtained from Jebro Incorporated of Sioux City, IA, and Monarch (PG 58 – 28) was obtained from Monarch Oil Incorporated of Omaha, NE. Kraton and Firestone were the two different brands of polymer tested. Kraton is a styrene-butadiene-styrene (SBS) polymer composed of blocks of styrene and butadiene, which was obtained from Kraton Polymers US LLC. The Firestone polymer was Stereon 841A, a high efficiency styrene-butadiene (SB) multi-block thermoplastic elastomer in pellet form, provided by Firestone polymers of Akron,

Ohio. Two different brands of crumb rubber products were tested as part of the research. Jai Tire Industries in Denver, Colorado provided one of the crumb rubber materials. The other crumb rubber was provided by EnTire Recycling Incorporated, which processes its rubber using cryogenic methods. The binders (Flint Hills, Jebro and Monarch) used in this research were first characterized using a standard set of Superpave binder tests. Results of these tests are shown in Table 3.1.

The three asphalt binders were then interacted with various polymer and crumb rubber combinations under various conditions to measure changes in rheological properties. Different methods were used to mix the binder with polymer and crumb rubber material. Binder, crumb rubber and polymer were mixed with a high shear mixer, a low speed mixer or by hand. Interaction variations included the binder being mixed with crumb rubber material only, with polymer only or with a combination of both. The temperature of the sample was kept constant at each stage during the interaction period. The method of heating the material was either on an oven maintained at constant temperature or using a heating mantle connected to a bench-type controller and temperature probe. Samples were taken at various intervals and tested using Dynamic Shear Rheometer (DSR) for G* and sin δ. The final binder product was passed through the Rolling Thin-Film Oven (RTFO) and Pressure Aging Vessel (PAV) to simulate aging and tested for the appropriate distress parameters.

Table 3.1 Results of Superpave Tests on Unmodified Binders.

(3.1a)

Binder Source	Grade	Viscosity Testing – Original Material			
		Viscosity (cPa)	Torque (%)	Shear Stress ($dyne/cm^2$)	Shear Rate (s^{-1})
Flint Hills	58-28	354.2	2.8	24.1	6.8
Monarch	58-28	345.8	2.8	23.5	6.8
Jebro	52-34	245.8	2.0	16.7	6.8

(3.1b)

Binder Source	Grade	D.S.R Testing – Original Material				R.T.F.O.				P.A.V.			
		Temp (°C)	Phase Angle (δ)	Shear Modulus (G*) KPa	G*/sin δ, Kpa	Temp (°C)	Phase Angle (δ)	Shear Modulus (G*) KPa	G*/sin δ, Kpa	Temp (°C)	Phase Angle (δ)	Shear Modulus (G*) KPa	G*/sin δ, Kpa
Flint Hills	58-28	58	86.6	1.604	1.607	58	83.6	3.157	3.177	19	46.5	4472.6	3246.5
										16	43.5	6837.5	4706.3
Monarch	58-28	58	87.3	1.396	1.398	58	85.1	2.901	2.912	19	47.3	5092.4	3741.2
Jebro	52-34	52	87.0	1.209	1.211	52	83.1	2.705	2.725	13	50.3	3140.0	2414.7
										10	47.0	5248.1	3836.1

(3.1c)

Binder Source	Grade	B.B.R. Testing – P.A.V.				
		Temp (°C)	Load (mN)	Deflection (mm)	Measured Stiffness (Mpa)	m-value
Flint Hills	58-28	-18	979.7	0.305	262.0676	0.3313
Monarch	58-28	-18	982.3	0.267	300.3737	0.3103
Jebro	52-34	-24	979.8	0.290	276.7763	0.3242

3.4 Interaction Experiments

3.4.1 Preliminary Experiment

3.4.1.1 Asphalt-Rubber Preliminary Testing

In the first series of experiments, asphalt binder was mixed with crumb rubber only. All three of asphalt binder types were mixed with crumbed rubber materials from Jai Tire and EnTire. Interaction temperature was controlled at 180^0 C (356^0 F). Six hundred grams of asphalt binder material were poured into one-liter cans and mixed with 10% crumb rubber by weight. An oven maintained at constant temperature was used to sustain the interaction temperature. The binder was mixed using a spatula at frequent intervals. Samples were taken at one hour, two hours, and at four hours. The samples were tested using Dynamic Shear Rheometer at 10 radians per second. The four-hour sample was processed through the RTFO and PAV to simulate aging. RTFO and PAV aged materials were then tested using the DSR at corresponding temperatures. Table 3.2 shows the binder cement, rubber material, interaction time, temperature, and method used.

Table 3.2 Binders, Crumb Rubber Sources, Interaction Temperatures, Methods, and Interaction Times.

Asphalt	Crumb Rubber	Interaction Temp (°C)	Interaction Method	Interaction Time
Jebro (52 – 34)	10 % Jai Tire	180 °C	Oven / Spatula	4 hours
	10 % Entire Recycling	180 °C	Oven / Spatula	4 hours
Monarch (58-28)	10 % Jai Tire	180 °C	Oven / Spatula	4 hours
	10 % Entire Recycling	180 °C	Oven / Spatula	4 hours
Flint Hills (58– 28)	10 % Jai Tire	180 °C	Oven / Spatula	4 hours
	10 % Entire Recycling	180 °C	Oven / Spatula	4 hours

3.4.1.2 Asphalt-Polymer Interactions

Next asphalt binder was mixed with three or four percent polymer by weight. The binder for the experiments included Flint Hills (PG 58-28) and Jebro (PG 52-34). Six hundred grams of binder were placed in a one liter can and three percent or four percent (by weight) of the assigned polymer was added. The blend was mixed at high speed (2500 rpm) for forty minutes. The speed was then reduced to 300 rpm and kept constant for six hours. Flint Hills (PG 58-28) was interacted with three percent Kraton and three percent Firestone polymers while Jebro (PG 52-34) was interacted with four percent Kraton and four percent Firestone polymers. Samples were heated with a mantle (Omega Glas - col 100B – TM 614) connected to a bench-type controller (Omega MCS - 2110). The temperature was maintained at 392^0 F (200^0 C). Four experiments were conducted using this procedure, as shown in Table 3.3.

Table 3.3 Polymers Used with Interaction Temperatures, Methods and Time.

Binder	Percent Polymer	Interaction Temp (°C)	Interaction Method	Interaction Time (hrs)
58 - 28 (Flint Hills)	3 % Kraton	200	Mixer	6
	3 % Firestone	200	Mixer	6
52 - 34 (Jebro)	4 % Kraton	200	Mixer	6
	4 % Firestone	200	Mixer	6

The asphalt polymer interaction samples were then processed through the RTFO and PAV to simulate aging. The original and processed material was subsequently tested using the DSR, Bending Beam Rheometer and Rotational Viscometer. The results are shown in Table 3.4.

Table 3.4 Results of Superpave Testing on Polymer Modified Binders.

(3.4a)

Binder Source	**Grade**	**Polymer**	**Viscosity Testing**			
			Original Material			
			Viscosity (cPa)	**Torque (%)**	**Shear Stress (dyne/cm^2)**	**Shear Rate (s^{-1})**
Flint Hills	58-28	3% Firestone	1050.0	8.4	71.4	6.8
		3% Kraton	874.8	7.0	59.5	6.8
Jebro	52-34	4% Firestone	629.0	5.0	42.8	6.8
		4% Kraton	909.3	7.3	61.7	6.8

(3.4b)

Binder Source	**Grade**	**Polymer**	**B.B.R. Testing**				
			P.A.V.				
			Temp (°C)	**Load (MN)**	**Deflection (mm)**	**Measured Stiffness (MPa)**	**m-value**
Flint Hills	58-28	3% Firestone	-18	978.9	0.258	309.515	0.2817
			-12	979.9	0.525	152.417	0.3266
Jebro	52-34	4% Firestone	-24	979.9	0.302	265.130	0.3083
			-18	979.0	0.709	112.676	0.3513
Jebro	52-34	4% Kraton	-24	979.2	0.364	219.484	0.2930
			-18	977.8	0.707	112.843	0.3388

(3.4c)

Binder Source	Grade	Polymer	D.S.R Testing											
			Original Material				R.T.F.O.				P.A.V.			
			Temp (°C)	Phase Angle (δ)	Shear Modulus (G*) KPa	G*/sin δ, Kpa	Temp (°C)	Phase Angle (δ)	Shear Modulus (G*) KPa	G*/sin δ, Kpa	Temp (°C)	Phase Angle (δ)	Shear Modulus (G*) KPa	G*/sin δ, Kpa
Flint Hills	58-28	3% Firestone	58	71.4	8255.200	8.710	58	67.4	1581.5	17.136	19	40.0	5416600	3483.6
			64	73.7	4209.200	4.386	64	69.8	8005.1	8.531	16	37.5	7799000	4750.3
			70	76.3	2229.200	2.295	70	72.5	3920.2	4.110	13	35.0	10993000	6302.4
			76	79.0	1183.600	1.206	76	75.3	2117.6	2.189				
			82	82.1	637.080	0.643								
		3% Kraton	58	75.0	3589.700	3.716	58	69.2	8363.2	8.944	19	42.2	5256700	3533.5
			64	77.1	1862.900	1.911	64	71.6	4307.1	4.539	16	39.6	7792000	4968.8
			70	78.8	9947.900	1.014	70	74.5	2298.5	2.385	13	37.1	11385000	6860.4
			76	79.7	563.470	0.573	76	78.0	1225.9	1.253				
Jebro	52-34	4% Firestone	52	78.9	2513.900	2.562	52	70.7	6754.7	7.157	13	44.1	1982900	1381.1
			58	80.1	1298.300	1.318	58	75.0	3359.9	3.478	10	42.5	3048000	2057.7
			64	80.7	7217.300	0.731	64	78.9	1687.0	1.719	7	40.6	4664400	3033.5
											4	38.8	7022000	4397.4
Jebro	52-34	4% Kraton	52	67.1	4377.000	4.752	52	61.1	9731.6	11.115	13	43.0	1703100	1161.8
			58	68.8	2452.200	2.630	58	62.2	5530.4	6.254	10	41.4	2595600	1715.6
			64	70.4	1434.200	1.522	64	64.0	3153.1	3.508	7	39.9	3927100	2517.2
			70	71.3	850.480	0.898	70	66.2	1873.2	2.047	4	38.5	5821500	3623.7

3.4.2 Asphalt-Polymer Crumb Rubber Interactions

The third stage of this research measured properties of asphalt binder blended with crumb rubber only and with both polymer and crumb rubber. Flint Hills (PG 58-28) binder, EnTire Recycling crumb rubber, and Firestone polymer were the primary raw materials utilized, but a few trials utilized Jebro (PG 58-34) binder, Jai Tire crumb rubber, and Kraton polymer. Either 5% or 7.5% crumb rubber by weight as a percentage of asphalt binder was used, with 2% (or in some cases 3%) polymer by weight. The interactions were conducted in one-half gallon cans and one-gallon cans. Table 3.6 shows the coding assigned to different interaction conditions. A heating mantle (Glos-col 100B – 618) was used to heat the material and was connected to the bench type controller which maintained the temperature. A longer temperature probe (TJC 36 – 12") was required. A high shear mixer (HSM-100LM-2) was used to mix the binder with polymer and crumb rubber. The one-half gallon cans were filled with 1200 grams of asphalt binder while one-gallon cans were filled 1500 grams of binder. The first fourteen interactions used half-gallon cans; all later trials used one-gallon cans. More asphalt material was used in the later interactions to maintain a more stable temperature for crumb rubber asphalt interaction.

Interactions were conducted in different stages with different temperatures and mixing speeds. The first stage involved high speed mixing (30 Hz or 50 Hz) at high temperature with only crumb rubber and binder. A high-speed mixer imparts energy, which increases the temperature of the materials being mixed. At higher mixer speeds, fluctuations in mix temperature were observed. The temperature was allowed to increase only slightly before the controller was shut down. The duration of the first stage was shortened to minimize binder property changes resulting from the increase in temperature.

The second stage involved the mixing of polymer at lower speed and temperature for forty minutes. The third and fourth stages involved shearing at 10 Hz over an extended period. Flint Hills binder was blended with 7.5% crumb rubber from EnTire using two different methods. In the first method the binder and crumb rubber were blended at 25 Hz for two hours at 392^{o} F. The blend was then sheared for an additional six hours at 30 Hz with temperature ranging between 392^{o} and 420^{o} F.

In the second method, the first stage involved mixing at 30 Hz for two hours at 392^{o} – 420^{o} F; mixing continued at 10 Hz for next six hours at 392^{o} F. The binder was blended with 10% EnTire, 7.5% Jai Tire, and 10% Jai Tire crumb rubber in similar fashion. The binder was mixed with maximum possible shear applied continuously during the two-hour period. The mix was then maintained in the temperature range of 392^{o} – 450^{o} F while mixing continued at 10 Hz for six hours.

The next six interactions involved binder being mixed with different percentages of polymer and crumb rubber material. The first stage involved mixing of crumbed rubber at 50 Hz between 330^{o} – 430^{o} F for twenty minutes. In the second stage, the polymer

was mixed at 30 Hz for forty minutes at a temperature between 392° – 402° F. The mix was then continuously sheared at 10 Hz (392° F) for four hours.

Later interactions involved Flint Hills binder, 5% EnTire crumb rubber, and 2% Firestone polymer with different methods of processing. In interaction 13, crumb rubber was added at 50 Hz for forty minutes (at 302° – 414° F). The next interaction involved mixing the crumb rubber at 30 Hz for two hours at 347° F. The second stage for both the interactions was identical with mixing at 25 Hz and 347° F for two hours. Interaction 15

Table 3.5 Code Description.

Position	Data	code	Meaning
1	Asphalt cement source		
		F	Flint Hills (58-28)
		J58	Modified Jebro (58-34)
		J	Jebro (52-34)
2	CRM Source, size and percentage	E10	10 % Entire
		J7	7.5 % Jai Tire
		E5(40-60)	5 % of Entire material passing #40 sieve and retained on #60 sieve
3	(Mixing Speed in the main interaction)	50Hz	50 cycle/sec
4	Mixing Temperature in the main interaction	200C	200°C
5	SBS type and percentage	f2	2% FireStone
		K3	3% Kraton
6	Different Interaction Parameters	A, B or C	See tables in Appendix for exact parameters
Interaction Temperature			temperature between 160° C -175° C
			temperature between 175° C - 200° C
			temperature 220° C
Interaction Time	10 minutes		Tank Material (Unaged)
	original		
	δ	G*	Values measured at 10th minute
	30 Hz - 200°c		Those conditions are up to end of the 10th minute

Table 3.6 Interaction Codes.

Mix No.	Code	Sample
1	F-E7-30H-200C-00-A	Flint Hills + 7.5 % Entire
2	F-E7-30H-200C-00-B	Flint Hills + 7.5 % Entire
3	F-E10-30H-200C-00	Flint Hills + 10 % Entire
4	F-J7-30H-200C-00	Flint Hills + 7.5 % Jai Tire
5	F-J10-30H-200C-00	Flint Hills + 10 % Jai Tire
6	F-E7-40H-200C-00	Flint Hills + 7.5 % Entire
7	F-E5-50H-200C-00-A	Flint Hills + 5 % Entire
8/20	F-E5-50H-200C-f2-A	Flint Hills + 5 % Entire + 2 % Firestone
9/21	F-E5-50H-200C-f3	Flint Hills + 5 % Entire + 3 % Firestone
10	F-E7-50H-200C-f2	Flint Hills + 7.5 % Entire + 2 % Firestone
11	F-E0-50H-200C-f2	Flint Hills + 2% Firestone
12/23	F-E5-50H-200C-K2	Flint Hills + 5 % Entire + 2 % Kraton
13/24	F-E5-50H-200C-f2-B	Flint Hills + 5 % Entire + 2 % Firestone
14/25	F-E5-30H-200C-f2	Flint Hills + 5 % Entire + 2 % Firestone
15	F-E5-50H-200C-00-B	Flint Hills + 5 % Entire
16	J58-E5-50H-160C-00	Jebro 58 - 34 + 5 % Entire
17	J58-E5-30H-175C-00	Jebro 58 - 34 + 5 % Entire
18/26	F-J10-50H-160C-f2	Flint Hills + 5 % Jai Tire+ 2 % Firestone
19	J58-J5-50H-160C-00	Jebro 58 - 34 + 5 % Jai Tire
27	F-E5(40-60)-50H-200C-f2-A	Flint Hills + 5 % Entire(E40-60) + 2 % Firestone
28	F-E5(30-40)-50H-200C-f2-A F-E5(60-80)-50H-200C-f2-A	Flint Hills + 5 % Entire(E30-40) + 2 % Firestone Flint Hills + 5 % Entire (E60-80) + 2 % Firestone
29	F-E5(60-80)-50H-200C-f2-A	Flint Hills + 5 % Entire(E60-80) + 2 % Firestone
30	F-E5(80-200)-50H-200C-f2A	Flint Hills + 5 % Entire(E80-200) + 2 % Firestone
31	F-E5(20-30)-50H-200C-f2	Flint Hills + 5 % Entire(E20-30) + 2 % Firestone
32	F-J5(60-80)-50H-200C-f2	Flint Hills + 5 % Entire(J60-80) + 2 % Firestone
33	F-J5(40-60)-50H-200C-f2	Flint Hills + 5 % Entire(J40-60) + 2 % Firestone
34	F-J5(80-200)-50H-200C-f2	Flint Hills + 5 % Entire(J80-200) + 2 % Firestone
35	F-J5(30-40)-50H-200C-f2	Flint Hills + 5 % Entire(J30-40) + 2 % Firestone
36	J-E5-50H-200C-f2	Jebro 52 - 34 + 5 % Entire + 2 % Firestone
37	J-E5-30H-200C-00	Jebro 52 - 34 + 5 % Entire

was similar to interaction 13 but polymer was omitted. In Interactions 16 and 17, modified Jebro (58 – 34) binder was mixed with 5% EnTire crumb rubber using same procedure used in Interactions 13 and 14.

In interaction 18, Flint Hills binder was blended with 5% Jai Tire crumb rubber plus 2% Firestone polymer and mixed at 30 Hz and 302^{o} F for forty minutes. The materials were then mixed at 25 Hz (347^{o} F) for four hours and twenty minutes. In Interaction 19, the same procedure was repeated for Jebro (58-34) and 5% Jai Tire crumb rubber. In Interactions 20 – 26, selected mixes (identical to those used in Interactions 8, 9, 10, 12, 13, 14, and 18) were sheared for additional five hours at 10 Hz and 320^{o} F.

Interaction 27 to 35 involved Flint Hills binder with 2% Firestone polymer and various sieve sizes of EnTire and Jai tire crumb rubber. For these interactions, 60-80E designates that an EnTire crumb rubber was used which passed the No. 60 sieve but was retained on the No. 80 sieve. In the first stage, the crumb rubber was added to the binder and sheared at 50Hz between 392^{o} – 420^{o} F for twenty minutes. The second stage involved adding polymer to the mix and shearing at 25 Hz and 392^{o} F for forty minutes. Additional shearing was continued at 10 Hz and 392^{o} F for four hours.

Mixes 36 and 37 consisted of Jebro (52 – 34) binder mixed with 5% EnTire crumb rubber (Exp 36) and 5% EnTire plus 2% Firestone polymer (Exp 37). The first stage involved mixing at 50 Hz (392^{o} – 420^{o} F) for forty minutes. Mixing continued at 30 Hz (392^{o} F) for twenty minutes in the second stage with additional 10 Hz shearing at 392^{o} F for four hours as the last stage. The final products from all interactions were subjected to the cigar tube test (ASTM D-5976) to measure the percent separation. Selected samples were processed through the RTFO and PAV. Materials were then tested using the Bending Beam Rheometer and the Dynamic Shear Rheometer to characterize the asphalt binder properties.

3.4.3 CRM Binder Properties

Experiments outlined within this section were all conducted with the same combination of Flint Hills binder and 5% EnTire crumb rubber plus 2% Firestone polymer. Various gradations of EnTire crumb rubber separated by sieving were used. Figure 3-4 shows the gradations of the CRM materials. A high shear mixer was used in combination with the heating mantle and a controller was used to maintain the temperature. All samples were placed in one gallon cans, so longer temperature probes were needed (TJC – 36 - 12 in.). To control the increase in temperature resulting from mixing, a fan was used to cool the mix during high speed shearing.

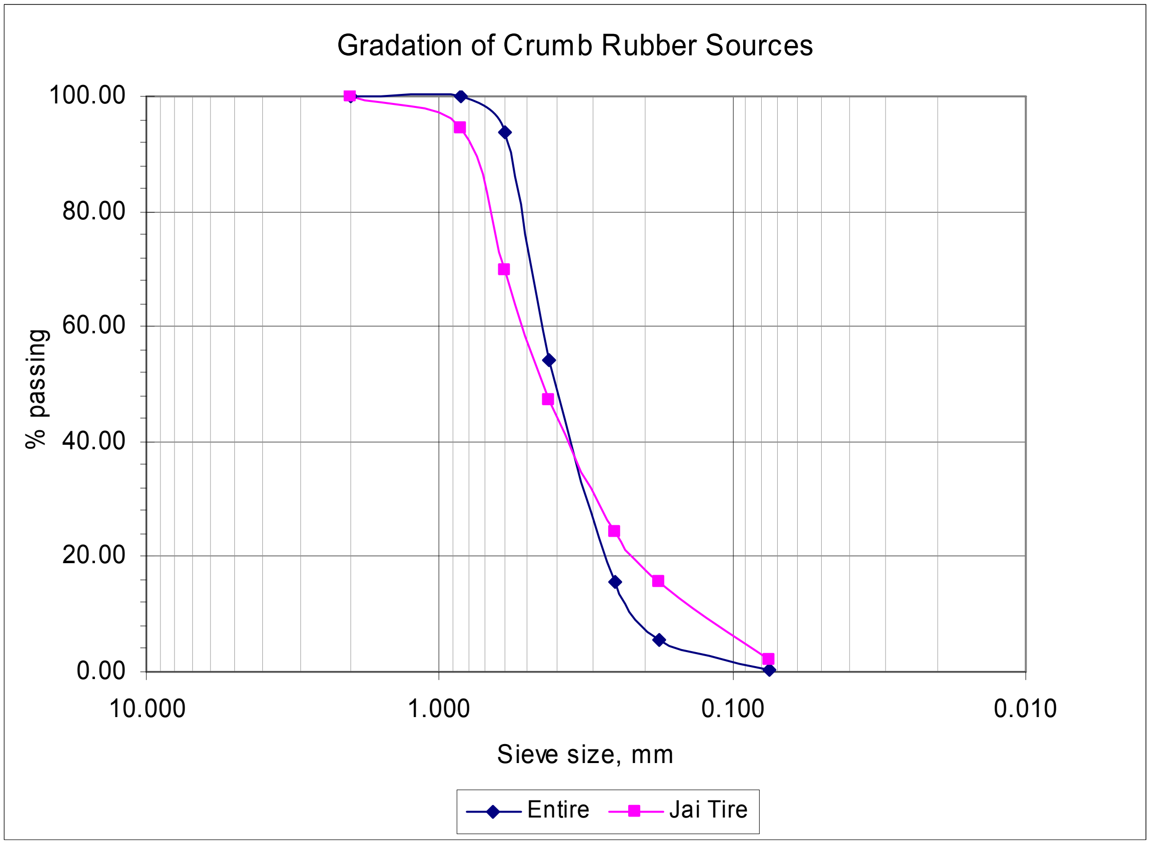


Figure 3-4 Gradation of Crumb Rubber Materials.

All samples were pre-sheared before processing. During pre-shear, the binder was mixed with crumb rubber for ten minutes at 10 Hz at a temperature of 170° C. The experiment was conducted in three stages. The first stage was performed at high shear and temperature while the binder was mixed with the crumb rubber. The blend was sheared at either 30 Hz or 50 Hz for forty minutes and the temperature was controlled at three levels 170° C, 200° C, and 220° C. The second stage was common to all experiments. Here the mix was sheared at 30 Hz for thirty minutes at a temperature of 170° C. The speed was then reduced to 10 Hz while the temperature was maintained at 170° C. Samples were taken at ten minute, twenty minute, forty minute, four hour and eight hour intervals. Samples were tested in DSR for G* and sin δ with 10 radians per second frequency at a temperature of 58° C. The four hour and eight hour samples were analyzed using the cigar tube test to measure percent separation. Experiments No. 56, 57 and 58 were sheared for six hours at 10 Hz at a temperature of 170° C before starting high shear mixing. Samples in these cases were taken at six hours, six hour and ten minutes, six hours and twenty minutes, six hours and forty minutes, ten hours, and fourteen hours.

3.5 Superpave Asphalt Binder Tests

3.5.1 Rolling Thin Film Oven Test

The rolling thin-film oven test (RTFOT) is a conditioning procedure that simulates the age hardening asphalt undergoes during the production and construction of HMA. This test exposes films of binder to heat and air to determine the effect of these conditions on a moving film of asphalt and to evaluate the resistance to aging during the production and construction of HMA Pavement. The procedure is detailed in ASTM D2872. Thirty-five grams ± 0.5 grams of the binder is poured into a specially designed bottle. This exact quantity is specified to make sure that the binder forms a thin film on the bottle's sides. Eight bottles are placed in a vertical rack, which is rotated to continually expose fresh films to hot air. This test is conducted in the oven at 163^{o} C for eighty-five minutes with 4,000 ml/min of air blown across the bottles. The residue from six bottles is combined into a single container and hand stirred to ensure homogeneity. The contents of the other two bottles were weighed and used to determine percent loss of material.

3.5.2 Pressure Aging Vessel

The pressure aging vessel (PAV) is used to simulate long-term aging, observed in five to ten years of pavement service. Fifty grams ± 0.5 grams of residue from RTFO-aged binder is placed in a pan. Pans were subjected to 100^{o} C temperature and a pressure of 2.1 MPa for a period for twenty hours. The residue obtained is then de-gassed in the vacuum oven for thirty minutes before testing in the DSR.

3.5.3 Bending Beam Rheometer

The bending beam rheometer (BBR) is used to assess asphalt binder stiffness at very low temperatures. The test uses engineering beam theory to measure the stiffness of a small asphalt beam sample under a creep load. A creep load is used to simulate the stresses that gradually build up in a pavement when temperature drops. Creep stiffness and m-value are the two parameters evaluated. Creep stiffness is a measure of how asphalt resists constant loading while the m-value is a measure of how the asphalt stiffness changes as loads are applied. Testing temperature ranges from -0^{0} C to -36^{0} C. In this test, a beam of asphalt binder 125 mm long, 12.5 mm wide, and 6.25 mm thick is placed in a low-temperature bath for 60 minutes to establish its temperature at the desired test temperature. The beam is then placed on two simple supports having a span of 100 mm. After initial loading and conditioning, a constant load of 980 mN is applied at beam center for 240 seconds and deflection is measured with a transducer. Load and deflection versus time curves are continuously generated during the test for inspection. Creep stiffness and creep rate are normally calculated by software.

3.5.4 Rotational Viscometer

The rotational viscosity is used to evaluate high temperature workability of binders. High temperature binder viscosity is measured to ensure that the asphalt is sufficiently fluid when pumping and mixing. Viscosity obtained from a Brookfield Viscometer is normally referred as "Brookfield viscosity". The test is performed according to ASTM D 4402. Superpave specifications require that the test be conducted at 130^0 C using a #27 spindle.

3.5.5 Cigar Tube Test

Modified binder was tested for thermal stability using the cigar tube test (CTT). Detailed procedures for sample preparation are provided in ASTM D 5976. In this test, thin aluminum cigar tubes were filled with polymer-modified binder and allowed to stand in the oven at 163° C (325° F) for forty-eight hours. Samples were then removed and immediately placed in the freezer for a minimum of four hours before removed and cut into three equal parts. Separation was determined by comparing the difference in DSR values (G*/sin δ, in Kpa) between the top and bottom samples from the same tube. Percent separation was then calculated using the following equation:

$$\text{Separation, \%} = \frac{(G^*/\sin\delta)_{\max} - (G^*/\sin\delta)_{avg}}{(G^*/\sin\delta)_{avg}} \times 100 \qquad \text{(Eq. 3-1)}$$

Where
G* = shear modulus,
δ = phase angle
$(G^*/\sin\delta)_{max}$ = higher value of either the top or the bottom portion of the tube,
$(G^*/\sin\delta)_{avg}$ is the average value of the two portions.

Separation of asphalt and polymers during hot storage can also be evaluated using the ring and ball test. Samples are prepared in accordance with ASTM D 5976 and differences in the softening point temperature between samples taken from the top and bottom portions of a sealed tube are reported in degrees Celsius.

3.5.6 Dynamic Shear Rheometer

The dynamic shear rheometer (DSR) is used to characterize the viscous and elastic behavior of asphalt binders. This is done by measuring the complex shear modulus (G*) and phase angle (δ) of asphalt binders. G* is a measure of the total resistance of a material to deforming when repeatedly sheared. Phase angle (δ) is an indicator of the relative amounts of recoverable and non-recoverable deformation. To determine the high-temperature performance grade, *G*/sin* δ is determined for the original binder and RTFO-aged blend to determine high temperature grade. Asphalt samples 2 mm thick are tested between two parallel plates. The bottom plate is fixed while the upper place

oscillates at a frequency of 10 radians per second to simulate traffic loading traffic. G* and δ are normally calculated by software with the rheometer. Minimum acceptable values of G*/sin δ for Superpave binders is 1.0 at the designated temperature when testing unaged (tank) binder.

3.6 Non-Superpave Asphalt Binder Tests Used

Ring and Ball Test: Separation of the modified binder can be tested on a ring and ball apparatus to determine the softening point of modified binder under provisions of ASTM D 36 (95). Two bitumen samples cast in shouldered brass rings are heated in a liquid bath (distilled water) at a constant rate while supporting a steel ball each. The temperature at which the bitumen sample softens enough to allow the ball fall a distance of 25 mm (1 inch) is reported as softening point. The steel balls used are 9.5 mm in diameter, each having a mass of 3.5 ± 0.05 g.

3.7 Summary

This chapter described in detail the equipment and methods used to conduct research on performance graded asphalt binders as well as CRM binders. Superpave tests were completed on performance graded binders alone, performance graded binders with polymer added, performance graded binders with crumb rubber added and performance graded binders with both polymer and crumb rubber added. Chapter 4 will report on and discuss the results of the experiments outlined in this chapter.

CHAPTER FOUR

RESULTS AND DISCUSSION

This chapter examines the interaction process to determine if binders with specific performance-related properties can be produced and outlines the general effects of different materials and interaction conditions on CRM binder properties. This chapter also presents the results of a limited experiment on the storage interactions of different asphalt performance graded binders and crumb rubber materials. Full testing results are presented in the appendix.

4.1 Preliminary Study

The preliminary study was conducted to determine the reactivity of asphalt with crumb rubber and to select binder combinations for the next series of experiments. Crumb rubber particles swell by absorbing components from the asphalt phase into the polymer chains to form a gel-like material. As different asphalts have different fractions and components, it was anticipated that rubber particles would swell differently when interacted with different types of asphalt.

Figures 4-1 through 4-8 show the extent of binder modification during preliminary interactions using three different grades of asphalt, Jebro (PG 52-34), Monarch (PG 58-28), and Flint Hills (PG 58-28) with two different sources of crumb rubber, Entire and Jai Tire. Asphalt cement grades were shown to have more effect on performance related properties of CRM binders than crumb rubber source. A major factor affecting the product was found to be the rubber content. The interaction conditions of time and temperature were shown to affect the developed properties of CRM binders. The effect of time is greatly dependent on the temperature.

Changing the rubber source changed the properties of the binders. Each crumb rubber source could achieve similar properties at different times and temperatures. However, the differences in modification caused by different rubber sources were not as significant as those caused with different grades of binders.

The concentration of rubber alters how much of the asphalt's light components are absorbed at the beginning of the interaction process and the quantity of rubber components that can be released later in the process during depolymerization. CRM asphalt has a considerable amount of material fillers. Thus, higher concentrations of crumb rubber (as compared to other binder modifiers) are required to achieve the same level of property modification. Figures 4-1 through 4-8 show the effectiveness of crumb rubber in modifying the binder performance-related properties, G^* and δ., Higher concentrations of crumb rubber provided significantly greater degrees of modification. The results suggest that CRM binders from different sources will behave differently under different interaction conditions. Thus, it is important to accurately characterize the interaction process over both the short and long terms using a large cross section of

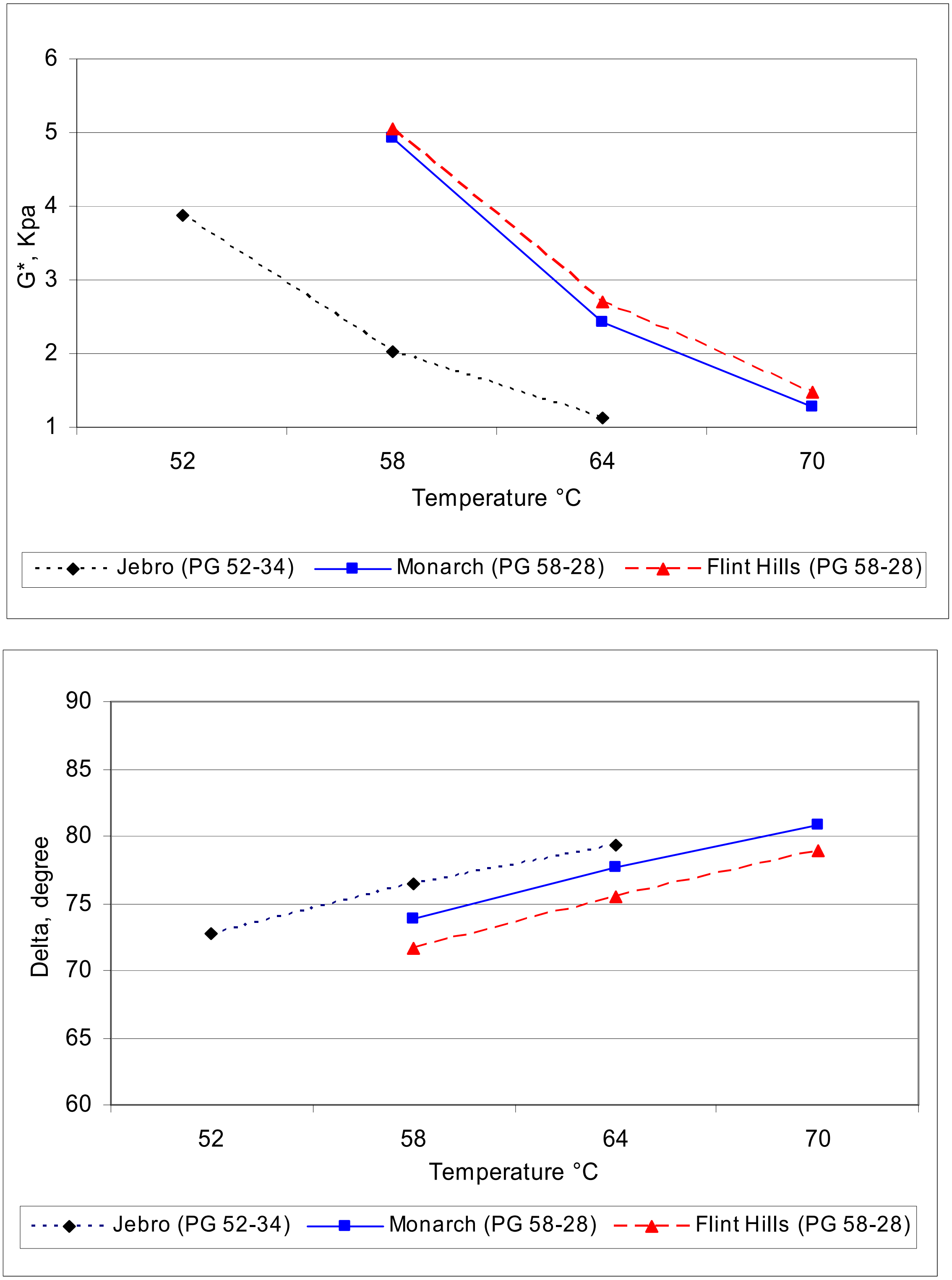
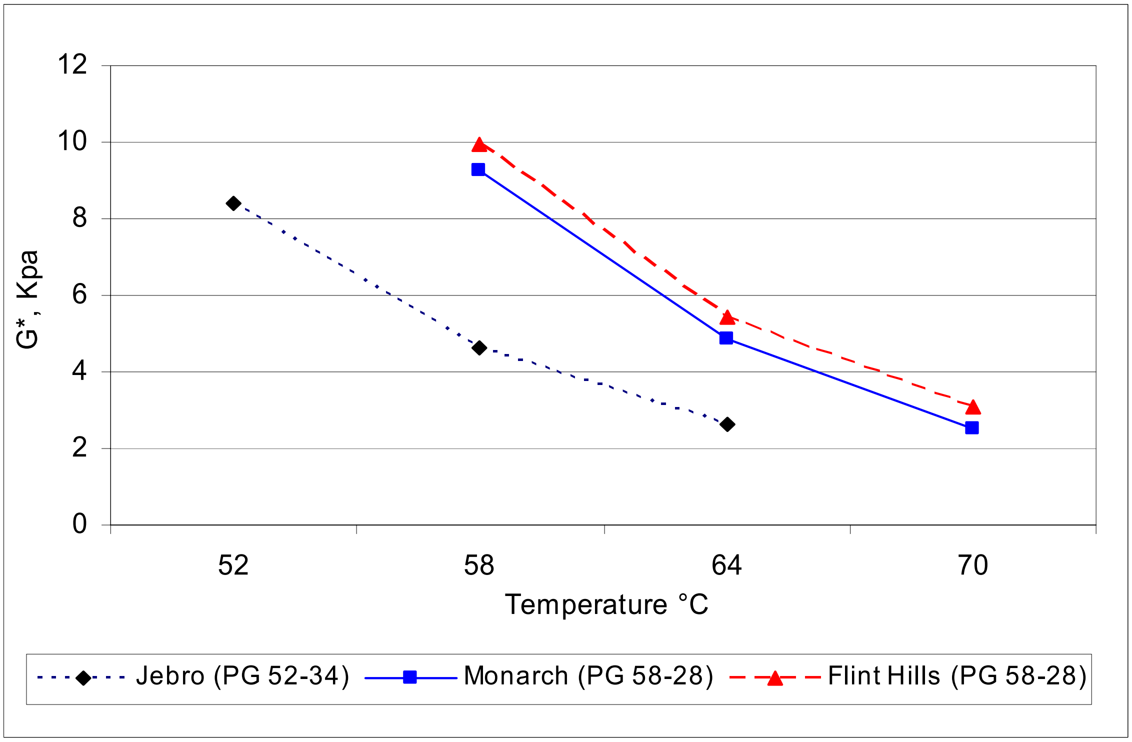


Figure 4-1-a Jai Tire CRM – Tank.



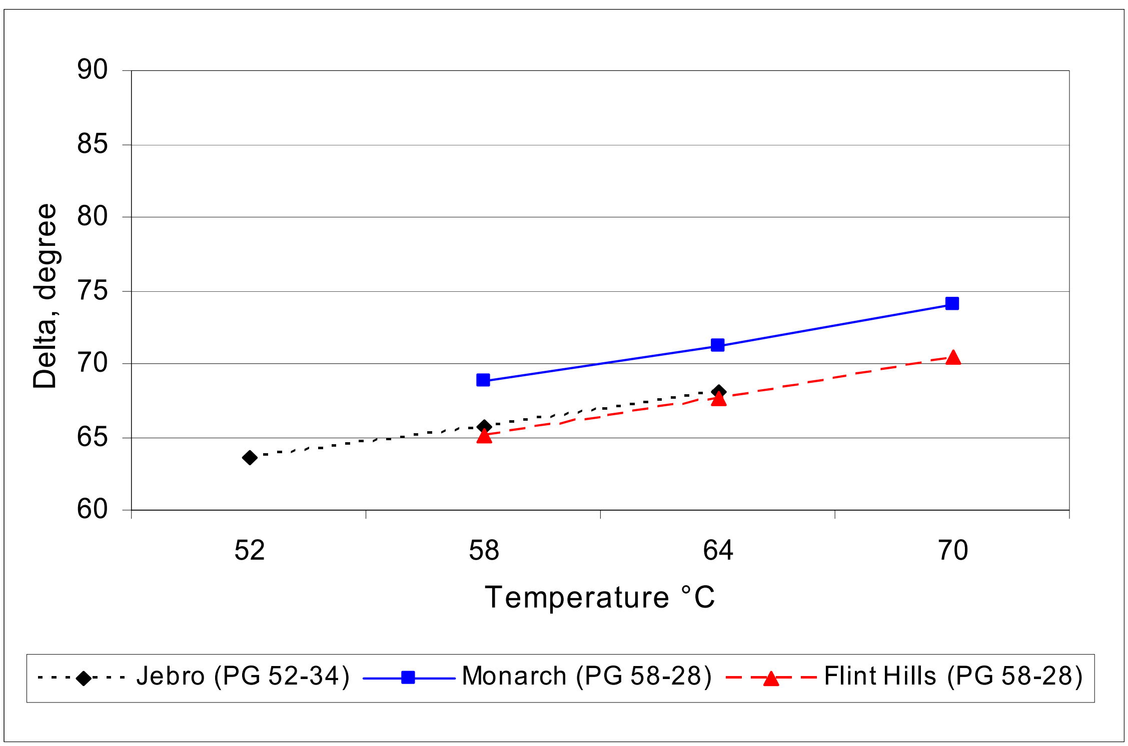
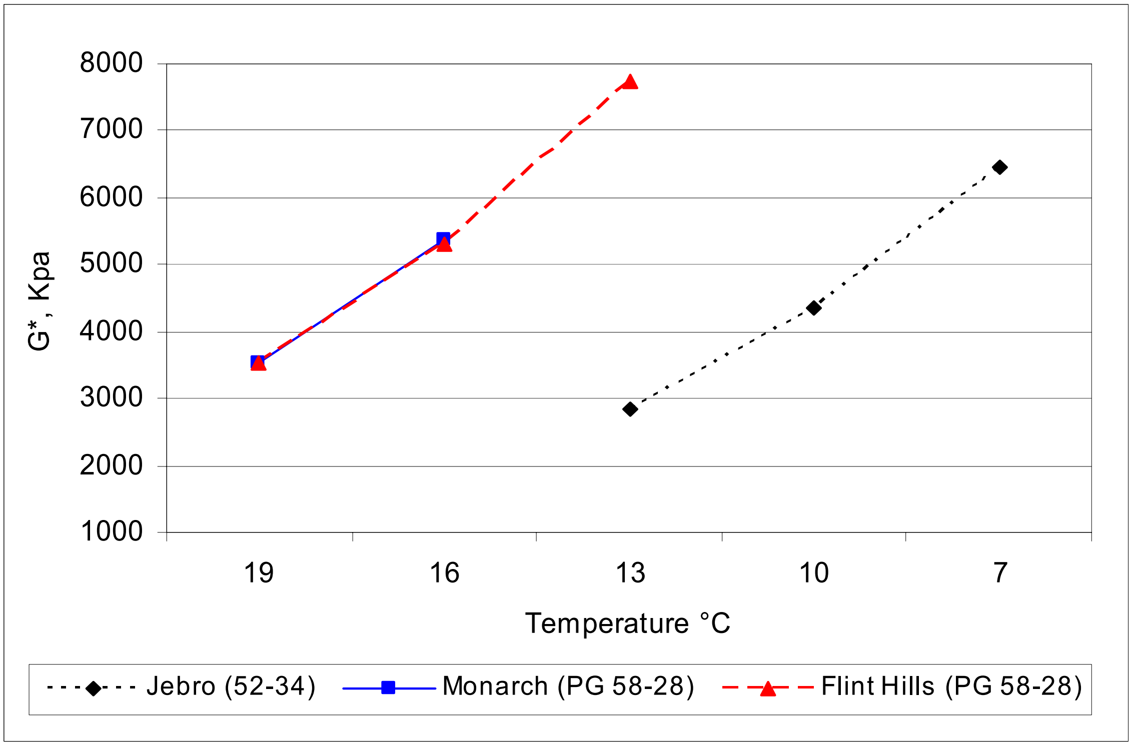


Figure 4-1-b Jai Tire CRM – R.T.F.O.



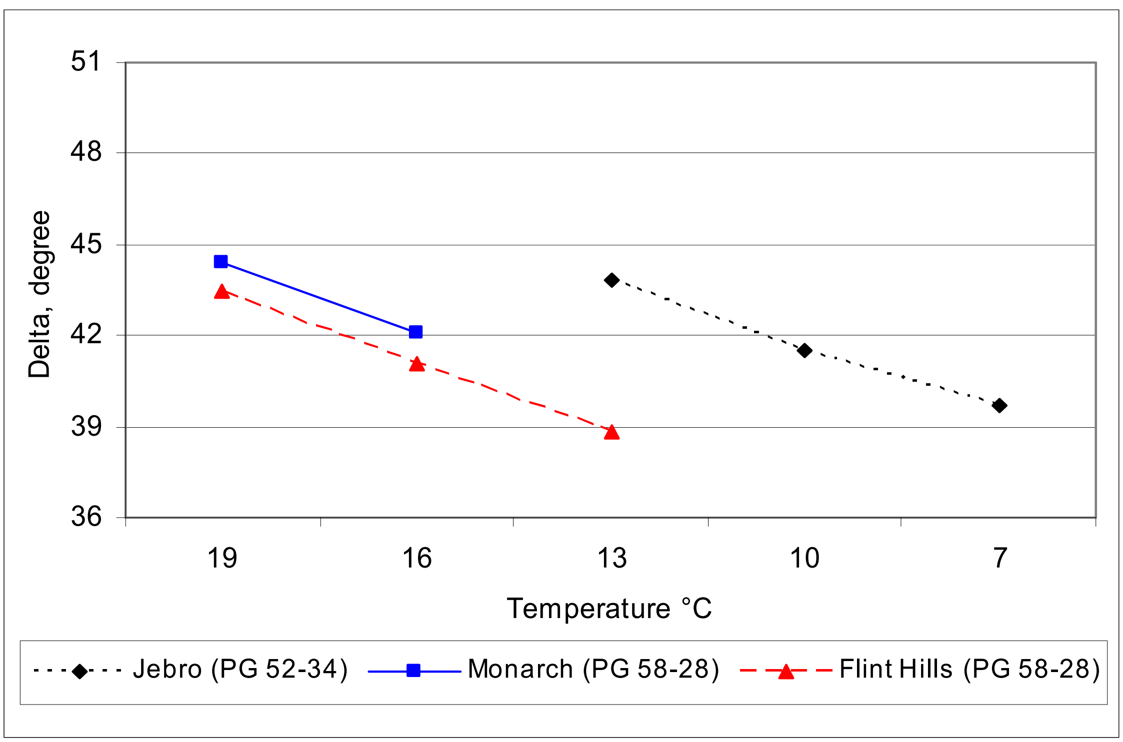
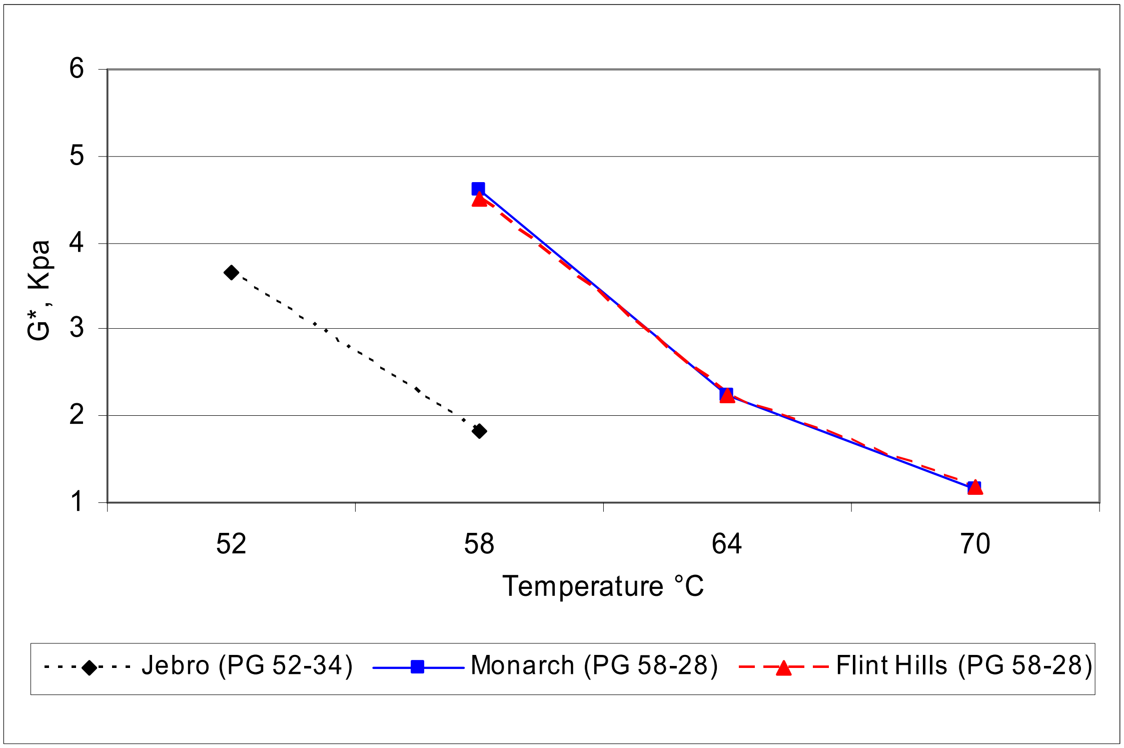


Figure 4-1-c Jai Tire CRM – P.A.V.



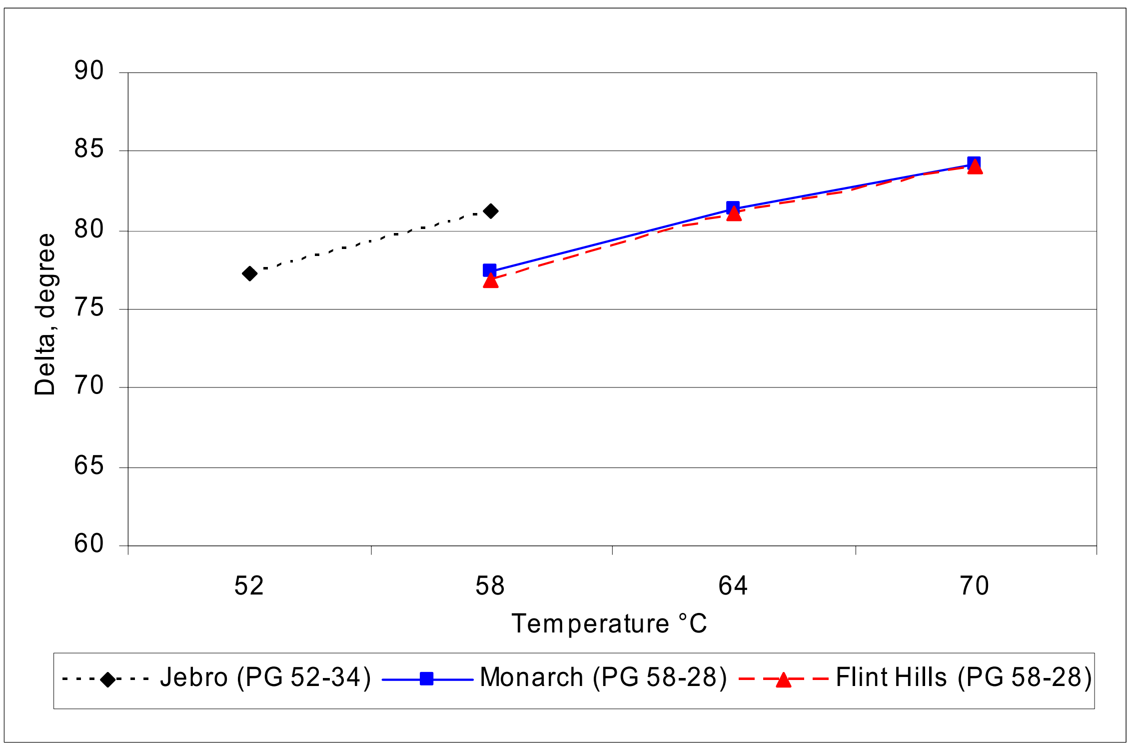


Figure 4-2-a Entire Recycling - Tank.

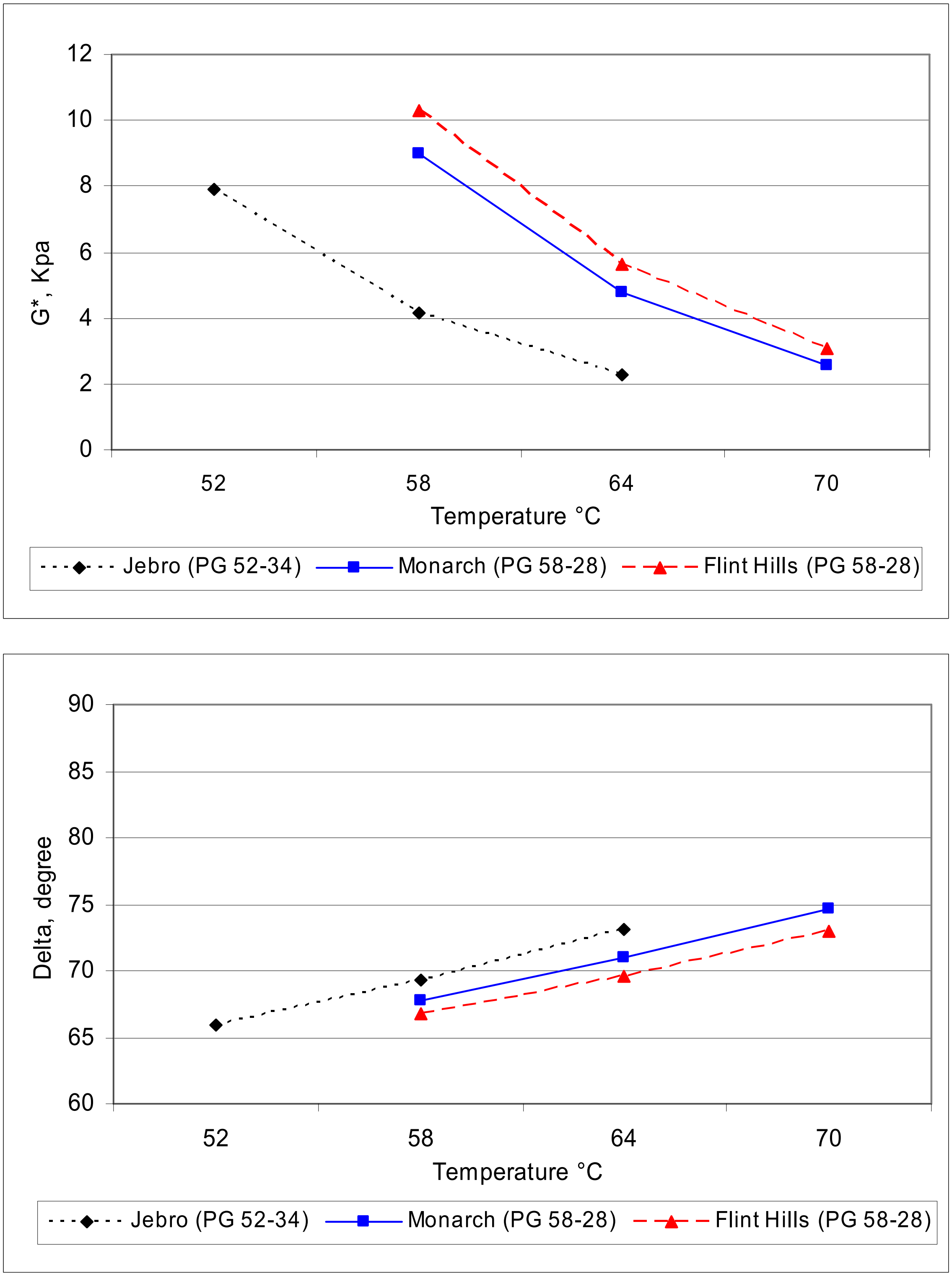
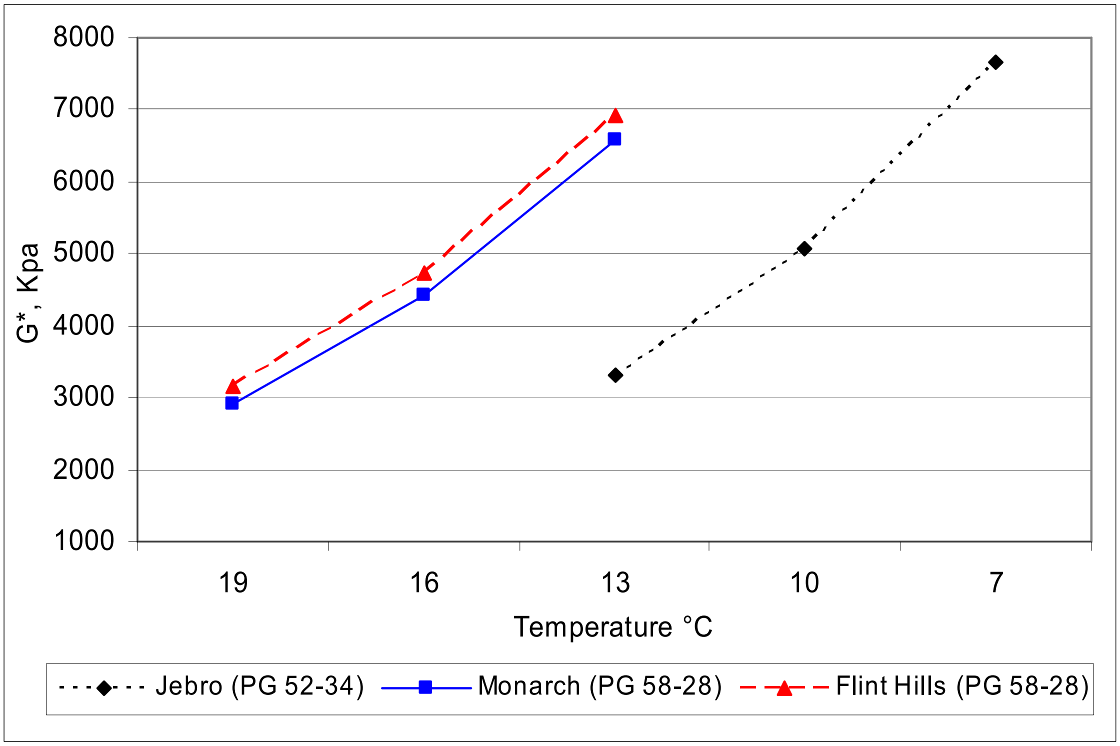


Figure 4-2-b Entire Recycling – R.T.F.O.



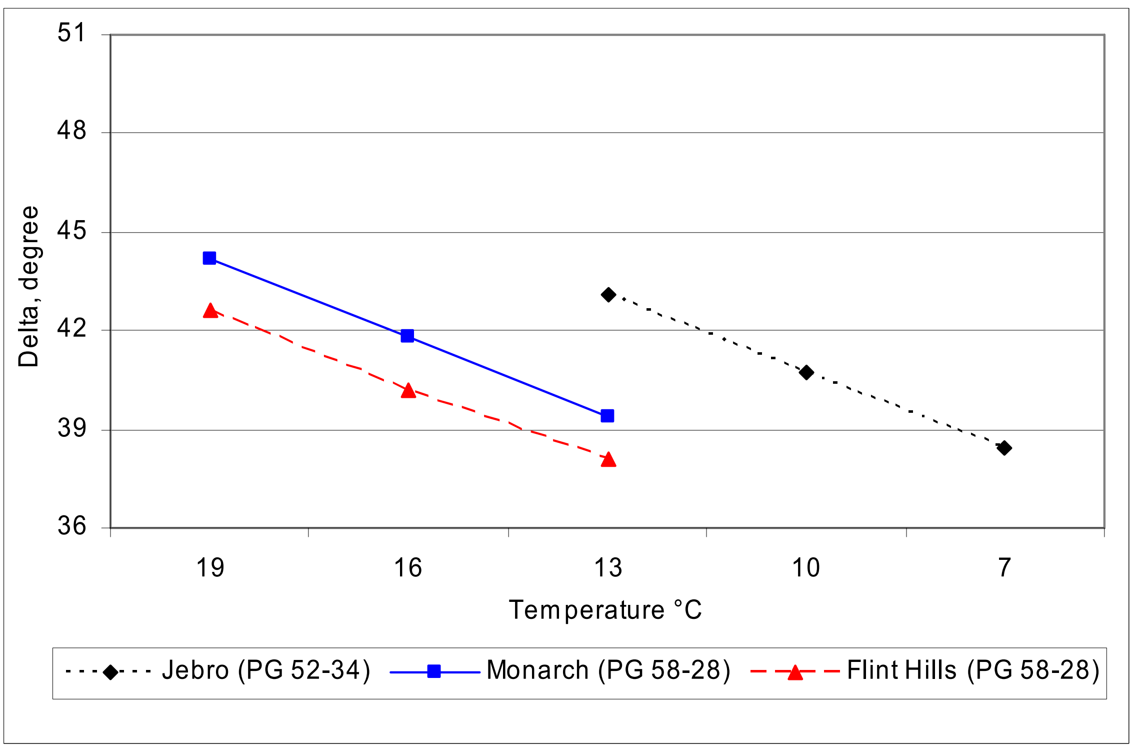
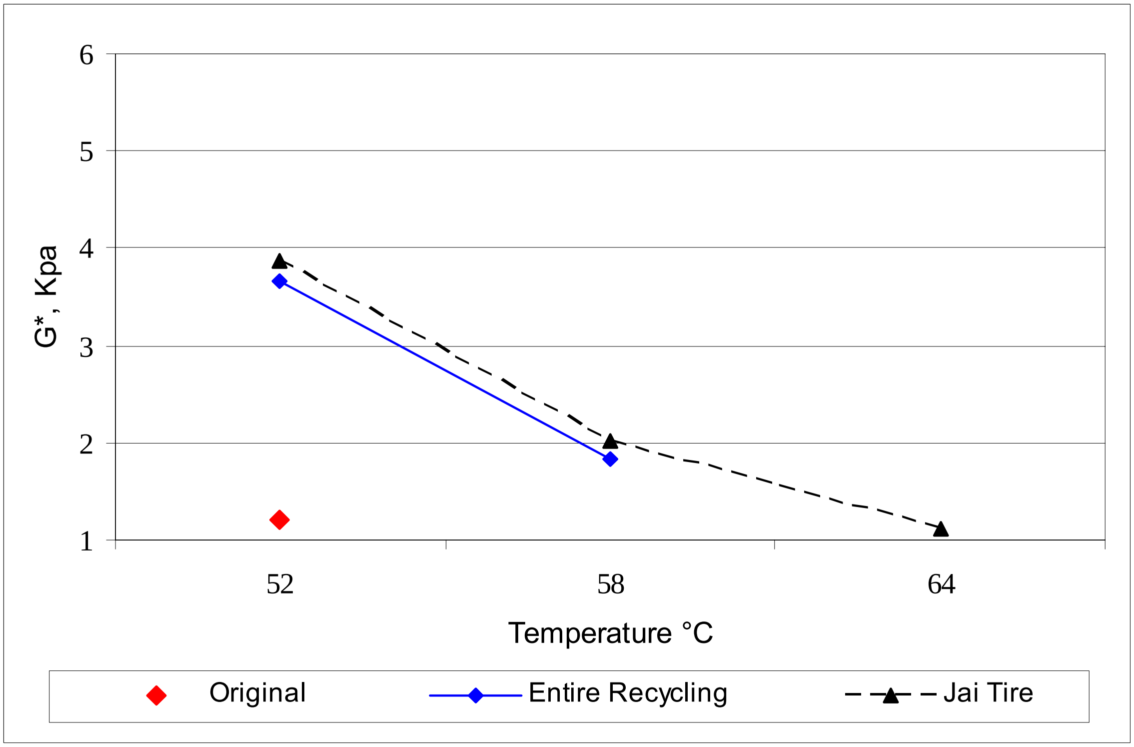


Figure 4-2-c Entire Recycling – P.A.V.



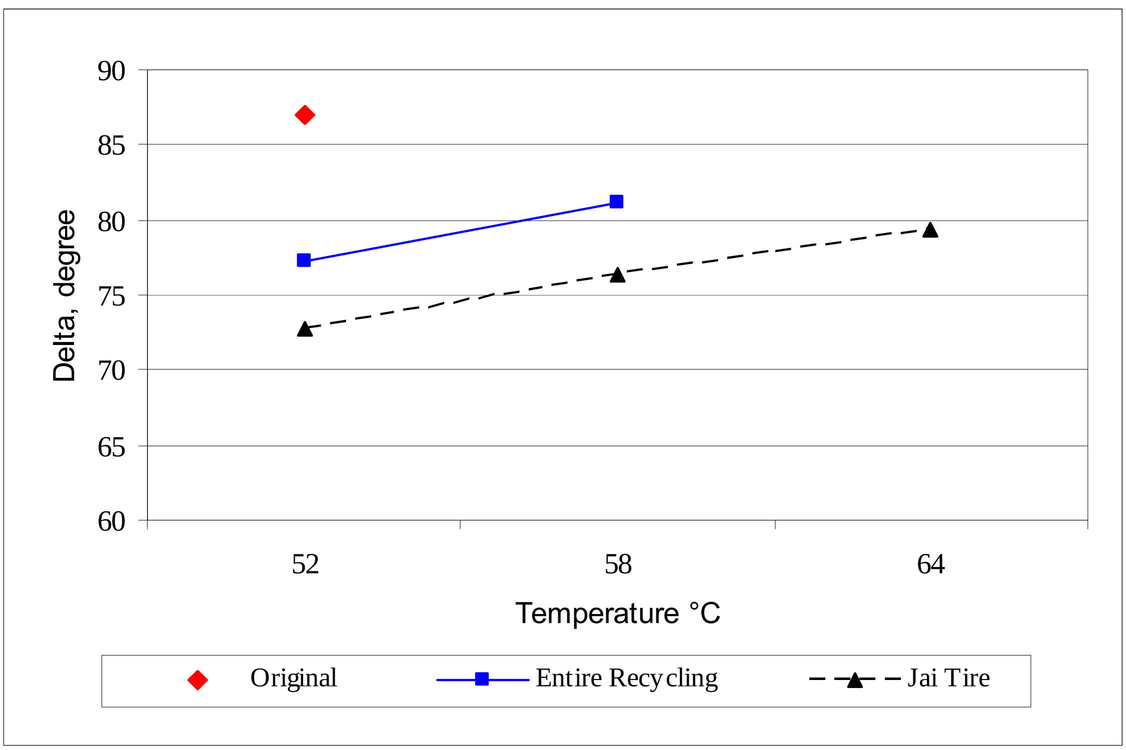
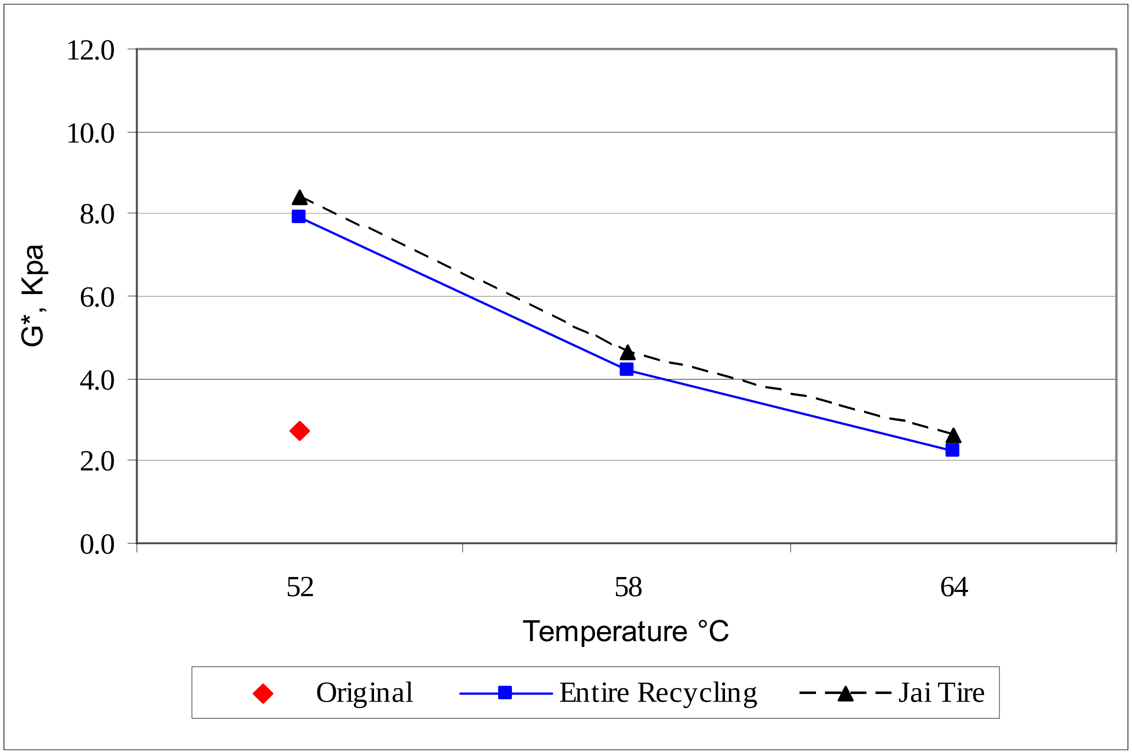


Figure 4-3-a Jebro (PG 52-34) AC – Tank.



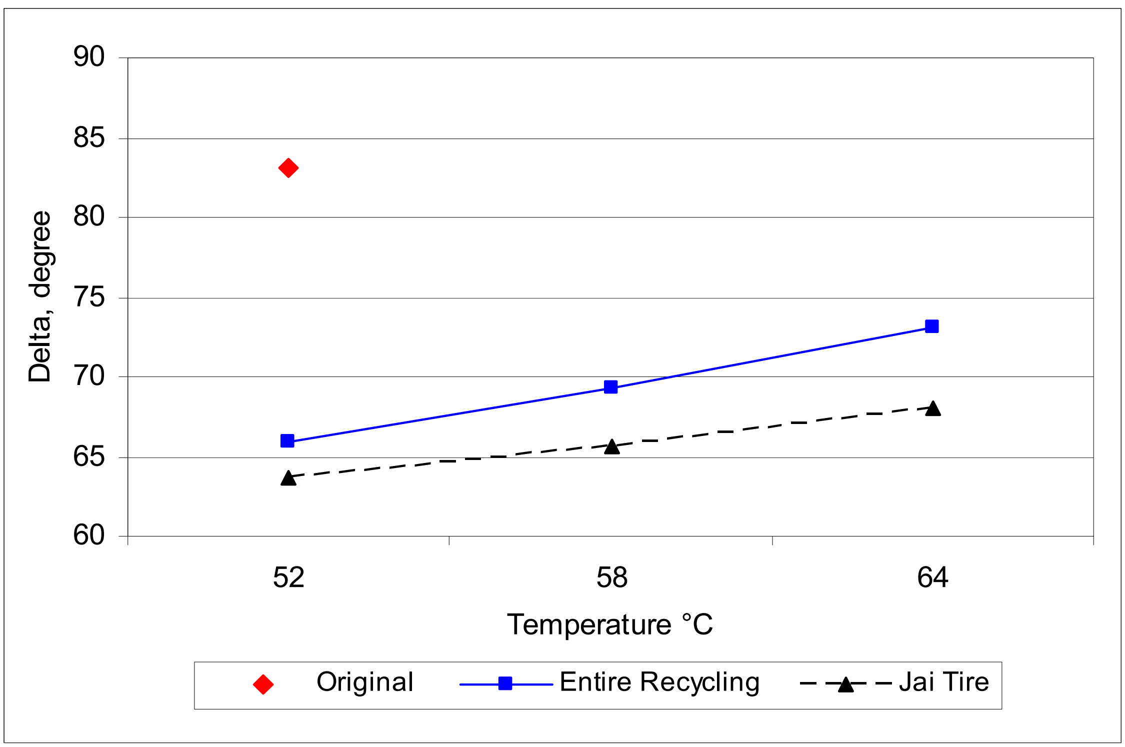
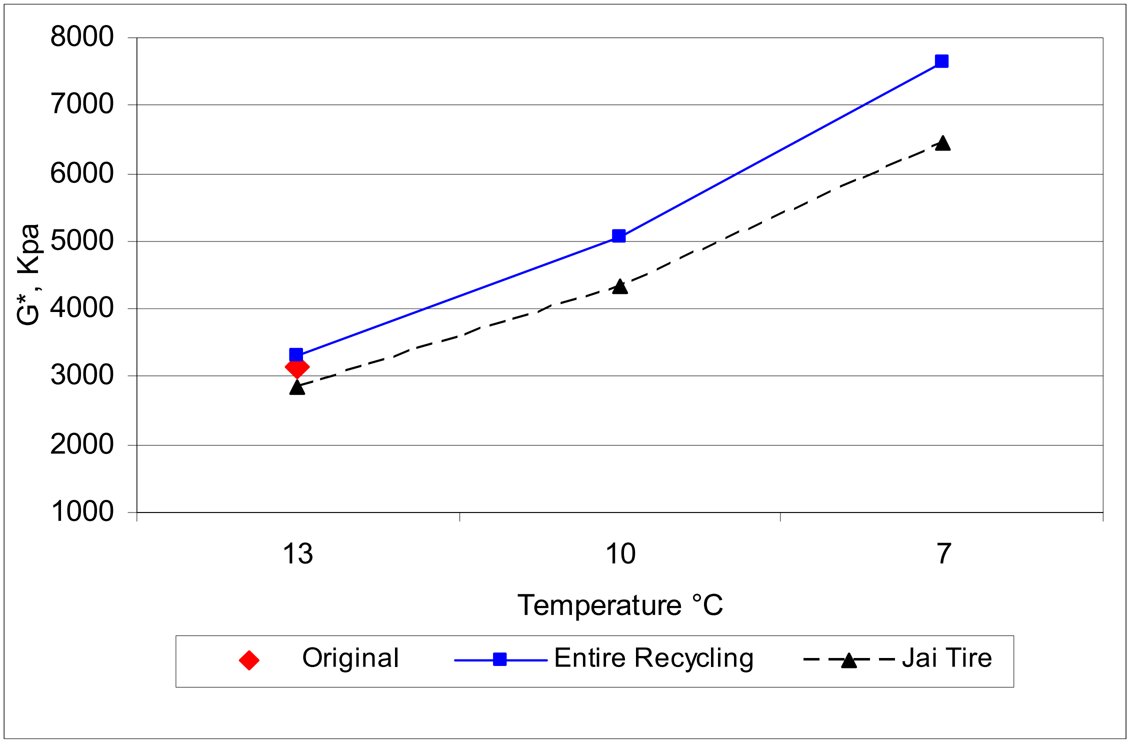


Figure 4-3-b Jebro (PG 52-34) AC – R.T.F.O.



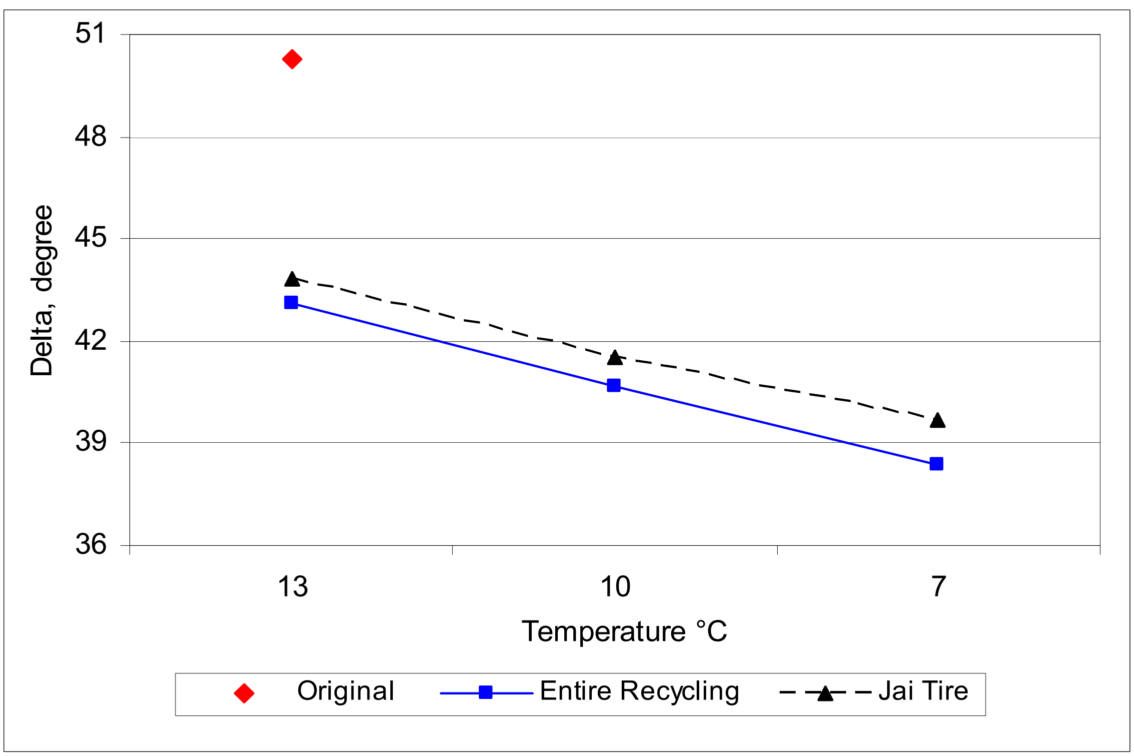


Figure 4-3-c Jebro (PG 52-34) AC – P.A.V.

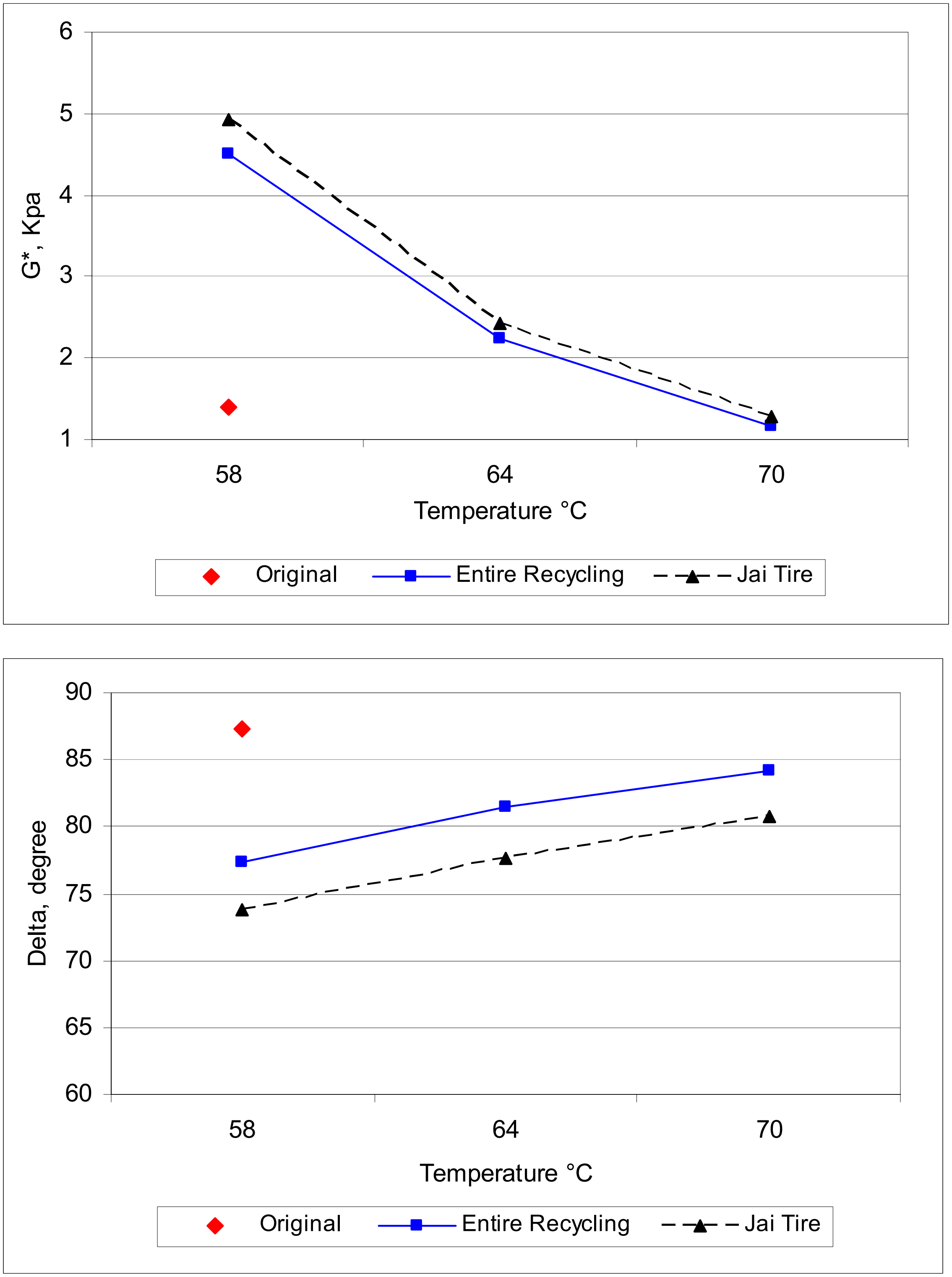
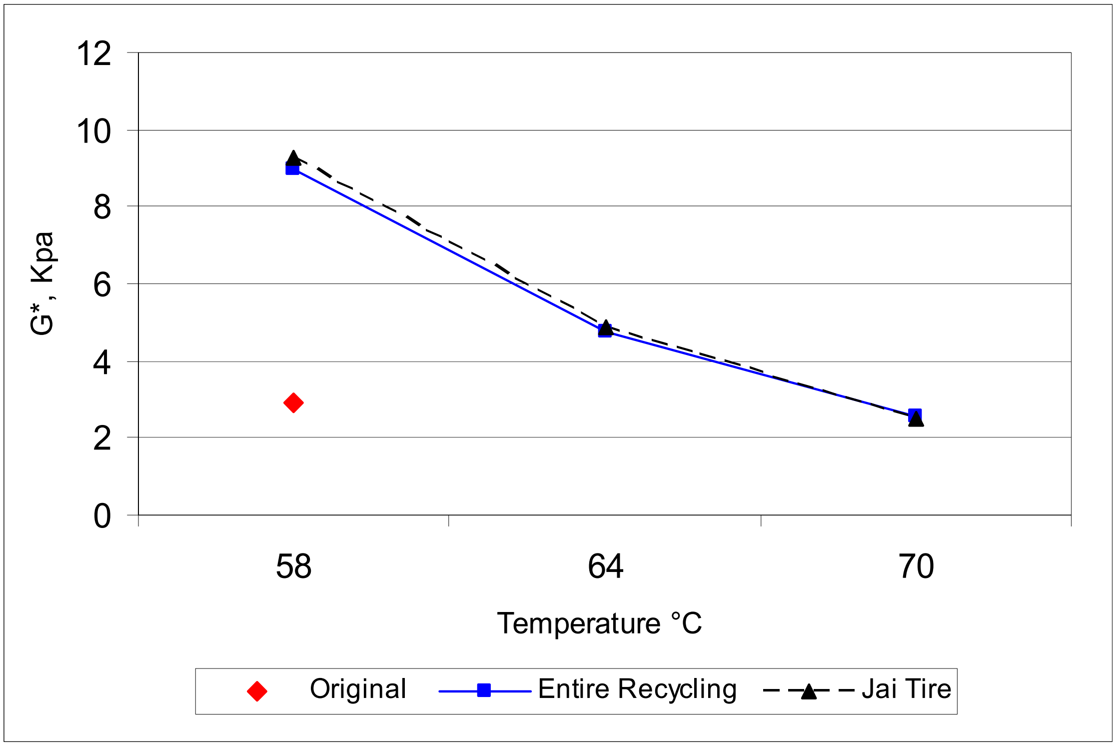


Figure 4-4-a Monarch (PG 58-28) AC – Tank.



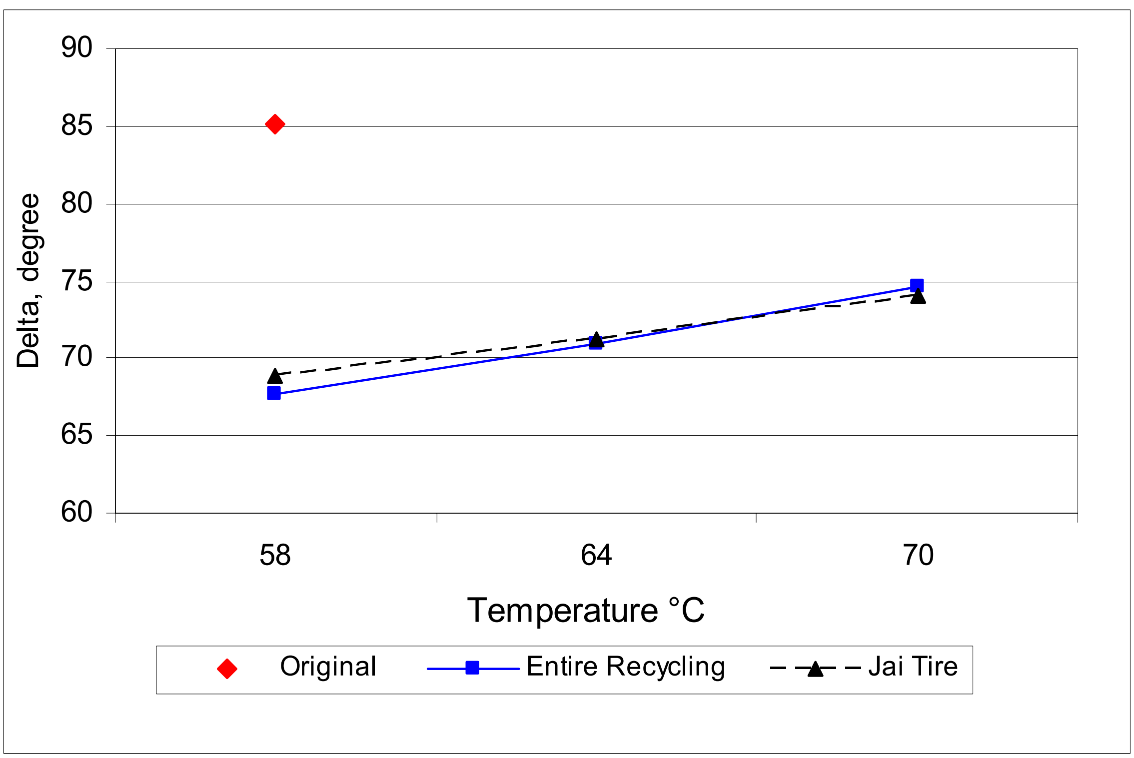
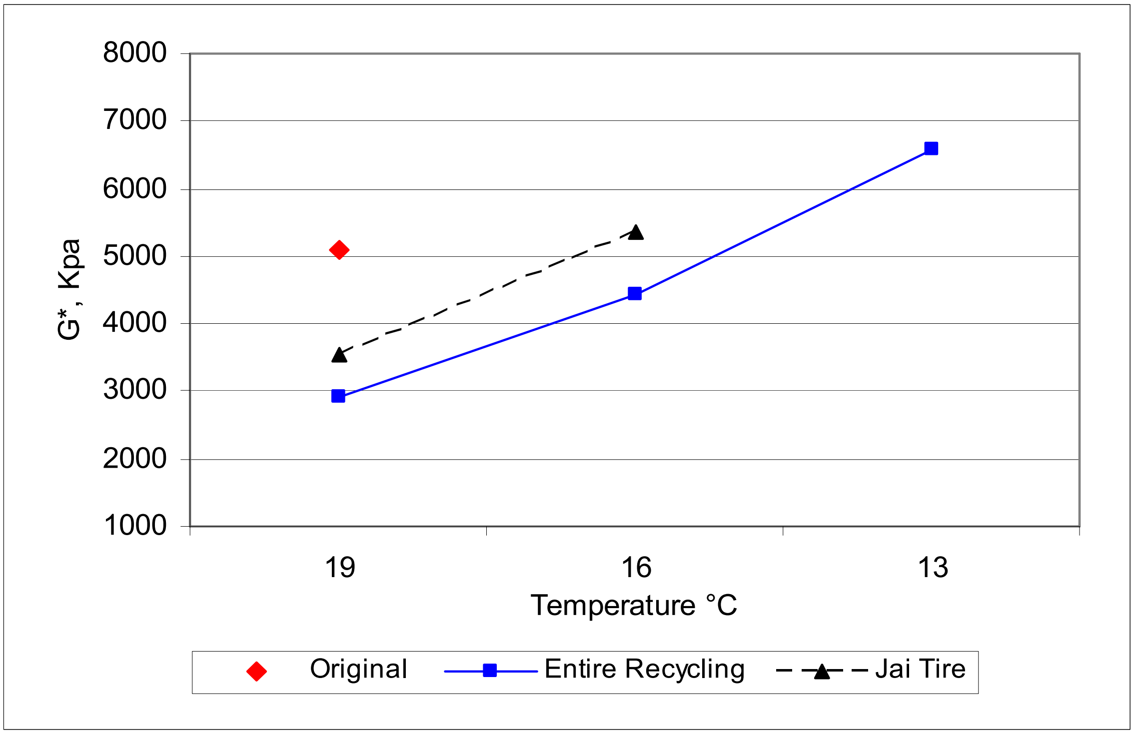


Figure 4-4-b Monarch (PG 58-28) AC – R.T.F.O.



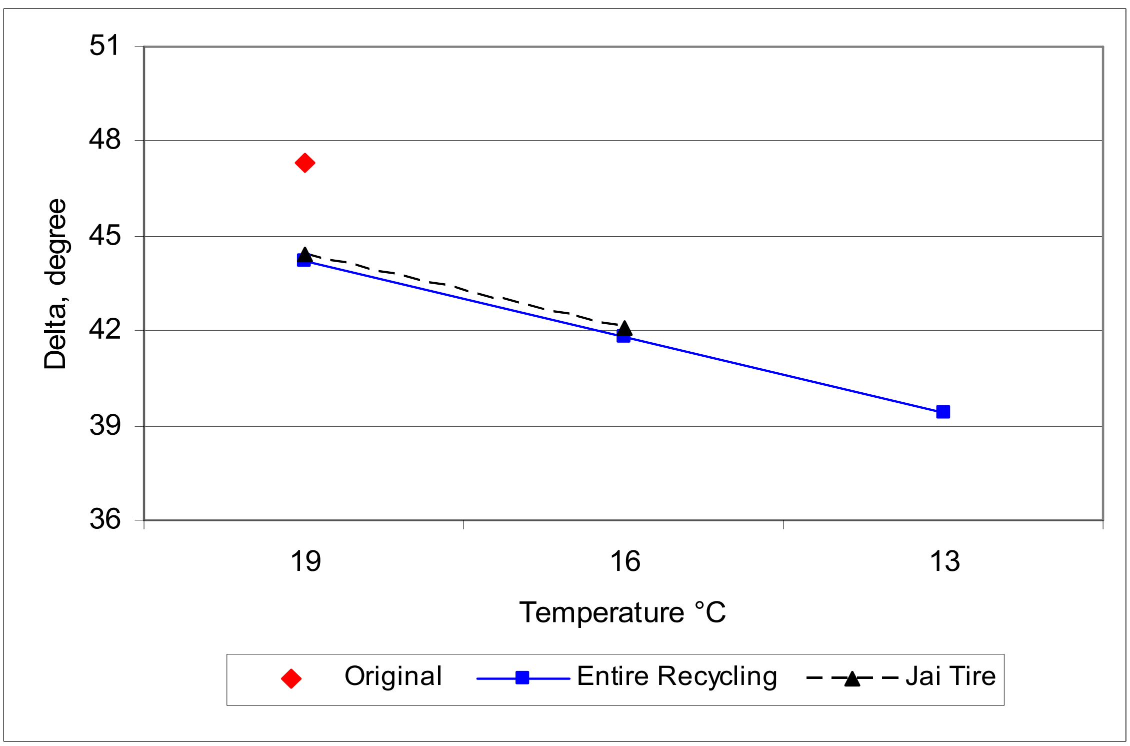
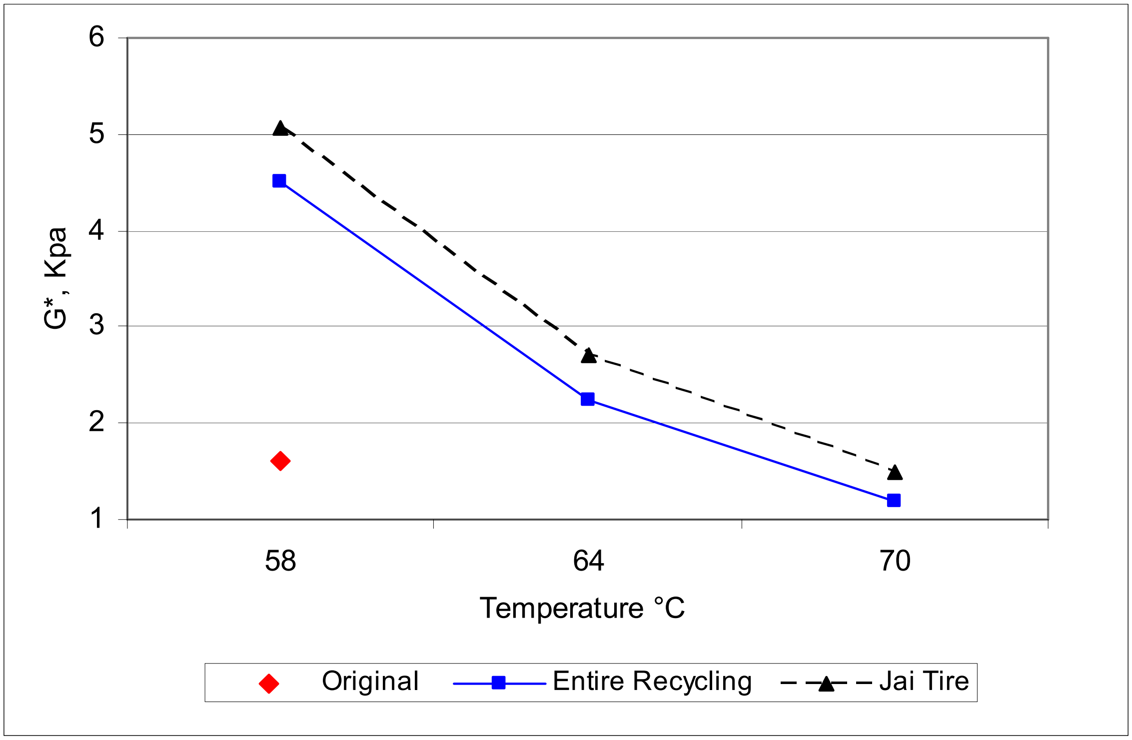


Figure 4-4-c Monarch (PG 58-28) AC – P.A.V.



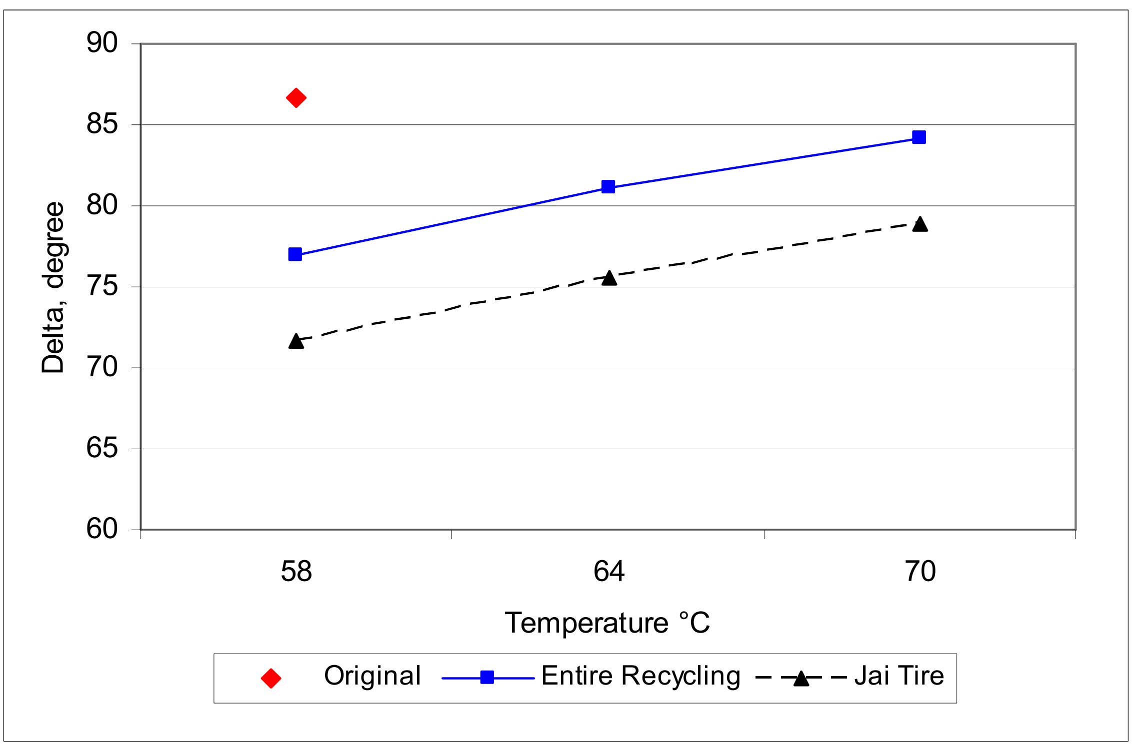
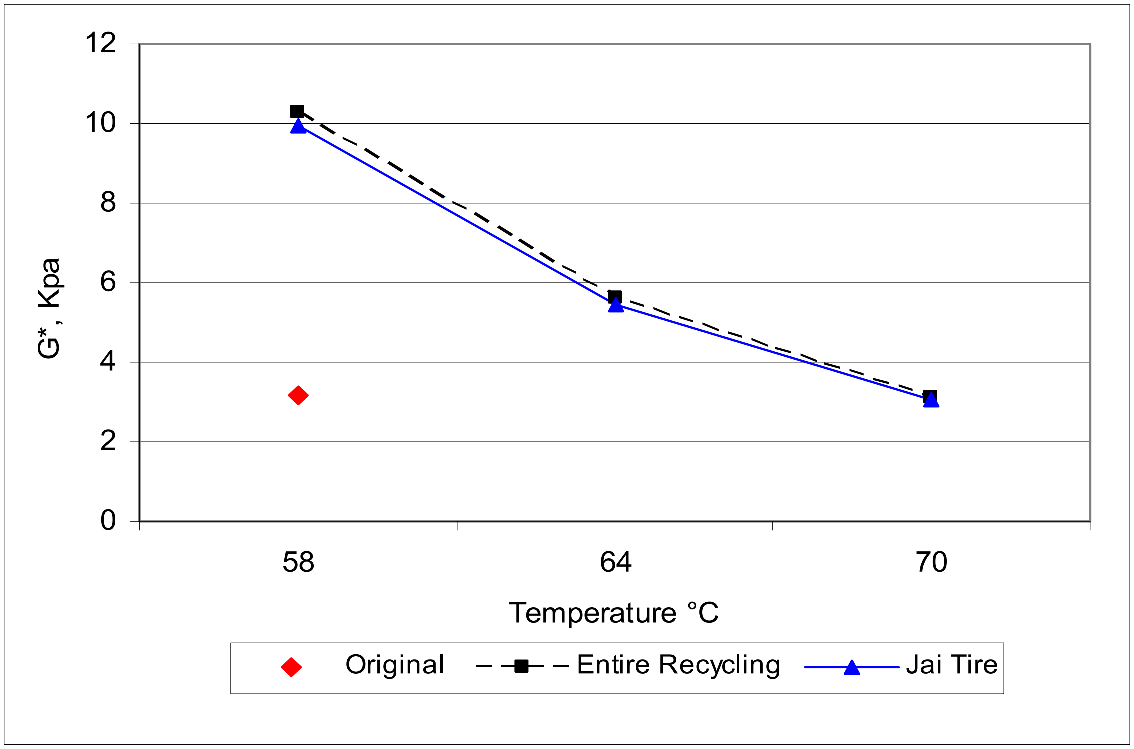


Figure 4-5-a Flint Hills (PG 58-28) AC – Tank.



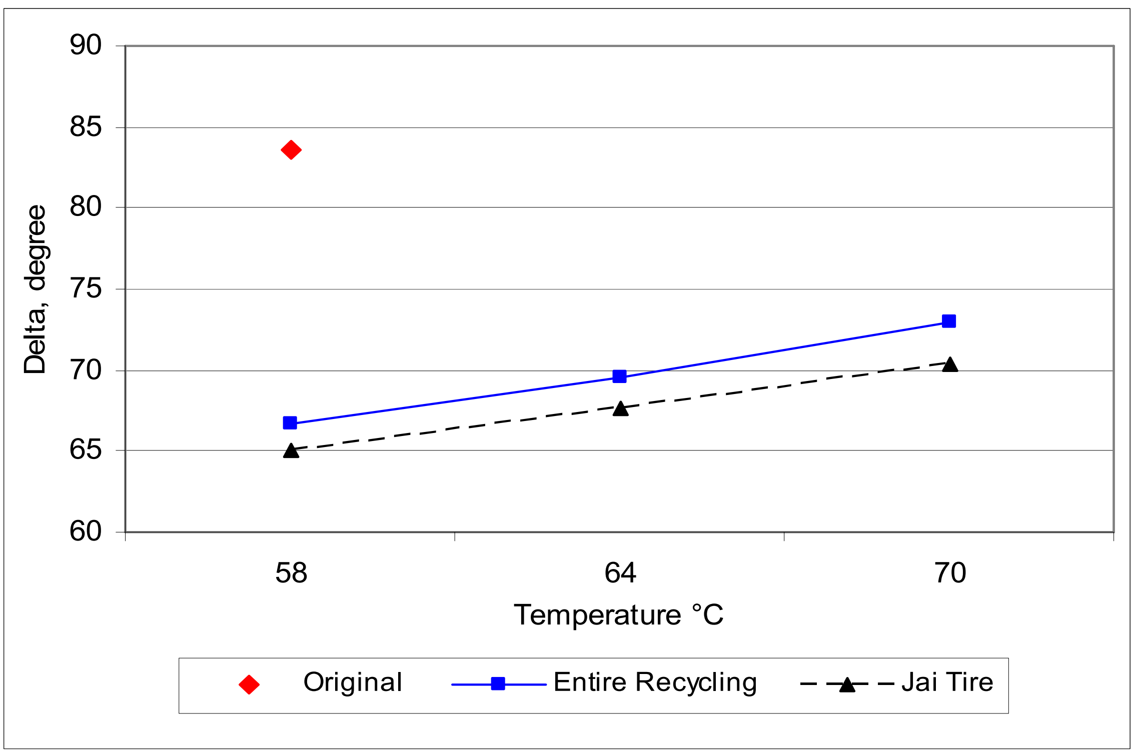
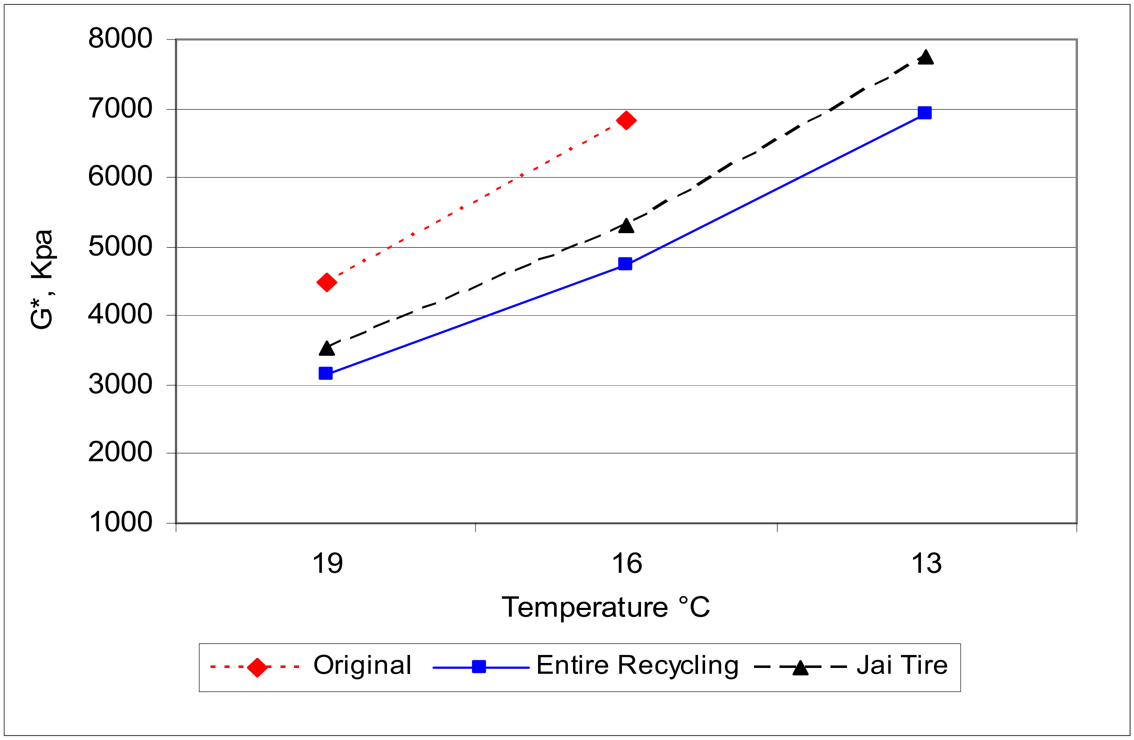


Figure 4-5-b Flint Hills (PG 58-28) AC – R.T.F.O.



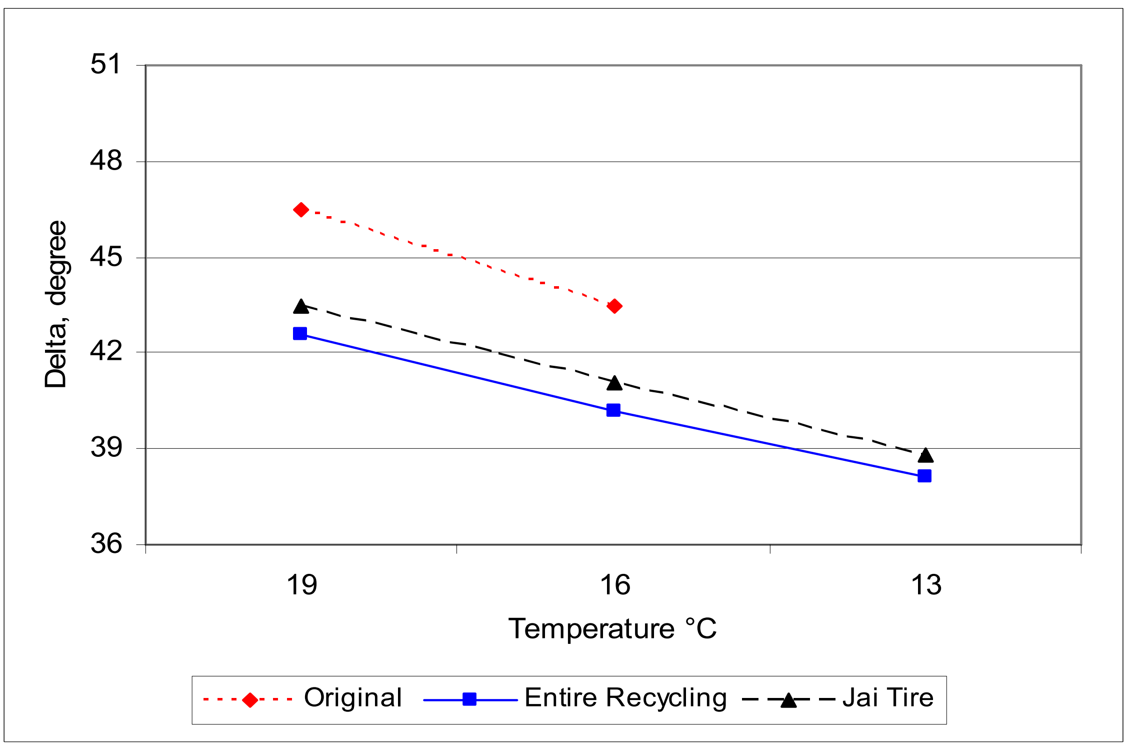
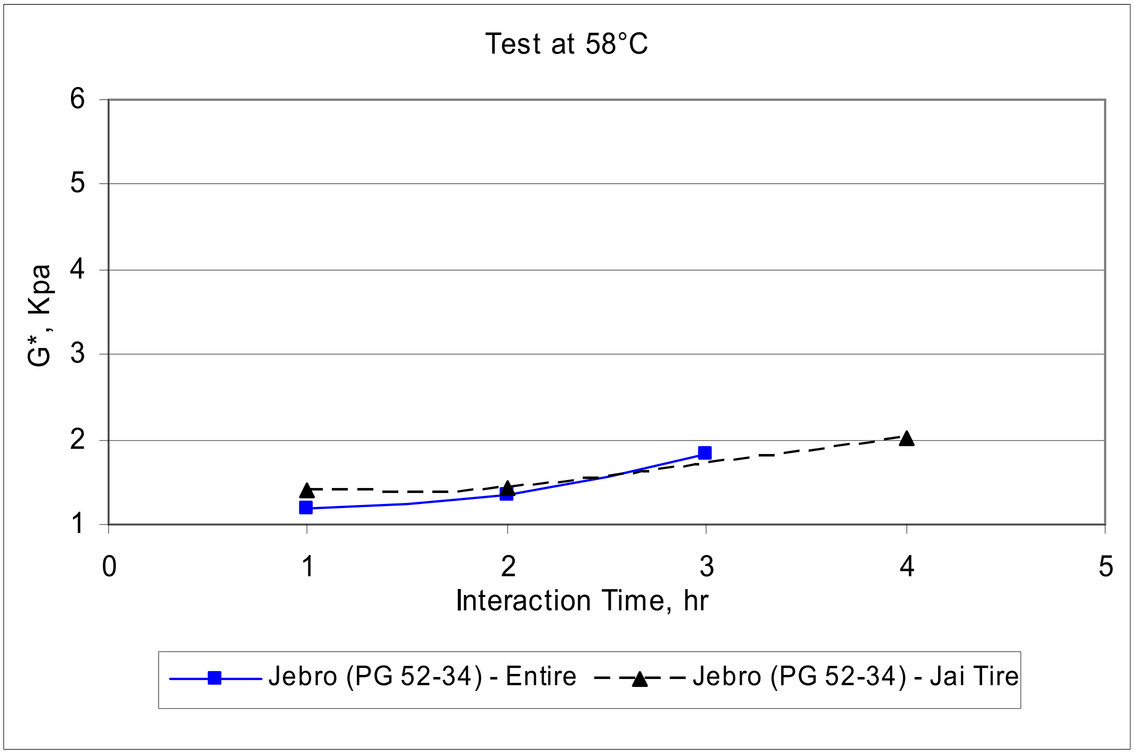


Figure 4-5-c Flint Hills (PG 58-28) AC – P.A.V.



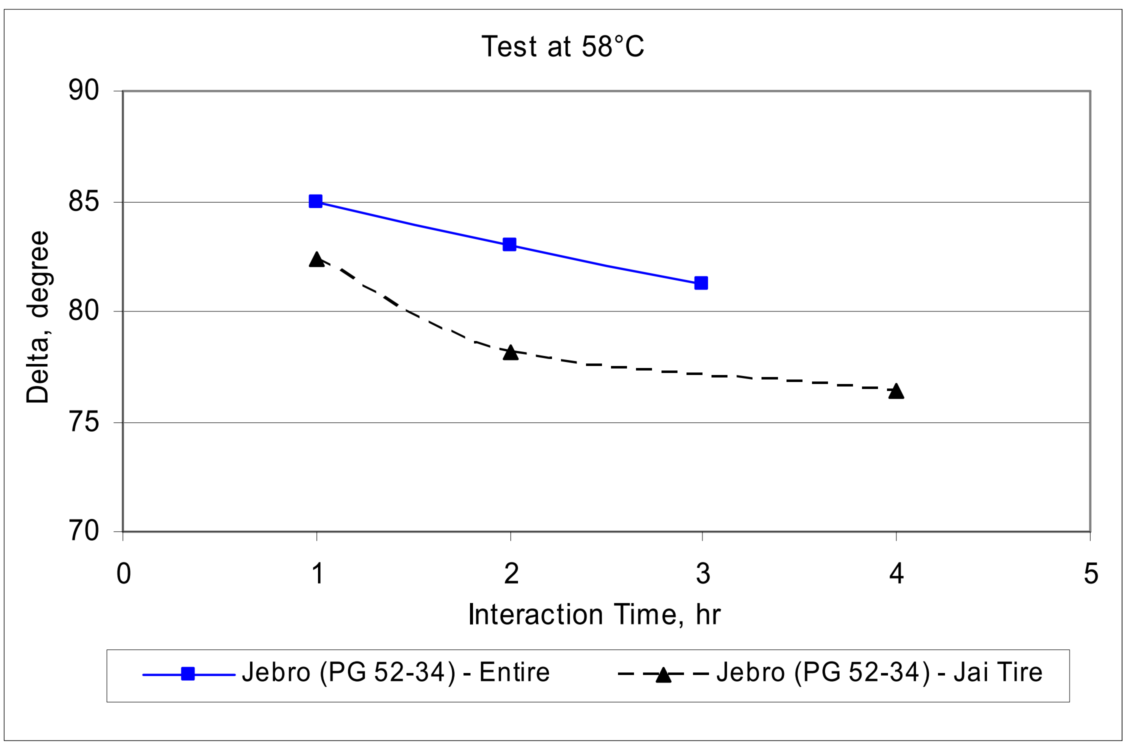
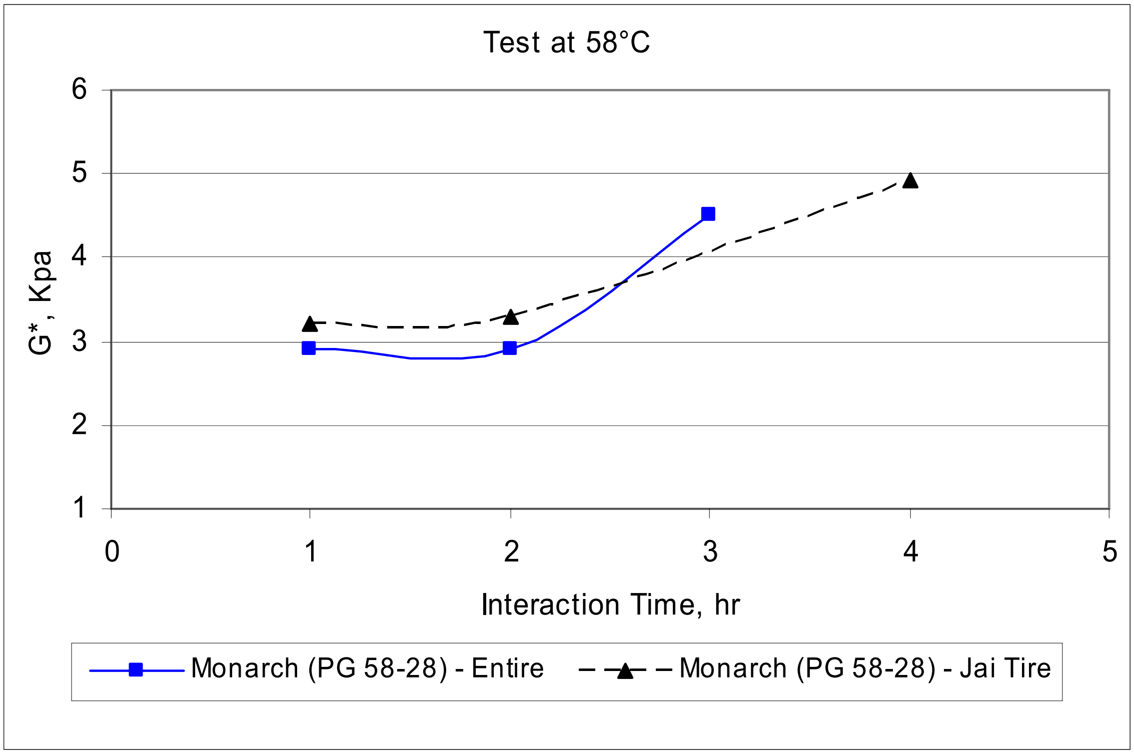


Figure 4-6 Jebro (PG 52-34).



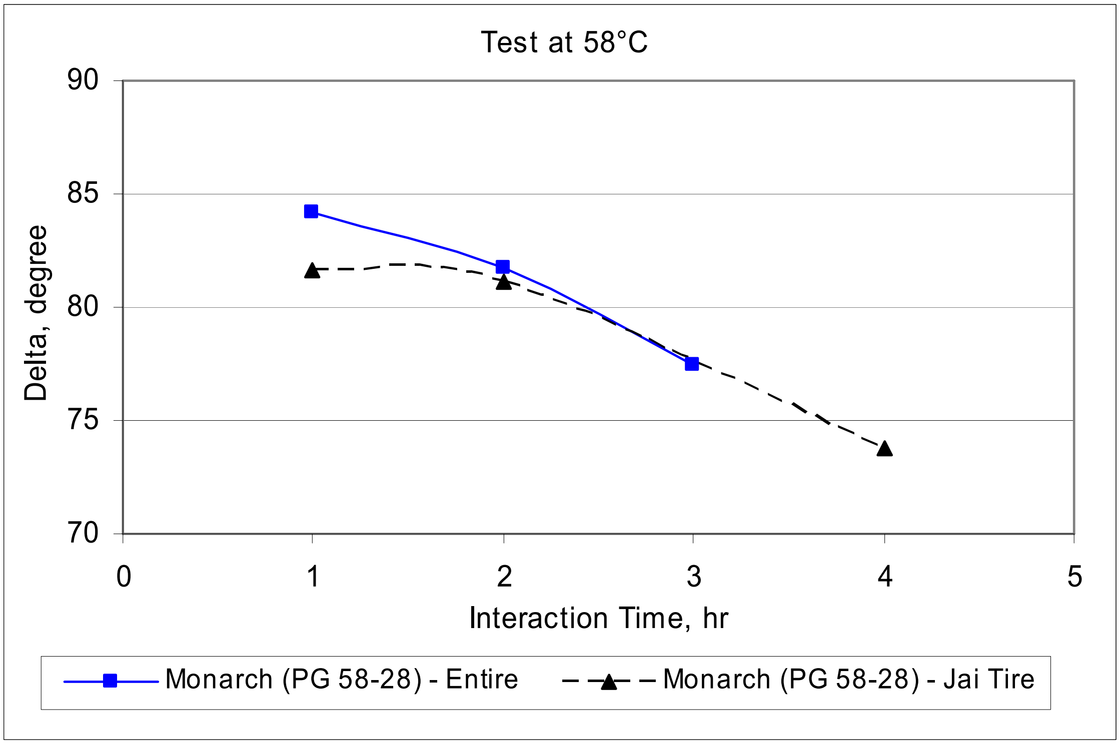
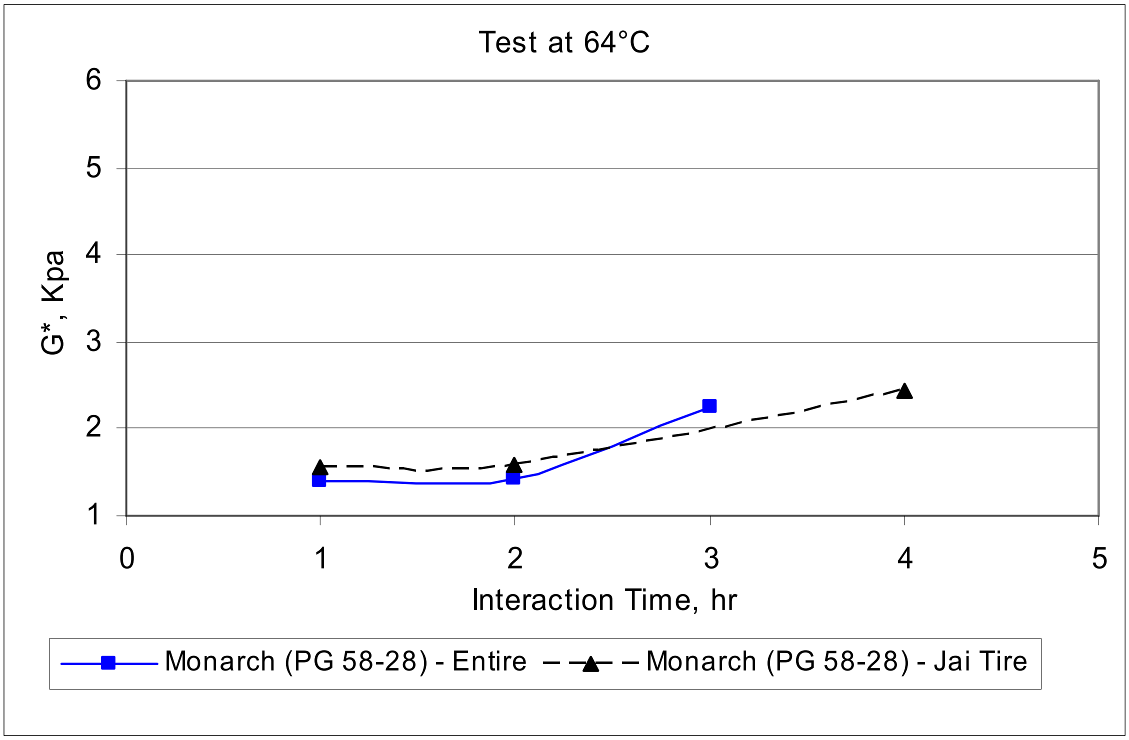


Figure 4-7-a Monarch (PG 58-28).



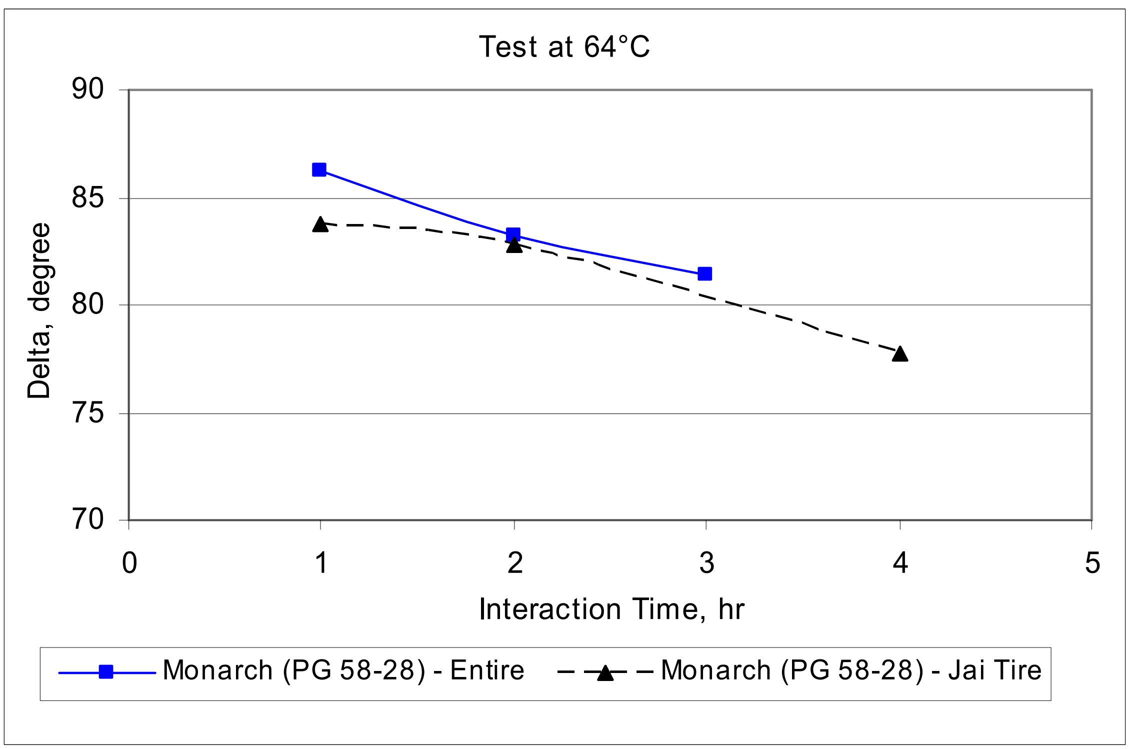
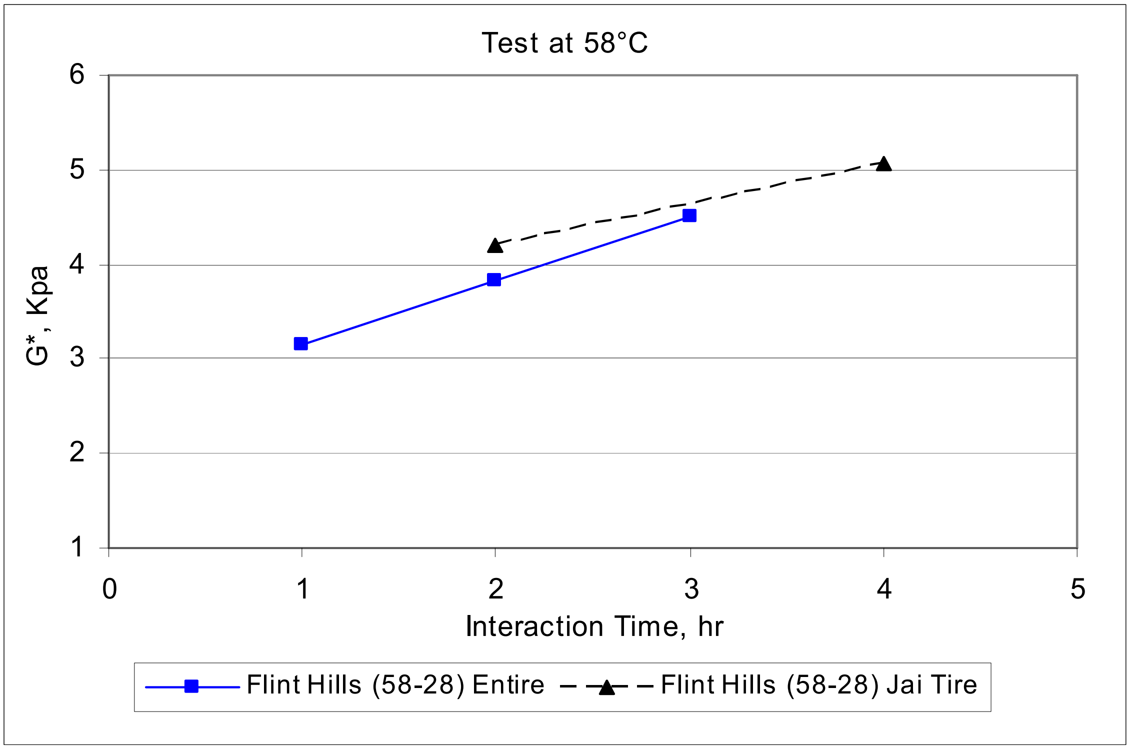


Figure 4-7-b Monarch (PG 58-28).



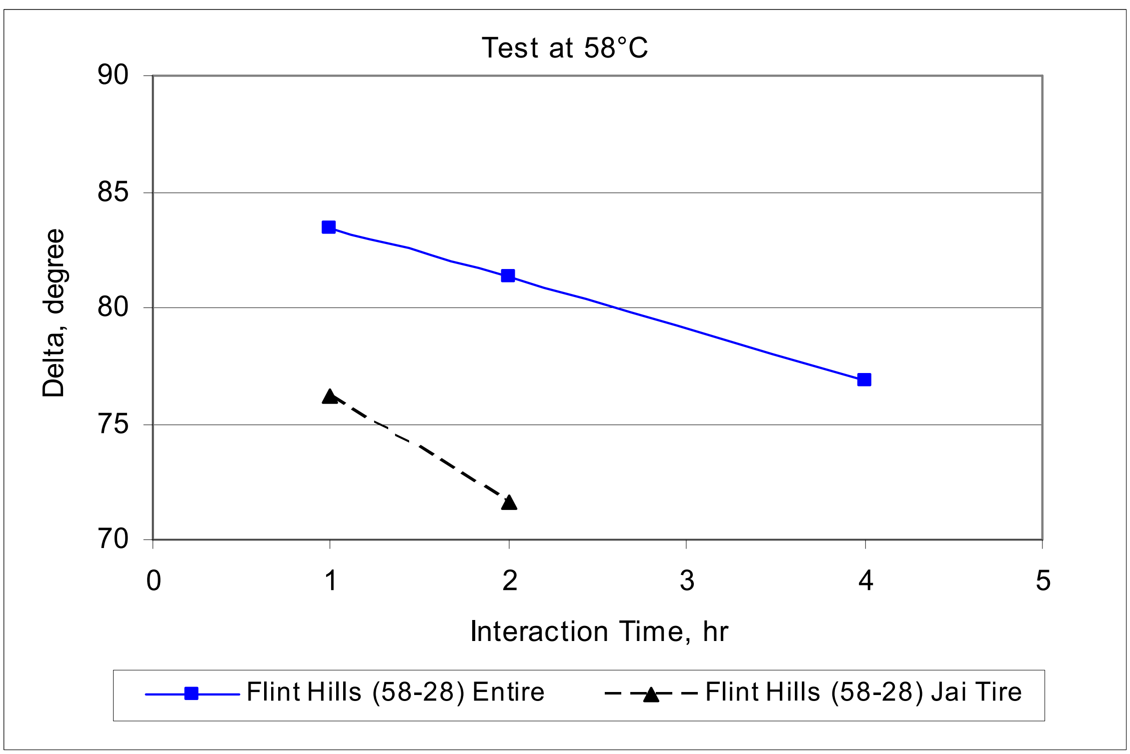
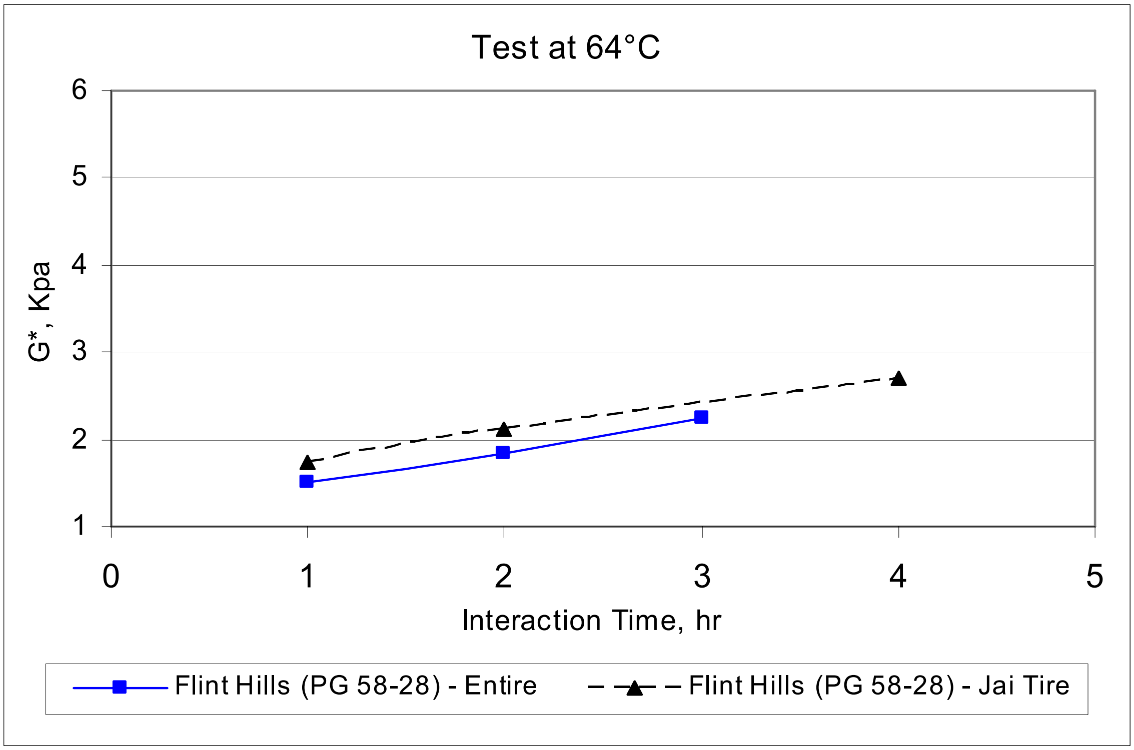


Figure 4-8-a Flint Hills (PG 58-28).



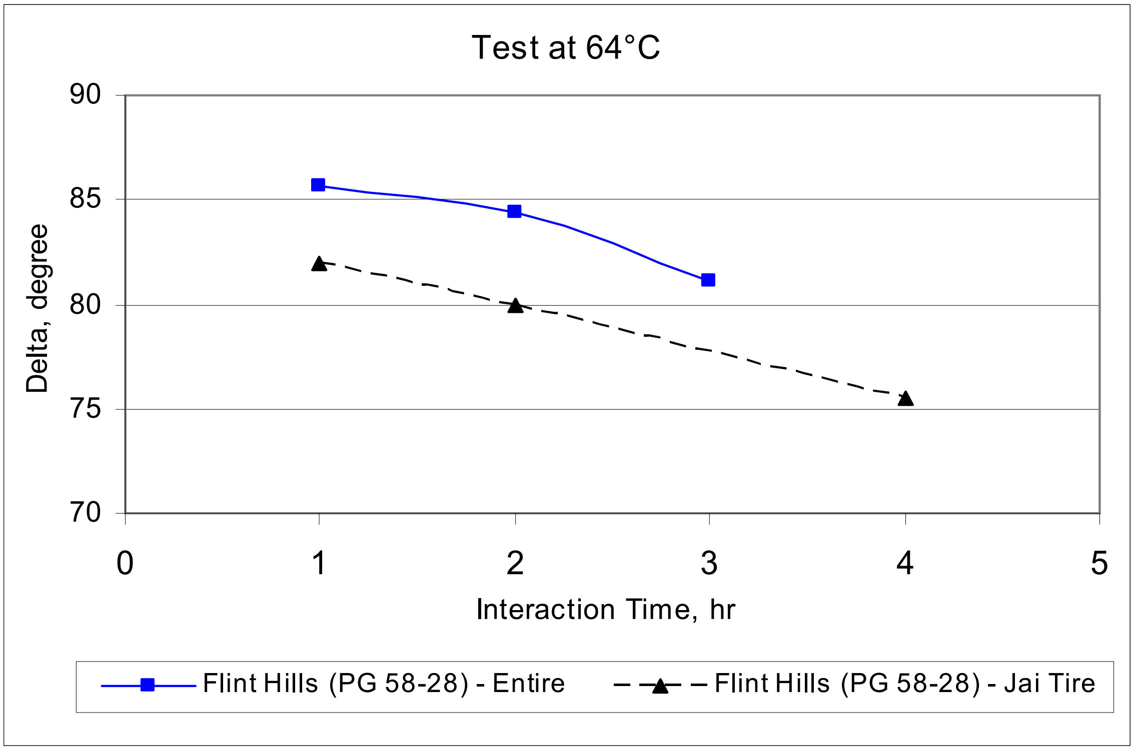


Figure 4-8-b Flint Hills (PG 58-28).

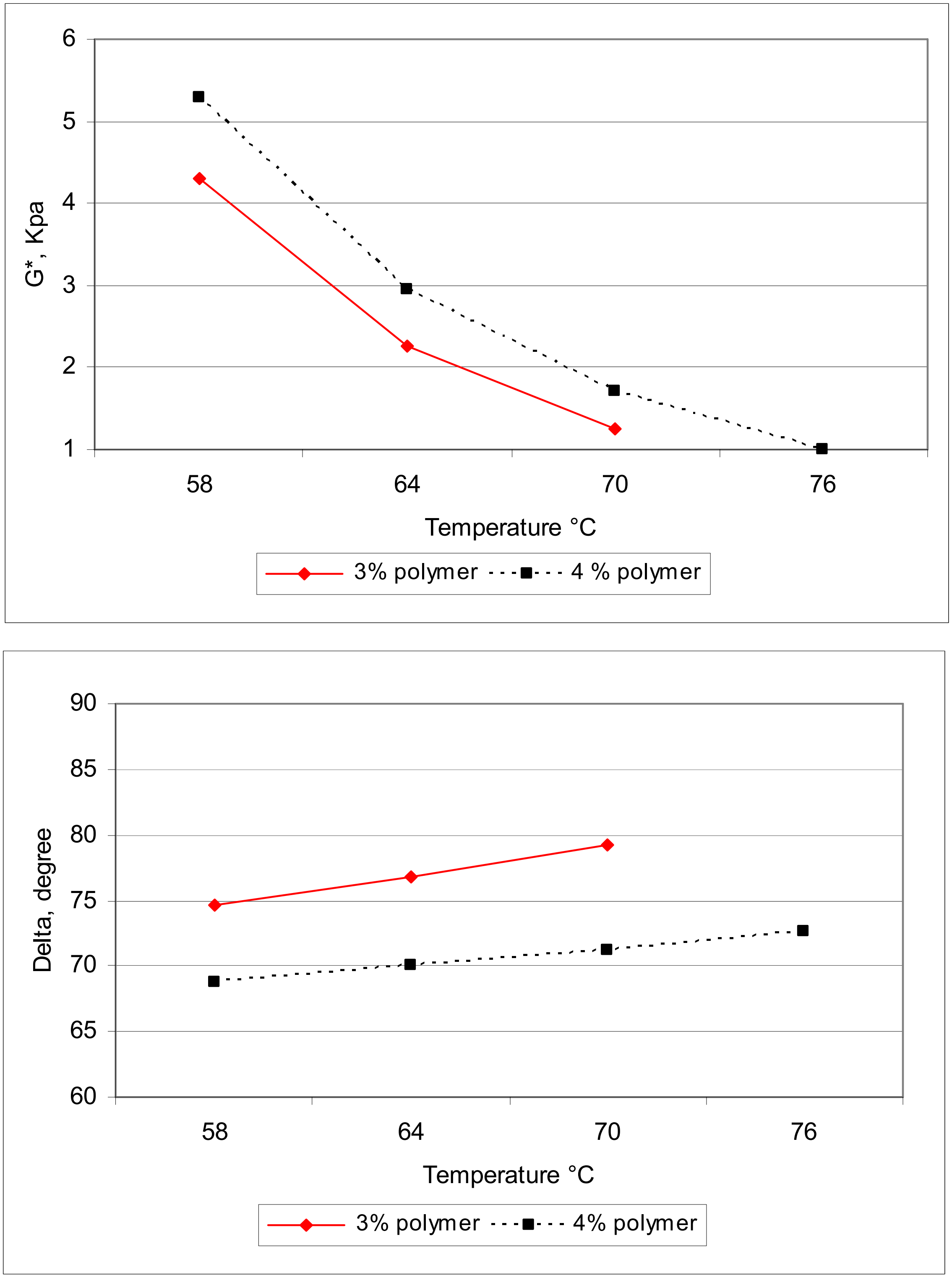


Figure 4-9-a Flint Hills (PG 58-28) with Firestone Polymer – Tank.

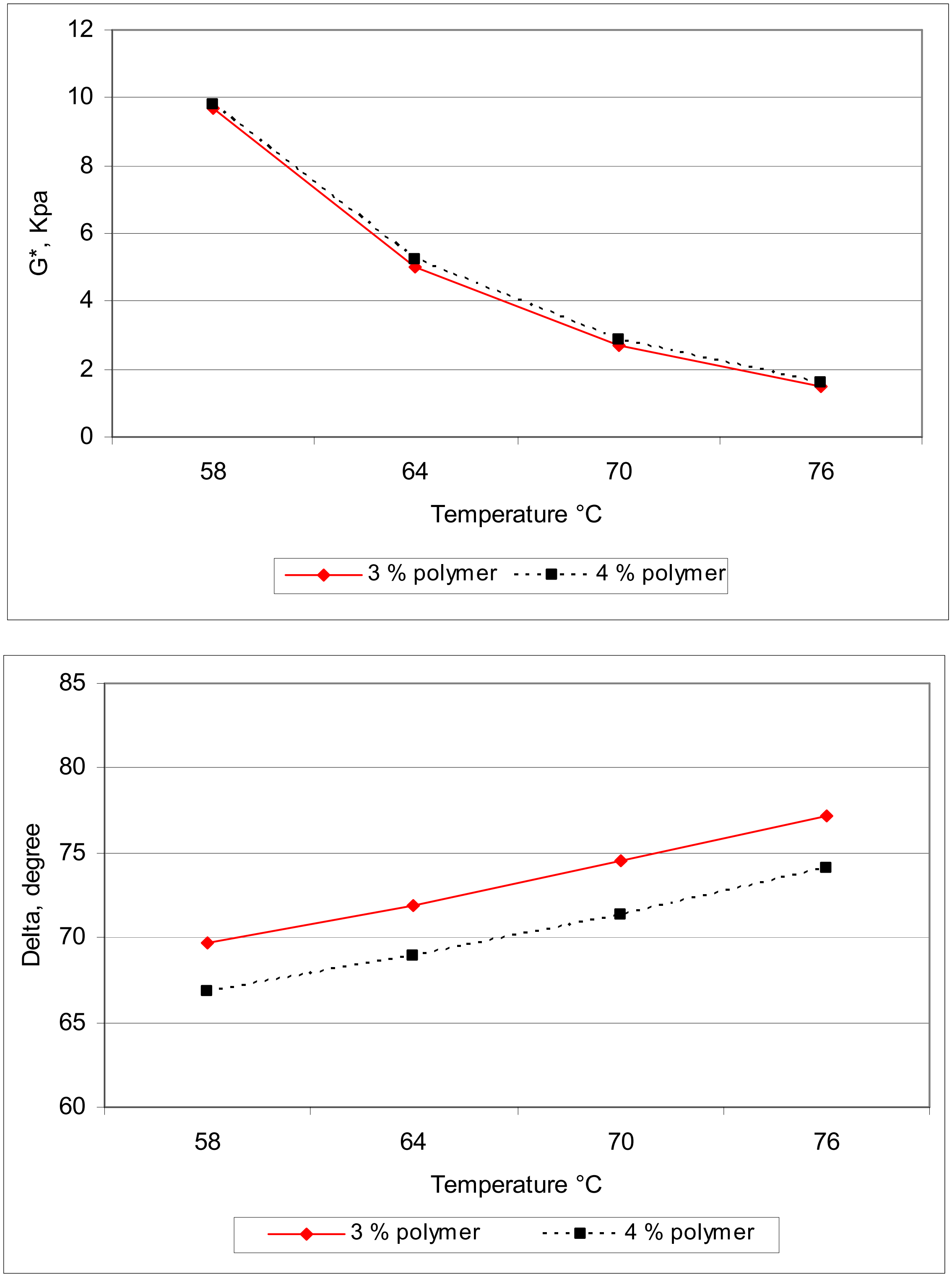
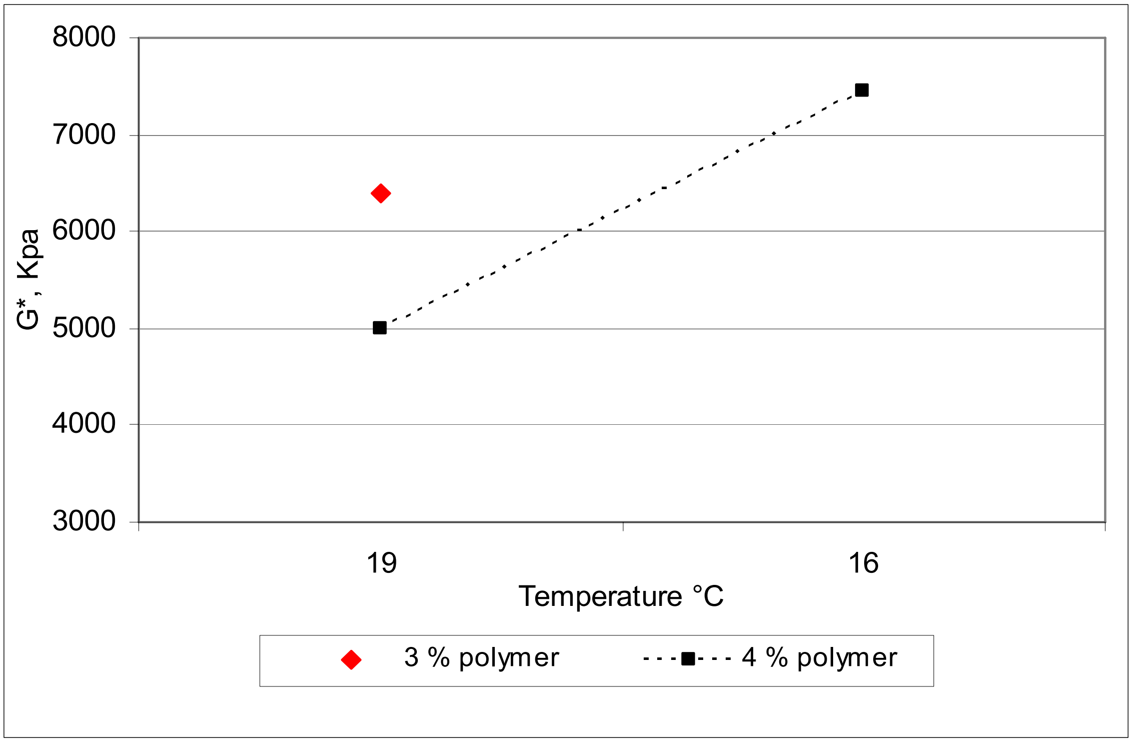


Figure 4-9-b Flint Hills (PG 58-28) with Firestone Polymer – R.T.F.O.



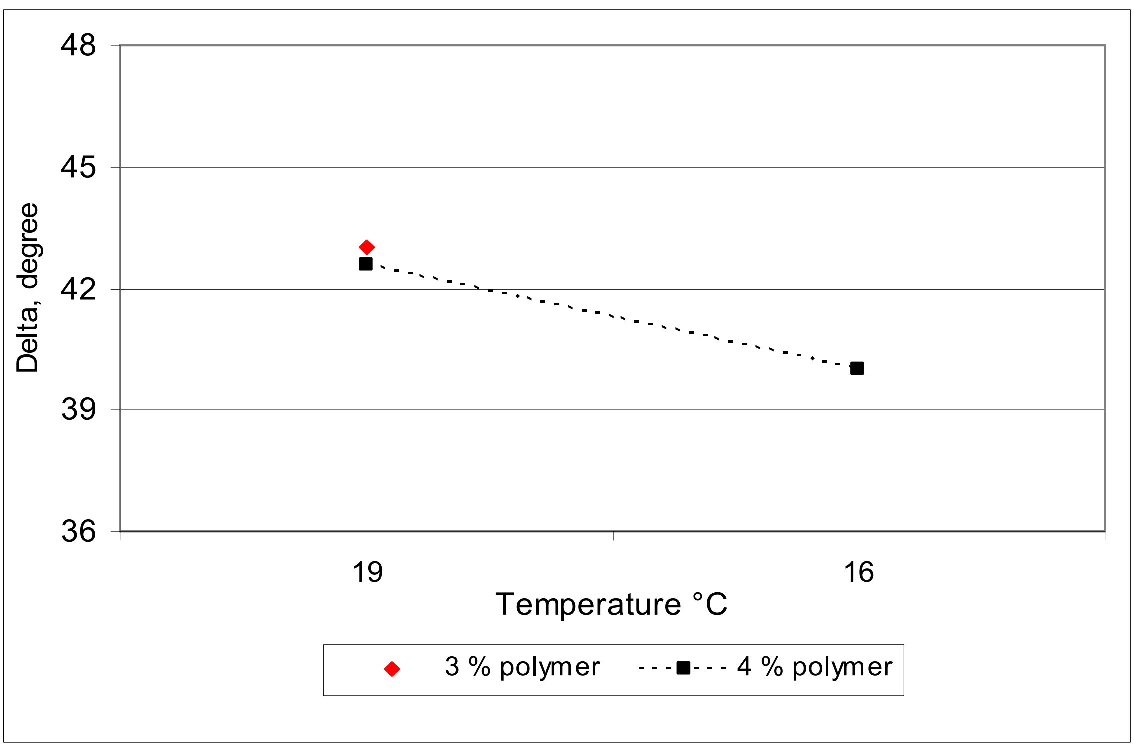
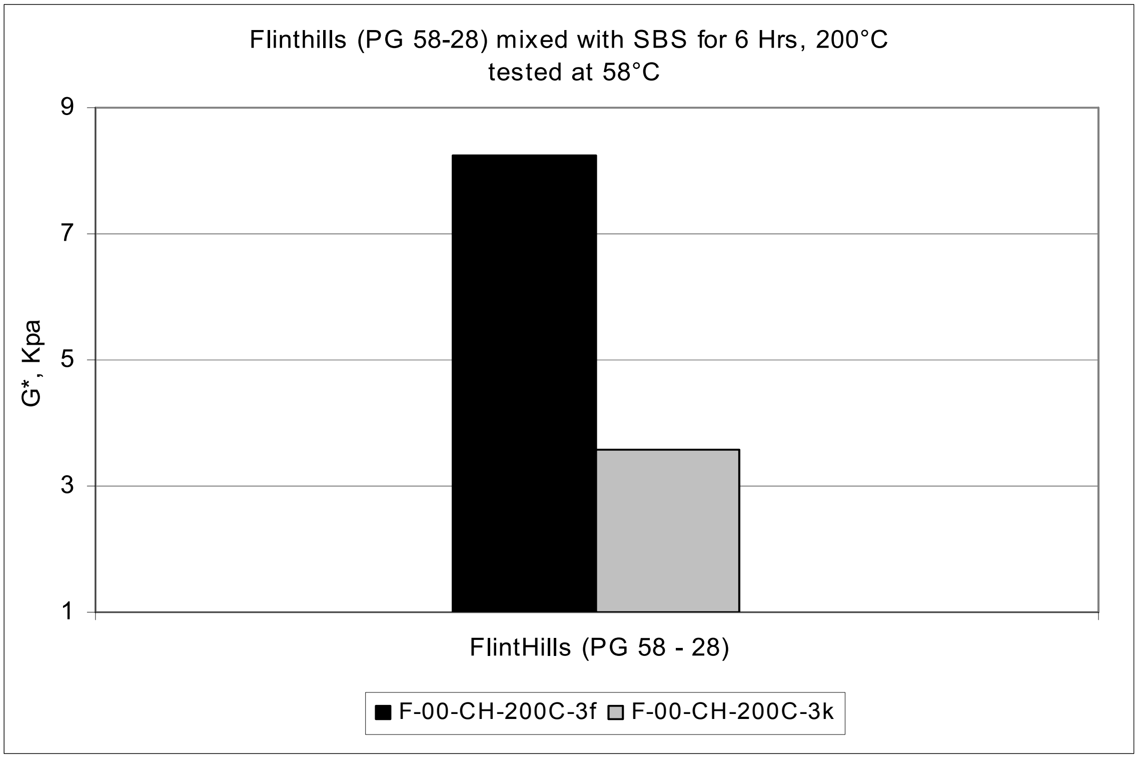


Figure 4-9-c Flint Hills (PG 58-28) with Firestone Polymer – P.A.V.



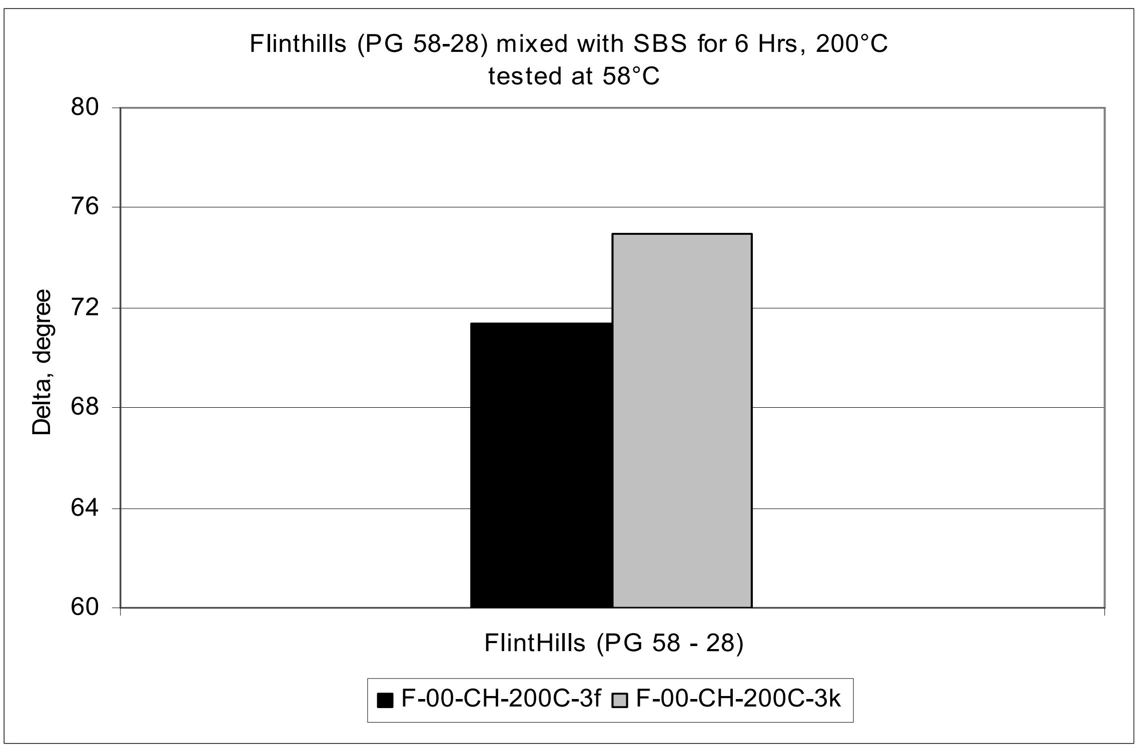
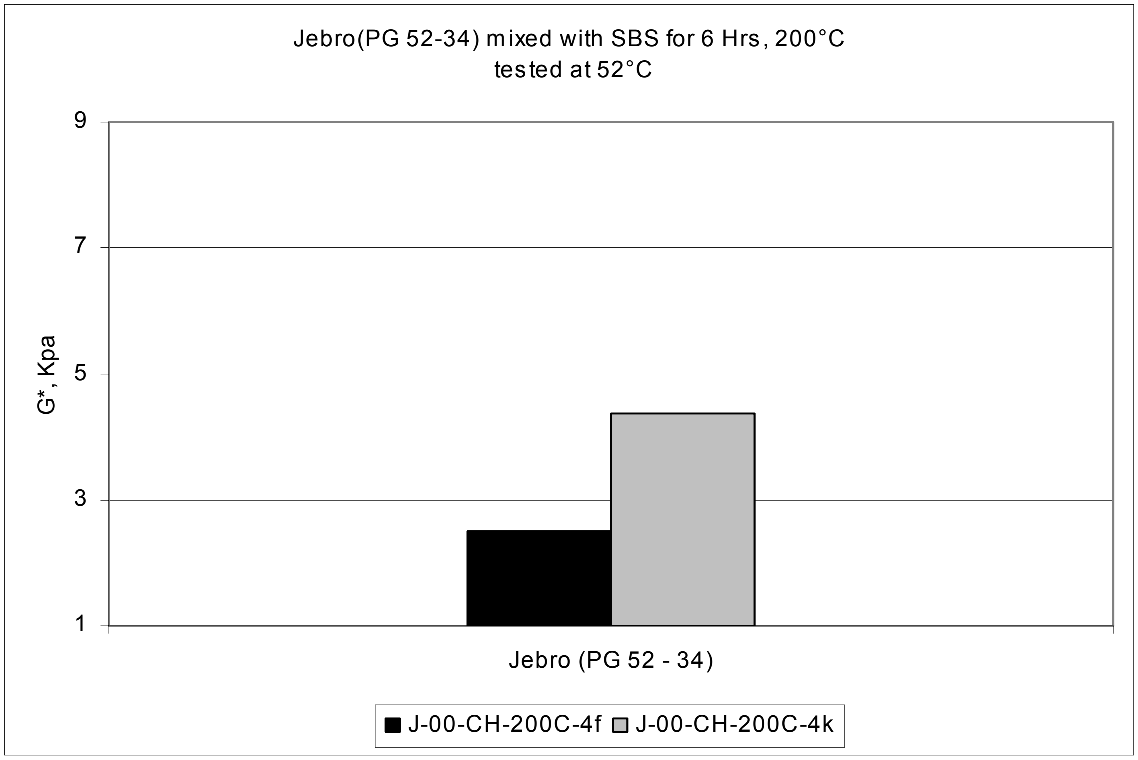


Figure 4-10 Flint Hills (PG 58-28) with 3% Polymer.



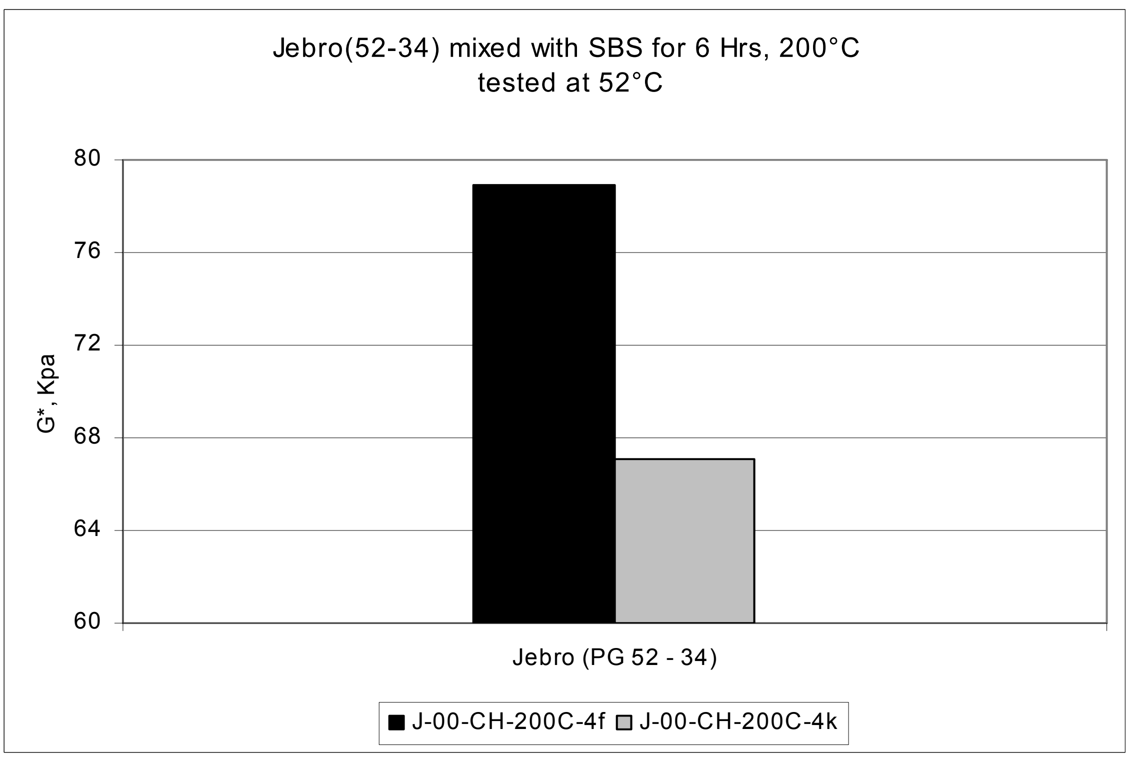


Figure 4-11 Jebro (PG 52-34) with 4% Polymer.

properties to provide a more detailed description of what can be accomplished with different combinations to produce an acceptable end result. The binder combinations tested in this study include additional rubber gradations and extended interaction conditions. This research could easily be extended into a more detailed study monitoring property progression during the initial interaction periods.

Controlling pavement performance through monitoring of CRM binder properties during production should be based upon DSR testing. Reliance on DSR data requires demonstration of the repeatability of DSR tests on CRM binders produced through the interaction process. Figures 4-1 through 4-8 show the variability of several asphalt-rubber combinations. Individual samples of the same material were taken from the same asphalt-rubber interaction and tested separately. The results were identical. Then samples were collected from two different interactions and tested. The results were very similar. Both long-term and short-term interactions are shown in Figures 4-1 through 4-8. Considering normal testing variability, the graphs demonstrate good repeatability, indicating that DSR testing procedures are valid.

4.2 Polymer Modifications

Figures 4-9 through 4-11 show modifications obtained using two different polymer modifiers from two different manufacturers, Firestone® and Kraton®. Effectiveness of the two modifiers depends on the polymer content and varied for each asphalt source. The results show that a 3% to 4% of SBS is sufficient to modify the PG 58 binder grade to a PG 70 grade. However, 3% to 4% SBS also affects the low temperature grade, changing it from -28 to -22. Softer asphalt, Jebro PG 52-34 maintained its low temperature properties with addition of polymer but changes in its high temperature properties were not as significant. Modifying PG 52-34 with 4% polymer improved the high temperature grade to PG 64 but reduced the low temperature grade to -28.

4.3 Property Development Experiment

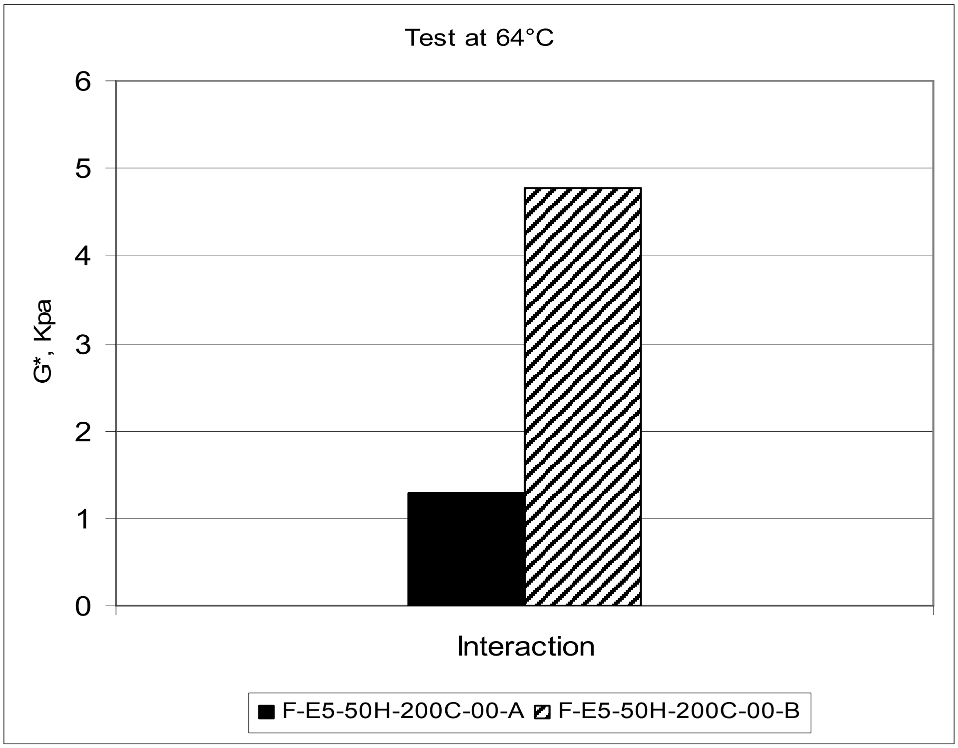
One objective of this research was to investigate basic performance-related properties of CRM binders similar to those specified using the Superpave system. Experiments examined the development of binder properties under precisely controlled interaction conditions immediately and for several hours after mixing the crumb rubber with asphalt. It showed that most property modifications could occur as early as the first 30 minutes of interaction and illustrated that most property modification occurs during short-term interactions within the first few minutes of interaction time. While this experiment focuses on the short-term interactions between asphalt and rubber, selected long-term interactions (up to several hours) were utilized to further characterize binder behavior under variations of interaction conditions. Two different CRM material sources, (Entire and Jai Tire) and one asphalt source (Flint Hills PG 58-28) were used in the reactivity interactions. Each crumb rubber was interacted as it was received from the manufacturer with particle size gradations unmodified by sieving. Figure 3.4 showed the

particle size distributions of each CRM source. The two crumb rubber sources cover the approximate range of crumb rubber gradations commonly marketed today. Effect of the crumb rubber particle size on the developed properties is discussed in section 4.4.

Figures 4 -12 through 4 -19 illustrate the development of G* and δ properties plus the results of separation testing. Crumb rubber concentration was shown to be an effective factor in property development, more effective than crumb rubber source or method of processing. Rubber concentration must be optimized for other binder properties (i.e. stability). Crumb rubber was tested at three concentrations (5%, 7.5%, and 10%) to show the effects that rubber concentration had on the developed properties. Results of the 7.5% and 10% CRM are shown in Figures 4 -12 to 4 -19.

Comparing the effect of crumb rubber concentration from different sources illustrates changes in developed properties as interaction conditions change. Higher crumb rubber concentrations have more effect on the high temperature properties than on low temperature properties. Increasing crumb rubber concentration increases the amount of the light asphalt fractions absorbed by rubber particles, stiffening the binder more than a low crumb rubber concentration. Increasing the crumb rubber concentration congests the binder matrix with swollen rubber particles. While this increases the modification of the developed property, it also produces a binder that is more affected by the high interaction temperature as swollen rubber particles depolymerize. The differences in material source behavior noted earlier were not altered by varying crumb rubber concentrations. For the 10% crumb rubber concentration, the values of δ were higher throughout the interaction period. The variations in property development were very limited at higher crumb rubber concentrations compared to lower concentrations for both crumb rubber sources. Higher crumb rubber concentration produces lower δ than the other two concentrations. The extraction of the light asphalt fractions through rubber particle swelling may be a factor affecting the development of the phase angle.

Stability testing demonstrates that 5% CRM produces acceptable separation test results and produces, in conjunction with 2% SBS, Superpave properties. The “5% CRM + 2% SBS” upgraded the high temperature of the PG 58-28 two grades, creating PG 70 binder. This is almost identical to the effect of 4% SBS with PG 58-28. Higher CRM concentrations (above 5%) produce more significant modifications to binder properties, but stability and workability of the binder will not be as desirable as with 5% crumb rubber. In most cases, a change of negative one grade resulted in low temperature properties of binders when CR concentrations above 5% were tested. The same effect was noticed with SBS only modifications. PG 58-28 asphalt interacted with 5% CRM and 2% SBS results in PG 70-22. PG 52-34 interacted with 5% CRM and 2% SBS results in PG 64-28.



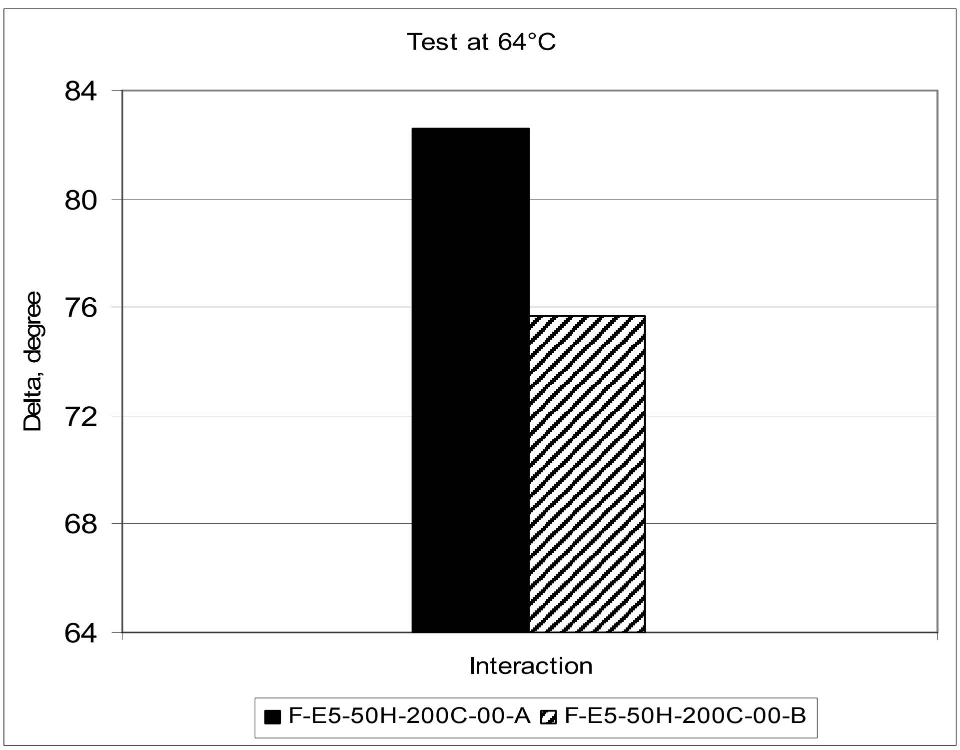


Figure 4-12-a Property Development of Flint Hills (PG 58-28) AC + 5% Entire CRM using Different Interaction Procedures.

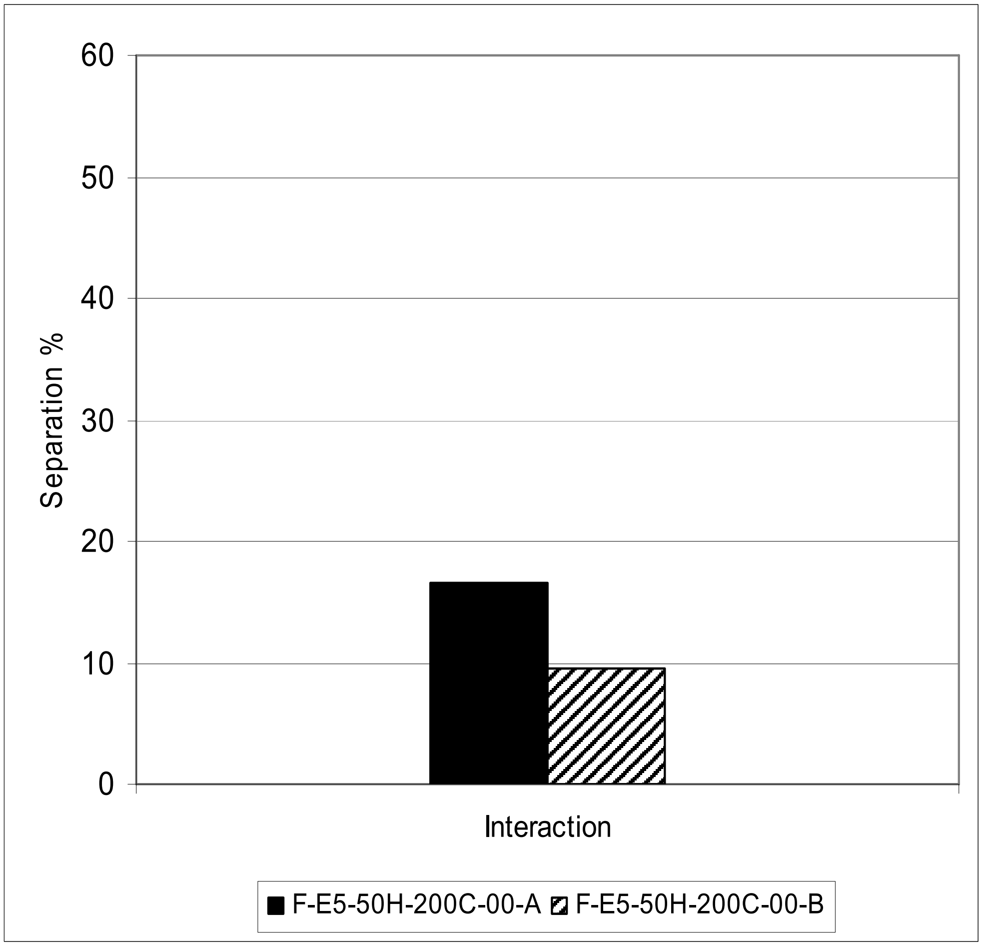
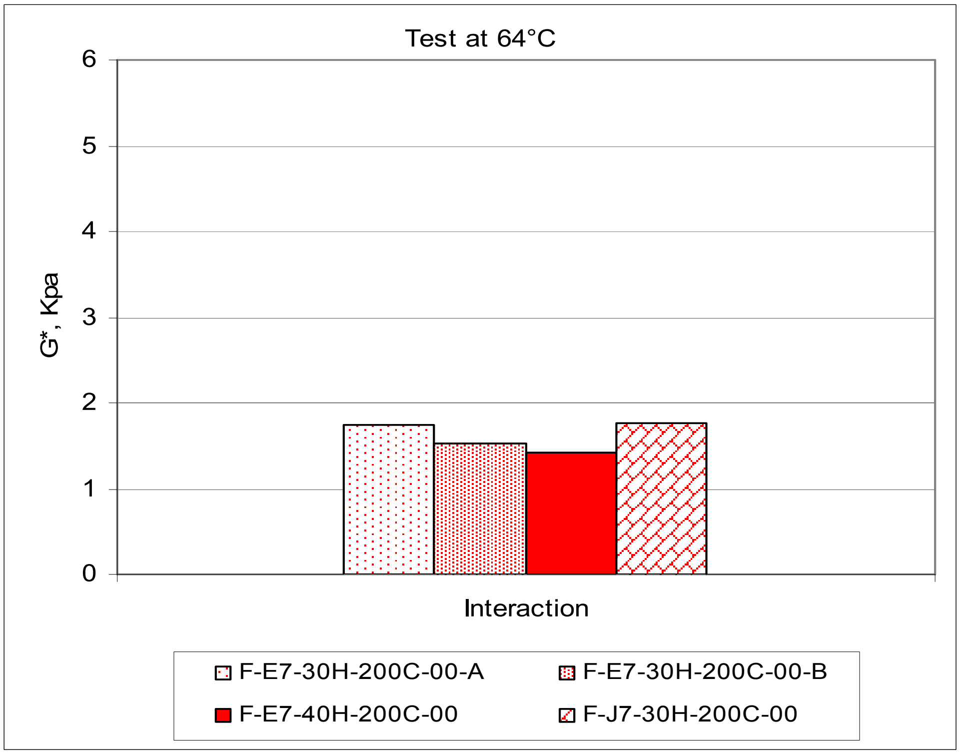


Figure 4-12-b Separation Percentage of Flint Hills (PG 58-28) AC + 5% CRM using Different Interaction Procedures.



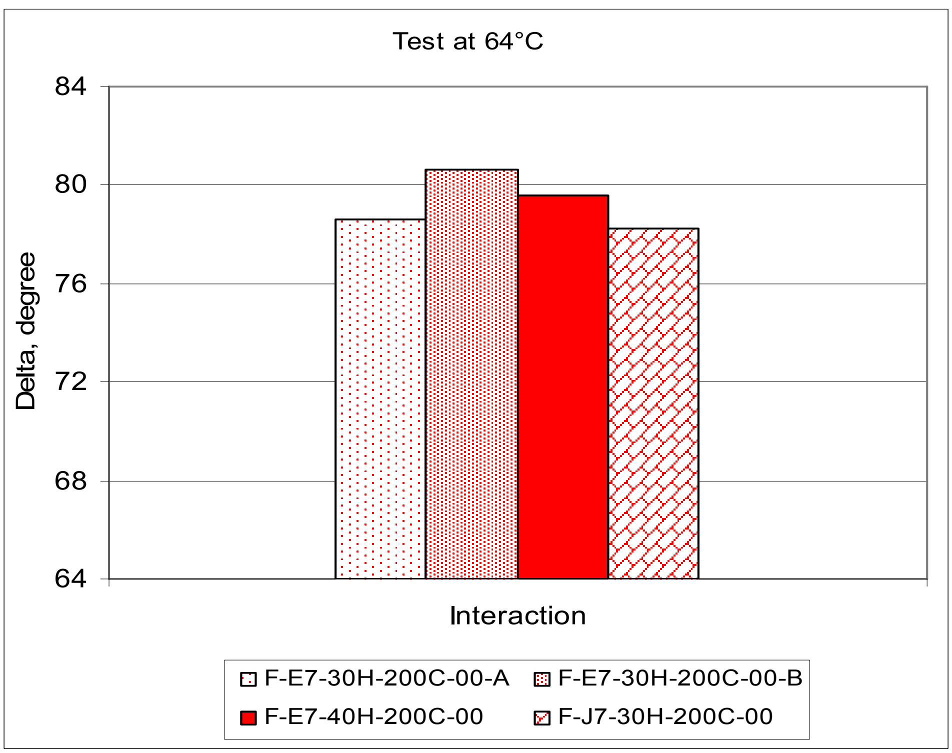


Figure 4-13-a Property Development of Flint Hills (PG 58-28) AC + 7.5% CRM using Different Interaction Procedures.

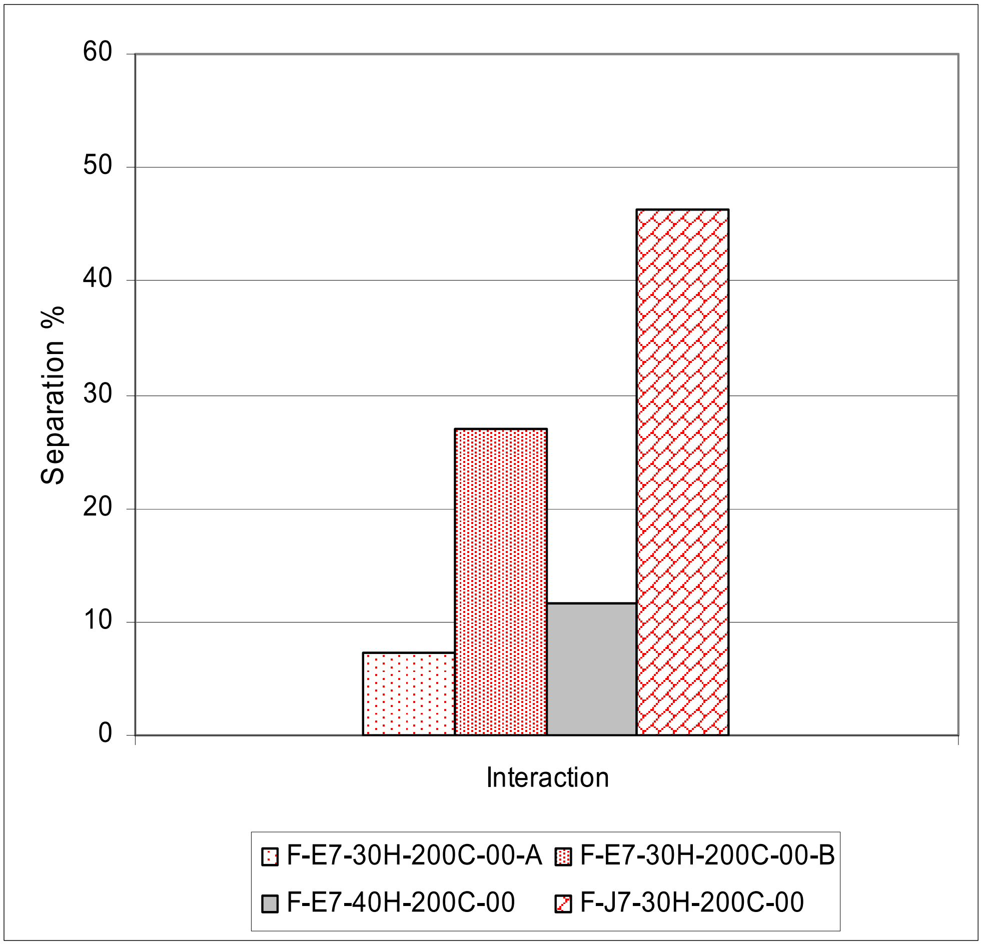
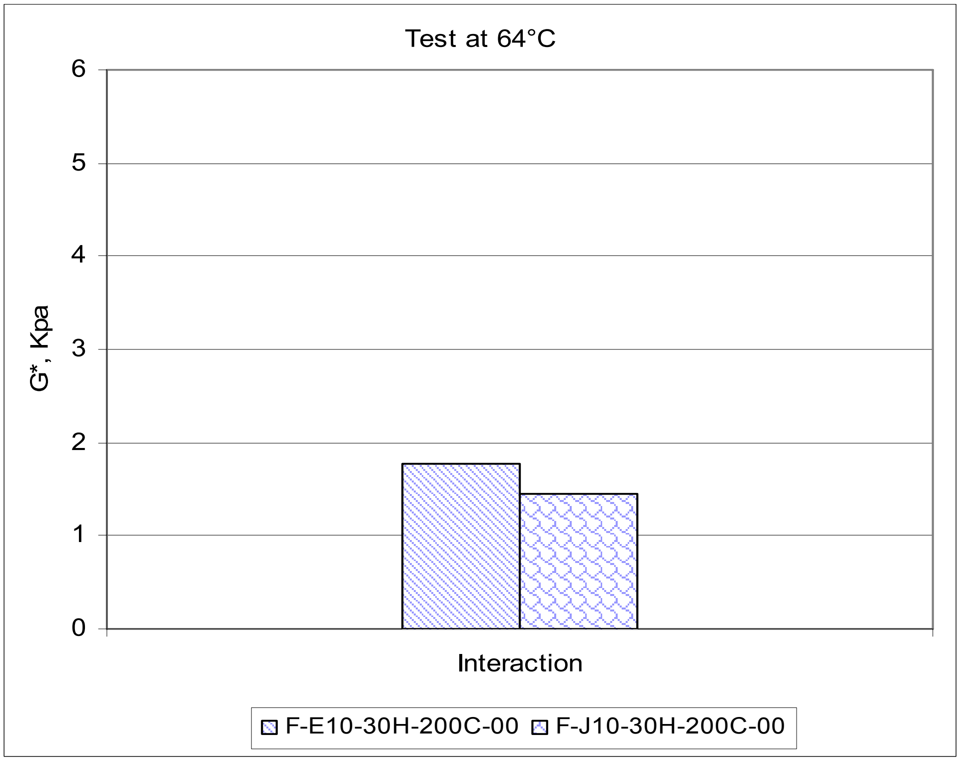


Figure 4-13-b Separation Percentage of Flint Hills (PG 58-28) AC + 7.5% CRM using Different Interaction Procedures.



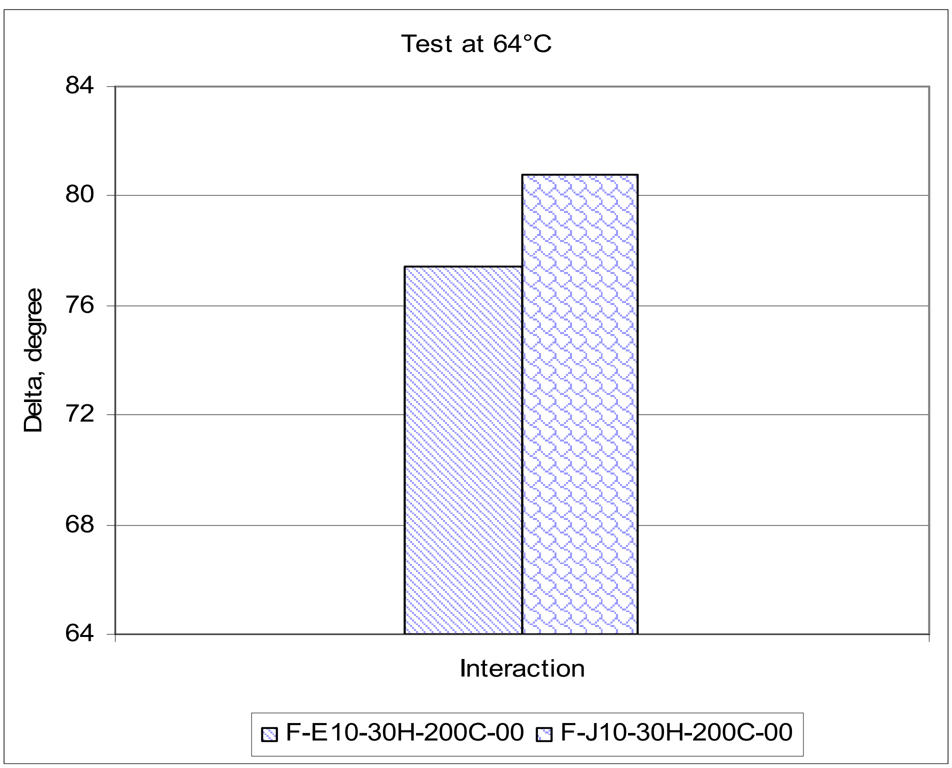


Figure 4-14-a Property Development of Flint Hills (PG 58-28) AC + 7.5% CRM using Different Interaction Procedures.

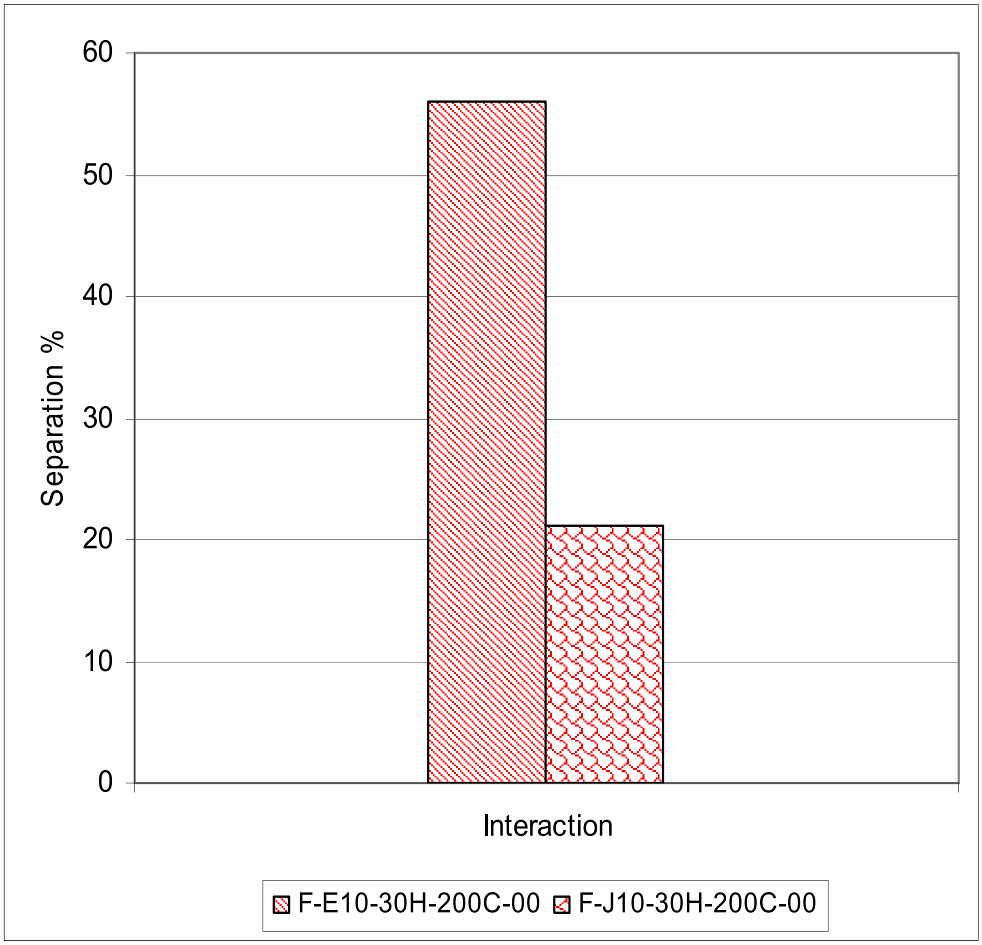
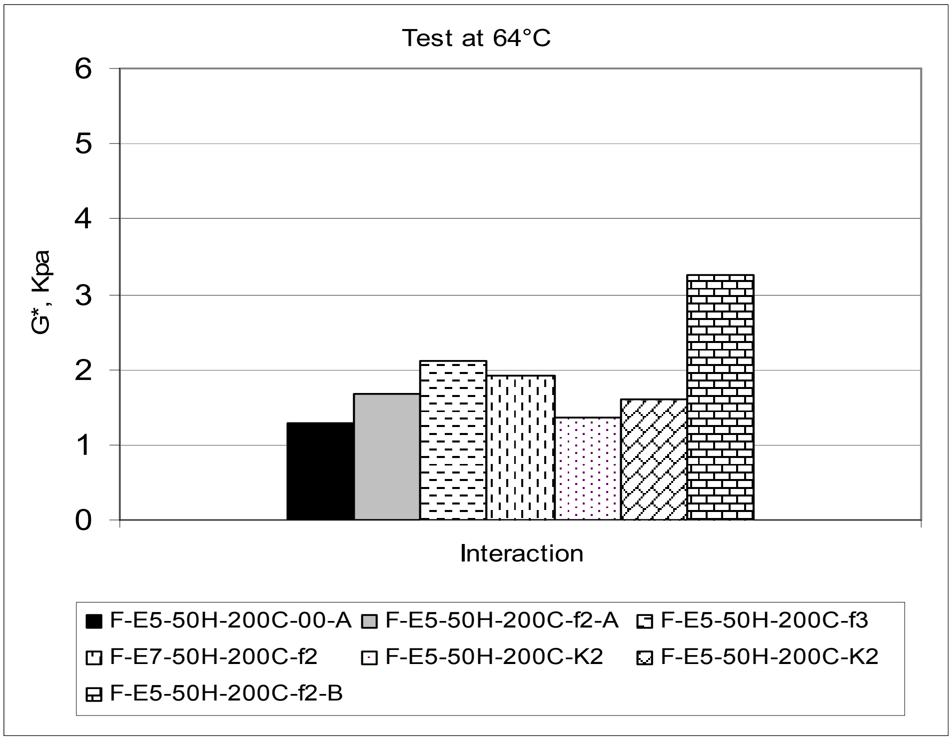


Figure 4-14-b Separation Percentage of Flint Hills (PG 58-28) AC + 7.5% CRM using Different Interaction Procedures.



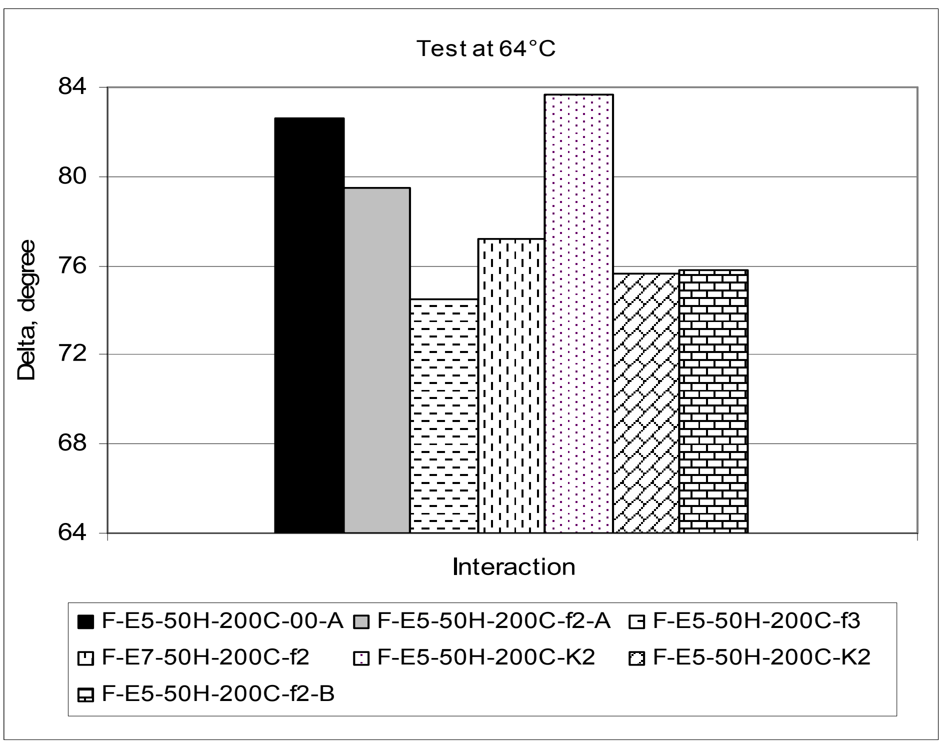


Figure 4-15-a Property Development of Flint Hills (PG 58-28) using the Same Interaction Procedures for Different Percentages of CRM and Different SBS.

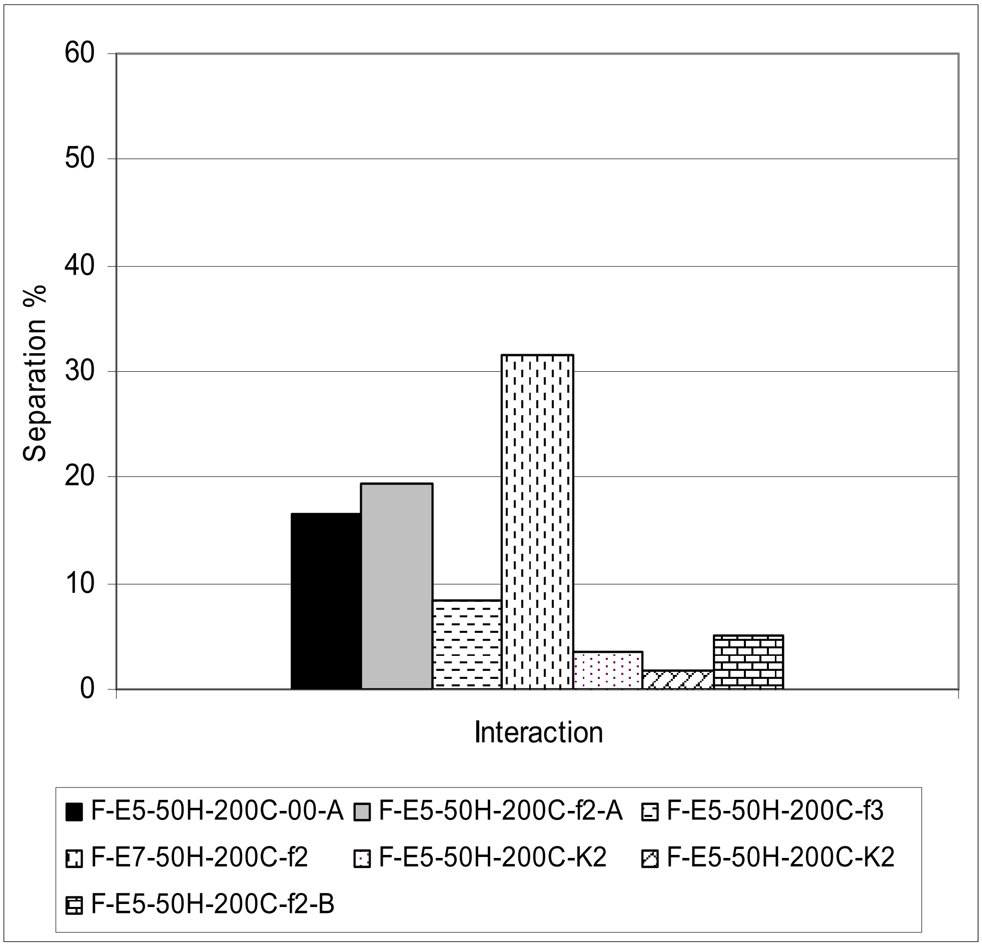
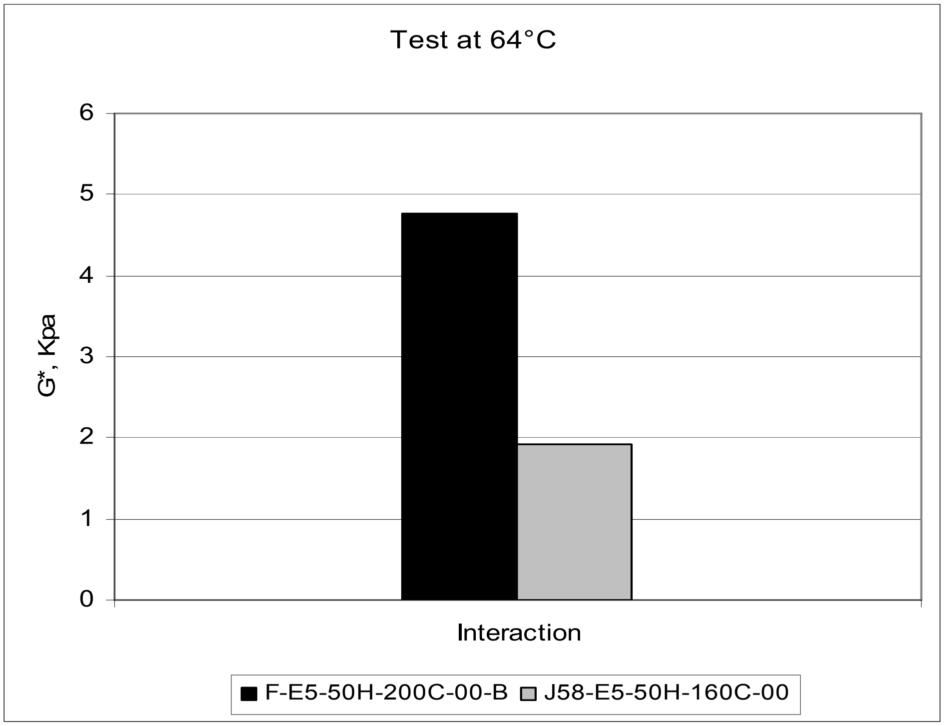


Figure 4-15-b Separation Percentage of Flint Hills (PG 58-28) AC using the Same Interaction Procedures for Different Percentages of CRM and Different SBS.



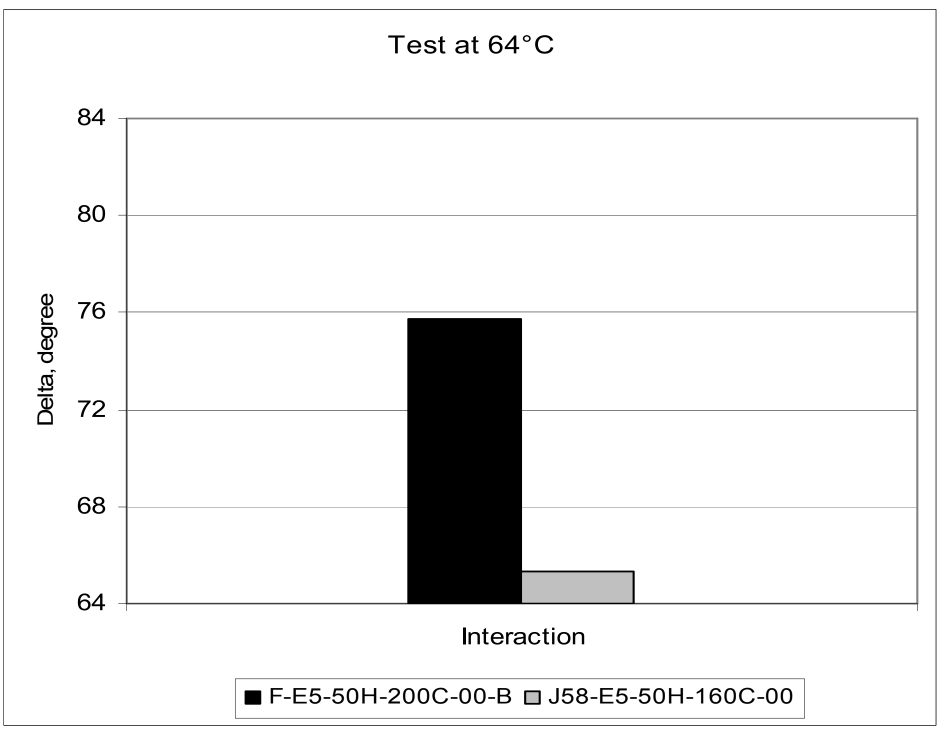


Figure 4-16-a Property Development of Flint Hills (PG 58-28) and Jebro (PG 58-34) AC.

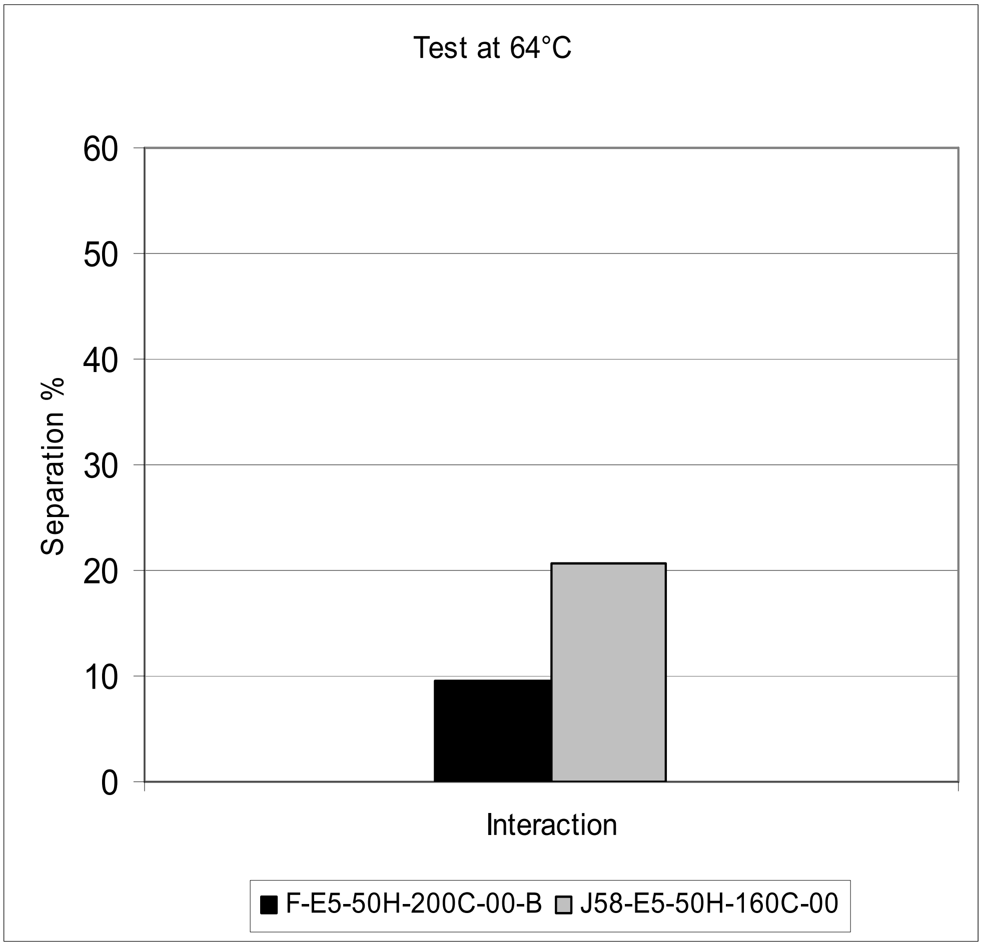
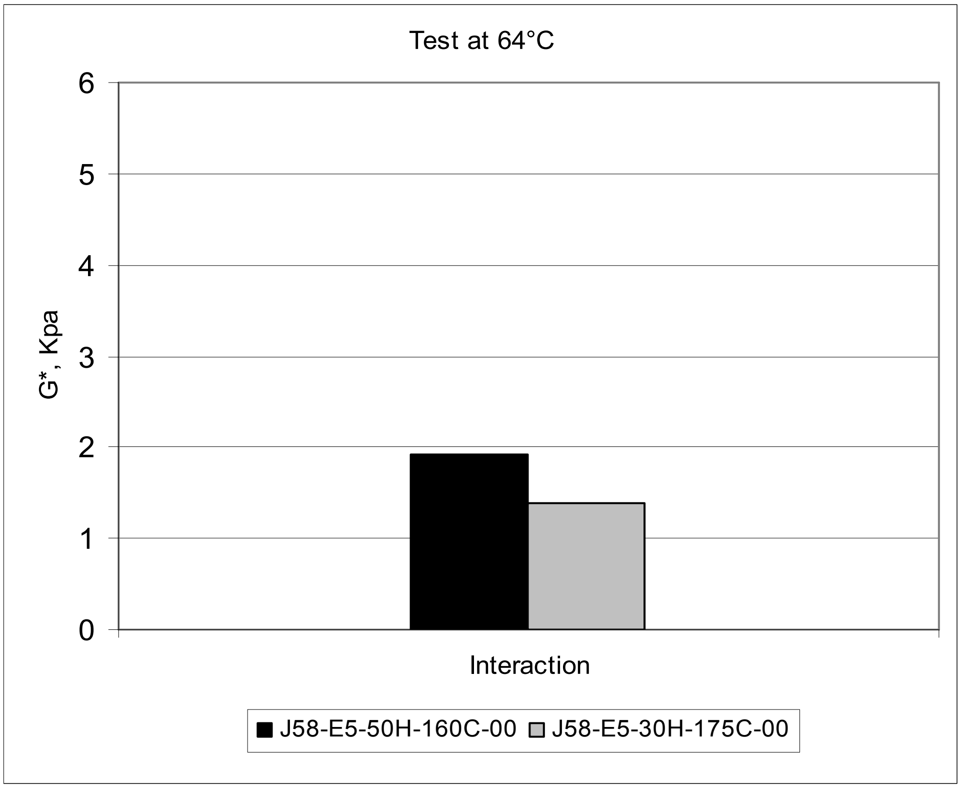


Figure 4-16-b Separation Percentage of Flint Hills (PG 58-28) and Jebro (PG 58-34) AC.



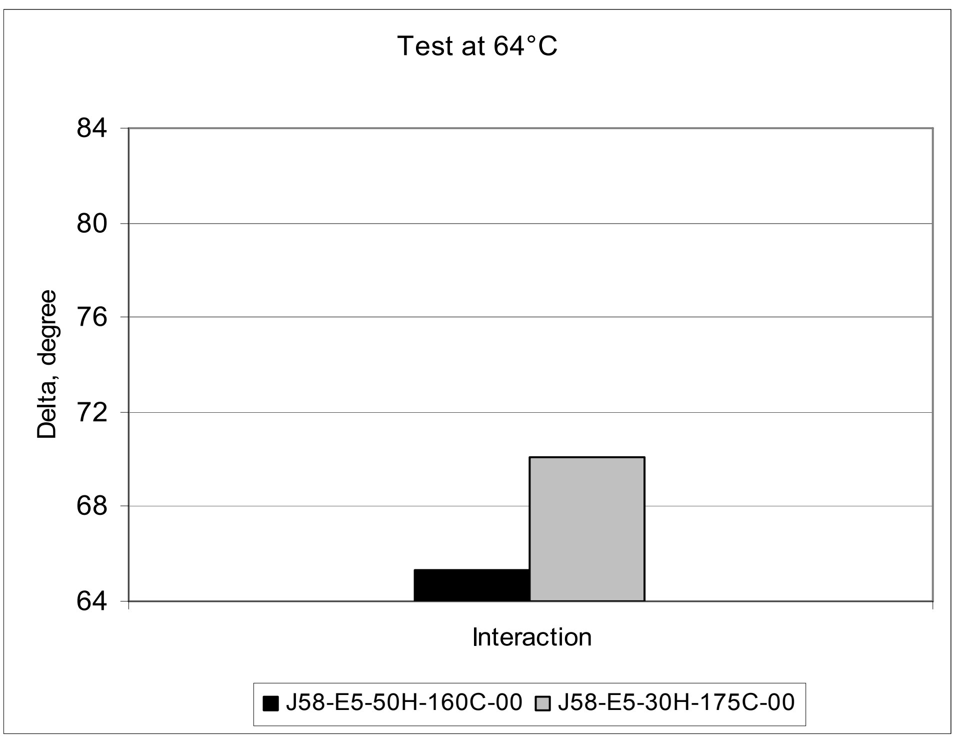


Figure 4-17-a Property Development of Jebro (PG 58-34) AC + 5% CRM.

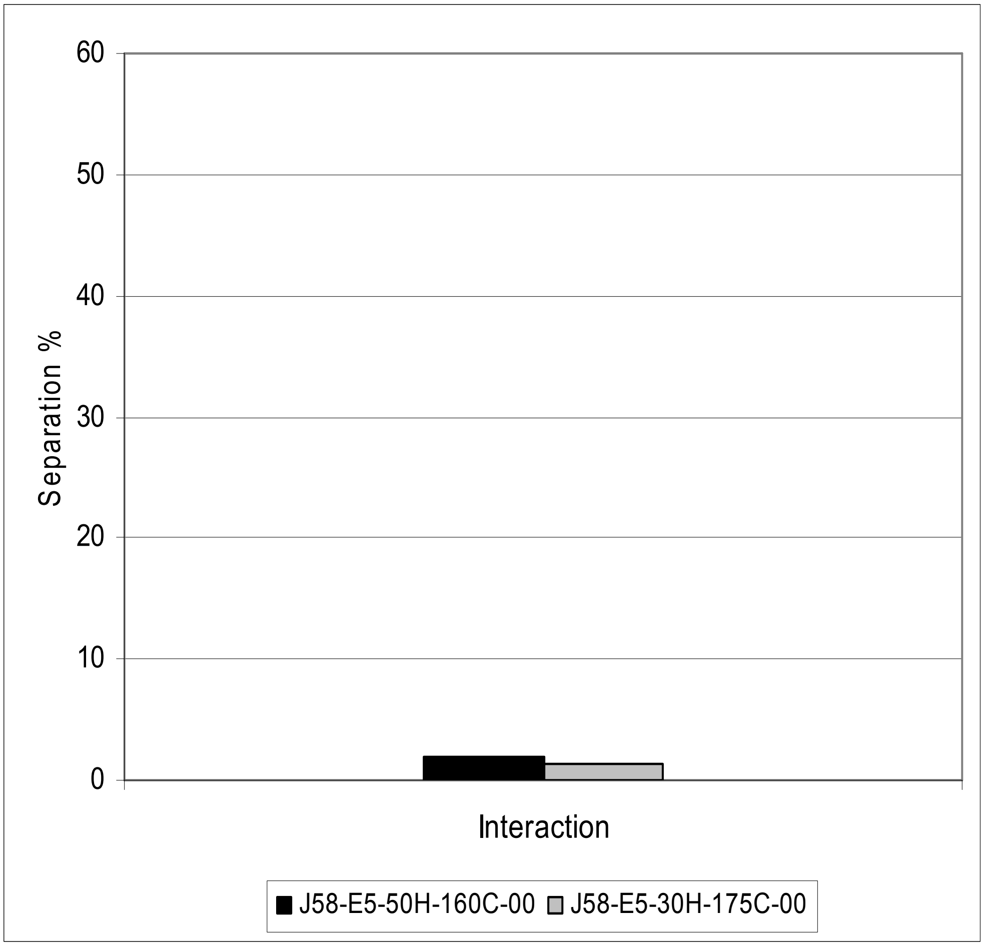
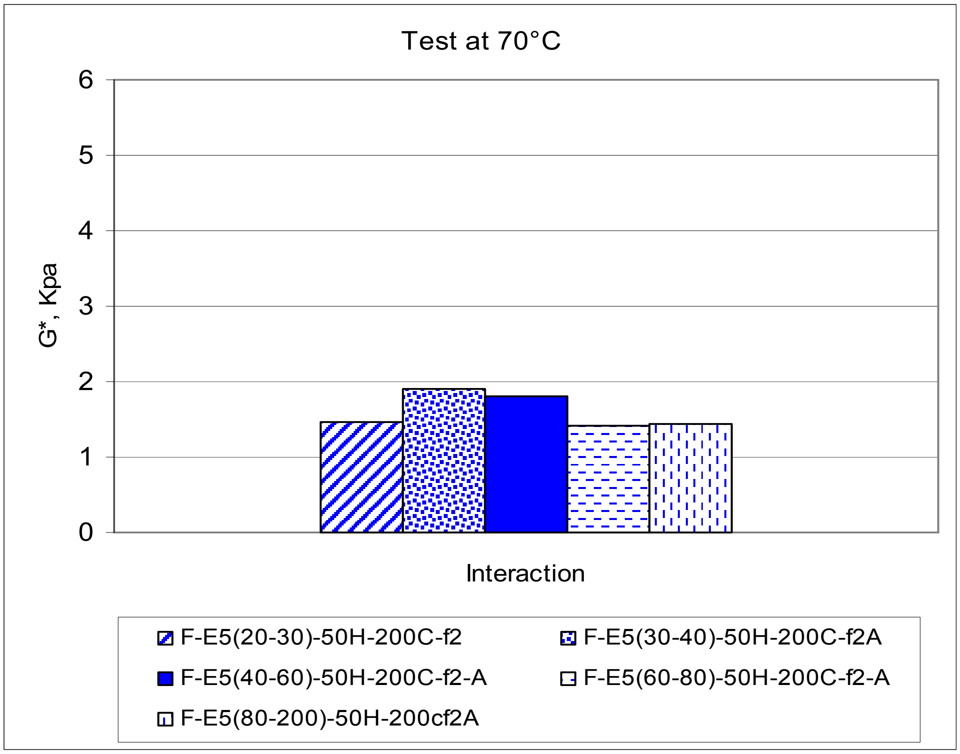


Figure 4-17-b Separation Percentage of Jebro (PG 58-34) AC + 5% CRM.



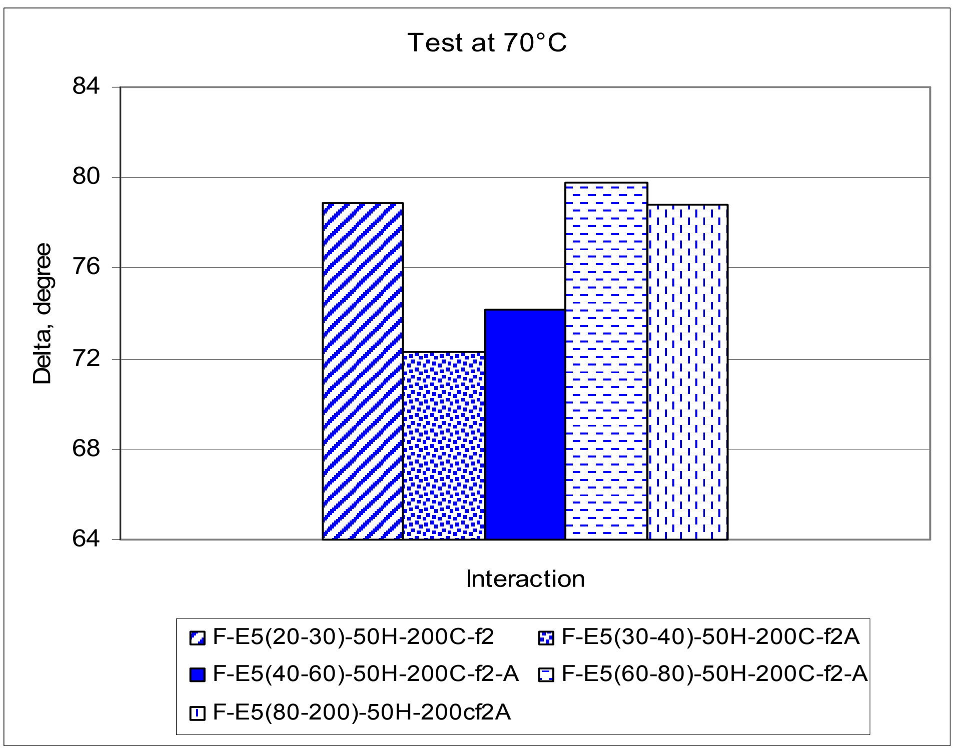


Figure 4-18-a Property Development of Flint Hills (PG 58-28) AC + 5% Entire CRM for Different Particle Sizes.

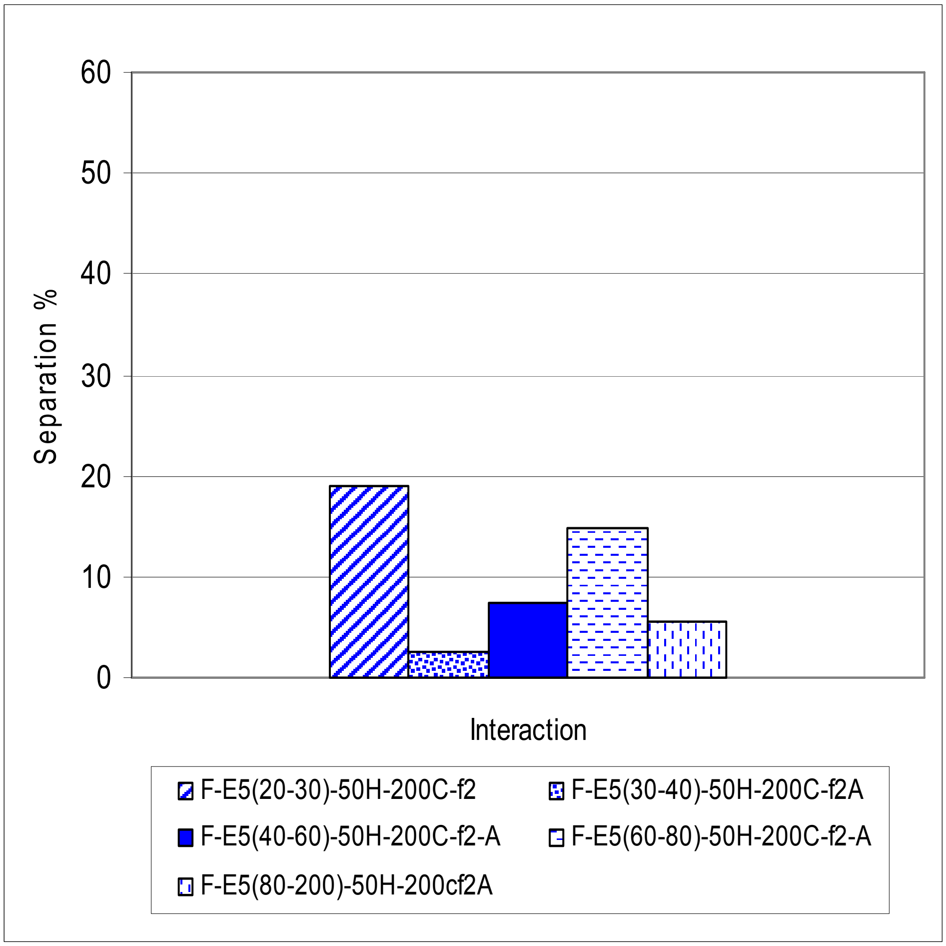


Figure 4-18-b Separation Percentage of Flint Hills (PG 58-28) AC + 5% Entire CRM for Different Particle Sizes.

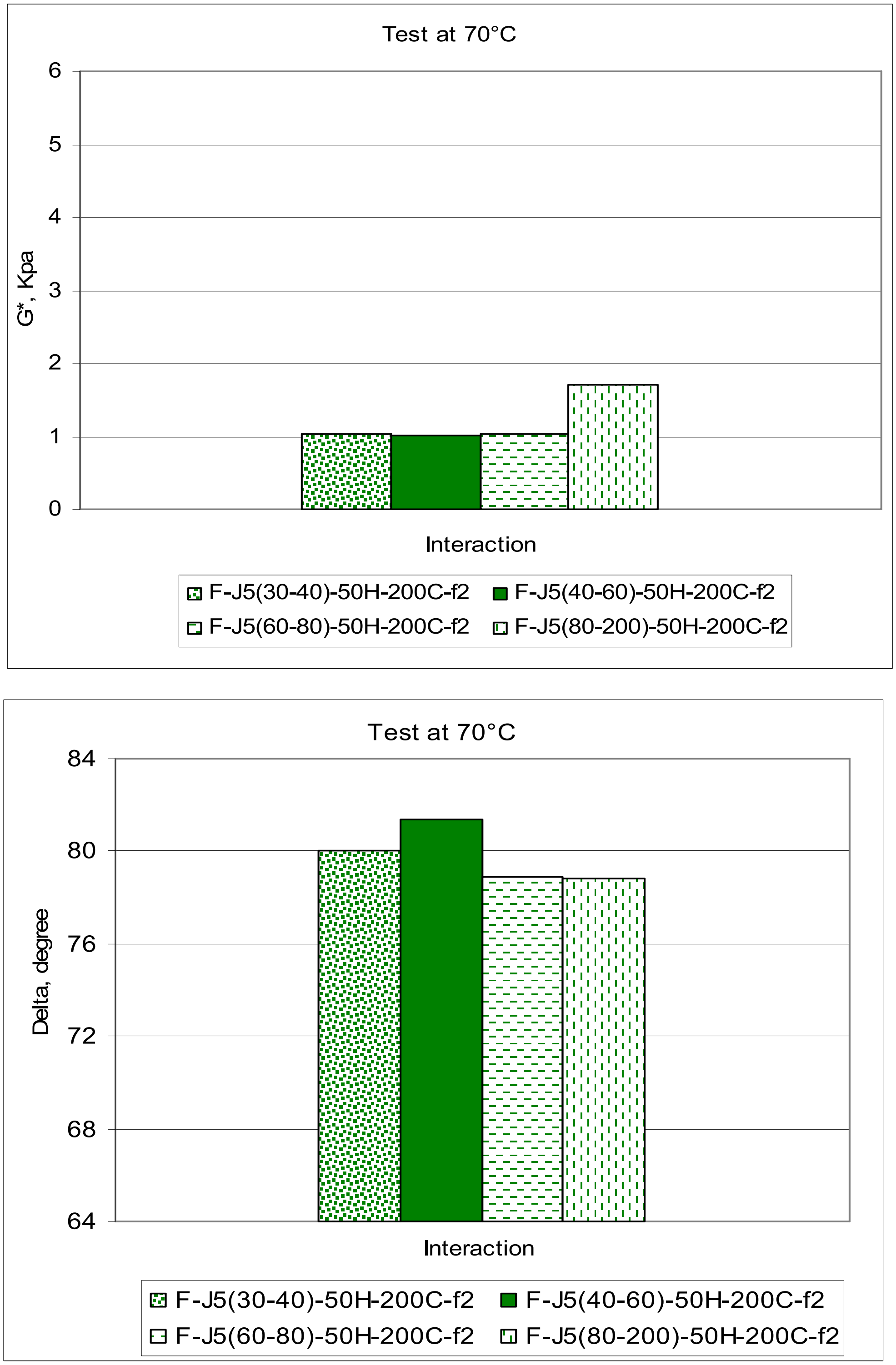


Figure 4-19-a Property Development of Flint Hills (PG 58-28) AC + 5% Jai Tire CRM for Different Particle Sizes.

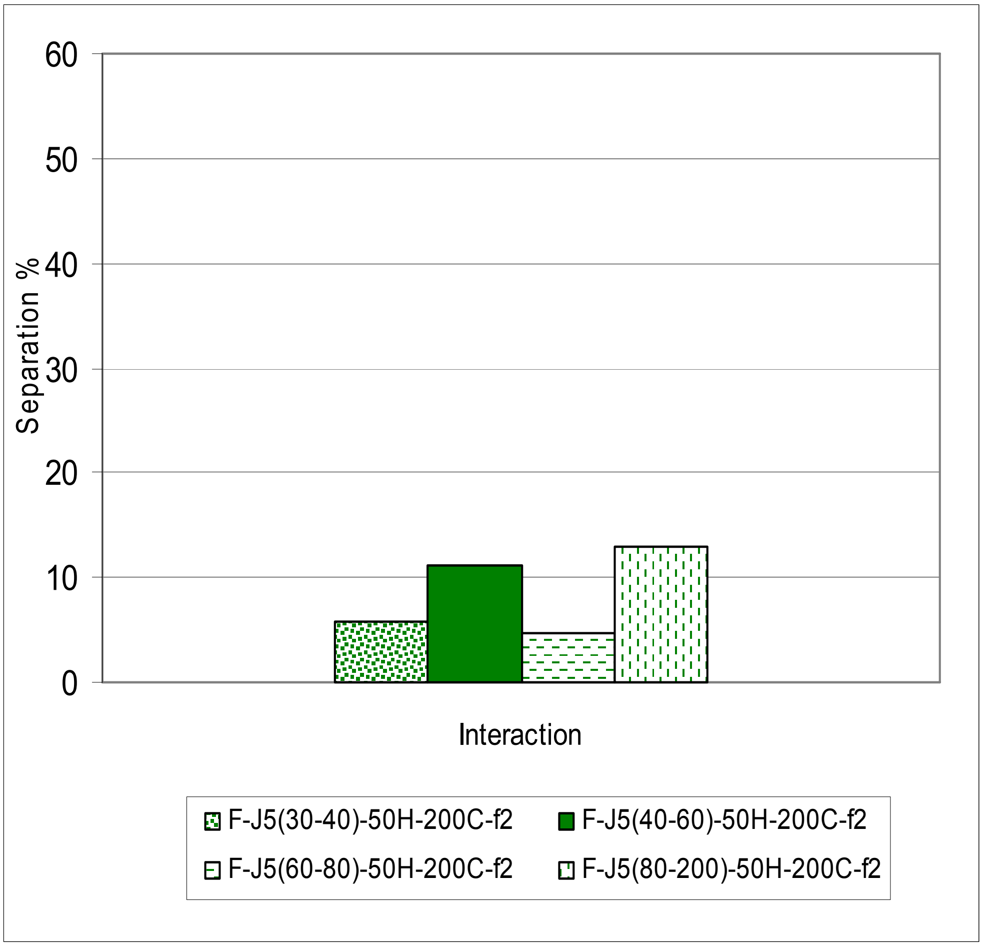


Figure 4-19-b Separation Percentage of Flint Hills (PG 58-28) AC + 5% Jai Tire CRM for Different Particle Sizes.

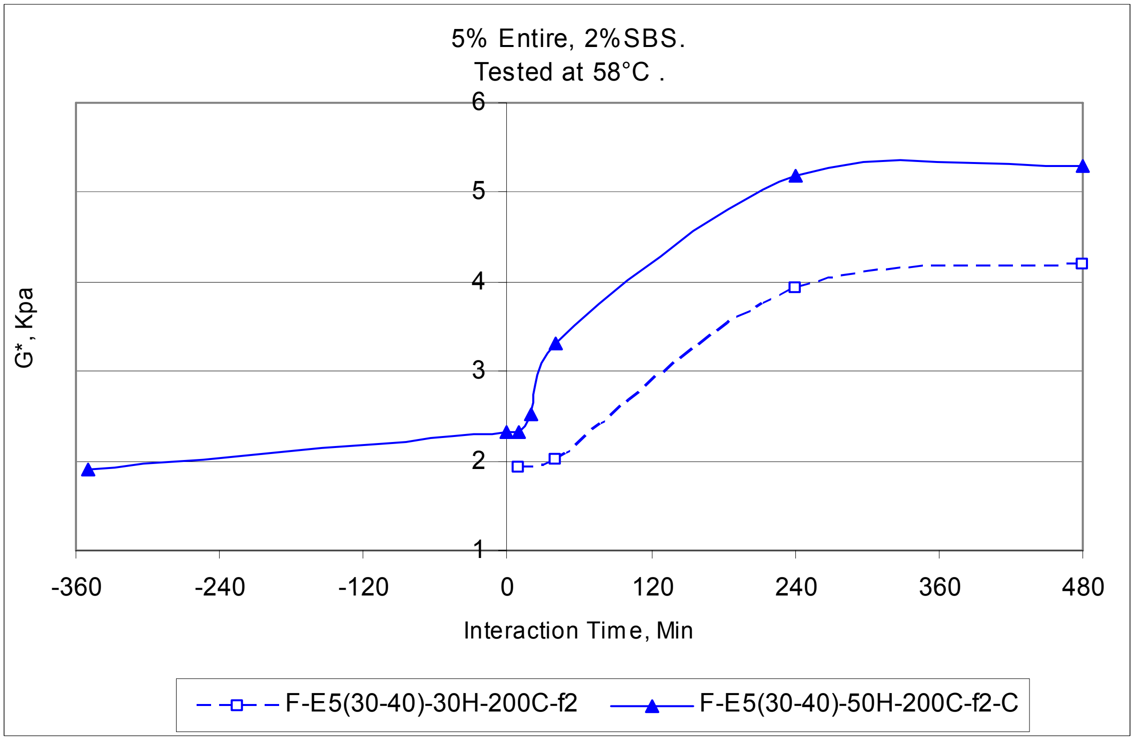
The results suggest that, with additional research, the low temperature rating with 5% CR plus 2% SBS can be controlled so that the low temperature grade remains unchanged. This is indicated by the slight change in low temperature properties of the 5% CR plus 2% SBS when compared to SBS only modification.

Stability of the modified binders was measured using the “Cigar-Tube” test and two binder separation tests. The DSR test was used to measure all performance graded binders while the Ring and Ball test was used only for modified binders without polymers. The results of the separation test using the DSR parameters are expressed in percent differences in tested properties between top and bottom of the testing tube. Separation of SBS only modifications ranged between 1% and 5%. Separation of the 5% CR plus 2% SBS binders ranged from 7% to 12%. This separation level is considered acceptable and is recommended for CRM binder specifications. The factors most affecting the separation test are interaction temperature and shearing energy, expressed in the frequency of shearing. Controls on the interaction process will be discussed further in section 4.5.

4.4 CRM Property Experiment

This experiment showed that different CRM gradations behave differently as the monitored properties develop, and that property development is influenced by material properties and interaction variables. Two main physical properties, particle size and surface area, were considered in the production of CRM asphalt binder. These two parameters affect the binder properties. This section discusses the relation between these two crumb rubber properties and the interaction conditions of time and temperature.

This section presents the differences in behavior between binders made with CRM of different particle sizes subjected to the different interaction conditions. Figures 4-20 through 4-37 show the results associated with two different CR particle size gradations. The data show that changes in binder properties reflect changes in the gel-like structure developed during the interaction process. Particle swelling stiffens the binder by decreasing the inter-particle distance and by decreasing the liquid phase of the binder.



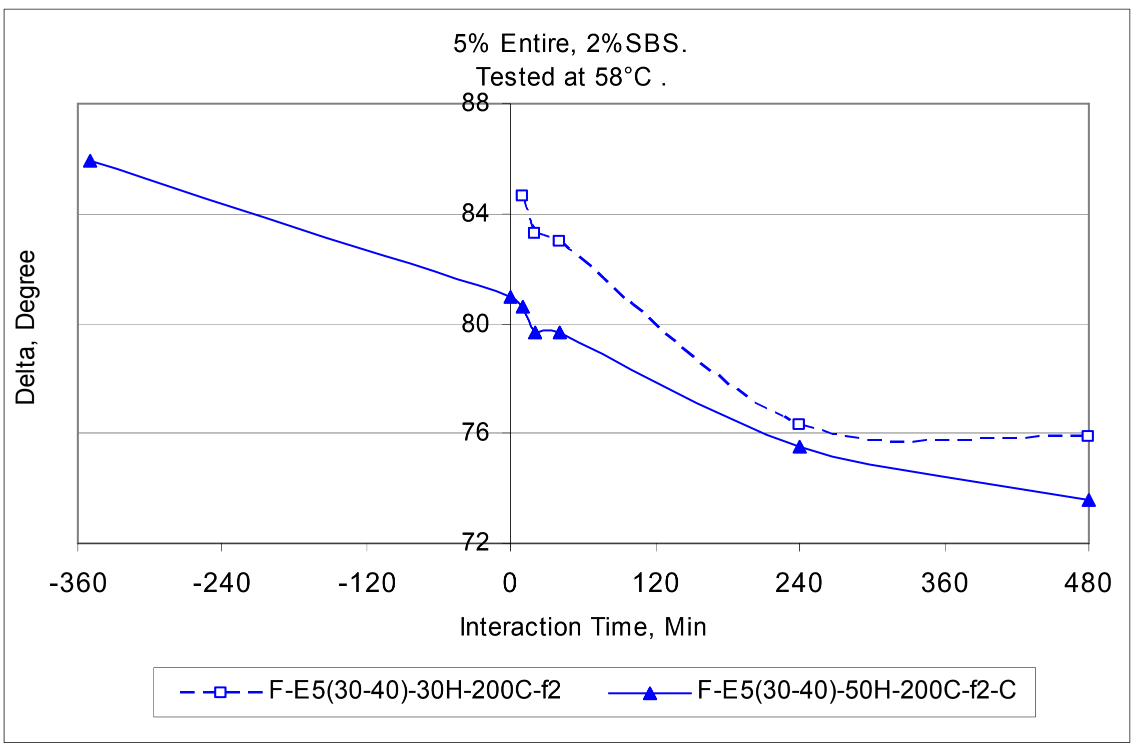


Figure 4-20-a Property Development of EnTire CRM, Size 30-40.

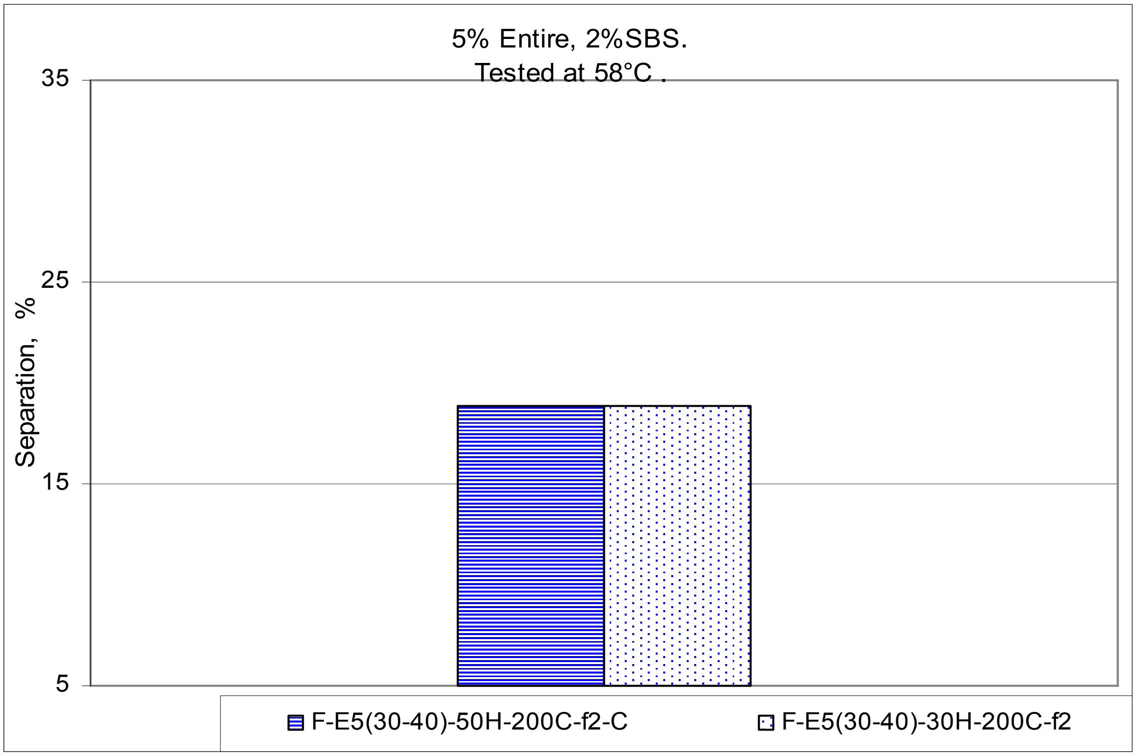
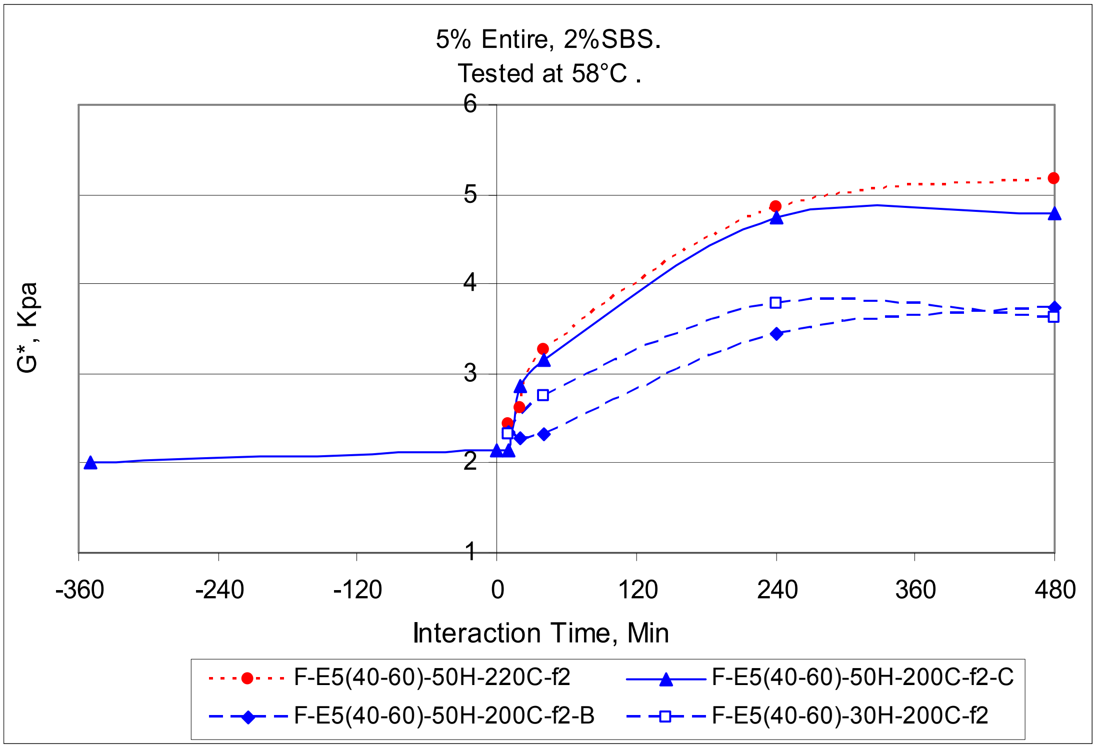


Figure 4-20-b Separation Percentage of EnTire CRM, size 30-40.



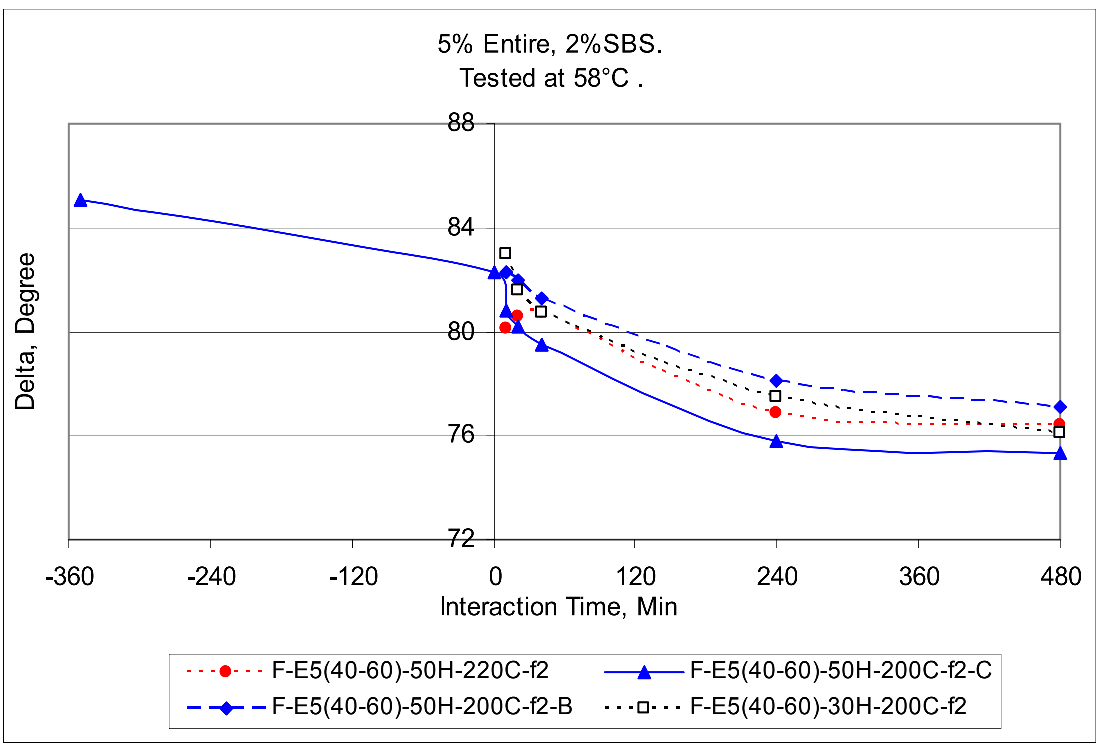


Figure 4-21-a Property Development of EnTire CRM, Size 40-60.

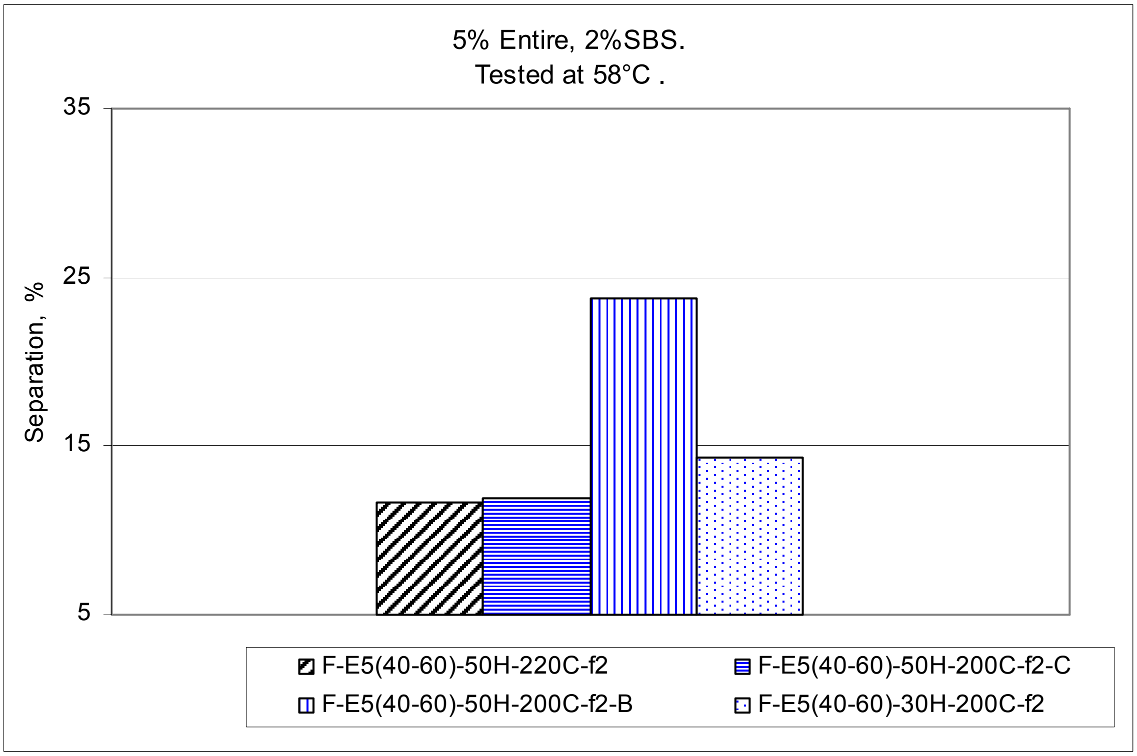
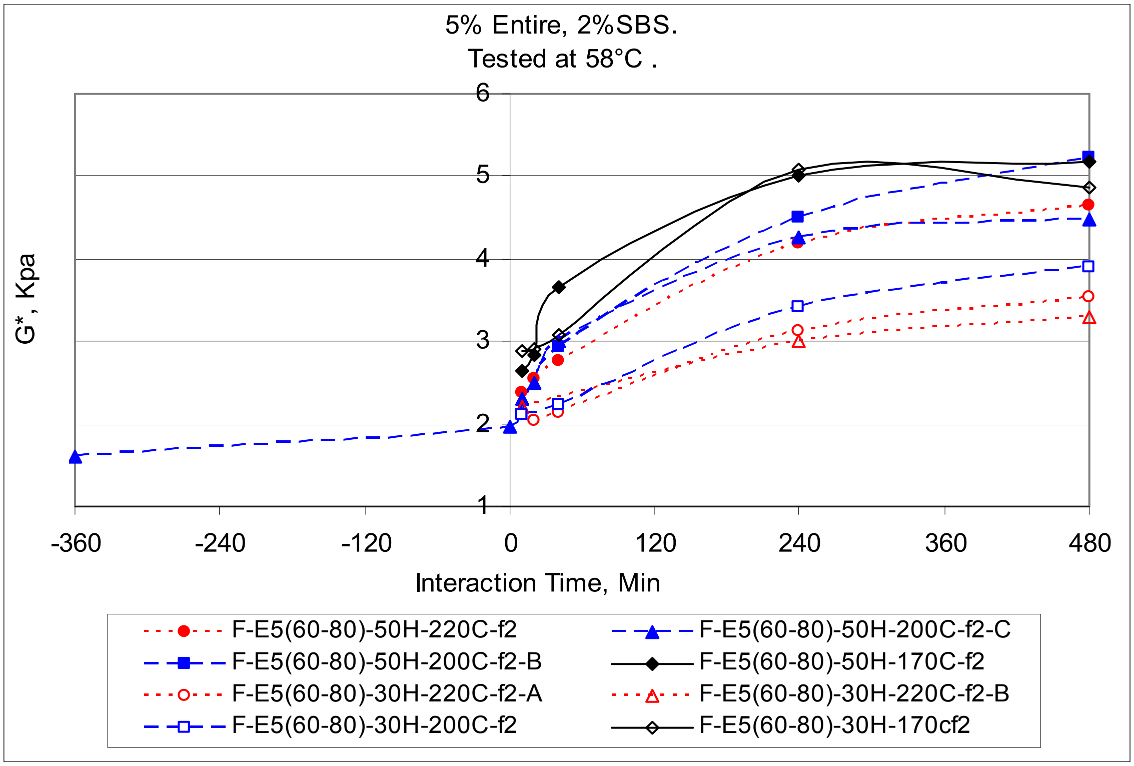


Figure 4-21-b Separation Percentage of EnTire CRM, size 40-60.



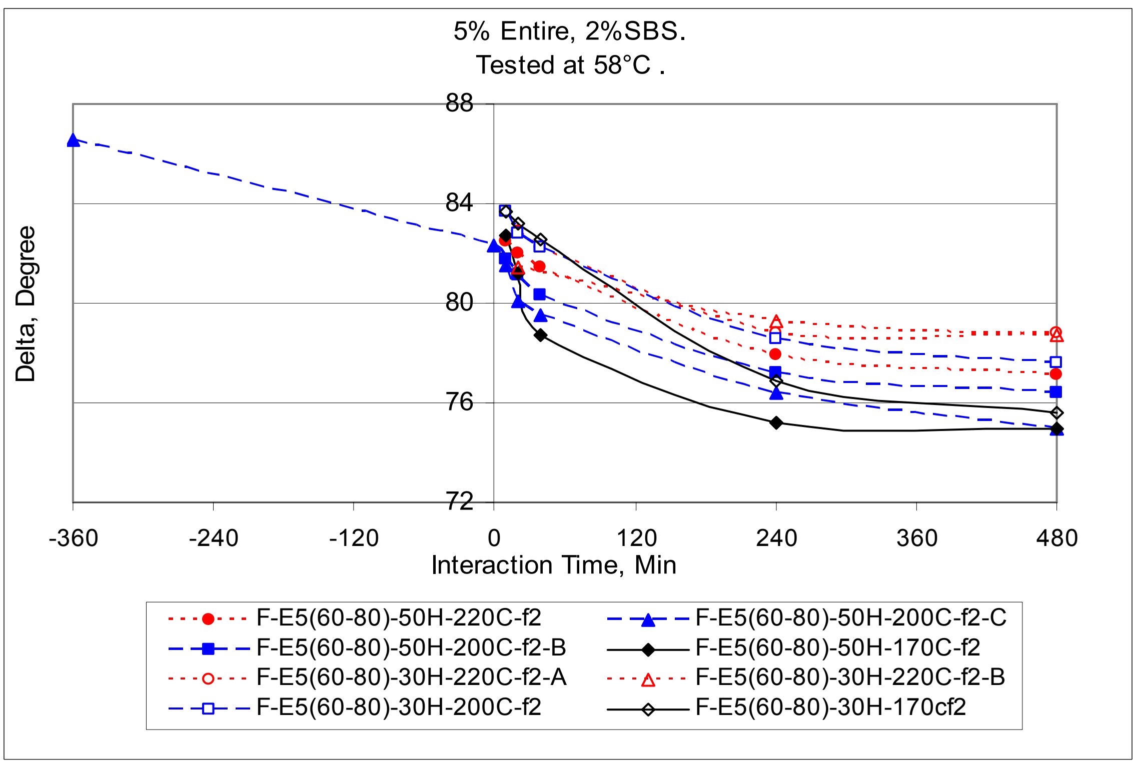


Figure 4-22-a Property Development of EnTire CRM, Size 60-80.

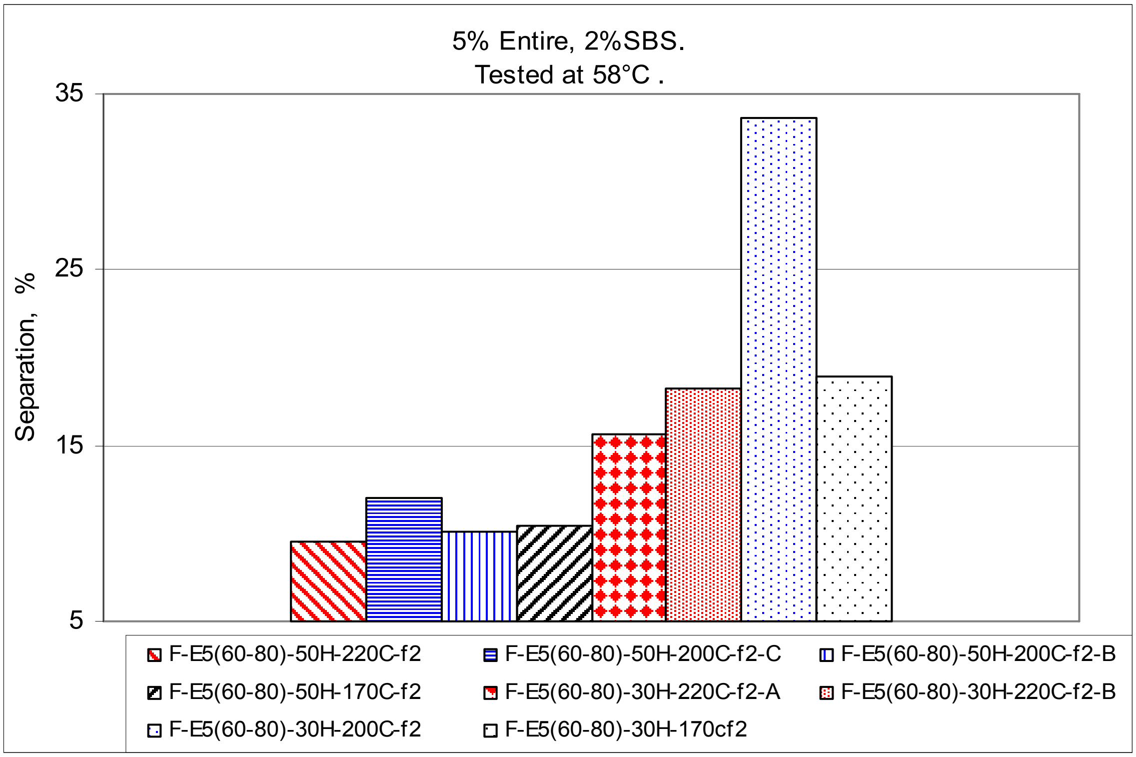
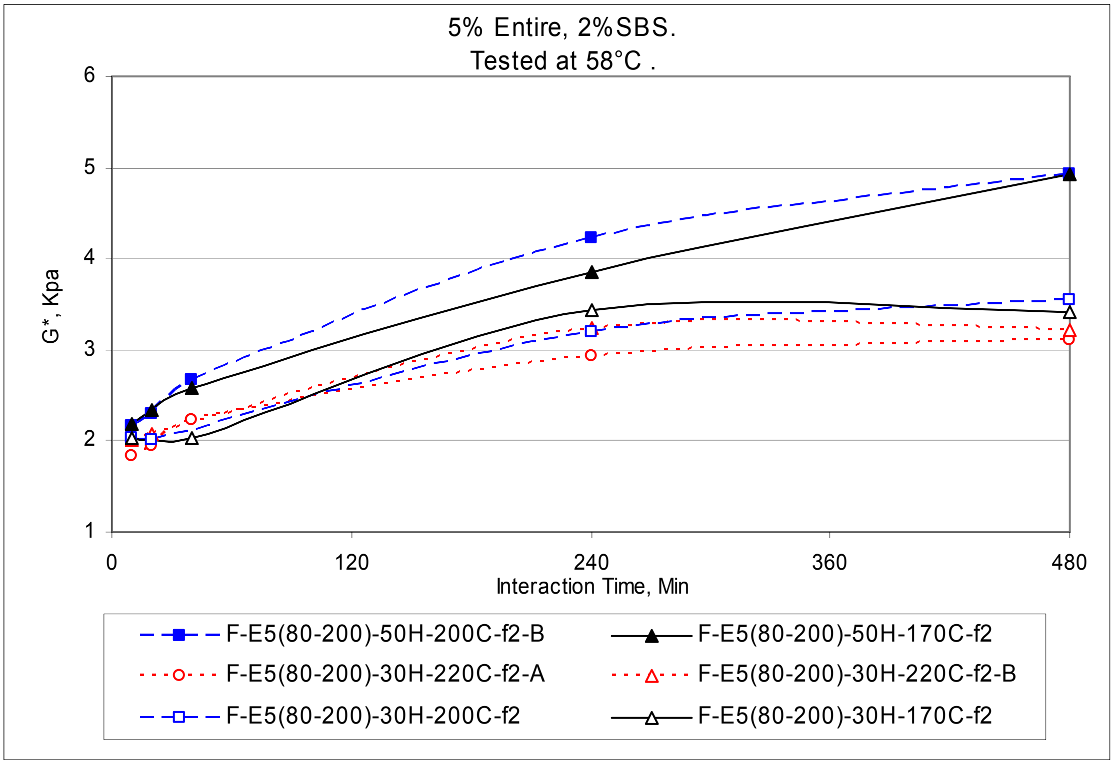


Figure 4-22-b Separation Percentage of EnTire CRM, size 60-80.



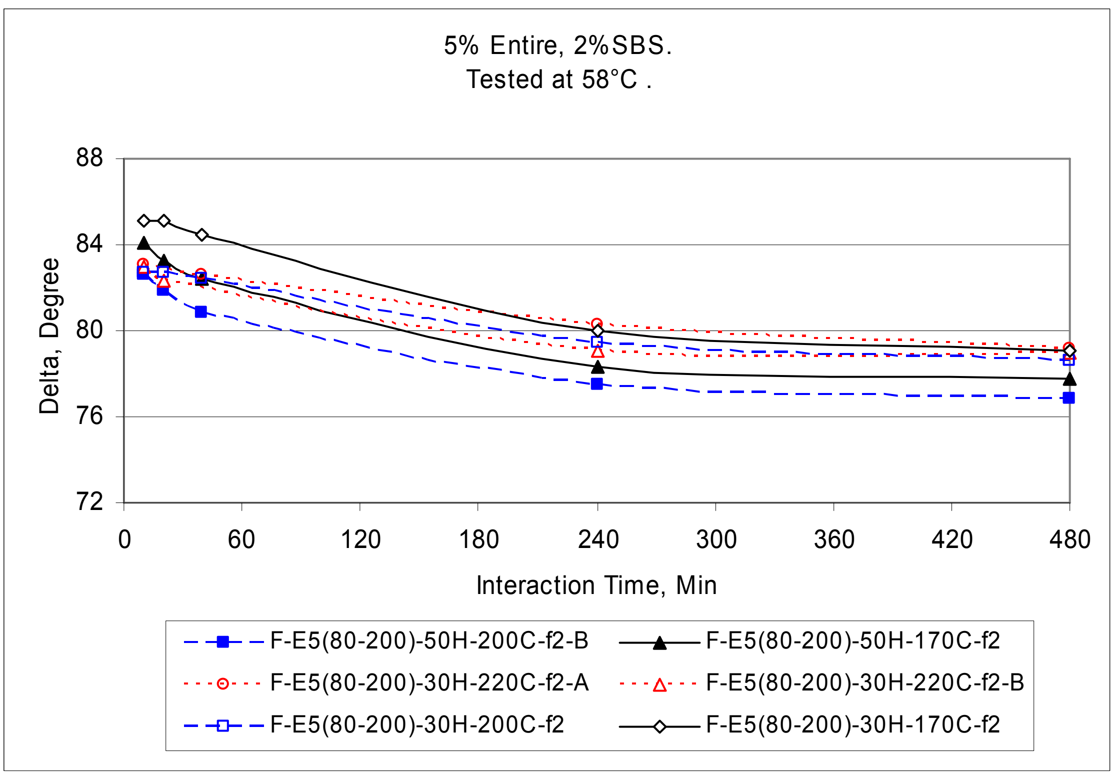


Figure 4-23-a Property Development of EnTire CRM, Size 80-200.

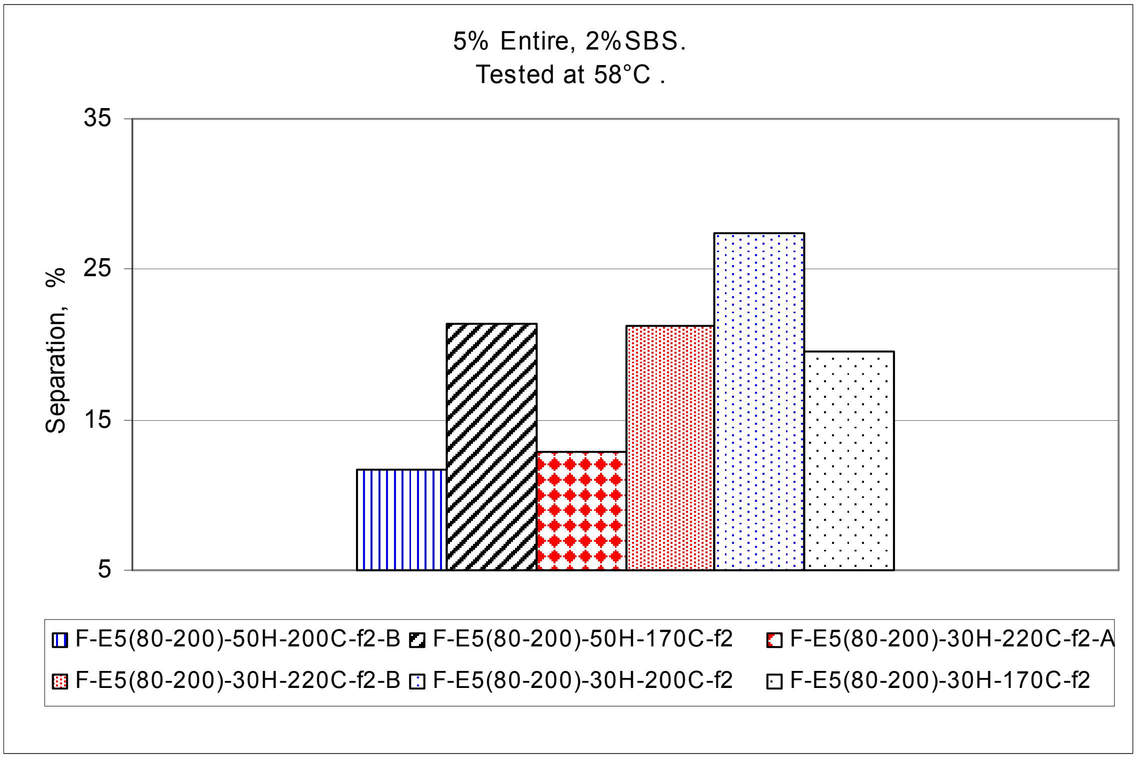
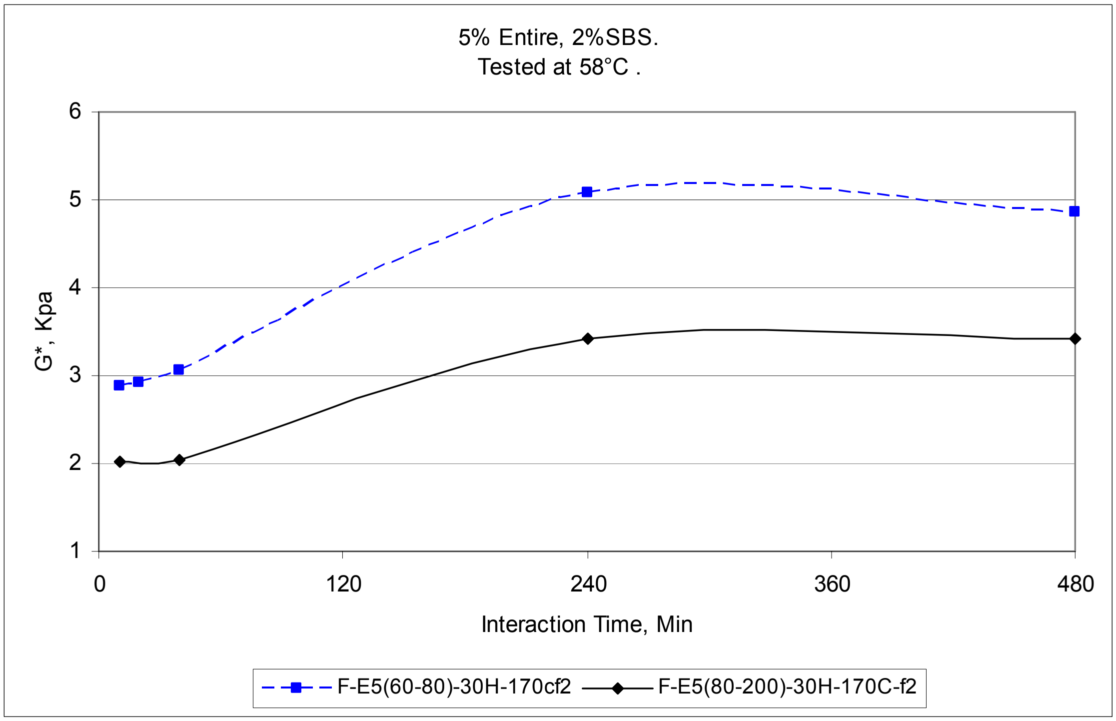


Figure 4-23-b Separation Percentage of EnTire CRM, size 80-200.



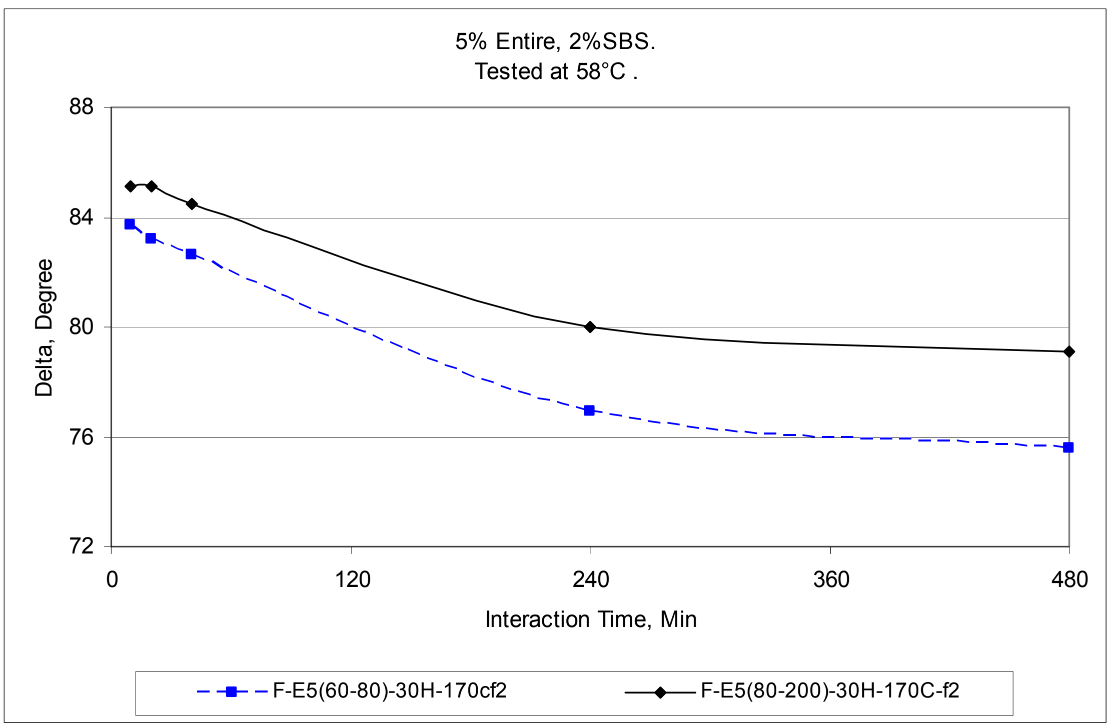


Figure 4-24-a Property Development of EnTire CRM. Main Interaction: 30 Hz at 170 °C for 40 minutes.

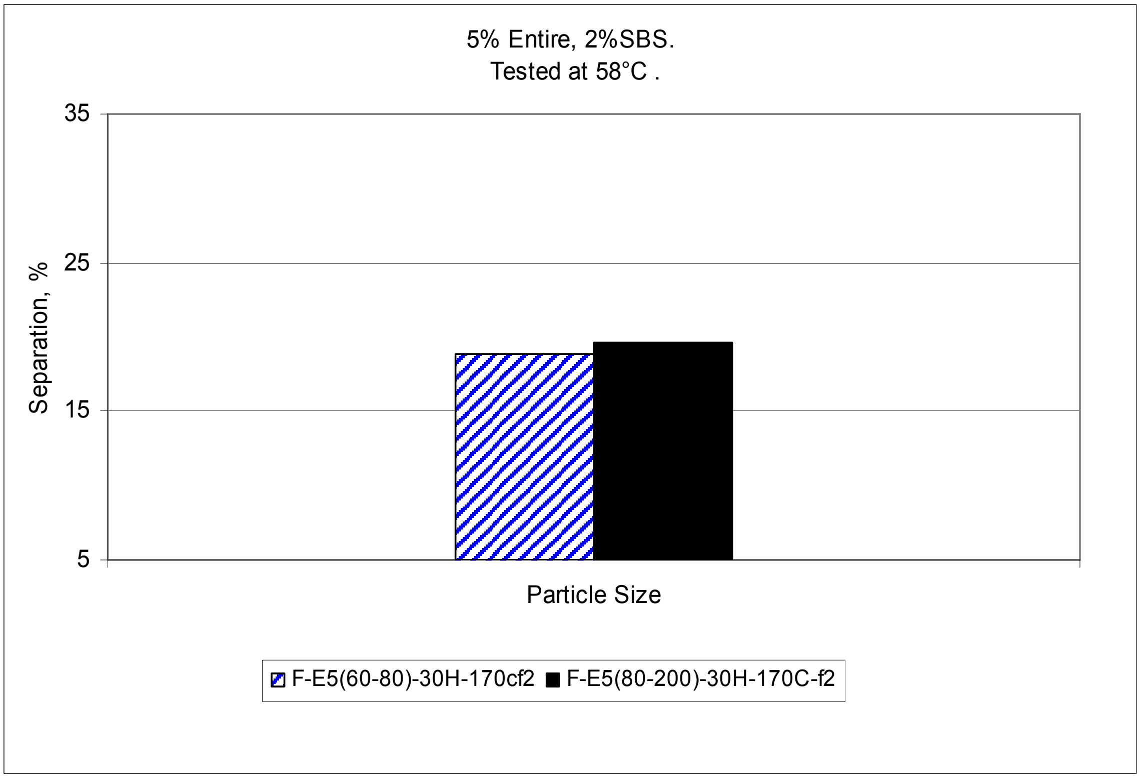
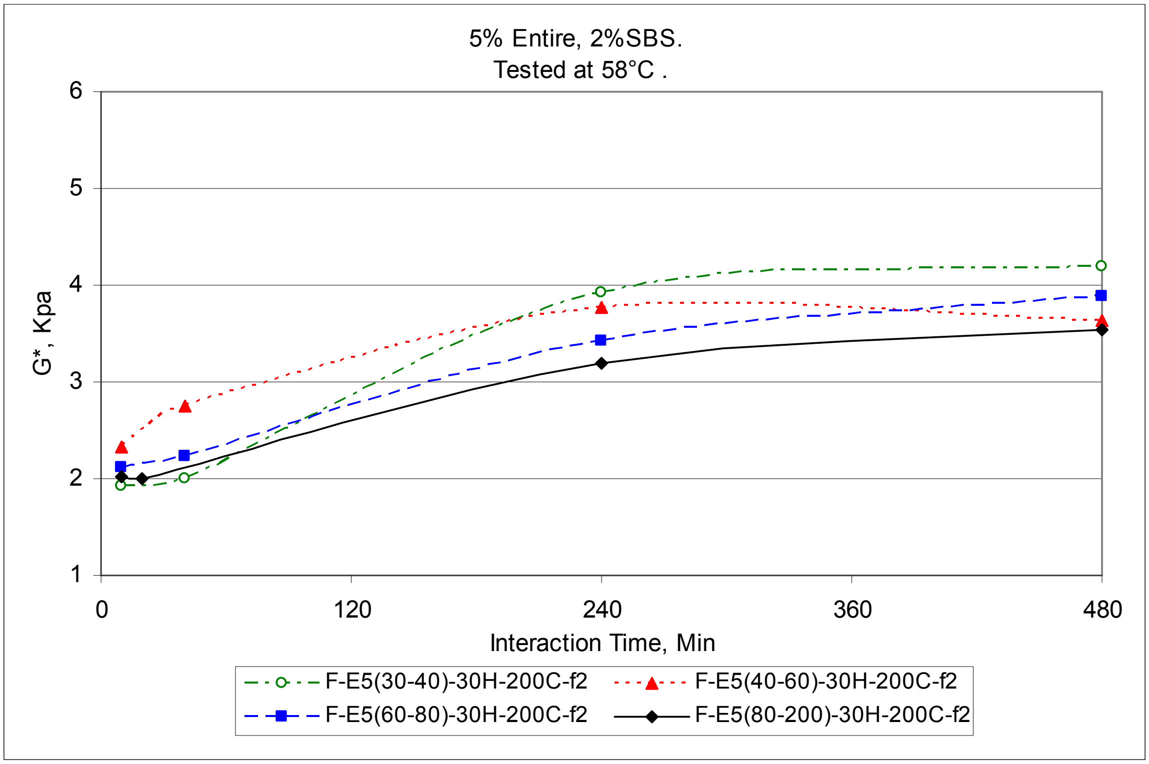


Figure 4-24-b Separation Percentage of EnTire CRM. Main Interaction: 30 Hz at 170 °C for 40 minutes.



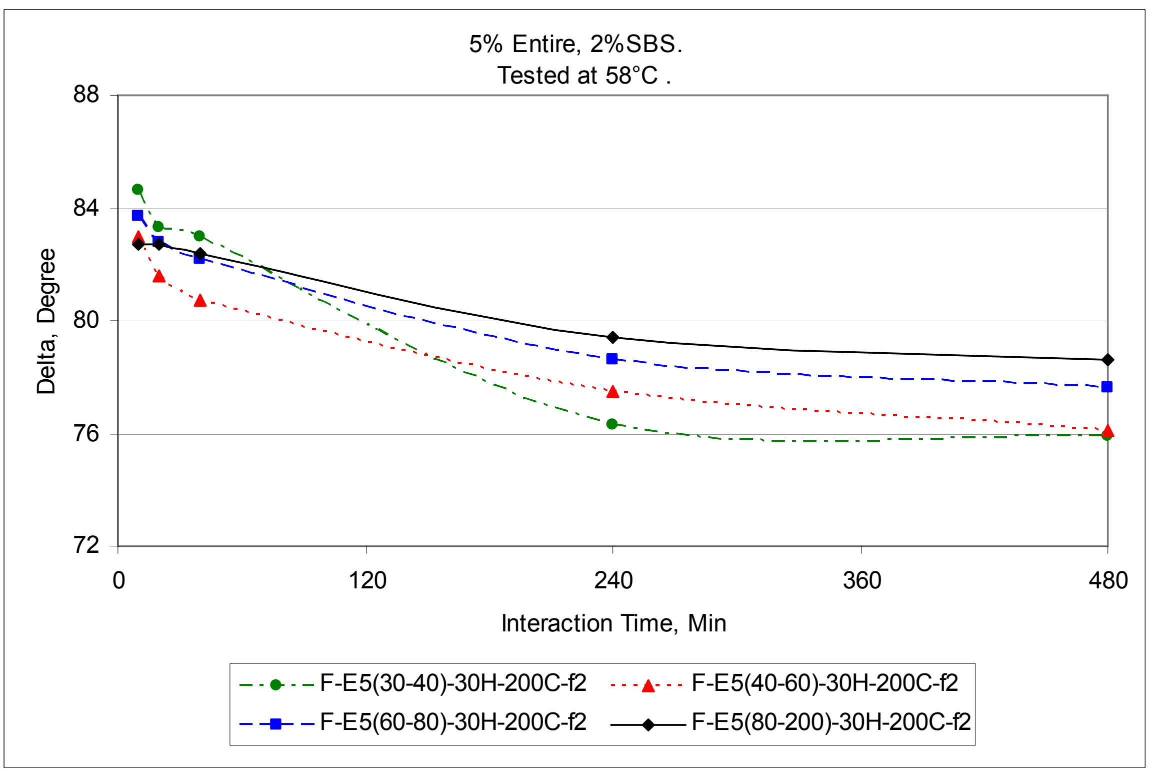


Figure 4-25-a Property Development of EnTire CRM. Main Interaction: 30 Hz at 200 °C for 40 minutes.

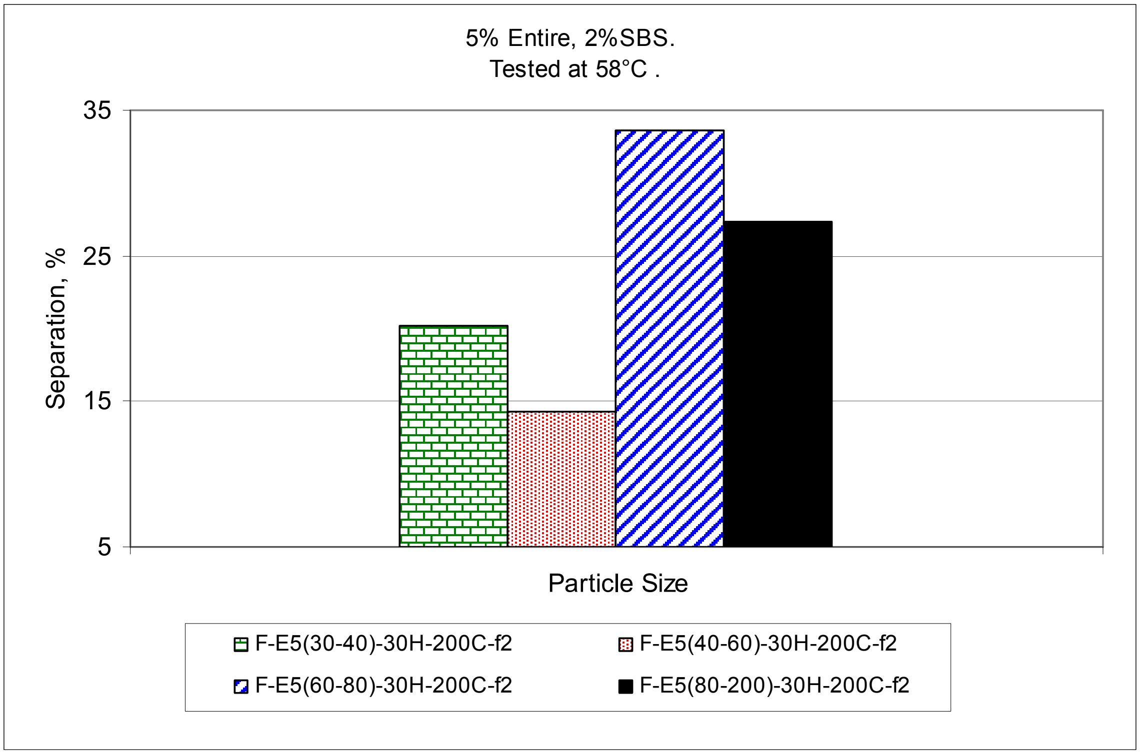
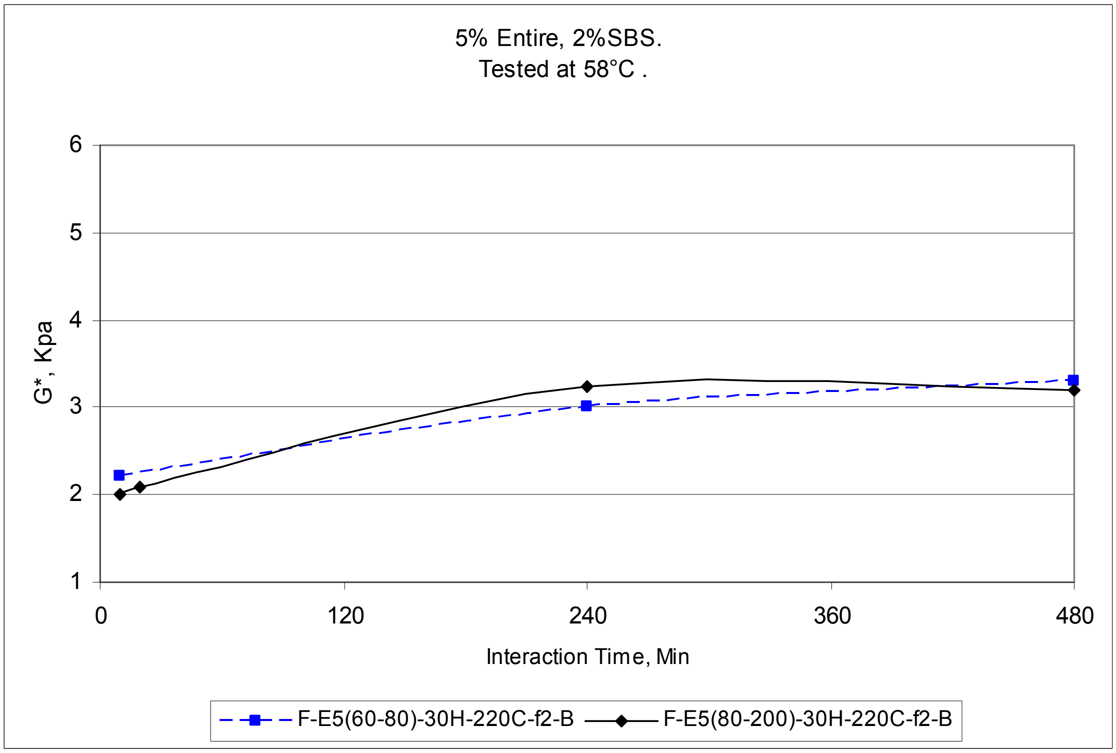


Figure 4-25-b Separation Percentage of EnTire CRM. Main Interaction: 30 Hz at 200 °C for 40 minutes.



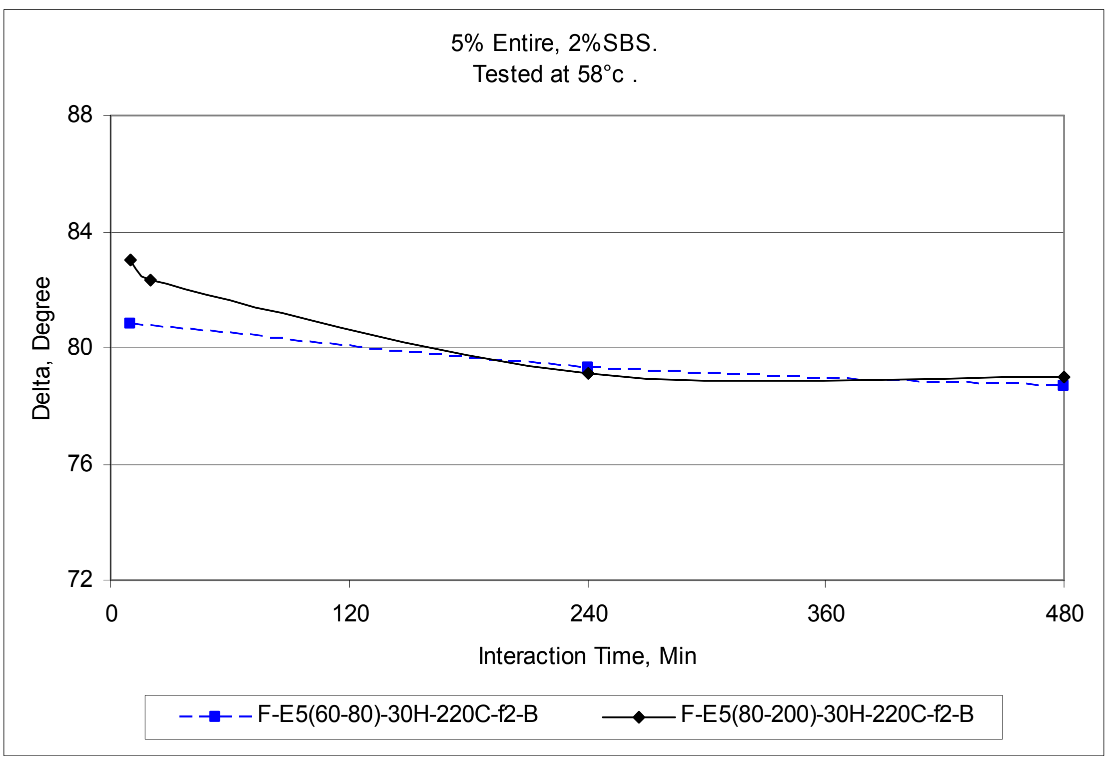


Figure 4-26-a Property Development of EnTire CRM. Main Interaction: 30 Hz at 220 °C for 20 minutes.

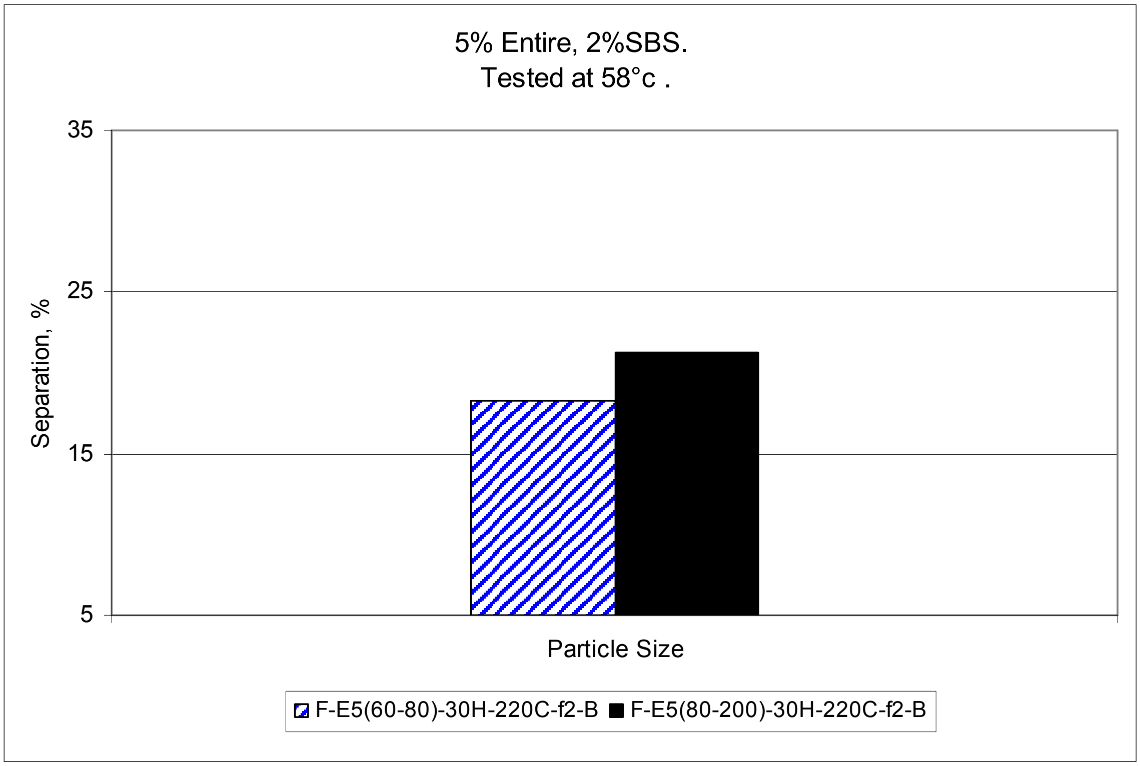
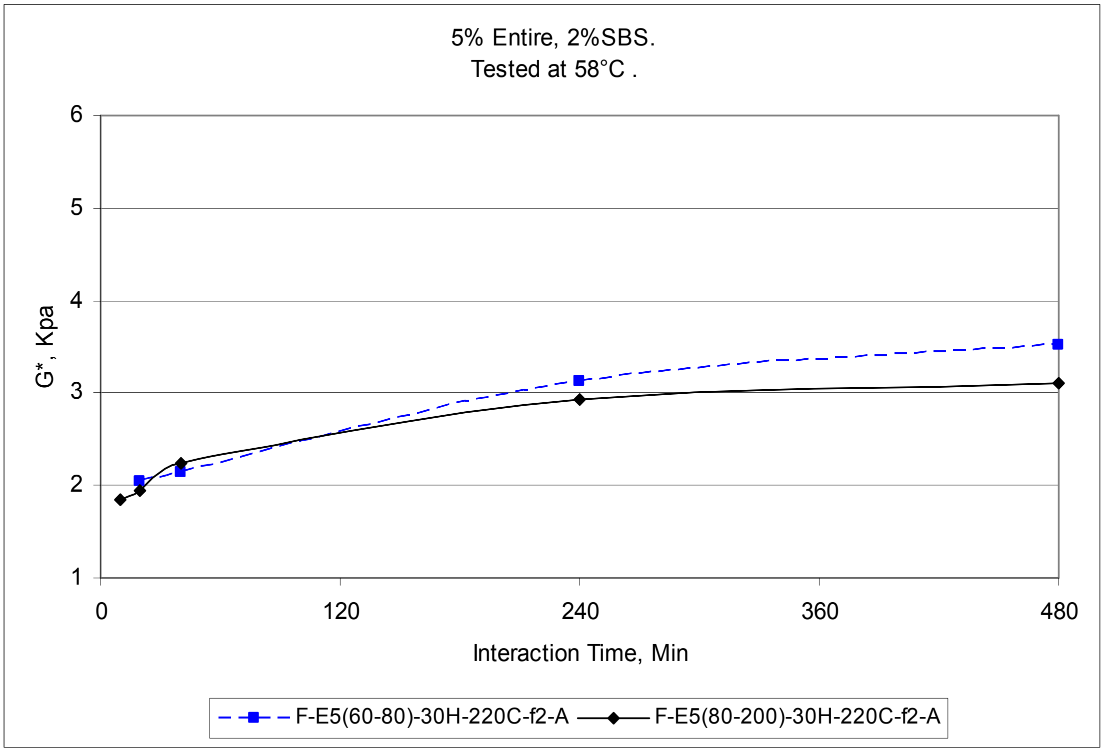


Figure 4-26-b Separation Percentage of EnTire CRM. Main Interaction: 30 Hz at 220 °C for 20 minutes.



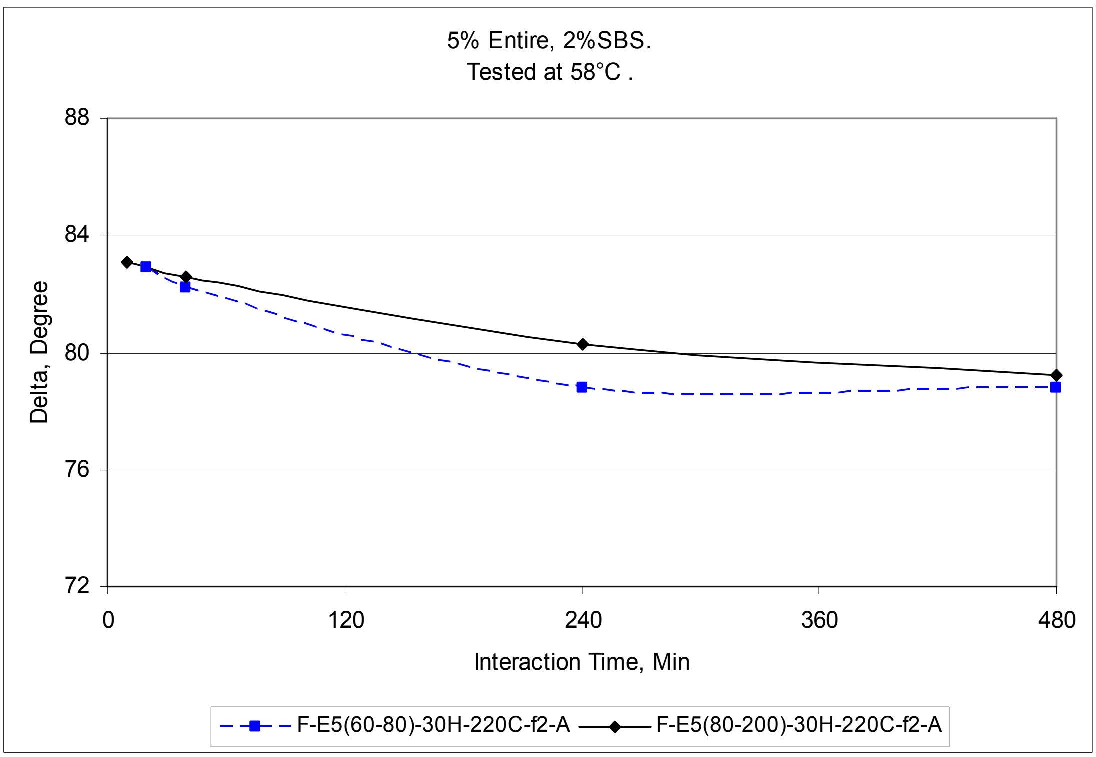


Figure 4-27-a Property Development of EnTire CRM. Main Interaction: 30 Hz at 220 °C for 40 minutes.

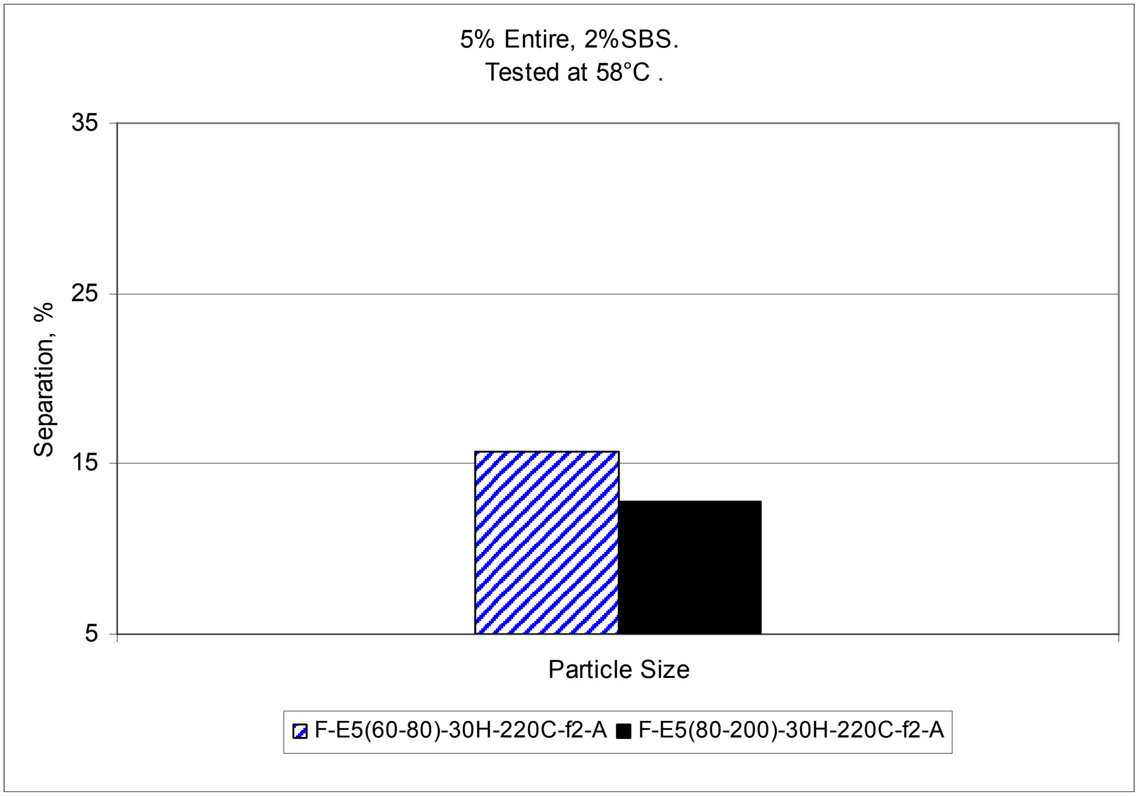
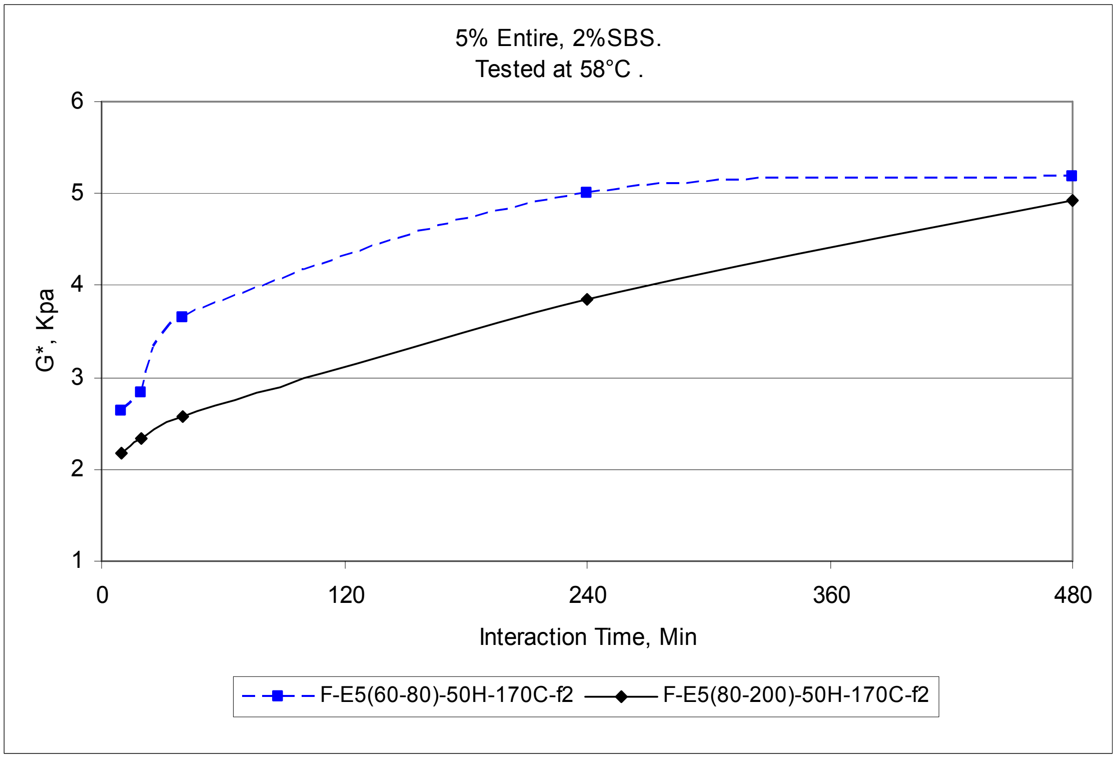


Figure 4-27-b Separation Percentage of EnTire CRM. Main Interaction: 30 Hz at 220 °C for 40 minutes.



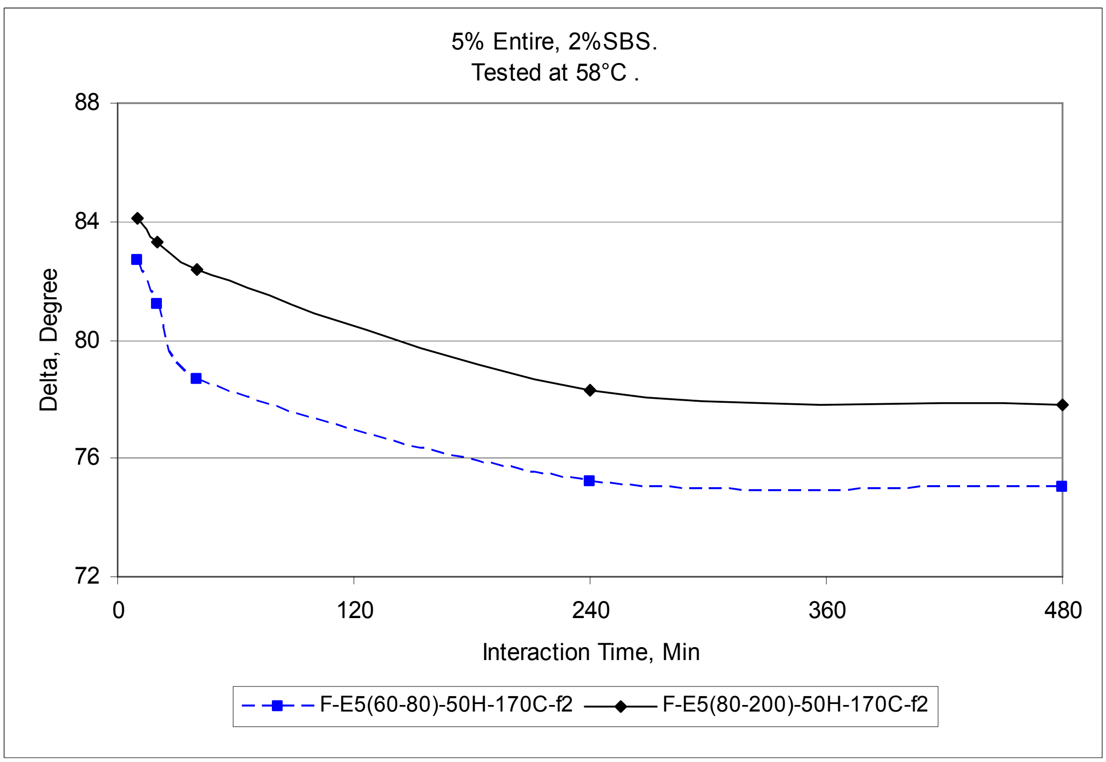


Figure 4-28-a Property Development of EnTire CRM. Main Interaction: 50 Hz at 170 °C for 40 minutes.

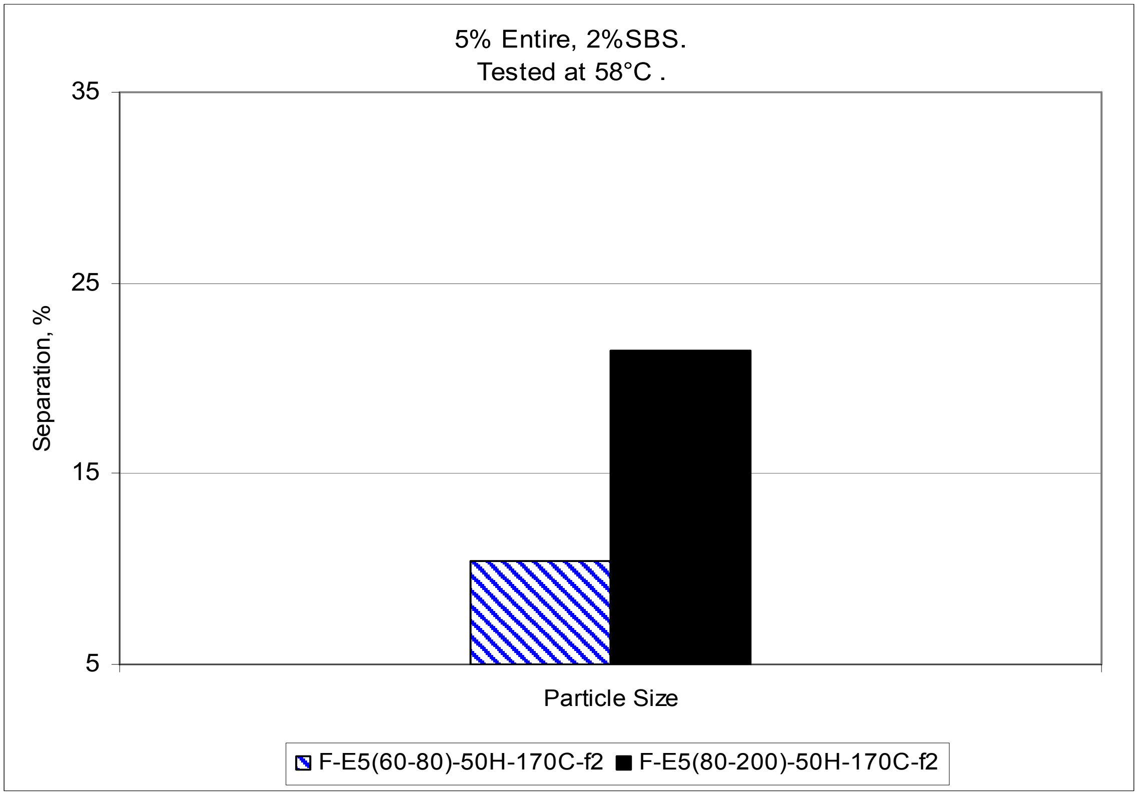


Figure 4-28-b Separation Percentage of EnTire CRM. Main Interaction: 50 Hz at 170 °C for 40 minutes.

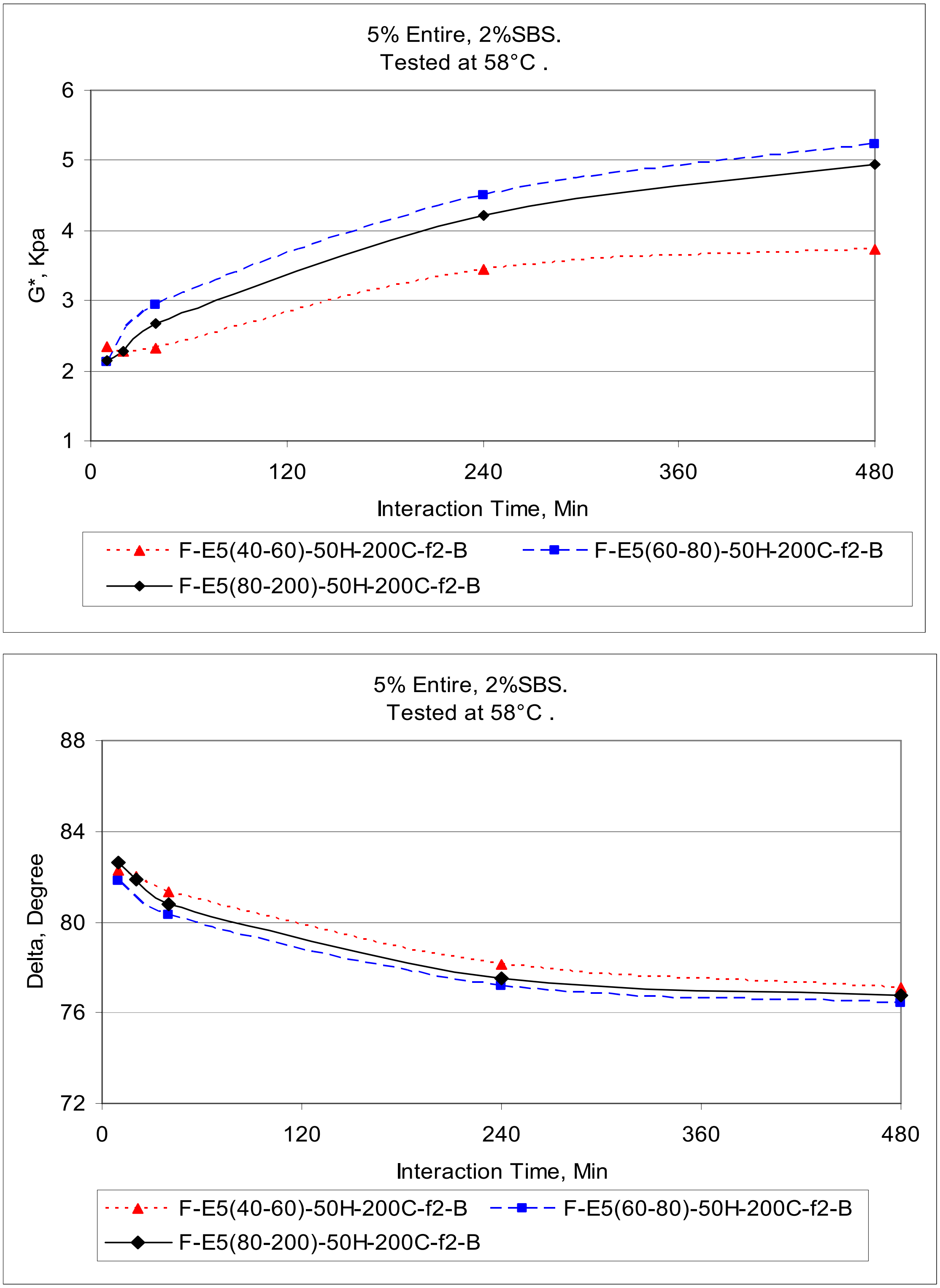


Figure 4-29-a Property Development of EnTire CRM. Main Interaction: 50 Hz at 200 °C for 40 minutes.

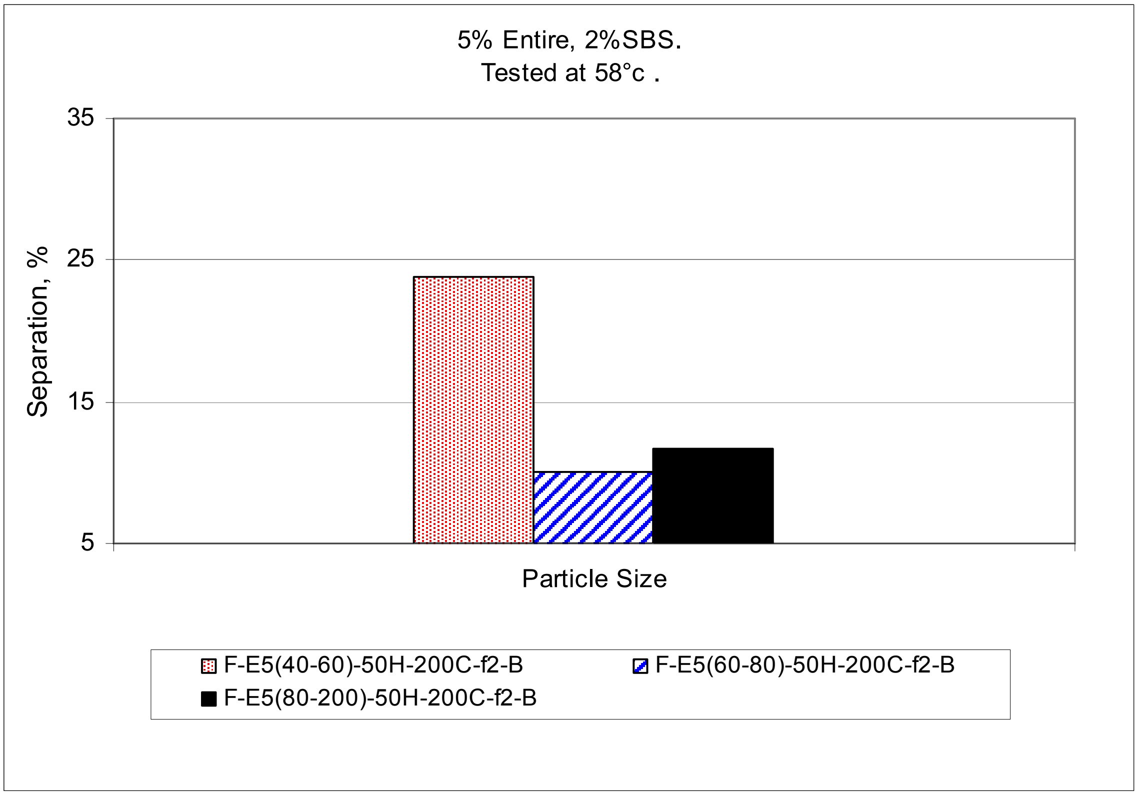
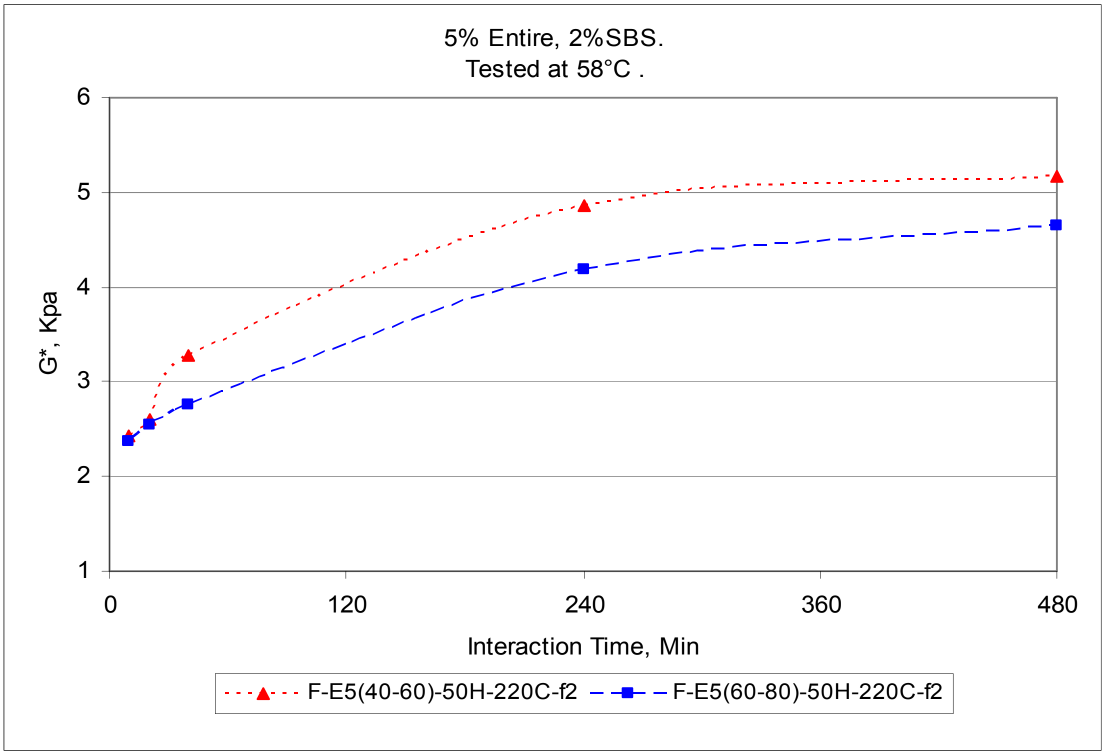


Figure 4-29-b Separation Percentage of EnTire CRM. Main Interaction: 50 Hz at 200 °C for 40 minutes.



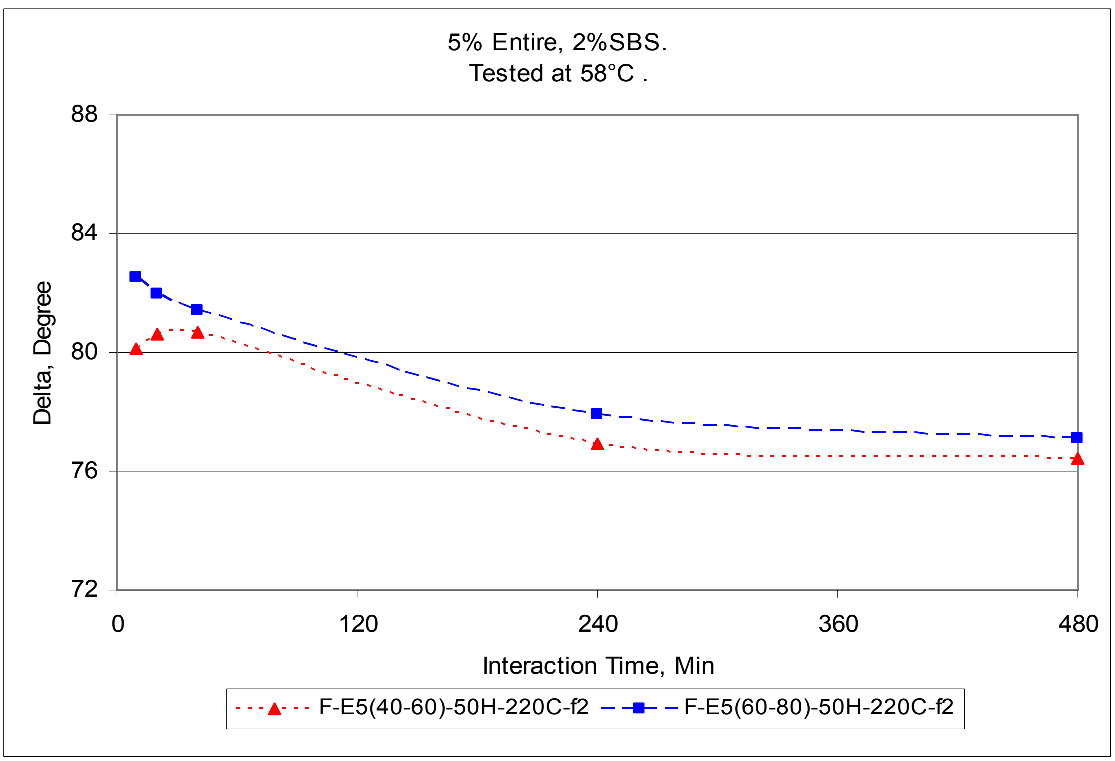


Figure 4-30-a Property Development of EnTire CRM. Main Interaction: 50 Hz at 220 °C for 40 minutes.

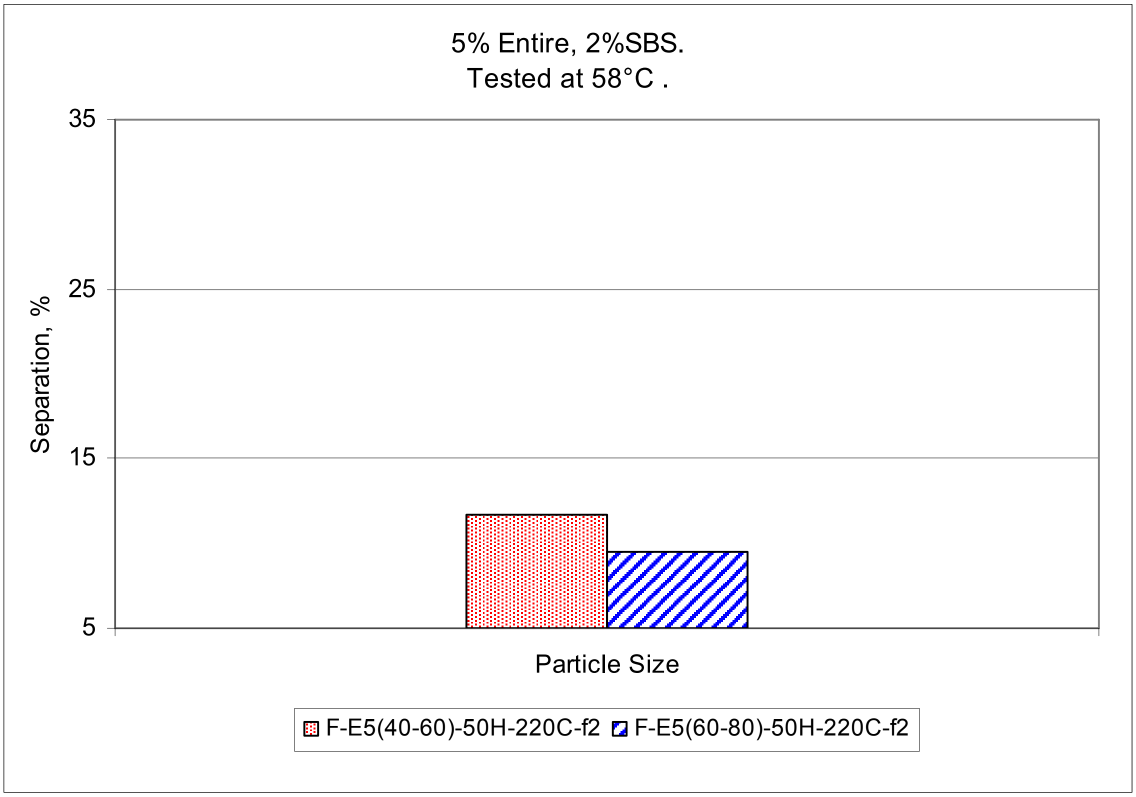
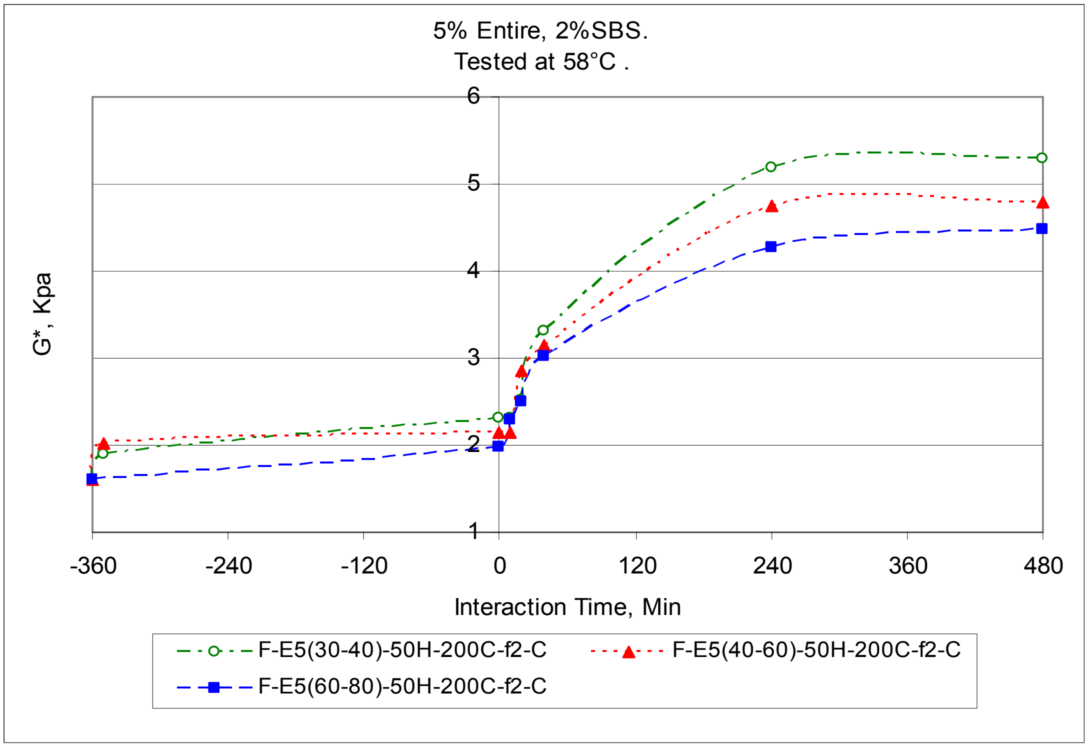


Figure 4-30-b Separation Percentage of EnTire CRM. Main Interaction: 50 Hz at 220 °C for 40 minutes.



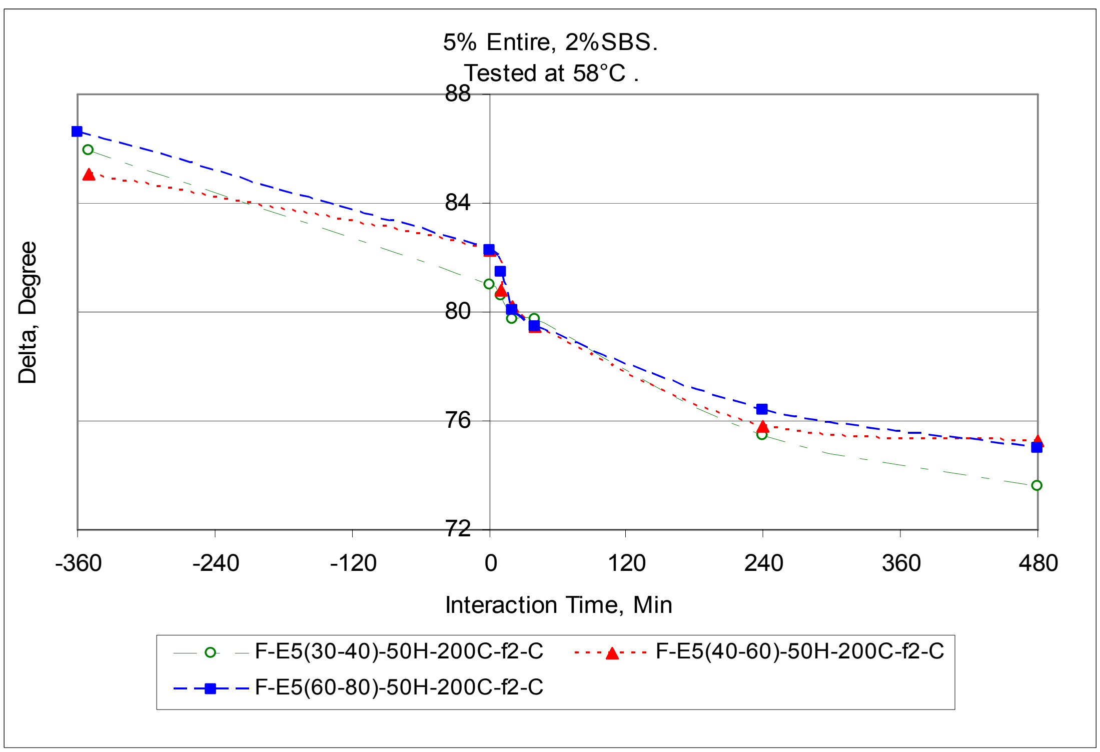


Figure 4-31-a Property Development of EnTire CRM. Main Interaction: presoaking for 6 hrs, 50 Hz at 220 °C for 40 minutes.

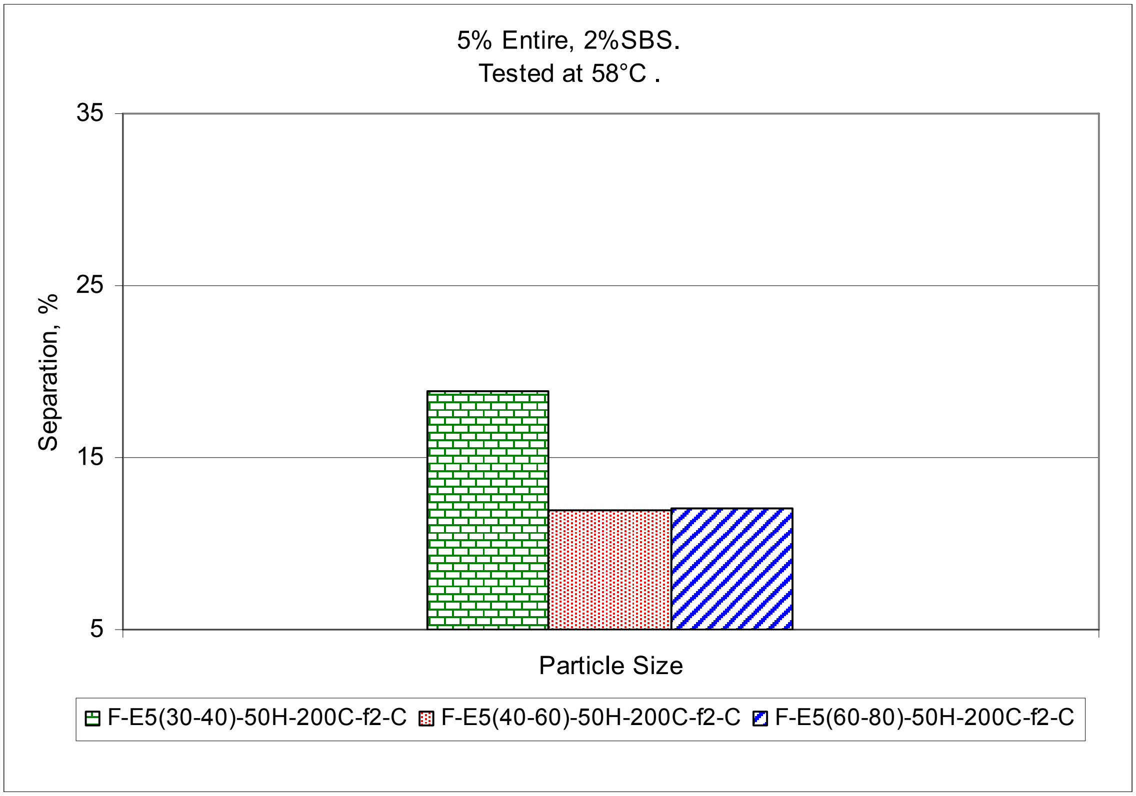
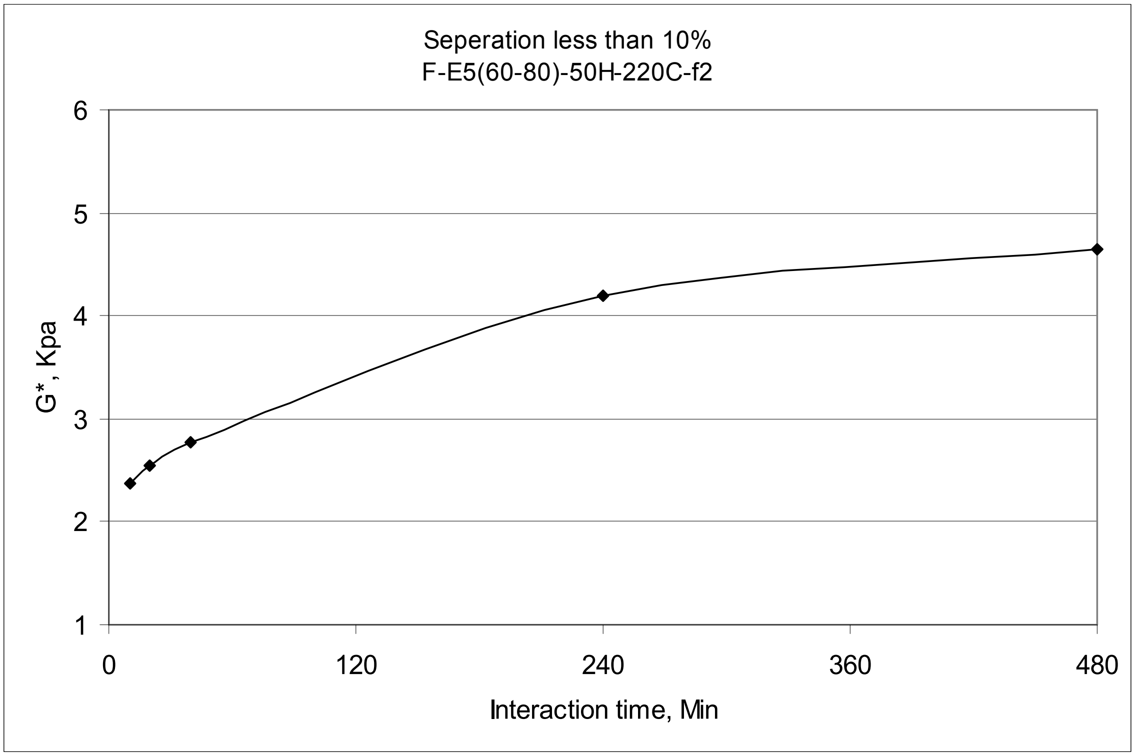


Figure 4-31-b Separation Percentage of EnTire CRM. Main Interaction: presoaking for 6 hrs, 50 Hz at 220 °C for 40 minutes.



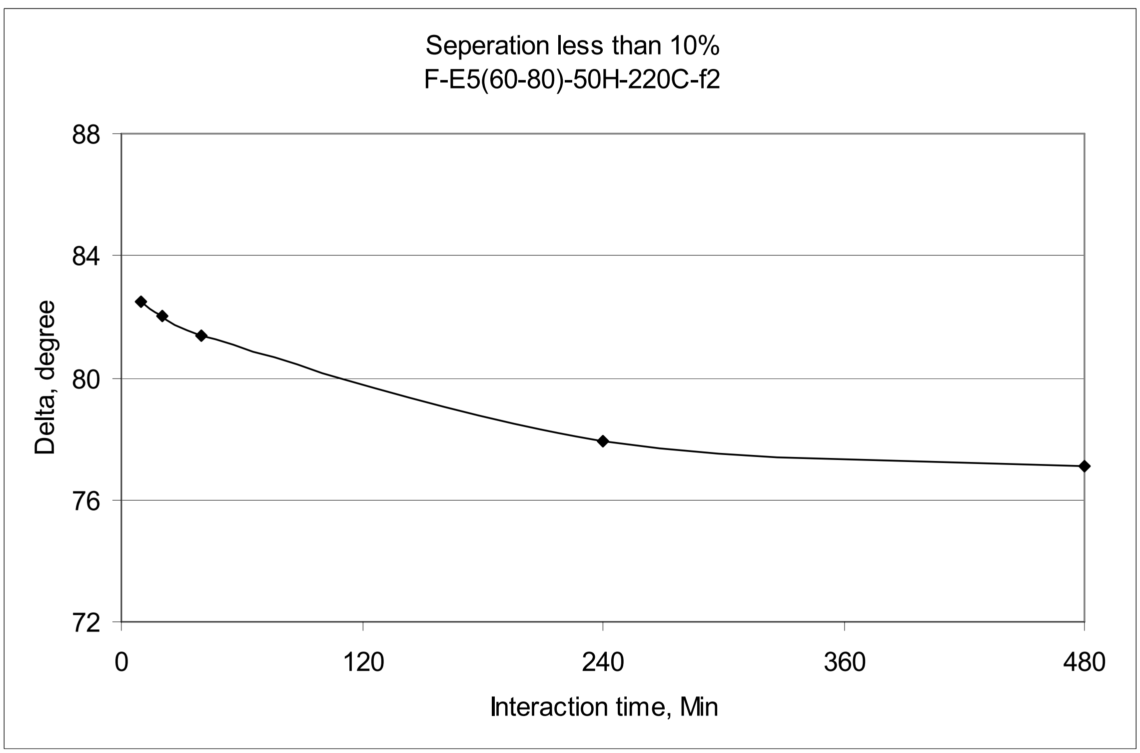
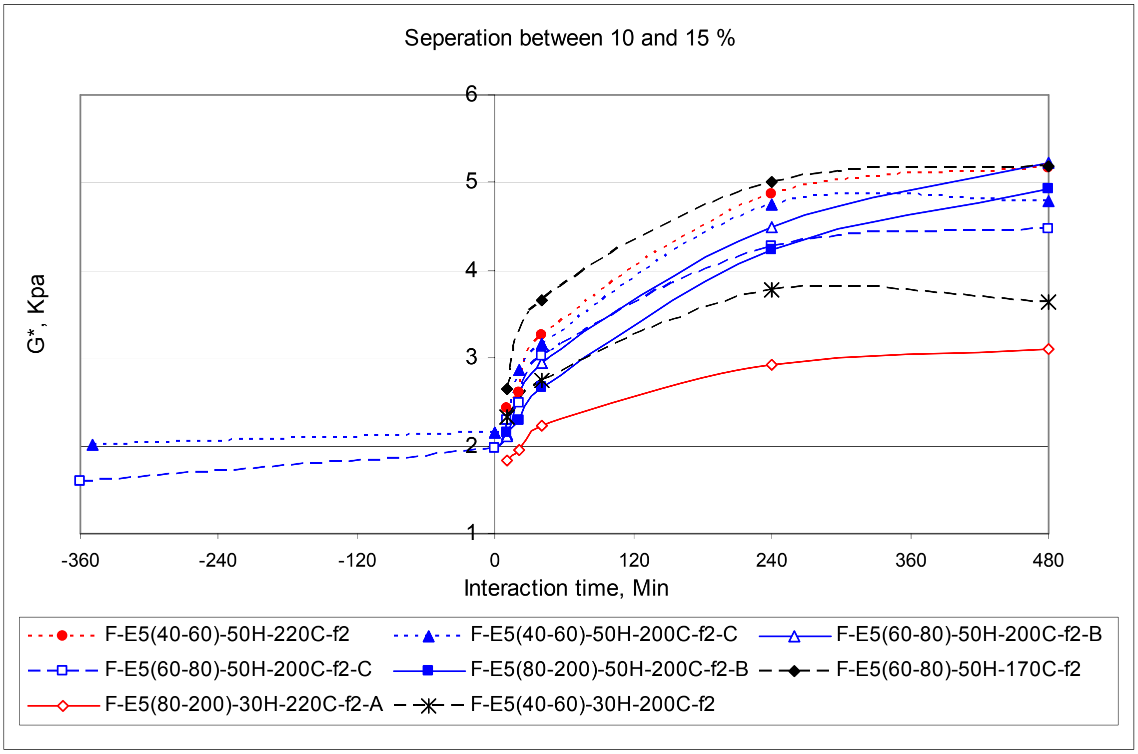


Figure 4-32 Property Development for Binders with Separation less than 10%.



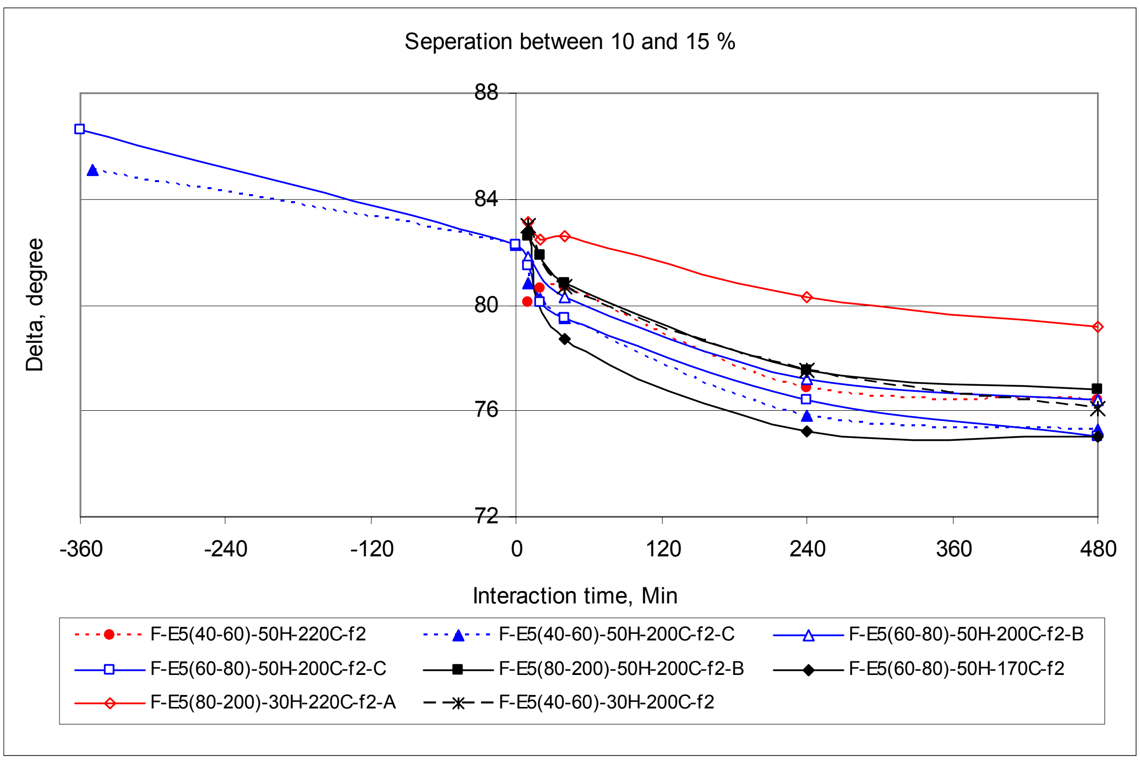


Figure 4-33 Property Development for Binders with Separation between 10% and 15%.

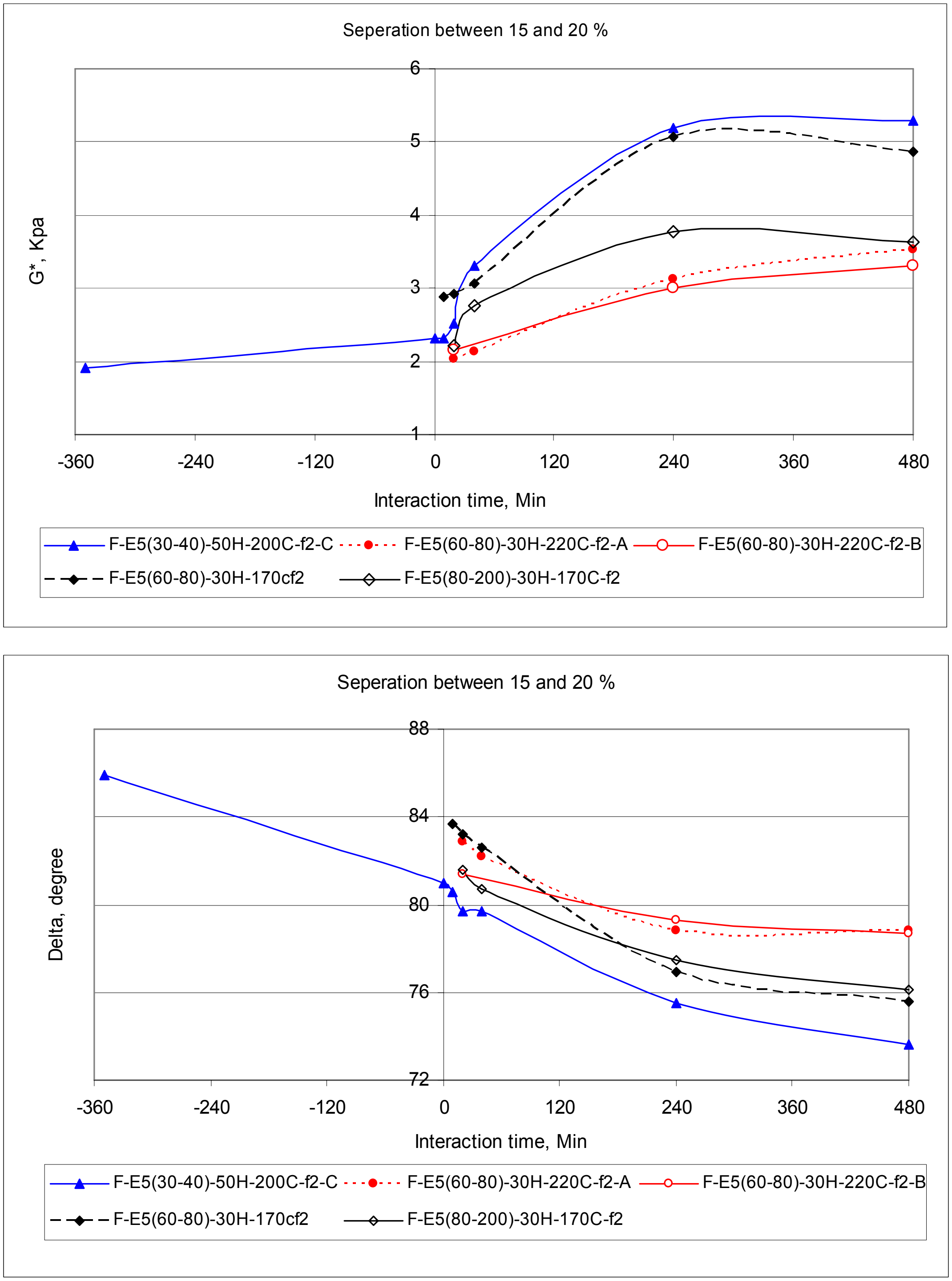


Figure 4-34 Property Development for Binders with Separation between 15% and 20%.

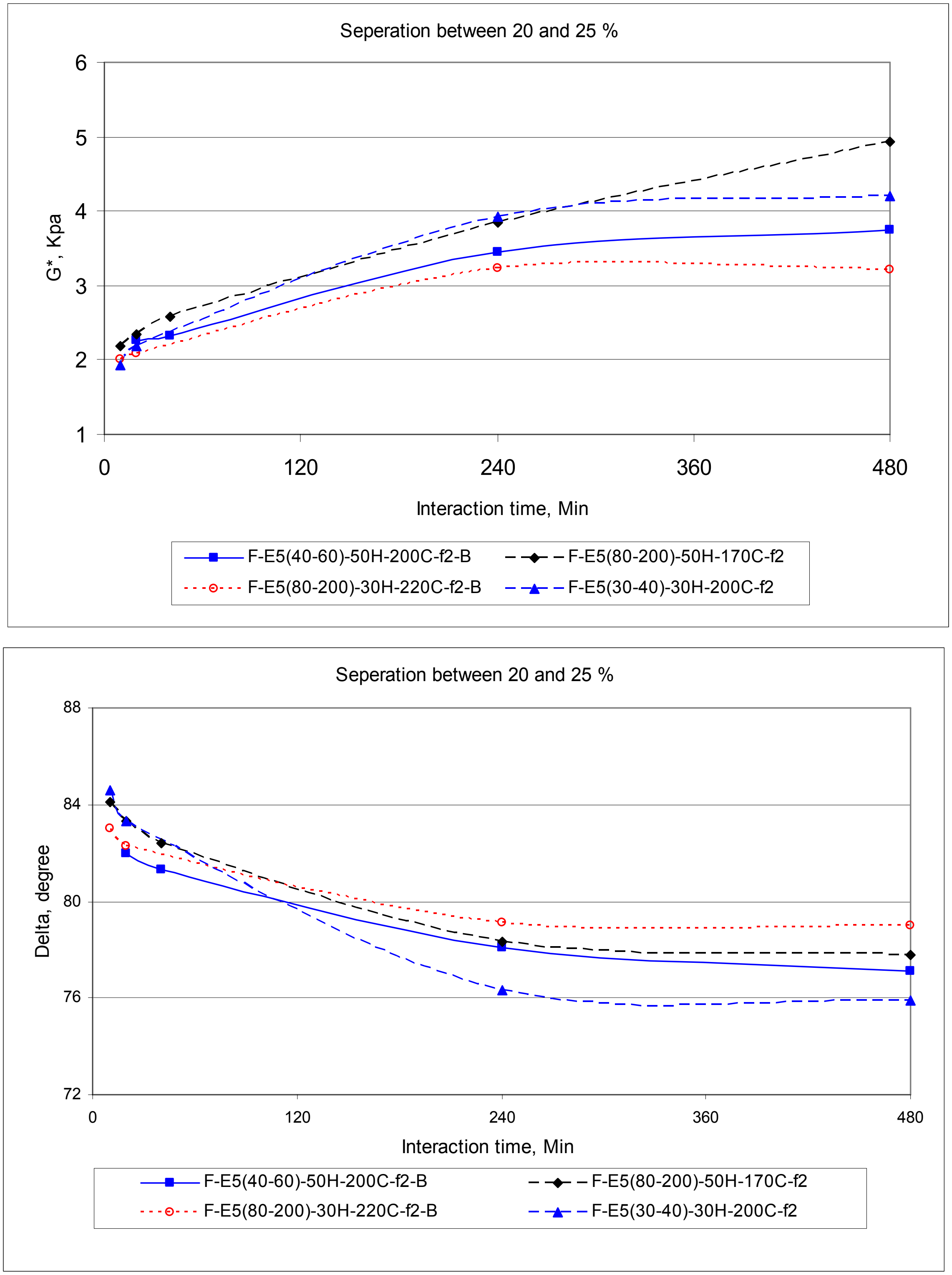
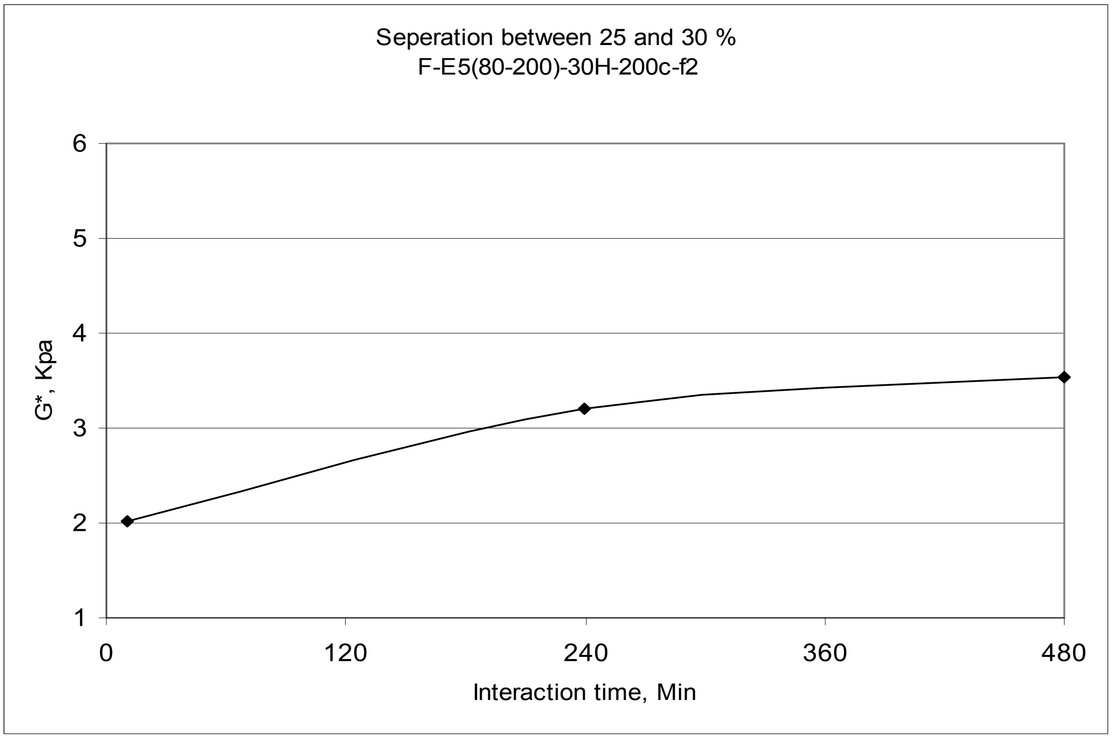


Figure 4-35 Property Development for Binders with Separation between 20% and 25%.



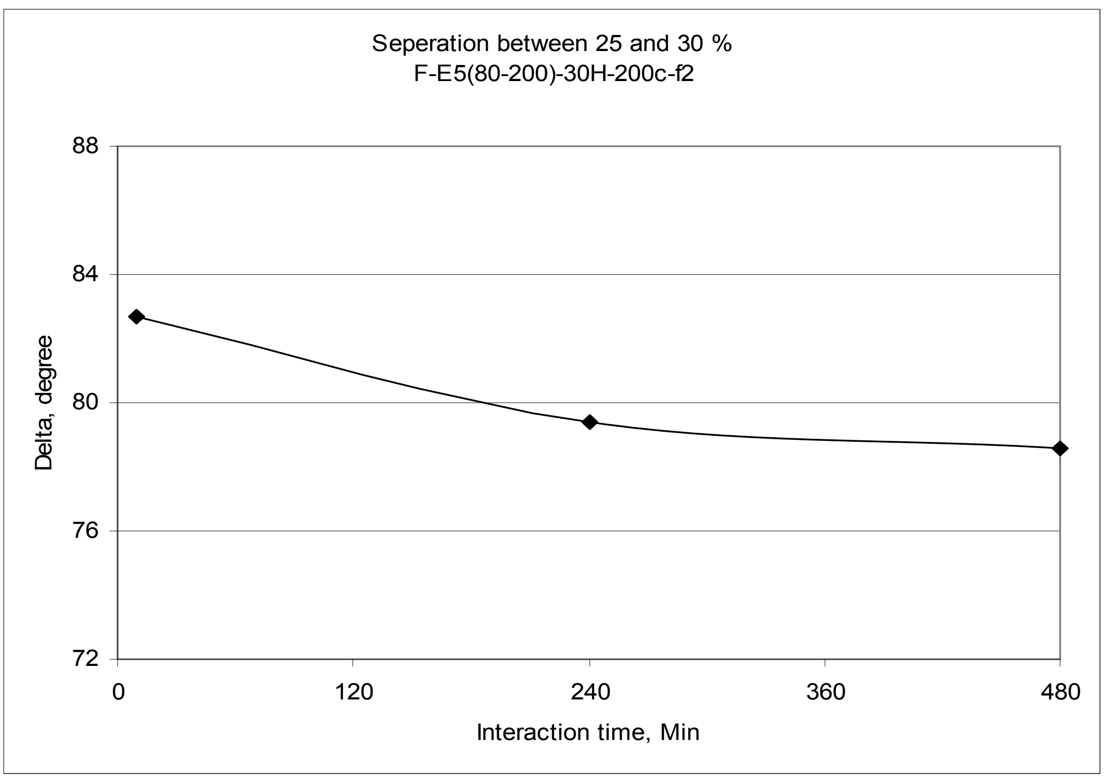
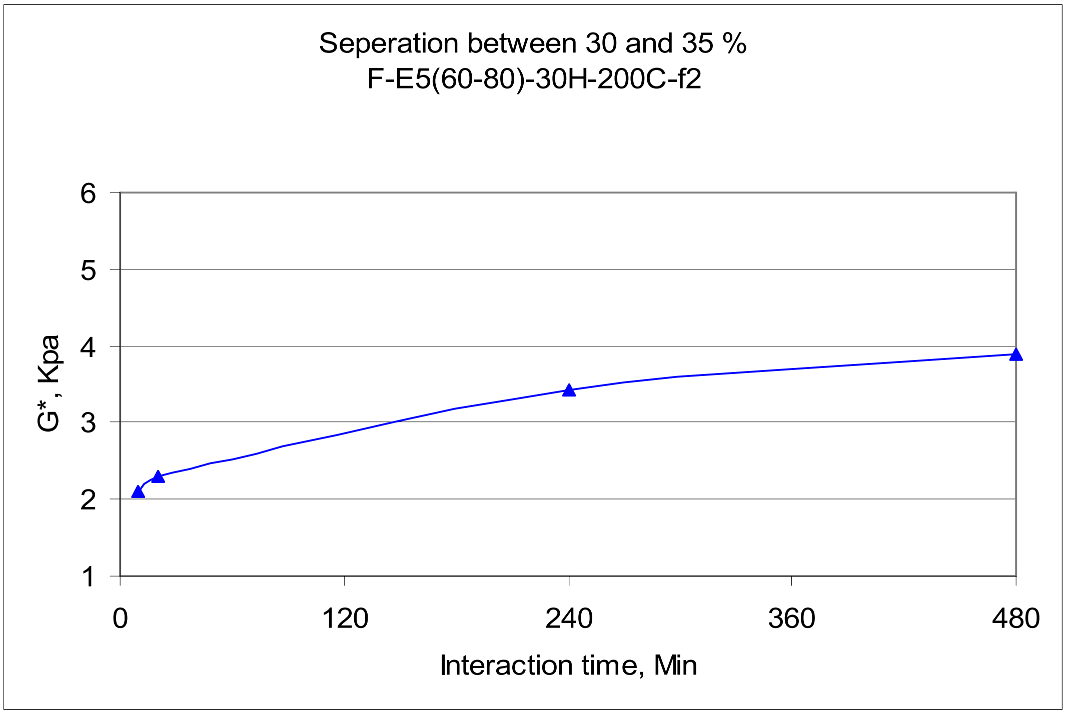


Figure 4-36 Property Development for Binders with Separation between 25% and 30%.



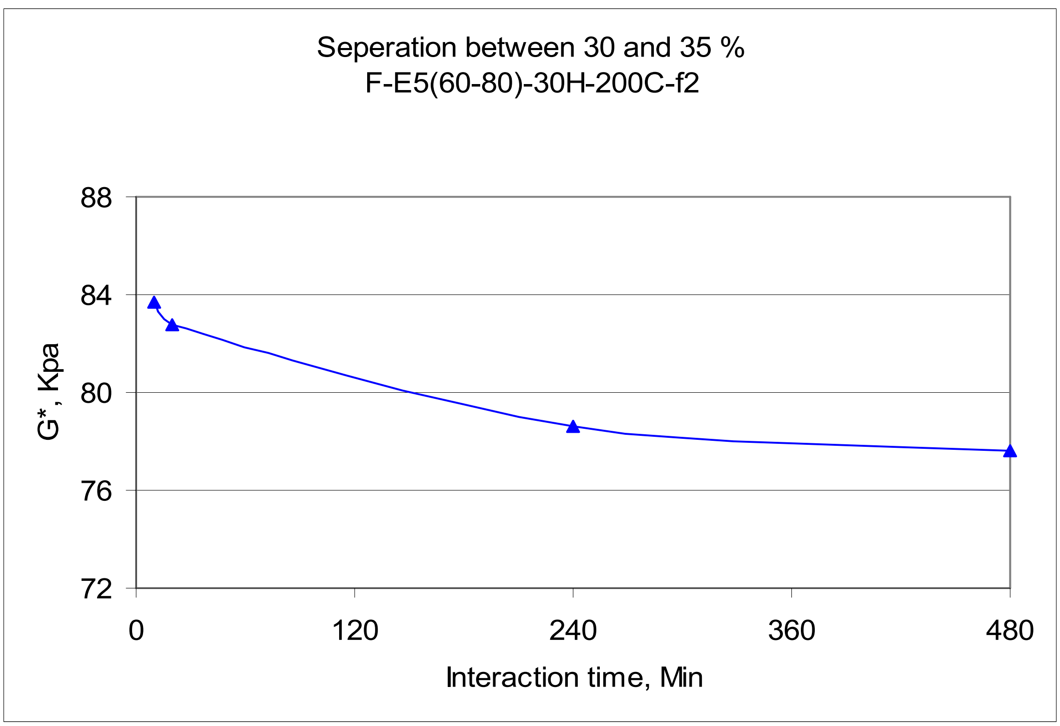


Figure 4-37 Property Development for Binders with Separation between 30% and 35%.

Depolymerization decomposes the binder network into lower molecular weight components. Depolymerization starts very early at high interaction temperatures and continues toward full destruction of the polymer network if the binder is exposed to very high temperature for extended periods of time.

Literature (58) suggests that there are differences in the interaction activities between fine and coarse particle sizes. Swelling activity is greatly affected by the particle size. Different particle sizes are in different interaction stages at any one time, mainly because fine particles achieve their maximum swelling faster than coarse particles and begin to depolymerize earlier. Modification of the liquid phase is also affected by the size of the particles. Because of their high surface area, fine particles absorb more light asphalt components in a shorter period of time, leaving the liquid phase of the binder stiffer. When rubber particles significantly depolymerize after time at high interaction temperature, the liquid phase of a binder made with fine material will be stiffer than the liquid phase of a binder made with a coarse material. Using high shear rate (or high frequency) mixer reduces the particle size of coarse crumb rubber and allows the interaction process to progress with greater speed.

4.5 Controlling the Interaction Process

CRM binders have a unique nature because they consist of a liquid phase containing swollen particles. The liquid phase and suspended particles form the binder structure that controls the binder response to stress. Modifications to the liquid phase are produced as crumb rubber particles absorb lighter fractions during swelling and release rubber components during depolymerization. This combination produces a stiffer liquid phase with more elastic components. Swelling of rubber particles during asphalt absorption affects the binder matrix by decreasing the distance between particles, thereby stiffening the binder. When interacting asphalt with crumb rubber at a given temperature, both actions occur near the beginning of the process, causing modification of both G* and δ. At some point during the interaction process, swelling is replaced by depolymerization. Depolymerization starts releasing rubber components back into the liquid phase, causing a decrease in the G* value while δ continues to modify. If interaction temperature is high or enough time passes, depolymerization will continue, causing destruction of the binder network; modification to δ will then be lost. Both material and interaction process variables affect the timing and the extent of the liquid phase and the matrix modifications.

Rubber sources show insignificant different effects on binder behavior. These differences are less significant at lower interaction temperatures than they are at higher temperatures. The effect of the interaction conditions on the development of the high temperature properties can be summarized as follows:

- The interaction temperature controls the activity of the interaction process. There are two main activities within the interaction process: swelling and depolymerization.

Interaction temperature affects the process by controlling the time when swelling is replaced with depolymerization.

- Shearing energy can be very effective in controlling crumb rubber particle sizes during the interaction process. Higher shear turns coarser particles into fine particles and help stabilize CRM binder properties.

Crumb rubber concentration is a factor that controls developed binder properties. Higher crumb rubber concentrations have more effect on both the matrix and the liquid phase than lower concentrations. A higher crumb rubber concentration increases the amount of light asphalt fractions absorbed by rubber particles, stiffening the liquid phase more than a lower crumb rubber concentration. Increasing the crumb rubber concentration also makes the binder matrix more congested with swollen rubber particles. While this increases the modification of the developed properties, it also produces a binder that is more affected by the high interaction temperature as swollen particles depolymerize. Higher rubber concentrations did not affect the interaction conditions required for the development of G* and δ with any crumb rubber source to the same extent as lower crumb rubber concentrations.

Extending the interaction process at low temperature (160°C -170°C) had no significant effect on the developed binder properties. This included pre-soaking of crumb rubber in asphalt prior to high shearing and high temperature processing.

4.6 Binder Stability Testing

Stability of the CRM binders was calculated based upon the results of two tests following the Cigar Tube Test processing, the Dynamic Shear Rheometer (DSR) and the standard Ring and Ball (R&B) test. The following test results were obtained:

Asphalt + SBS

DSR Separation:
(1-5)% for 3% SBS

R&B Separation:
(0-1) °C for 3% SBS

Asphalt + CRM

DSR Separation:
(10-15)% for 5% CRM
(20-30)% for 7.5% CRM
(30-40)% for 10% CRM

R&B Separation:
(1-2) °C for 5% CRM
(3-5) °C for 7.5% CRM

(7+) °C for 10% CRM

Asphalt + CRM + SBS

DSR Separation:
(7-12)% for 5% CRM +2% SBS
(30+)% for 7.5% CRM +2% SBS

R&B Separation:
(1-2) °C for 5% CRM +2% SBS
(3+) °C for 7.5% CRM +2% SBS

Minimum stability requirements are commonly based on project specifications. Acceptable values normally range between 10% and 15% difference in G*/sin δ values from the DSR test and a difference of one to three degrees Celsius in softening point measured using the R & B test.

4.7 Elastic Recovery Testing

Elastic recovery describes the ability of an asphalt binder to elongate when tension is applied and to recover its original shape when the tension is released. This property is important in both fatigue and rutting resistance. Elastic recovery is a property that is indicative of the quality of polymer components in asphalt binders. ASTM D 6084 procedures are used to test the elasticity of an asphalt binder. The percent recovery was determined after pulling the sample to an elongation of 10 cm, stopping the elongation, immediately cutting the test specimen into two halves, and allowing it to sit undisturbed for a period of 60 minutes in the testing machine. Elastic recovery test results on selected binders are listed in Table 4-1.

4-8 Summary of the Test Results

Figures 4-38 and 4-39 summarize the interaction results and indicate the acceptable interactions of different variables with respect to the high temperature parameter of Superpave binder specifications.

Table 4 -1 Elastic recovery test results.

Sample Id	Material type	Test Method	Test Temperature	Recovery %
F-E5(30-40)-30H-200C-f2	Tank	ASTM D6084-97	25 °C	44
F-E5(40-60)-50H-200C-f2-B				47
F-E5(40-60)-30H-200C-f2				44
F-E5(60-80)-50H-200C-f2-B				47
F-E5(60-80)-50H-170C-f2				54
F-E5(80-200)-50H-200C-f2-B				48
F-E5(30-40)-30H-200C-f2	RTFO			59
F-E5(40-60)-50H-200C-f2-B				59
F-E5(60-80)-50H-200C-f2-B				55
F-E5(80-200)-50H-200C-f2-B				62

.

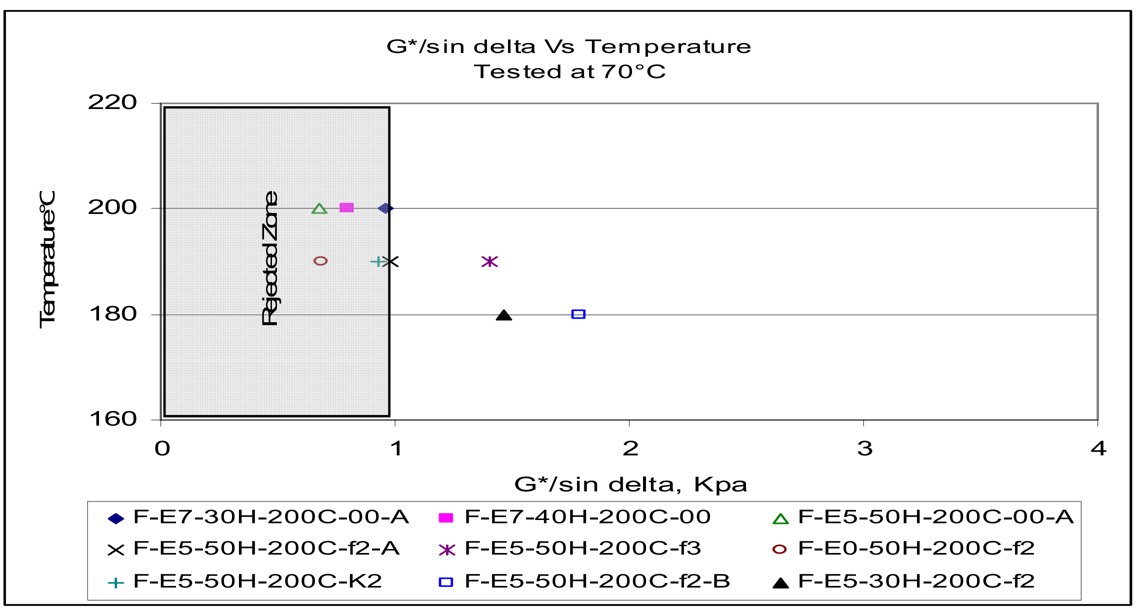


Figure 4-38-a G*/Sin Delta versus Mixing Temperature.

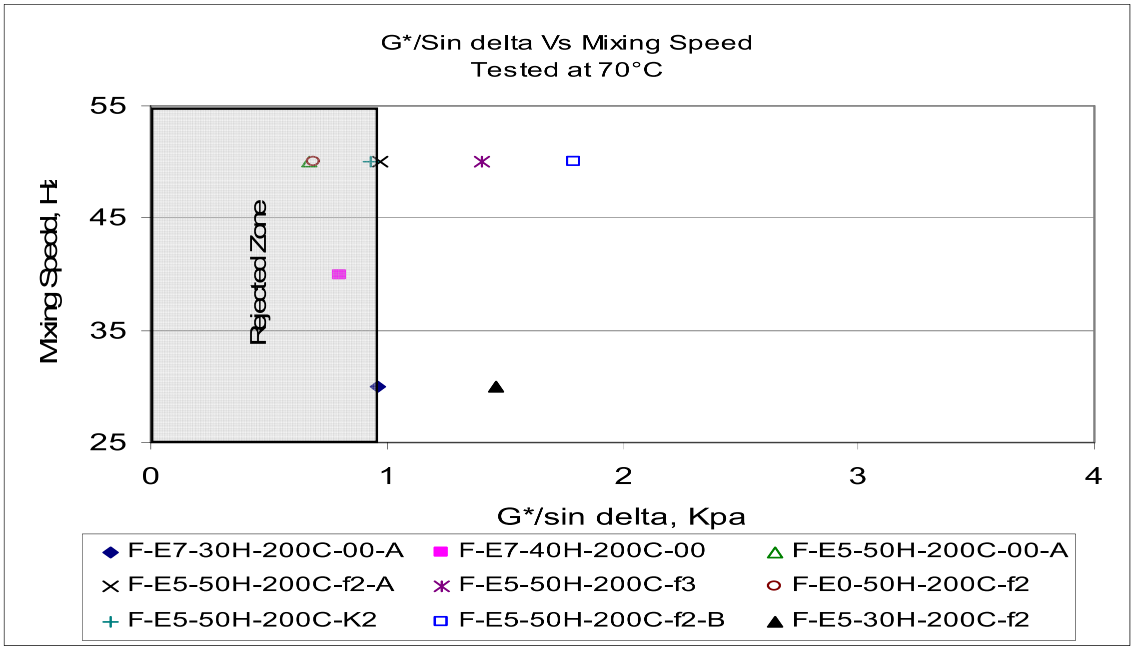


Figure 4-38-b G*/Sin Delta versus Mixing Speed.

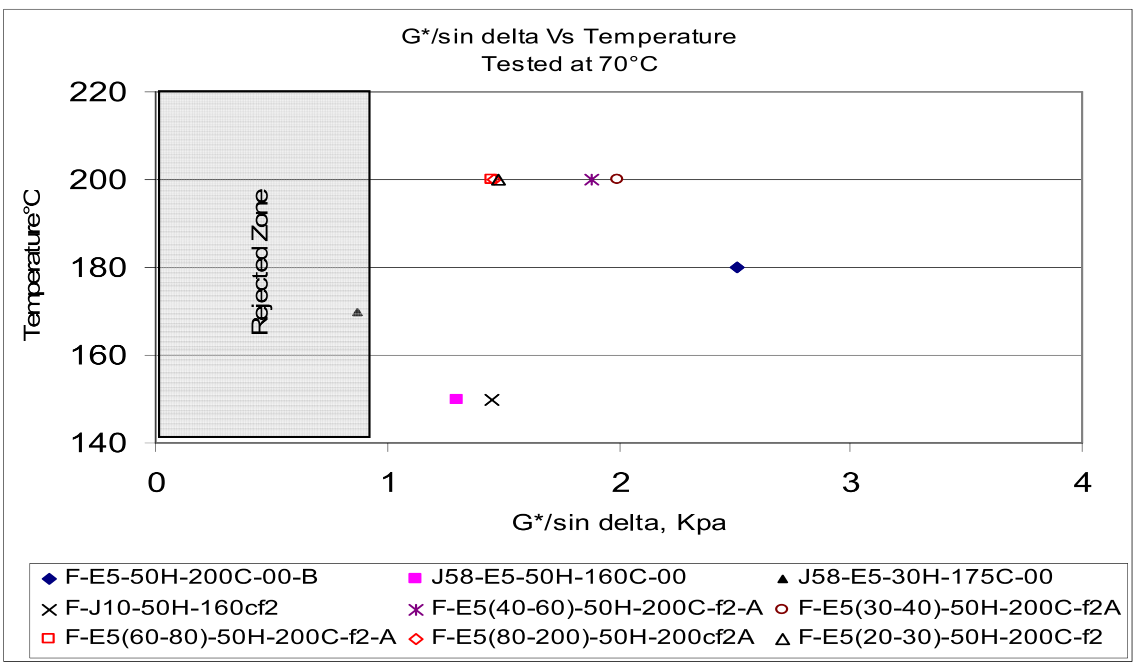


Figure 4-39-a G*/Sin Delta versus Mixing Temperature.

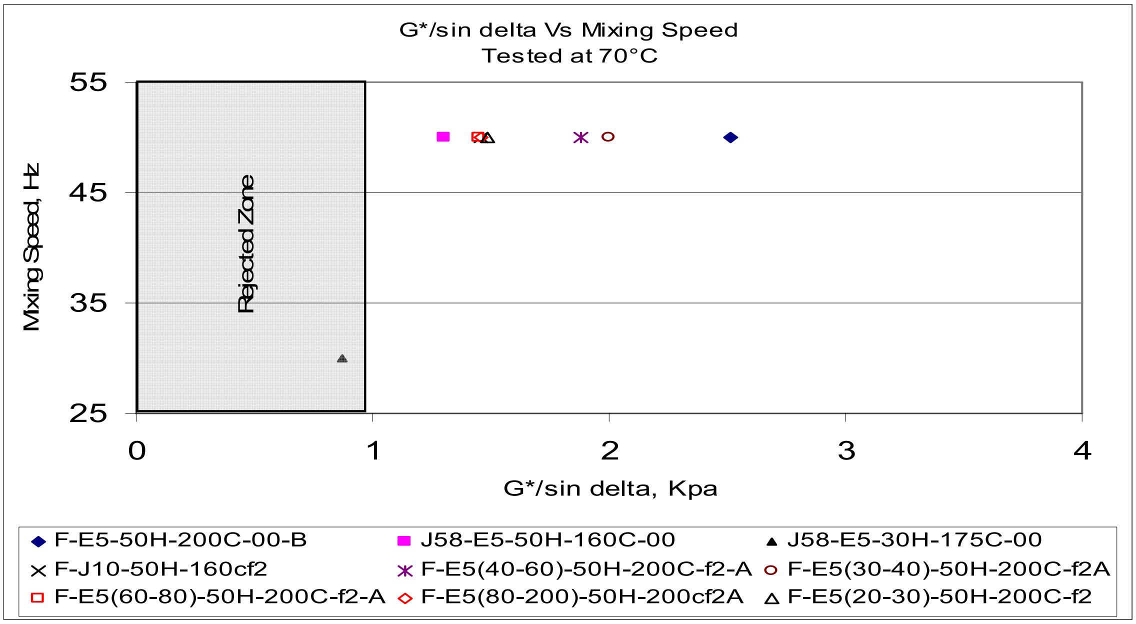


Figure 4-39-b G*/Sin Delta versus Mixing Speed.

CHAPTER FIVE

SUMMARY AND RECOMMENDATIONS

5.1 Introduction

CRM asphalt has been used successfully in a variety of pavement applications. CRM asphalt has the potential to make use of a significantly larger number of scrap tires as modified asphalt binder for HMA pavement. The factor that most adversely influences the demand for CRM asphalt in pavement applications is the use of traditional asphalt testing methods to characterize binder production. Traditional testing methods measure non-fundamental properties of the binder and are not suitable for detecting changes in binder properties related to pavement performance. Traditional testing methods produce contradictions with regard to the effects of material properties on binder performance properties. A more fundamental characterization of the binder production process should be used so that specific performance related properties can be produced by regulating the interaction process. Superpave testing has demonstrated that material properties and interaction variables affect the resulting binder performance properties. Superpave characterization also provides insight into the changes in binder composition that develop during the interaction mechanism.

5.2 Summary of Test Results

As demonstrated by this research, factors that affect binder properties are material variables and interaction variables. Material variables include binder properties and crumb rubber properties. This study tested crumb rubber from two different production processes with different particle size gradations to demonstrate specific property modifications which could be achieved during the production of performance graded asphalt binders.

Interaction process variables include time and temperature. Interaction time was tested for both short term and intermediate term applications. Interaction temperature was studied at three levels, low, intermediate and high. Crumb rubber concentration was also evaluated at three levels, 5%, 7.5% and 10%.

CRM binders have a unique structure consisting of a liquid phase and swollen particles. Both components contribute to the properties of the modified binder. The changes that develop during interaction depend mainly on extraction of the binder's more volatile fractions, which causes swelling of the rubber particles, and then by depolymerization of the swollen particles. Each material property has some effect on the changes that develop in both the liquid phase and the swollen particles. DSR testing of separation, based upon differences in values ($G^*/\sin \delta$ in kPa) from Cigar Tube Test samples, provides a more refined understanding of the mechanisms which can be used to modify the asphalt binder. The ability of the DSR to measure parameters which can be used to regulate modified binder property development was illustrated in Chapter 4. Measuring

changes in fundamental properties that reflect changes in the binder structure is more appropriate than conventional testing methods because fundamental properties can be directly correlated to pavement performance. The use of Superpave testing procedures will allow monitoring of pavement performance properties during the CRM binder production process.

This study focused on the production of CRM binders and their properties prior to mixing with aggregates. Parameters of the interaction process used in developing performance related specifications of CRM binders can be varied by selecting different combinations of material properties and interaction variables. For example:

- Interaction temperature controls progression of interaction activities. For a specific asphalt rubber combination, lower interaction temperature results in particle swelling that will continue for a relatively longer period of time while higher interaction temperature results in particle swelling for only a short period of time followed by depolymerization.
- The effect of interaction time at different temperatures shows there is an initial period when most of the modified binder properties develop followed by a stabilizing period. The duration of the initial period varies depending on CR material properties such as rubber source and particle size.
- Shearing energy controls crumb rubber particle size; higher energy converts coarser particles into finer particles. Finer particles help stabilize the binder production process.
- Higher crumb rubber concentrations have significant effects on the matrix and the liquid phase of the binder. Higher crumb rubber concentrations congest the matrix with swollen particles at low interaction temperatures. At higher interaction temperatures, a higher concentration of smaller particles depolymerizes more quickly, which produces greater changes in modified binder properties. Higher crumb rubber concentrations also absorb more binder volatiles, stiffening the liquid phase more than lower crumb rubber concentrations. Higher crumb rubber concentration did not significantly modify the interaction conditions required for the development of G^* and δ when compared to lower CR concentrations.
- The high temperature properties, G^* and δ, were not developed by the same process. The increase in G^* is mainly due to particle swelling. The decrease in δ continues even during the early stages of depolymerization, indicating that swelling is not the sole factor affecting the development of δ. Component exchange between asphalt and rubber in the early depolymerization stages stiffens the binder liquid because depolymerization (of rubber) adds more elasticity to

the binder. Depolymerization of rubber appears to have the largest effect on modifying the phase angle.

- Binder stability can be achieved through high speed mixing and/or through extending the interaction time, up to a maximum of about eight hours.

The results of research on low temperature properties suggest that modifications to the low temperature properties produced by interaction conditions are limited compared to modifications of high temperature properties. In most cases, low temperature grades changed one negative grade as a result of addition of crumb rubber plus polymer modifiers. The same results were derived when using only polymer modifiers. Earlier work had suggested that crumb rubber without polymer additives can significantly improve the low temperature properties of asphalt binders.

5.3 Recommendations Regarding Testing for CRM Asphalt Applications

Superpave methods have been adopted by many state highway agencies because tests performed on combinations of binder and mineral aggregate can be used to predict pavement performance. These procedures have proven especially valuable when designing pavements for high traffic volume situations. Superpave procedures incorporate pavement performance prediction directly into the asphalt mix design process. However, Superpave testing procedures require an array of equipment above and beyond that required by more traditional testing procedures. Although lower traffic volume applications can often be adequately served by less rigorous testing procedures, it is recommended that Superpave testing be considered for use when designing CRM pavements for any road. Recent research efforts have focused on adapting Superpave technology for use on lower volume roads.

The results of this research suggest that incorporating crumb rubber into an asphalt binder under the interaction conditions specified at a rubber content of 10% or higher in the presence of SBS polymer can result in problems with workability of the modified binder. Incorporating crumb rubber into an asphalt binder at a rubber content of 10% or higher without SBS polymer may result in problems with stability of the modified binder. It is therefore recommended that a maximum of 5% crumb rubber be incorporated into a binder when producing crumb rubber modified asphalt. Results also suggest that roughness/smoothness of the crumb particle surfaces, type of rubber used and production method have less significant effects than rubber concentration on the final product. The only parameter that needs to be specified with regard to crumb rubber properties for CRM asphalt is particle size gradation (i.e. 100% of material will pass #40 sieve).

The authors recommend the establishment of two levels of CRM binders, a Polymer Enhanced (PE) CRM binder containing both crumb rubber (5%) and polymer (2%) and a CRM Binder consisting of asphalt and crumb rubber only. Separation of PE CRM binders should be measured using DSR testing on samples prepared using the Cigar

Tube Test. Maximum acceptable separation values for DSR tested material should not exceed 10% to 15%. The use of Superpave mix design procedures specifying performance graded asphalt binder plus 5% crumb rubber and 2% SBS will produce a pavement with properties similar to performance graded asphalt binder with 3.5% to 4% SBS polymer added. The resulting pavement will exhibit most of the performance improvements normally associated with polymer modified asphalt mixes. Superpave testing procedures and frequency of testing currently prescribed by the NDOR for performance graded pavements should be used for PE CRM binder mixes.

The alternative mix design specifying performance graded asphalt plus 5% crumb rubber will significantly improve more traditional HMA pavement performance. The resulting pavement will also exhibit many of the improvements associated with polymer modified mixes. Stability testing for binders with crumb rubber as the only modifier can be based upon the Ring and Ball test. A difference of between 1^{o} C to 3^{o} C in the softening temperature between the top and bottom portions of samples prepared using the Cigar Tube Test is the recommended maximum acceptable value.

The NDOR should also require elastic recovery testing during crumb rubber binder production to ensure adequate polymer content. Unaged (tank) material should have an elastic recovery not less than 45% while aged material (residue from RTFO or PAV) should exhibit elastic recovery of 55% or greater.

The NDOR should consider implementing the current Superpave quality control and quality assurance procedures for all future projects involving crumb rubber modified asphalt. Research concludes that, with a limit of 5% crumb rubber, binder processing and handling will be comparable to that experienced with the polymer-modified binders already in use while pavement performance will improve significantly.

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APPENDIX

Table A-2 Code Description

Position	Data	code	Meaning
1	Asphalt cement source		
		F	Flint Hills (58-28)
		J58	Modified Jebro (58-34)
		J	Jebro (52-34)
2	CRM Source, size and percentage	E10	10 % Entire
		J7	7.5 % Jai Tire
		E5(40-60)	5 % of Entire Material passing #40 sieve and retained on #60 sieve
3	(Mixing Speed in the main interaction)	50Hz	50 cycle/sec
4	Mixing Temperature in the main interaction	200C	200°C
5	SBS type and percentage	f2	2 % FireStone
		K3	3 % Kraton
6	Different Interaction Parameters	A, B or C	See tables in Appendix for exact parameters
Interaction Temperature			temperature between 160° C -175° C
			temperature between 175° C -200° C
			temperature 220° C
Interaction Time	10 (minutes)		Tank material (unaged)
	Original		
	δ	G*	Those values measured at 10th minute
	30 Hz - 200°c		Those conditions are up to end of the 10th minute

Table A-2 Test Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
Code	Description	Test Temp. °c	orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
			δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep%	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa
F-E7-30H-200C-00-A	FlintHill+ 7.5 % Entire- 25 Hz, 2 hr, 392°F- 30 Hz for 6 hr, 392- 420° F	64	30 Hz - 200°c									test					30 Hz - 200°c												30 Hz - 200°c	
		70										80.5	0.94	79.1	1.14											77.7	1.18			
F-E7-30H-200C-00-B	FlintHill + 7.5 % Entire, 30 Hz, 2 hr, 392 - 430°F -10 Hz, 6 hr, 392° F	64	30 Hz - 200°c											10 Hz - 200°c			10 Hz - 200°c									10 Hz - 200°c			10 Hz - 200°c	
		70										79.7	0.95																	
		19	30 Hz - 200°c																											
		-18	30 Hz - 200°c																											
F-E10-30H-200C-00	Flint Hills + 10 % Entire, 30 Hz, 2 hr,392 - 460°F-10 Hz, 6 hr,392° F	64	30 Hz - 200°c											10 Hz - 200°c			10 Hz - 200°c									10 Hz - 200°c			10 Hz - 200°c	
		70										77.6	1.28																	
F-J7-30H-200C-00	Flint Hills + 7.5 % JaiTire, 30 Hz, 2 hr,392 - 420°F-10 Hz, 6 hr,392° F	64	30 Hz - 200°c											10 Hz - 200°c			10 Hz - 200°c									10 Hz - 200°c			10 Hz - 200°c	
		70										80.7	0.8																	
F-J10-30H-200C-00	Flint Hills + 10 % J-Tire, 30 Hz, 2 hr, 392 - 470°F-10 Hz, 6 hr, 392°F	64	30 Hz - 200°c											10 Hz - 200°c			10 Hz - 200°c									10 Hz - 200°c			10 Hz - 200°c	
		70										82.3	0.69																	
F-E7-40H-200C-00	FlintHill+7.5 % Entire at High shear 2 hr, 392-450°F-10 Hz, 6 hr, 392°F	64	40 Hz - 200°c											10 Hz - 200°c			10 Hz - 200°c									10 Hz - 200°c			10 Hz - 200°c	
		70										83.6	0.76																	
F-E5-50H-200C-00-A	Flint Hills + 5 % Entire, 50 Hz, 20 min, 330 - 430°F-30 Hz, 40 min, 392 - 402°F-10 Hz, 4 hr, 392°F	64	50Hz- 165-220°c				30Hz-200°c				10Hz-200°c						82.6	1.28	16.6	78.3	3.27	10Hz-200°c				End				
		70															84.4	0.67		80.9	1.67									
		19															10Hz-200°c					42.8	5969							
		-18																						221.43	0.29					

Table A-2 (continuted) Test Results (370 to 840 minutes).

Time, Min			380		400		430	480									600									840		
Code	Description	Test Temp. °c	orig		orig			Orig			Rtfo		PAV		BBR		orig			RTFO		PAV.DSR		BBR		orig		
			δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	sep%
F-E7-30H-200C-00-A	FlintHill+ 7.5 % Entire- 25 Hz, 2 hr, 392°F- 30 Hz for 6 hr, 392-420° F	64	30 Hz - 200°c					78.6	1.75	7.28	30 Hz - 200°c					End												
		70						80.8	0.95																			
F-E7-30H-200C-00-B	FlintHill + 7.5 % Entire, 30 Hz, 2 hr, 392 - 430°F - 10 Hz, 6 hr, 392° F	64	10 Hz - 200°c					80.6	1.53	27	77.8	2.71	10 Hz - 200°c			End												
		70						82.4	0.84		80.2	1.39																
		19						10 Hz - 200°c					44.3	5565														
		-18											10 Hz - 200°c		238	0.29												
F-E10-30H-200C-00	Flint Hills + 10 % Entire, 30 Hz, 2 hr,392 - 460°F-10 Hz, 6 hr,392° F	64	10 Hz - 200°c					77.4	1.77	56.1	End																	
		70						79.6	1.01																			
F-J7-30H-200C-00	Flint Hills + 7.5 % JaiTire, 30 Hz, 2 hr,392 - 420°F-10 Hz, 6 hr,392° F	64	10 Hz - 200°c					78.2	1.77	46.2	End																	
		70						80.7	0.96																			
F-J10-30H-200C-00	Flint Hills + 10 % J- Tire, 30 Hz, 2 hr, 392 - 470°F-10 Hz, 6 hr, 392°F	64	10 Hz - 200°c					80.8	1.45	**21**	End																	
		70						81.4	0.79																			
F-E7-40H-200C-00	FlintHill+7.5 % Entire at High shear 2 hr, 392-450°F-10 Hz, 6 hr, 392°F	64	10 Hz - 200°c					79.6	1.43	11.7	End																	
		70						81.3	0.79																			
F-E5-50H-200C-00-A	Flint Hills + 5 % Entire, 50 Hz, 20 min, 330 - 430°F-30 Hz, 40 min, 392 - 402°F-10 Hz, 4 hr, 392°F	64																										
		70																										
		19																										
		-18																										

Table A-3 Test Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
			orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
Code	Description	Test Temp. °c	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep%	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa
F-E5-50H-200C-f2-A	FlintHill+ 5 % Entire, 50 Hz, 20 min, 330 - 430°F -2% Firestone, 30 Hz, 40 min, 392 - 402°F-10 Hz for 4 hr,392°F-10 Hz for 5 hr, 320°F	64															79.5	1.67	19.4											
		70									10Hz-200°c						82	0.88												
		19	50Hz- 165-220°c				30Hz-200°c													10Hz-200°c						10 Hz - 160°c				
		-18									10Hz-200°c																			
F-E5-50H-200C-f3	Flint Hills + 5 % Entire, 50 Hz, 20 min,330 - 430°F-3 % Firestone, 30 Hz, 40 min,392 - 402°F-10 Hz, 4 hr, 392°F-10 Hz, 5 hr,320°F	64	50Hz- 165-220°c				30Hz-200°c				10Hz-200°c						74.5	2.12	8.29	10Hz-200°c										
		70															77.2	1.2												
		16																								10 Hz - 160°c				
		-18	50Hz- 165-220°c				30Hz-200°c				10Hz-200°c									10Hz-200°c										
F-E7-50H-200C-f2	Flint Hills + 7.5 % Entire at 50 Hz, 20 min, 330 - 430°F-2 % Firestone, 30 Hz, 40 min,392-402°F- 10 Hz, 4 hr, 392°F-10 Hz, 5 hr, 320°F	64					30Hz-200°c				10Hz-200°c						77.2	1.92	31.5											
		70															79.9	1.06		73.3	3.1									
		16	50Hz- 165-220°c														10Hz-200°c					43.1	9378			10 Hz - 160°c				
		-18					30Hz-200°c				10Hz-200°c						10Hz-200°c							221.43	0.29					
F-E0-50H-200C-f2	Flint Hills + 2% Firestone, 50 Hz, 20 min,330 - 430°F-30 Hz, 40 min,392 - 402°F-10 Hz, 4 hr, 392°F	64															83.7	1.37		77.5	3.15				End					
		70															85.8	0.69	3.62	80.6	1.53	10Hz-200°c								
		19	50Hz- 165-220°c				30Hz-200°c				10Hz-200°c											42.3	6228							
		-18															10Hz-200°c							284.84	0.28					
F-E5-50H-200C-K2	Flint Hills + 5 % Entire, 50 Hz, 20 min, 330 -	64	50Hz- 165-220°c				30Hz-200°c				10Hz-200°c						75.6	1.6	1.83	10Hz-200°c						10 Hz - 160°c				

	430°F-2 % Kraton, 30 Hz, 40 min,392 - 402°F-10 Hz,4 hr, 392°F- 10 Hz, 5 hr, 320°F	70				77.5	0.91								
F-E5-50H-200C-f2-B	Flint Hills + 5 % Entire, 50 Hz,40 min,302 - 414°F-2 % Firestone, 25 Hz, 4hr,347°F-10 Hz 5 hr ,320°F	64	50Hz- 150-220°c	30Hz-200°c	10Hz-200°c	75.8	3.26	5							10 Hz - 160°c
		70				78.9	1.76		73.3	4.21					
		19									40.2	6441			
		-18											292.1	0.26	
F-E5-30H-200C-f2	Flint Hills + 5 % Entire, 30 Hz,40 min,302 - 414°F-2 % Firestone, 25 Hz, 4hr,347°F-10 Hz 5 hr ,320°F	64	30Hz- 165-220°c	30Hz-200°c	10Hz-200°c	76.3	2.7	5.45							10 Hz - 160°c
		70				79.5	1.44		73.7	2.98					
		19									44.1	13887			
		-18											246.23	0.27	

Table A-3 (continued)   Test Results (370 to 840 minutes).

Time, Min			380		400		430	480									600									840		
			orig		orig			Orig			Rtfo		PAV		BBR		orig			RTFO		PAV.DSR		BBR		orig		
Code	Description	Test Temp. °c	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	sep%
F-E5-50H-200C-f2-A	FlintHill+ 5 % Entire, 50 Hz, 20 min, 330 - 430°F -2% Firestone, 30 Hz, 40 min, 392 - 402°F-10 Hz for 4 hr,392°F-10 Hz for 5 hr, 320°F	64	10 Hz - 160°c														78.7	1.78	14	74.6	3.44	10 Hz - 160°c			End			
		70															81.1	0.97		77.5	1.8							
		19															10 Hz - 160°c					42.6	4911					
		-18																						250	0.298			
F-E5-50H-200C-f3	Flint Hills + 5 % Entire, 50 Hz, 20 min,330 - 430°F-3 % Firestone, 30 Hz, 40 min,392 - 402°F-10 Hz, 4 hr, 392°F-10 Hz, 5 hr,320°F	64	10 Hz - 160°c														73.4	2.42	17.7	69.7	3.17	10 Hz - 160°c			End			
		70															75.8	1.36		72.6	1.77							
		16															10 Hz - 160°c					39.7	6769					
		-18																						236	0.288			
F-E7-50H-200C-f2	Flint Hills + 7.5 % Entire at 50 Hz, 20 min, 330 - 430°F-2 % Firestone, 30 Hz, 40 min,392-402°F- 10 Hz, 4 hr, 392°F-10 Hz, 5 hr, 320°F	64	10 Hz - 160°c														79	1.27	34.4			10 Hz - 160°c			End			
		70															79	1.27		69.1	3.62							
		16															10 Hz - 160°c					39.6	7519					
		-18																						237	0.279			
F-E0-50H-200C-f2	Flint Hills + 2% Firestone, 50 Hz, 20 min,330 - 430°F-30 Hz, 40 min,392 - 402°F-10 Hz, 4 hr, 392°F	64																										
		70																										
		19																										
		-18																										
F-E5-50H-200C-K2	Flint Hills + 5 % Entire, 50 Hz, 20 min, 330 - 430°F-2 % Kraton, 30 Hz, 40 min,392 - 402°F-10 Hz,4 hr, 392°F- 10 Hz, 5 hr, 320°F	64	10 Hz - 160°c																	10 Hz - 160°c					End			
		70															77	1.14	6.39									
F-E5-50H-200C-f2-B	Flint Hills + 5 % Entire, 50 Hz,40 min,302 - 414°F-2 % Firestone, 25 Hz, 4hr,347°F-10 Hz 5 hr ,320°F	64	10 Hz - 160°c																6.18	10 Hz - 160°c					End			
		70															78.8	1.71										
		19															10 Hz - 160°c											
		-18																										
F-E5-30H-200C-f2	Flint Hills + 5 % Entire, 30 Hz,40 min,302 - 414°F-2 % Firestone, 25 Hz, 4hr,347°F-10 Hz 5 hr ,320°F	64	10 Hz - 160°c																5.08	72.1	4.85	10 Hz - 160°c			End			
		70															75	1.71		75.1	2.6							
		19															10 Hz - 160°c					40.9	7035					
		-18																						266	0.27			

Table A-4 Test Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
			orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
Code	Description	Test Temp. °c	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep %	δ	G*, Kpa	Sep %	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep %	δ	G*, Kpa
F-E5-50H-200C-00-B	Flint Hills + 5 % Entire, 50 Hz, 40 min, 302 - 414°F- 33 Hz, 4hr, 347° F	64	50Hz- 150-220°c						33Hz-175°c for 4 hr								75.7	4.77	9.51						End					
		70															78.5	2.46		70.8	5.33									
		19							33Hz-175°c for 4 hr													38.6	8121							
		-18															33Hz-175°c							295.53	0.27					
J58-E5-50H-160C-00	Jebro(58 - 34) + 5 % Entire, 50 Hz, 40 min, 313°F- 25 Hz, 4hr+ 20 min, 347°F	64	50Hz- 165-220°c						25Hz-175°c								65.3	1.92	20.7			25Hz-175°c			End					
		70															67.1	1.2		62.4	2.47									
		19	50Hz- 165-220°c						25Hz-175°c								25Hz-175°c					42.2	6830							
		-18																						128.52	0.31					
J58-E5-30H-175C-00	Jebro(58 - 34) + 5 % Entire, 30 Hz, 2hr, 347°F-25 Hz, 4hr, 347°F	64	30Hz- 175°c									25Hz-175°c														70.1	1.39	11.4	End	
		70																								71.5	0.83			
F-J10-50H-160cf2	Flint Hills + 5 % Jai Tire, 50 Hz, 40 min,302°F- 2% Firestone, 25 Hz, 4hr +20 min, 347°F- 10 Hz, 5hr, 320°F	64	50Hz- 160°c						25Hz-175°c								75.3	3.15	25Hz-175°c							10 Hz - 160°c				
		70															78.2	1.73												
J58-J5-50H-160C-00	Jebro(58 - 34) + 5 % JaiTire, 50 Hz, 40 min, 302°F- 25 Hz, 4hr+ 20 min, 347°F	70	50Hz- 160°c						25Hz-175°c								66.7	0.99	24.1	25Hz-175°c						End				
F-E5(40-60)-50H-200C-f2-A	FlintHill + 5 % Entire(40-60) at 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F- 10 Hz, 4 hr, 392° F.	70	50Hz- 200°c-215°c				25Hz- 200°c				10Hz-200°c						74.2	1.81	7.35	End										
F-E5(30-40)-50H-200C-f2A	FlintHill + 5 % Entire(30-40), 50 Hz, 20 min,392 - 420°F-2 % FireStone, 25 Hz, 40 min,392°F- 10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c				25Hz- 200°c				10Hz-200°c						72.3	1.9	2.49	End										

Table A-4 (continuted) Test Results (370 to 840 minutes).

Time, Min			380		400		430	480									600									840		
Code	Description	Test Temp. °c	orig		orig			Orig			Rtfo		PAV		BBR		orig			RTFO		PAV.DSR		BBR		orig		
			δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	sep%
F-E5-50H-200C-00-B	Flint Hills + 5 % Entire, 50 Hz, 40 min, 302 - 414°F- 33 Hz, 4hr, 347° F	64																										
		70																										
		19																										
		-18																										
J58-E5-50H-160C-00	Jebro(58 - 34) + 5 % Entire, 50 Hz, 40 min, 313°F- 25 Hz, 4hr+ 20 min, 347°F	64																										
		70																										
		19																										
		-18																										
J58-E5-30H-175C-00	Jebro(58 - 34) + 5 % Entire, 30 Hz, 2hr, 347°F-25 Hz, 4hr, 347°F	64																										
		70																										
F-J10-50H-160cf2	Flint Hills + 5 % Jai Tire, 50 Hz, 40 min,302°F- 2% Firestone, 25 Hz, 4hr +20 min, 347°F- 10 Hz, 5hr, 320°F	64	10 Hz - 160°c																15.8	10 Hz - 160°c						End		
		70															76.9	1.42										
J58-J5-50H-160C-00	Jebro(58 - 34) + 5 % JaiTire, 50 Hz, 40 min, 302°F- 25 Hz, 4hr+ 20 min, 347°F	70																										
F-E5(40-60)-50H-200C-f2-A	FlintHill + 5 % Entire(40-60) at 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F- 10 Hz, 4 hr, 392° F.	70																										
F-E5(30-40)-50H-200C-f2A	FlintHill + 5 % Entire(30-40), 50 Hz, 20 min,392 - 420°F-2 % FireStone, 25 Hz, 40 min,392°F- 10 Hz, 4 hr, 392° F	70																										

Table A-5 Test Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
Code	Description	Test Temp. °c	orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
			δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep%	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa
F-E5(60-80)-50H-200C-f2-A	FlintHill + 5 % Entire(60-80), 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c				25Hz- 200°c				10Hz-200°c						79.8	1.43	14.8	75.7	2.46				End					
		16																				40.1	6994							
		-18																						258.49	0.28					
F-E5(80-200)-50H-200cf2A	FlintHill + 5 % Entire(80-200), 50 Hz, 20 min, 392 - 420°F - 2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c				25Hz- 200°c					10Hz-200°c					78.8	1.43	5.54	End										
F-E5(20-30)-50H-200C-f2	FlintHill + 5 % Entire(20-30), 50 Hz, 20 min, 392 - 420°F- 2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c				25Hz- 200°c					10Hz-200°c					78.9	1.45	19	End										
F-J5(60-80)-50H-200C-f2	FlintHill + 5 % Jaitire(60-80), 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c				25Hz- 200°c					10Hz-200°c					78.9	1.04	4.62	End										

F-J5(40-60)-50H-200C-f2	FlintHill + 5 % Jaitire(40-60), 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c	25Hz- 200°c		10Hz-200°c	81.4	1.01	11.1	End										
F-J5(80-200)-50H-200C-f2	FlintHill + 5 % Jaitire(80-200), 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz, 4 hr, 392° F	70	50Hz- 200°c-215°c	25Hz- 200°c		10Hz-200°c	78.8	1.71	13	End										
F-J5(30-40)-50H-200C-f2	FlintHill + 5 % Jaitire(30-40), 50 Hz, 20 min, 392 - 420°F-2 % FireStone, 25 Hz, 40 min, 392°F-10 Hz,4 hr, 392° F	70	50Hz- 200°c-215°c	25Hz- 200°c		10Hz-200°c	80	1.03	5.9	End										

Table A-6 Test Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
			orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
Code	Description	Test Temp. °c	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep %	δ	G*, Kpa	Sep %	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep %	δ	G*, Kpa
J-E5-50H-200C-f2	Jebro(52 - 34)+ 5 % Entire, 50 Hz, 40 min, 392 - 420°F- 2 % Firestone, 30 Hz, 20 min, 392°F- 10 Hz, 4 hr, 392°F	64	50Hz- 200°c-215°c						30Hz-200°c		10Hz-200°c						76.1	1.26	5.91	68.2	3.23				End					
		70															78.8	0.7		71.1	1.81									
		16									10Hz-200°c											41.4	3982							
		-18																						118.16	0.31					
J-E5-30H-200C-00	Jebro(52 - 34) + 5 % Entire, 50 Hz, 40 min, 392 - 420°F- 30 Hz, 20 min, 392°F- 10 Hz,4 hr, 392°F	64	50Hz- 200°c-215°c						30Hz-200°c		10Hz-200°c						81.5	0.89	12.6	76.5	1.94				End					
		13																				42.3	4355							
		-18																						105.69	0.32					
F-E5(30-40)-30H-200C-f2	FlintHill+5%Entire(30-40), 30HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	84.6	1.93	83.3	2.19	83	2	30Hz- 170°c			10Hz-170°c		76.3	3.92	33.9	10Hz- 170°c													
F-E5(40-60)-50H-200C-f2-B	FlintHill+5%Entire(40-60), 50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	82.3	2.35	82	2.27	81.3	2.32	30Hz- 170°c					78.1	3.45	17.2	10Hz- 170°c													
F-E5(40-60)-30H-200C-f2	FlintHill+5%Entire(40-60), 30HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	83	2.32	81.6	2.22	80.7	2.76	30Hz- 170°c			10Hz-170°c		77.5	3.77	16.5	10Hz- 170°c													
F-E5(60-80)-50H-200C-f2-B	FlintHill+5%Entire(60-80), 50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	81.8	2.12	81.1	2.11	80.3	2.94	30Hz- 170°c					77.2	4.5	5.89	10Hz- 170°c													

Table A-6 (continued) Test Results (370 to 840 minutes).

Time, Min			380		400		430	480									600									840		
Code	Description	Test Temp. °c	orig		orig			Orig			Rtfo		PAV		BBR		orig			RTFO		PAV.DSR		BBR		orig		
			δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	sep%
J-E5-50H-200C-f2	Jebro(52 - 34)+ 5 % Entire, 50 Hz, 40 min, 392 - 420°F- 2 % Firestone, 30 Hz, 20 min, 392°F- 10 Hz, 4 hr, 392°F	64																										
		70																										
		16																										
		-18																										
J-E5-30H-200C-00	Jebro(52 - 34) + 5 % Entire, 50 Hz, 40 min, 392 - 420°F- 30 Hz, 20 min, 392°F- 10 Hz,4 hr, 392°F	64																										
		13																										
		-18																										
F-E5(30-40)-30H-200C-f2	FlintHill+5%Entire(30-40), 30HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					75.9	4.2	20.2	End																	
F-E5(40-60)-50H-200C-f2-B	FlintHill+5%Entire(40-60), 50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					77.1	3.74	23.8	End																	
F-E5(40-60)-30H-200C-f2	FlintHill+5%Entire(40-60), 30HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					76.1	3.63	14.3	End																	
F-E5(60-80)-50H-200C-f2-B	FlintHill+5%Entire(60-80), 50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					76.4	5.23	10.1	End																	

Table A-7 Test Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
Code	Description	Test Temp. °c	orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
			δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep%	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa
F-E5(60-80)-30H-170cf2	FlintHill+5%Entire(60-80), 30HZ, 170°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	83.7	2.88	83.2	2.92	82.6	3.07	30Hz- 170°c			10Hz-170°c		76.9	5.07	20	10Hz- 170°c													
F-E5(60-80)-50H-220C-f2	FlintHill+5%Entire(60-80), 50HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	82.5	2.37	82	2.55	81.4	2.77	30Hz- 170°c			10Hz-170°c		77.9	4.19	4.76	10Hz- 170°c													
F-E5(60-80)-30H-220C-f2-A	FlintHill+5%Entire(60-80), 30HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	81.5	2.18	82.9	2.04	82.2	2.14	30Hz- 170°c			10Hz-170°c		78.8	3.12	2.49	10Hz- 170°c													
F-E5(80-200)-50H-200C-f2-B	FlintHill+5%Entire(80-200), 50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	82.6	2.15	81.9	2.29	80.8	2.67	30Hz- 170°c			10Hz-170°c		77.5	4.23	22	10Hz- 170°c													
F-E5(80-200)-30H-200C-f2	FlintHill+5%Entire(80-200), 30HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	82.7	2.02	82.7	2	82.4	1.95	30Hz- 170°c			10Hz-170°c		79.4	3.2	20.1	10Hz- 170°c													
F-E5(80-200)-50H-170C-f2	FlintHill+5%Entire(80-200), 50HZ, 170°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	84.1	2.18	83.3	2.34	82.4	2.58	30Hz- 170°c			10Hz-170°c		78.3	3.84	16.6	10Hz- 170°c													
F-E5(80-200)-30H-170C-f2	FlintHill+5%Entire(80-200), 30HZ, 170°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	85.1	2.02	85.1	1.92	84.5	2.03	30Hz- 170°c			10Hz-170°c		80	3.43	31.3	10Hz- 170°c													
F-E5(80-200)-30H-220C-f2-A	FlintHill+5%Entire(80-200), 30HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	83.1	1.84	82.5	1.95	82.6	2.23	30Hz- 170°c			10Hz-170°c		80.3	2.93	2.59	10Hz- 170°c													

Table A-7 (continued)   Test Results (370 to 840 minutes).

Time, Min			380		400		430	480									600									840		
Code	Description	Test Temp. °c	orig		orig			Orig			Rtfo		PAV		BBR		orig			RTFO		PAV.DSR		BBR		orig		
			δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	sep%
F-E5(60-80)-30H-170cf2	FlintHill+5%Entire(60-80), 30HZ, 170°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					75.6	4.86	18.9	End																	
F-E5(60-80)-50H-220C-f2	FlintHill+5%Entire(60-80), 50HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					77.1	4.64	9.53	End																	
F-E5(60-80)-30H-220C-f2-A	FlintHill+5%Entire(60-80), 30HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					78.8	3.53	15.7	End																	
F-E5(80-200)-50H-200C-f2-B	FlintHill+5%Entire(80-200), 50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					76.8	4.93	11.7	End																	
F-E5(80-200)-30H-200C-f2	FlintHill+5%Entire(80-200), 30HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					78.6	3.54	27.4	End																	
F-E5(80-200)-50H-170C-f2	FlintHill+5%Entire(80-200), 50HZ, 170°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					77.8	4.93	21.4	End																	
F-E5(80-200)-30H-170C-f2	FlintHill+5%Entire(80-200), 30HZ, 170°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					79.1	3.42	19.6	End																	
F-E5(80-200)-30H-220C-f2-A	FlintHill+5%Entire(80-200), 30HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					79.2	3.1	12.8	End																	

Table A-8 Testing Results (0 to 370 minutes).

Time, Min			10		20		40		60		70	120		240			300									360			370	
Code	Description	Test Temp. °c	orig		orig		orig		orig			orig		orig			orig.DSR			RTFO.DSR		pav.DSR		BBR		orig			orig	
			δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	δ	G*, Kpa	Sep%	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa
F-E5(60-80)-30H-220C-f2-B	FlintHill+5%Entire(60-80), 30HZ, 220°C, 20min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	80.8	2.22	81.4	2.16	30Hz- 170°c					10Hz-170°c		79.3	3.01	15.7	10Hz- 170°c													
F-E5(80-200)-30H-220C-f2-B	FlintHill+5%Entire(80-200), 30HZ, 220°C, 20min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	83	2.01	82.3	2.08	30Hz- 170°c					10Hz-170°c		79.1	3.24	23.2	10Hz- 170°c													
F-E5(40-60)-50H-220cf2	FlintHill+5%Entire(40-60), 50HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	80.1	2.43	80.6	2.61	80.7	3.27	30Hz- 170°c			10Hz-170°c		76.9	4.87	0.64	10Hz- 170°c													
F-E5(60-80)-50H-200C-f2-C	FlintHill+5%Entire(60-80),10Hz, 170°C, 6 hr -50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 14 hr	58	test		10Hz-170°c up to end of 6 hours then go t0 50 Hz at 200°C for 40 minutes																					82.3	1.98	50Hz- 200°c	81.5	2.29
F-E5(30-40)-50H-200C-f2-C	FlintHill+5%Entire(30-40),10Hz, 170°C, 6 hr -50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 14 hr	58	85.9	1.9	10Hz-170°c up to end of 6 hours then go t0 50 Hz at 200°C for 40 minutes																					81	2.32		80.6	2.32
F-E5(40-60)-50H-200C-f2-C	FlintHill+5%Entire(40-60),10Hz, 170°C, 6 hr -50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 14 hr	58	85.1	2.02	10Hz-170°c up to end of 6 hours then go t0 50 Hz at 200°C for 40 minutes																					82.3	2.15		80.8	2.14

Table A-8 (continued) Test Results (370 to 840 minutes).

Time, Min			380		400		430	480									600									840		
Code	Description	Test Temp. °c	orig		orig			Orig			Rtfo		PAV		BBR		orig			RTFO		PAV.DSR		BBR		orig		
			δ	G*, Kpa	δ	G*, Kpa		δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	Sep%	δ	G*, Kpa	δ	G*, Kpa	S, MPA	m	δ	G*, Kpa	sep%
F-E5(60-80)-30H-220C-f2-B	FlintHill+5%Entire(60-80), 30HZ, 220°C, 20min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					78.7	3.3	18.3	End																	
F-E5(80-200)-30H-220C-f2-B	FlintHill+5%Entire(80-200), 30HZ, 220°C, 20min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					79	3.21	21.3	End																	
F-E5(40-60)-50H-220cf2	FlintHill+5%Entire(40-60), 50HZ, 220°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 8 hr	58	10Hz- 170°c					76.4	5.16	11.7	End																	
F-E5(60-80)-50H-200C-f2-C	FlintHill+5%Entire(60-80),10Hz, 170°C, 6 hr -50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 14 hr	58	80.1	2.49	79.5	3.02	30Hz- 170°c	10Hz- 170°c									76.4	4.26	23.1	10Hz- 170°c						75	4.48	12
F-E5(30-40)-50H-200C-f2-C	FlintHill+5%Entire(30-40),10Hz, 170°C, 6 hr -50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 14 hr	58	79.7	2.52	79.7	3.32		10Hz- 170°c									75.5	5.19	12.3	10Hz- 170°c						73.6	5.3	18.8
F-E5(40-60)-50H-200C-f2-C	FlintHill+5%Entire(40-60),10Hz, 170°C, 6 hr -50HZ, 200°C, 40min - 2%FireStone, 30Hz, 170°C, 30 min - 10Hz,170°C up to 14 hr	58	80.2	2.86	79.5	3.15		10Hz- 170°c									75.8	4.74	11.8	10Hz- 170°c						75.3	4.8	32.2

Table A-9 Test Results (370 to 840 minutes).

Time. Min				60			120		240		360	
Code	Description	Test Temp. ºC		original			original		original		original	
				δ	G*		δ	G*	δ	G*	δ	G*
J-S10-0H-180C-00	Jebro+ 10% Sack- Mixed with spatula, 180ºC	58	Mixed with Spaula - 180ºC	82.4	1.399	Mixed with Spaula - 180ºC	78.1	1.441	76.4	2.029		
		64							79.3	1.117		
J-E10-0H-180C-00	Jebro+ 10% Entire- Mixed with spatula, 180ºC	58	Mixed with Spaula - 180ºC	85.0	1.178	Mixed with Spaula - 180ºC	85.0	1.344	81.2	1.823		
M-S10-0H-180C-00	Monarch+ 10% Sack- Mixed with spatula, 180ºC	58	Mixed with Spaula - 180ºC	81.6	3.220	Mixed with Spaula - 180ºC	81.1	3.300	73.8	4.926		
		64		83.8	1.556		83.5	1.592	77.7	2.427		
		70							80.8	1.271		
M-E10-0H-180C-00	Monarch+ 10% Entire- Mixed with spatula, 180ºC	58	Mixed with Spaula - 180ºC	84.2	2.892	Mixed with Spaula - 180ºC	81.7	2.913	77.4	4.510		
		64		86.2	1.392		83.2	1.429	81.4	2.249		
		70							84.2	1.159		
F-S10-0H-180C-00	Flint Hills+ 10% Sack- Mixed with spatula, 180ºC	58	Mixed with Spaula - 180ºC			Mixed with Spaula - 180ºC	76.2	4.201	71.6	5.058		
		64		82.0	1.724		79.9	2.111	75.5	2.712		
		70					82.5	1.123	78.9	1.479		
F-E10-0H-180C-00	Flint Hills+ 10% Entire- Mixed with spatula, 180ºC	58	Mixed with Spaula - 180ºC	83.4	3.156	Mixed with Spaula - 180ºC	81.3	3.818	76.9	4.498		
		64		85.7	1.518		84.4	1.833	81.1	2.236		
		70							84.1	1.178		
F-00-CH-200C-3f	FlintHill(58 - 28)+ 3%Fire stone	58	Sheared at 200°C								71.4	8.2552
F-00-CH-200C-3k	FlintHill(58 - 28)+ 3% Kraton stone	58	Sheared at 200°C								75	3.5897
J-00-CH-200C-4f	Jebro (52 - 34)+ 4%Firestone	58	Sheared at 200°C								78.9	2.5139
J-00-CH-200C-4k	Jebro (52 - 34)+ 4%Kraton	58	Sheared at 200°C								67.1	4.377