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The utilization of agriculturally derived lignin as an antioxidant in asphalt binder

by

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ABSTRACT

Oxidation is the primary cause of long-term aging in asphalt pavements. As a pavement oxidizes, it stiffens and can eventually crack. The use of an antioxidant as a performance enhancer in an asphalt binder could delay aging, thus increasing the life of an asphalt pavement. Lignin is a highly-available, well-studied antioxidant. A wet-mill ethanol plant produces several co-products, some of which contain lignin. The use of lignin from ethanol production could provide benefit to asphalt pavements while also giving more value to the co-products. Three lignin-containing co-products were each combined with four asphalt binders separately in varying amounts to determine the optimum amount of co-product that would provide the greatest benefit to the asphalt binders. The asphalt binder/co-product blends were evaluated according to Superpave specifications and performance graded on a continuous scale. The data indicates a stiffening effect of the binder caused by the addition of the co-products. The more co-product added, the greater the stiffening. Binder stiffening benefits the high temperature properties, while the low temperature binder properties are negatively affected. However, the low temperature stiffening effects are small, and in many cases not significant. The co-products have an overall effect of widening the temperature range of the binders. This suggests some antioxidant activity between the binder and the lignin. Testing with a fourth co-product with no lignin supports the idea that lignin acts as an antioxidant. The samples with no lignin aged significantly more than the samples with lignin. Infrared spectrometry also supports the idea that lignin acts as an antioxidant by decreases in some oxidative aging products.

1. INTRODUCTION

1.1 Background

The use of performance enhancers in asphalt binder is not a new concept. Many materials exist and are in wide use which improve the properties of asphalt binder. Thermoplastic copolymers improve asphalt binder by increasing high and low temperature rheological properties (Lucena *et al*. 2004). Chemicals such as hydrated lime and polyphosphoric acid also improve various properties of asphalt binder (Little and Peterson 2005, Edwards *et al*. 2006). However, there are no performance enhancers in widespread use which slow the oxidative aging of asphalt binder by acting as an antioxidant. Slowing oxidative aging of asphalt binder would have great potential benefit for hot-mix asphalt pavements. As a pavement ages, it stiffens and becomes more susceptible to failure from load and thermal stresses (Roberts *et al*. 1996, Liu *et al*. 1998). Slowing oxidative aging would increase the service life of a pavement. The public could benefit by saving money and having less construction inconveniences. However, an antioxidant for use in asphalt pavements would need to be available in large amounts and be cost effective.

Antioxidants have been studied in asphalt pavements, but none have proven to be practical for incorporation into the asphalt industry. Lignin, a known antioxidant, is the second most available biological polymer on earth (Dizhbite *et al*. 2004). Lignin is found in many sources such as timber, grass, and corn (Glasser and Sarkanen 1989). A kernel of corn contains a relatively small amount of lignin in the outer coating (Gulati *et al*. 1997). Corn has many uses, including its use in the growing ethanol industry. Currently, approximately 1.8 billion bushels of corn are processed annually for the production of ethanol, which uses 17 percent of the United State’s corn crop (Iowa Corn 2006). Present technology allows only the starch to be used to make ethanol, while over twenty five percent of the kernels are left for other uses (Bothast and Schlicher 2005). The main use of most ethanol co-products is livestock feed. However, with ethanol production forecasted to be about ten billion gallons in 2015 (Urbanchuk 2006), the amount of co-products produced will be so large that new uses will have to be found to ensure the success of the ethanol industry (Demirbas and Balat 2006). Utilizing the antioxidant potential of lignin derived from ethanol production could provide benefit to both the ethanol and asphalt industries. The potential synergies between the two large industries could possibly create many new economic opportunities. Also, using an organic, renewable performance modifier in asphalt pavements has an environmentally friendly appeal. “Green pavements” could possibly have the same impact on the construction materials industry as ethanol has been to the energy sector.

1.2 Objective

The main objective of this study is to evaluate the effects of lignin-containing ethanol co-products for use in asphalt binder. This is a first-phase study which evaluates whether the co-products have an overall positive or negative effect on the binders. Work was performed to analyze how the lignin-containing co-products chemically and physically interact with samples of asphalt binder.

1.3 Thesis Arrangement

This thesis first presents a literature review that summarizes the past use of antioxidants in asphalt binder. Asphalt oxidation is explained, along with how lignin acts as an antioxidant. State-of-the-art experimental methods were explored to determine the best possible method of analysis. Chapter 3 explains the experimental procedures used to analyze lignin-containing co-products as performance enhancers in asphalt binder. The experimental plan is also outlined and rationalized. The results of the various experiments are illustrated and summarized in Chapter 4. Chapter 5 is dedicated to the statistical analysis of the data. General trends are presented, which state how the co-products affect the various binders. The final chapter states the conclusions of the experiment. Recommendations are made, along with future work that can be performed to better understand the effect that lignin-containing co-products have on asphalt materials.

2. LITERATURE REVIEW

2.1 The Economy and Biofuels

The United States is currently working to establish a more bio-based economy (Demirbas and Balat 2006). A bio-based economy utilizes energy from renewable organic matter rather than fossil fuels. Biofuels have several advantages over fossil fuels. Biofuels are renewable, environmentally friendly, provide energy security, and present a large economic opportunity for the United States (Demirbas and Balat 2006).

The most developed and widely used biofuel on the market is corn-derived ethanol. Production of ethanol from corn is not a new technology. Ethanol was first introduced in the early 1900s for use in the first Ford Model Ts (Bothast and Schlicher 2005). The carburetor on the Model T had an adjustment valve that allowed for the use of ethanol. It was Henry Ford's vision to build an affordable vehicle that could be powered by a fuel that would boost the U.S. economy (Bothast and Schlicher 2005). Ethanol was used well into the 1930s. However, after World War II, gas and oil became so abundant and affordable that ethanol nearly disappeared from the market. It was not until the 1970s that ethanol came back into use. In 1973, political strife within the Middle East and the Organization of Petroleum Exporting Countries (OPEC) caused large gas and oil supply interruptions for the United States. That resulted in a renewed interest in renewable energy sources, particularly ethanol. In 1979, the U.S. produced approximately ten million gallons of ethanol (Bothast and Schlicher 2005). Interest in ethanol continued to grow as Congress passed the Clean Air Act of 1990. Legislation mandated that gasoline contain an oxygen source to reduce toxic emissions (Bothast and Schlicher 2005, Gulati *et al.* 1997). Oxygenates ethanol and methyl-tert-butyl-ether (MTBE) help reduce emissions, displace toxic components of gasoline, reduce emissions of volatile organic compounds, carbon monoxide, nitrogen oxides, and other toxics (Renewable Fuels Association 2004). However, MTBE is a known pollutant and has caused widespread contamination of water supplies (Renewable Fuels Association 2004). Approximately thirty percent of urban water wells have been negatively impacted by MTBE. Nationwide, it is the second most found chemical in groundwater next to formaldehyde (Renewable Fuels Association 2004). Ethanol is a much better alternative to MTBE because it is entirely biodegradable, and only twice the volume of ethanol is needed since it contains twice the amount of oxygen as MTBE (Bothast and Schlicher 2005). Due to the gradual phase out of MTBE and the greater demand for energy, ethanol production has grown dramatically from 2.8 billion gallons in 2003 to 4.9 billion gallons in 2006 (Bothast and Schlicher 2005). Ethanol production is forecasted to be approximately 9.8 billion gallons in 2015 (Urbanchuk 2006).

Not only is ethanol better for the environment and U.S. energy security, ethanol is good for the economy (Bothast and Schlicher 2005, Demirbas and Balat 2006, Renewable Fuels Association 2004). Rural America benefits greatly from the use of corn as a renewable fuel. Ethanol increases the demand for corn, which raises corn profits, putting more money in many economically depressed rural areas. Also, the increased corn prices help lower federal farm program costs. In 2006 alone, the ethanol industry was responsible for creating over 160,000 jobs and boosted U.S. household income by nearly 6.7 billion dollars (Iowa Corn 2006, Urbanchuk 2006). The average 100 million gallon per year ethanol plant annually produces 406 million dollars for the local economy, 223 billion of gross output, and supports nearly 1,600 jobs, increasing household income by 50 million dollars (Iowa Corn 2006, Urbanchuk 2006).

Ethanol production produces many different co-products (Bothast and Schlicher 2005). The type of co-products created depends upon the method of ethanol production. There are two different methods used to produce ethanol: dry mill and wet mill. The majority of ethanol plants in the United States are dry mill (66%) (Bothast and Schlicher 2005). Dry milling focuses on ethanol production and maximizing capital return (Bothast and Schlicher 2005). Dry milling involves milling the whole corn kernel, liquefying it to produce a mash, and then adding enzymes and yeast to produce ethanol (Bothast and Schlicher 2005). The ethanol is then distilled from the mixture. For every one bushel of corn (56 lbs), approximately 2.8 gallons of ethanol is produced (Bothast and Schlicher 2005, Iowa Corn 2006). Also, approximately 17 lbs of dried distillers grains are produced, which is the main co-product of dry milling. Dried distillers grains are predominately used as livestock feed (Bothast and Schlicher 2005, Iowa Corn 2006).

Wet milling is a much different process. Wet-mill ethanol plants are more of a biorefinary, which separate the corn kernel into different components before ethanol is made (Bothast and Schlicher 2005, Gulati *et al*. 1997). Figure 1 illustrates the differences between the wet-mill and dry-mill process. Approximately 70-72 percent of the kernel is starch, which is converted into ethanol (Gulati *et al*. 1997). The rest of the components are turned into many different co-products. For every one bushel of corn, approximately 2.5 gallons of ethanol are produced. Also, 1.6 pounds of corn oil, 2.6 pounds of gluten meal, and 13.5 pounds of gluten feed are produced (Bothast and Schlicher 2005). Different wet mill plants produce different co-products. Many new co-products are being developed that have many unexplored uses. Utilization of co-products from ethanol production is vital to the success

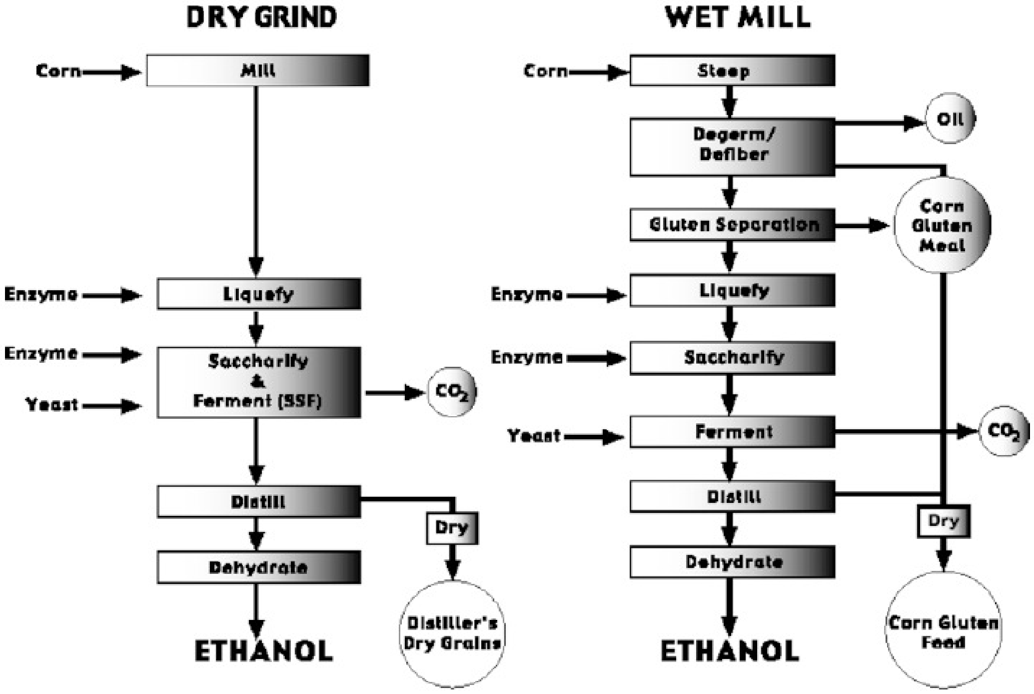

Figure 1. Different ethanol production methods (Bothast and Schlicher 2005)

and profitability of the ethanol industry (Bothast and Schlicher 2005, Van Dam and DeKlerk-Engles 2005). Many uses for various co-products have yet to be discovered.

The outer hull of the kernel contains the fiber which is composed of cellulose, hemicellose, and lignin (Gulati *et al*. 1997). Predominately, these are used to create corn gluten meal. The value of corn gluten fluctuates with the available supply and the cost of other competing animal feed sources (Bothast and Schlicher 2005). Profitability simply as an animal feed is not guaranteed, especially with the forecast for over ten billion gallons of ethanol is 2015. There will be an abundance of co-products with no identified markets with the current ethanol production forecast (Cooper 2005). A new use for the co-products is essential for the success of the ethanol industry.

The corn gluten meal contains lignin (Bothast and Schlicher 2005). Lignin is not known to have much value as a nutrient for animal feed. Utilizing the lignin as a chemical antioxidant provides the ethanol industry with more opportunities to be successful. If the lignin-containing co-products could be used as an antioxidant in asphalt pavements, both industries could greatly benefit.

2.2 Asphalt Oxidation

Asphalt oxidation is the main cause of long-term deterioration in asphalt pavements (Domke *et al*. 2000, Herrington *et al*. 1994, Liu *et al*. 1998). The complex reaction of the various chemicals present in asphalt with atmospheric oxygen cause aging throughout the life of the pavement. Oxidative aging causes the asphalt to harden and become brittle, which can eventually lead to pavement failure (Herrington *et al*. 1994, Ruan *et al*. 2003). Most of the aging that occurs in asphalt occurs during the process of blending the aggregate with the asphalt binder in the intense heat of an asphalt plant (Roberts *et al*. 1996). The rate of oxidative aging is primarily controlled by temperature. The oxidation rate of asphalt approximately doubles for every 10°C rise in temperature (Gawel and Baginksa 2004). During the short duration the asphalt is heated, the temperature can reach up to 135-165°C (Roberts *et al*. 1996). Substantial rheological changes can occur, such as increased viscosity and decreased ductility (Domke *et al*. 2000, Herrington *et al*. 1994, Liu *et al*. 1998). The asphalt binder in the mixture continues to age while the mix is being stored, transported, and eventually paved during construction (Roberts *et al*. 1996).

Long-term oxidative aging begins immediately after a pavement has been constructed (Roberts *et al*. 1996). Long-term oxidation occurs at a much slower rate than the initial oxidation of asphalt during mixing and construction. However, many experts believe long-term oxidative aging to be the cause for many pavement failures in the field (Domke *et al*. 2000, Herrington *et al*. 1994, Liu *et al*. 1998). Long-term aging causes a further large increase in stiffness and loss of ductility. Eventually, thermal and/or load stresses cause the pavement structure to crack. These cracks can unite, forming alligator cracks and eventually potholes or other pavement failures (Figure 2). Retardation of oxidative aging would maintain the elastic properties of the asphalt binder providing substantial benefit to the pavement structure.

Figure 2. Alligator cracks and pothole formation

Oxidative aging of asphalt is controlled by the chemical properties of the asphalt (Liu *et al*. 1998). Asphalt binders vary widely in chemical composition (Roberts *et al*. 1996). Their composition is determined by place of origin and the refining techniques used to produce the asphalt. Asphalt binder is derived from crude oil from places all around the world. The crude oil of each area greatly differs because of wide geological variability. This variability gives the asphalt binders widely differing chemical properties (Roberts *et al*. 1996). The important physical properties civil engineers depend on for strength and durability are directly influenced by the chemical properties.

Asphalt binder is mainly composed of carbon and hydrogen, with nitrogen and sulfur filling in the lower percentages (Peterson 1984). Trace amounts of heavy metals, such as vanadium and nickel, are also present. These elements combine to form the main fractions of asphalt cement: asphaltenes, saturates, naphthalene aromatics and polar aromatics (light and heavy resins, respectively). This separation method is known as Corbett's method and is the most widely used method to fractionate asphalt (Ruan *et al.* 2003). These four fractions chemically and physically interact with each other, forming a complex colloidal system. Asphaltenes and saturates are normally incompatible compounds, but they are brought together by the presence of aromatics. Each fraction provides different properties of asphalt. Asphaltenes are the main contributors of viscosity (hardening effects) while an abundance of aromatics and saturates decrease the ductility (elastic effects) (Ruan *et al*. 2003).

Different asphalts undergo oxidation at differing rates. Asphalts of different origin and distillation methods oxidize differently. Also, the presence of a polymer can also affect the aging rate (Ruan *et al*. 2003). Polymers, such as styrene-butadiene-styrene (SBS) and styrene-b-butadiene (SBR), are commonly used in high-volume roadways to increase the high and low temperature properties of asphalt (Ruan *et al*. 2003). Polymers work by providing increased viscosity at high temperatures and increased ductility at low temperatures (Ruan *et al*. 2003, Lucena *et al*. 2004). By increasing the high and low temperature properties, the service life of a pavement is increased by reducing the pavement's ability to form ruts or crack. When a polymer-modified binder undergoes oxidative aging, two simultaneous reactions occur. The binder undergoes oxidation while the polymer chemically degrades (Ruan *et al*. 2003). When a polymer-modified binder oxidizes, the asphaltene content of the binder increases. This causes decreased temperature susceptibility with regard to stiffness and viscosity. A decrease in the molecular size of the polymer produces the same effect. As a polymer is degraded in size, the polymer-asphalt interactions are reduced. Therefore, the asphalt's temperature susceptibility is a function of the combined effect of asphalt oxidation and polymer degradation (Ruan *et al*. 2003). Even the presence of a polymer cannot stop the deleterious effects of oxidative aging on an asphalt pavement (Ruan *et al*. 2003). There are many polymers available for use in asphalt pavements. Many polymers have the similar effects of decreasing an asphalt's temperature susceptibility. However, none of them truly act as an antioxidant by preventing aging from oxygen radicals (Lucena *et al*. 2004).

Chemical antioxidants have been studied in asphalt pavements. Chemicals such as lead diamyldithiocarbamate (LDADC), zinc kailkyldiathiophashate (ZDDP), zinc diabutyl dithiocarbamate (ZDBC), and napthenoid oil have been tested and proven to act as antioxidants in asphalt (Oliver 1995, Ouyang *et al*. 2006a, Ouyang *et al*. 2006b). The chemicals reduce the ability of free radicals to rapidly oxidize asphalt. However, these chemicals have not been incorporated into the asphalt paving industry. LDADC contains lead and is therefore an environmental concern. ZDDP is used as an antioxidant to prevent wear in extreme pressure additives in gear oil formulations, while ZDBC is commonly used as an accelerator in rubber formulation (Ouyang *et al*. 2006b). ZDDP and ZDBC are not cost effective and therefore are not likely to be used at the wide scale needed for asphalt pavement construction. Wood lignin has also been researched as an antioxidant in asphalt. Wood lignin proved to be a very promising performance enhancer (Bishara *et al*. 2005). At four and seven percent by weight, wood lignin provided benefit to the asphalt. The temperature performance grade significantly increased. An increased high temperature performance grade has a direct relationship to less rutting potential (Roberts *et al*. 1996). The low temperature performance grade of the binders was either not affected, or showed a limited variation from the control value (Bishara *et al*. 2005). Therefore, the lignin had an overall effect of widening the temperature range of the binders. However, wood lignin was never incorporated into the asphalt industry. Wood lignin is a waste product of the paper industry. Many engineers and industry members are not accustomed to using asphalt pavements as a "horizontal landfill." Since wood-derived lignin carries the negative stigma of being a waste product, its potential use in asphalt pavements is low. Also, the structure and behavior of wood lignin is widely dependent upon timber source, season of growth, moisture conditions, and other uncontrollable environmental variables (Terrel and Rimstritong 1979). This makes it difficult to produce a homogeneous lignin product. A homogenous lignin is needed so its properties can be predicted and pavements can be designed properly. Therefore, wood lignin failed due to its image as a waste product and due to its inconsistency as a material. However, the lignin itself did prove to benefit asphalt by acting as an antioxidant.

2.3 Lignin as an Antioxidant

Lignin is an extremely complex polymer that originates from the plant kingdom (Brauns 1952, Glasser and Sarkanen 1989). Lignin is an amorphous compound with no set chemical formula. Figure 3 illustrates the general structure of lignin. Lignin is not a constitutionally defined compound, but rather a collective term for groups of high molecular amorphous compounds that are chemically closely related (Brauns 1952). Lignin can technically be defined as the "incrusting material of a plant built mainly from phelypropane building stones, which are unhydrolyzable by acids, readily oxidizable, soluble in hot alkali and bisulfate, and readily condenses with phenols and thio compounds" (Brauns 1952). Lignin is a hydrocarbon and consists mainly of carbon, hydrogen, and oxygen (Brauns 1952). The chemical structure of lignin is highly aromatic in nature with many randomly attached methoxyle and hydroxyl groups. Lignin can also contain aromatic hydrogen atoms, carbonyl groups, and aliphatic double bonds. This illustrates the complex chemical structure of lignin. Lignin also has complex physical and chemical properties that vary with plant source, growth conditions, and extraction mechanism (Dizhbite *et al*. 2004).

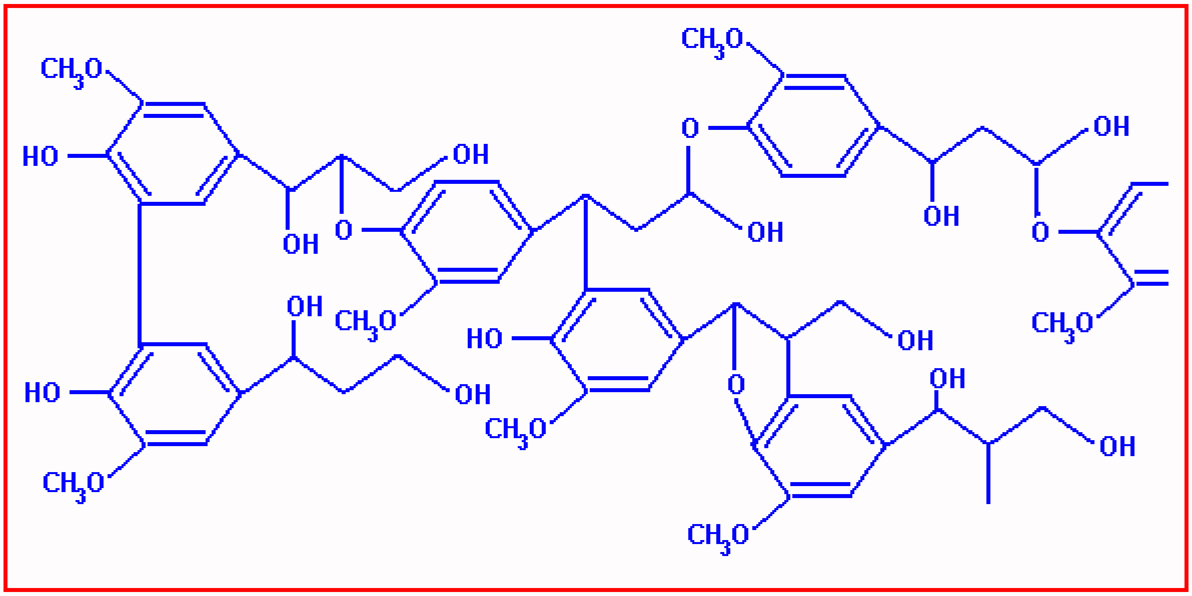

Figure 3. Chemical structure of lignin (Knee 2007)

One key chemical property that is evident from all lignins is its ability to act as an antioxidant. The antioxidant effects of lignins are derived from the scavenging action of their phenolic structures on oxygen containing free radicals (Dizhbite *et al*. 2004). Phenolic structures are benzene rings with attached hydroxyl groups. Benzene rings are six carbon structures with each carbon sharing a single and double covelant bond to another carbon. In a phenolic group, there can be one or more hydroxyl groups attached to the benzene ring. The ability of phenolic compounds to be antioxidants is the functional groups' ability to neutralize free radicals (Boeriu *et al*. 2004, Dizhbite *et al*. 2004, Glasser and Sarkanen 1989). Free radicals are known to actively break down substances by breaking apart the substance's chemical structure. Phenols can neutralize a free radical by either donating a proton or an electron (Dizhbite *et al*. 2004). Because of its structure, phenols are able to do both while remaining relatively stable. Lignins contain a large amount of phenolic groups, making them an effective antioxidant (Boeriu *et al*. 2004, Dizhbite *et al*. 2004).

There are many factors that can affect the antioxidant ability of lignin. The source of biological origin is the most important factor in determining the lignin structure (Dizhbite *et al*. 2004). Most fibrous plants contain large amounts of lignin. Worldwide, lignin is the second most abundant biological polymer next to cellulose (Dizhbite *et al*. 2004). Trees, grasses, and many agricultural plants contain large amounts of lignin in the plants' cell walls. Each plant is biologically and chemically different; therefore, the lignin obtained after extraction will be different. The extraction method is also very important in determining the lignin's antioxidant ability. Lignins can be extracted from the plant material by chemicals such as ethanol, acetone, acetic acid, methanol and propanol (Dizhbite *et al*. 2004). Each extraction method will produce a slightly different lignin, with each lignin having a slightly different antioxidant ability.

The lignin used for this study was obtained from the outer hull of the corn kernel. The outer hull was removed from the rest of the kernel by means of a chemical washing process. The lignin remaining after the washing process is relatively small in molecular weight compared to lignin derived from timber or grasses. However, the lignin contains large amounts of phenol structures, giving it excellent antioxidant ability. The lignin-containing co-products were combined with different asphalt binders to evaluate the effect the co-products have on asphalt binder. The next chapter explains the experimental methods used to evaluate the co-products in asphalt binder.

3. EXPERIMENTAL METHODS

3.1 Experimental Materials

Four asphalt binders were selected for this study. Two of the binders are local binders used in Iowa, while the other two binders are well-studied, nationally-used binders. The two local binders were obtained from a supplier in Tama, Iowa. One binder is a styrene-butadiene-styrene (SBS) polymer modified PG 58-22 (LPMB), while the other binder is unmodified and is a PG 64-16 (LB). The two well-known binders were obtained from the Federal Highway Administration's Materials Reference Library (MRL) in Sparks, Nevada. One binder is a PG 58-22 California Costal asphalt referenced as AAD-1. The other binder is a PG 64-16 West Texas Asphalt given the designation AAM-1. AAD-1 and AAM-1 vary greatly in their chemical composition. AAD-1 is known to be more susceptible to oxidative aging, while AAM-1 is known to be less susceptible to oxidative aging (Mortazavi and Moulthrop 1993). Table 1 illustrates the chemical composition of the two MRL binders.

Table 1. Chemical contrast of AAD-1 and AAM-1 (Mortazavi and Moulthrop 1993)

Component composition	AAD-1	AAM-1	Elemental composition	AAD-1	AAM-1
Asphaltenes	23.9	9.4	Carbon	81.6	86.8
Polar aromatics	41.3	50.3	Hydrogen	10.8	11.2
Napthene aromatics	25.1	41.9	Oxygen	0.9	0.5
Saturates	8.6	1.9	Sulfur	6.9	1.2

Three different lignin-containing co-products were used for this study. All co-products were obtained from the same source. Grain Processing Corporation (GPC) provided the co-products for this study. GPC operates a full-scale, wet-mill corn-based ethanol plant in Muscatine, Iowa. The three different co-products were obtained during the initial processing of the corn. Before the corn kernel is processed into ethanol, the outer hull is removed. The outer hull consists mainly of cellulose, hemi-cellulose, and lignin (Gulati et al. 1997). Through different processing methods, three different lignin-containing co-products were developed. Co-product A is an alkaline-washed corn hull while co-product B is an acid-modified corn hull. Co-product C is an unmodified corn hull. Co-products A and B contain slightly more lignin than co-product C. Each co-product was thoroughly dried to approximately ten percent moisture and ground into a fine powder. The co-products possess a lignin content ranging from ten to twelve percent, depending upon the method of production. The exact lignin content of each co-product is unknown. The remainders of the co-products are cellulose and hemi-cellulose, which are inert biological polymers. A fourth co-product was added later in the research. Co-product D was further modified by having the lignin chemically removed to leave cellulose and hemi-cellulose. This sample will act as a control since it contains no lignin and therefore has no antioxidant ability. Co-product D is used to help determine if the effects the lignin-containing co-products have on the binders are purely physical filler effects, or if there is antioxidant activity present. If treatment combinations with co-products A, B, and C are more beneficial than co-product D, then there is possibly some beneficial antioxidant activity between the lignin and the binder. Figure 4 illustrates the physical appearance of the co-products.

3.2 Experimental Plan

The experimental matrix is to test all four asphalt binders with all three lignin-containing co-products (A, B, and C) at varying amounts. Each binder was blended with co-products A, B, and C at three, six, and nine percent by weight. Nine percent was chosen as the high limit since previous studies show that the addition of approximately ten percent of lignin causes a negative effect on the binder (Bishara *et al*. 2005). Two of the binders (AAD-1 and LPMB) and were evaluated with the addition of twelve percent of co-products A, B, and C to see if any further benefit to the asphalt binders can be seen. Co-product D was tested with AAD-1 and AAM-1 at three, six, and nine percent to evaluate if the lignin-containing co-products are acting as an antioxidant and/or filler. Each binder was also tested without the addition of any co-products. Table 2 illustrates the treatment group combinations evaluated during the study. 52 treatment combinations were produced which underwent asphalt binder performance testing.

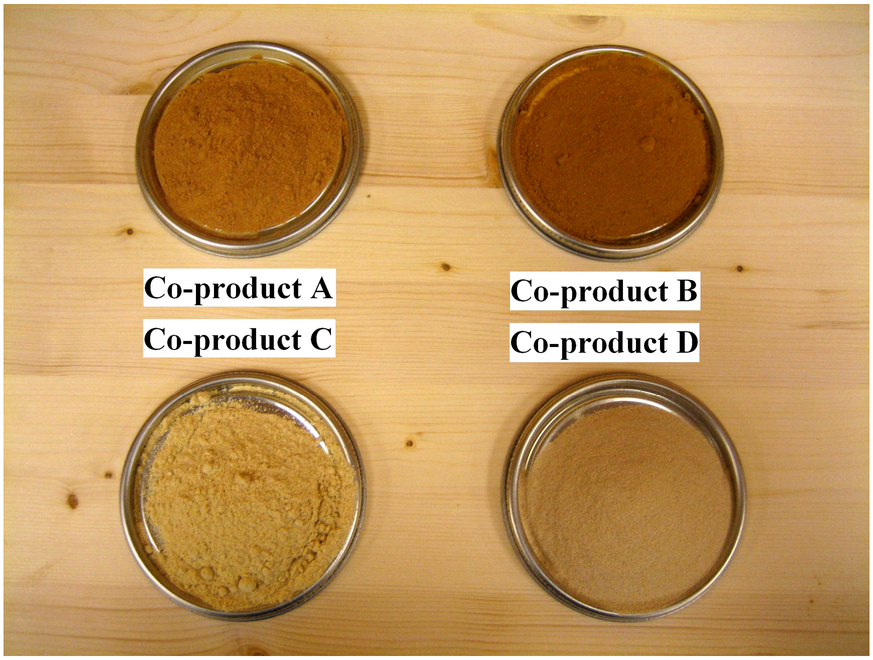

Figure 4. Wet-mill ethanol co-products used in study

Table 2. Treatment group combinations

	Binders			
Co-product	**AAD-1**	**AAM-1**	**LPMB**	**LB**
A	0, 3, 6, 9, 12	0, 3, 6, 9	0, 3, 6, 9, 12	0, 3, 6, 9
B	3, 6, 9, 12	3, 6, 9	3, 6, 9, 12	3, 6, 9
C	3, 6, 9, 12	3, 6, 9	3, 6, 9, 12	3, 6, 9
D	3, 6, 9	3, 6, 9	--	--

Numbers indicate amount of ligin added by percent weight

Each treatment combination was performance tested according to ASSHTO M 320 (2002) and ASTM D 6373 (1999). Initially, the asphalt binder was blended with the co-products using a high-speed sheer mill. The binder was heated to 155°C before the shear mill was started. Once the binder reached temperature, the shear mill was set to approximately 5000 rotations per minute for one hour. Once the binder and co-product were thoroughly blended, the blends immediately underwent performance testing.

3.3 Performance Testing

Performance grading an asphalt binder requires many steps and several separate testing procedures. Initially, each binder/co-product combination was tested using a dynamic shear rheometer (DSR) according to ASTM D 7175 (2005). A DSR characterizes the visco-elastic properties of a material by determining the complex modulus (G*) and phase angle (δ) of the sample. The complex modulus is the overall resistance of the asphalt to deformation when repeatedly sheared (McGennis *et al.* 1994). The complex modulus consists of two parts, storage modulus (G’) and elastic modulus (G”). At higher temperatures, asphalt behaves like a viscous material; at low temperatures, the asphalt behaves as an elastic material. However, at most intermediate temperatures, asphalt behaves as a visco-elastic material, exhibiting both viscous and elastic behavior. The phase angle determines how much the behavior of the complex modulus is viscous, and how much is elastic (McGennis *et al.* 1994). The relationship between G*, G’, G”, and δ is shown in Figure 5.

Each binder/co-product combination was tested unaged, immediately after mixing with a DSR. Each combination was tested at three different temperatures, with the temperatures depending upon the stated performance grade of the binders prior to blending

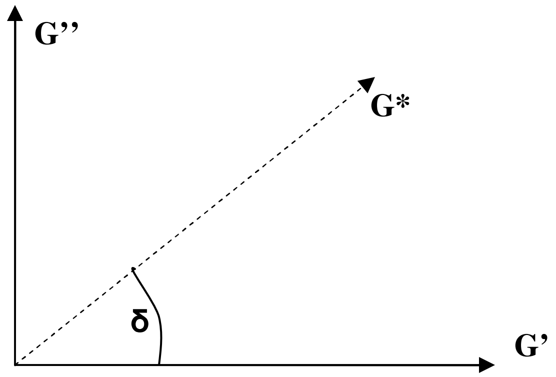

Figure 5. Components of the complex modulus

the co-products. The high temperature performance grade of each binder is in part determined with the unaged DSR test. A sample fails at a given temperature if the value of G*/sin(δ) is less than 1.00 kPa. If the G*/sin(δ) drops below the cutoff value, then the chance of rutting increases. The high temperature performance grade is set to control rutting (Bahia and Anderson 1995, The Asphalt Institute 2003). Typically, high temperature asphalt performance grades are determined in six degree increments. However, for this study, a continuous performance grade (PG) scale will be determined for each sample. For instance, instead of a PG 58 or PG 64, the samples will be given performance grades to the nearest 0.1°C. The three test temperatures were used to provide sufficient data to produce a regression line that determines the predicted temperature when the G*/sin(δ) value is equal to 1.00 kPa. Each binder was also tested in triplicate to provide a good estimation of the variation between the samples of the same treatment group.

Next, each binder/co-product combination was short-term aged in a rolling thin film oven (RTFO) according to ASTM D 2872 (2004). A RTFO ages a sample of asphalt for 85 minutes at 163.0°C. 35.0 grams of asphalt is added to small cylindrical bottles with an opening at one end. Eight bottles are loaded onto a carousel and rotated while being heated to produce thin films of asphalt binder. Hot air is blown into the bottles while they are being aged. This aging procedure mimics the aging a binder goes through while being mixed with aggregate and constructed in the field (Roberts *et al.* 1996, The Asphalt Institute 2003). During this process, the binder undergoes oxidative age hardening and has noticeably stiffer properties. The mass of the asphalt before aging and after aging was recorded to determine mass loss during aging. Mass loss may not exceed 1.0 percent according to Superpave specifications (The Asphalt Institute 2003).

After a sample was short-term aged in a RTFO, it was then again analyzed for high temperature properties with a DSR. The same three temperatures were used for each sample as were used during the unaged test. However, instead of having a failure criterion of 1.0 kPa as for the unaged blends, the failure criterion increases to 2.2 kPa due to the stiffening of the asphalt (The Asphalt Institute 2003). Another high temperature performance grade was determined using the RTFO residue of the binder/co-product blends. The lowest of the two high temperature performance grades (unaged or RTFO aged) was used for the final high temperature performance grade.

The remainder of the RTFO residue was further aged using a pressure aging vessel (PAV) according to ASTM D 6521 (2004). A PAV ages the asphalt with high pressure (2.1 MPa) and high heat (100°C) for 20 hours. The PAV simulates 5-10 years of in-service aging (Bahia and Anderson 1994). After the samples were aged, they were placed in a vacuum oven at 163°C for 30 minutes to remove entrapped air in the samples. The PAV aged asphalt is used to test the intermediate critical temperature with the DSR. Instead of using $G^*/\sin(\delta)$, $G^*\sin(\delta)$ is used because it better represents a control for the fatigue cracking phenomenon that occurs at intermediate temperatures. As a binder ages, it becomes more susceptible to fatigue cracking due to the oxidation of the binder (Bahia and Anderson 1995). The intermediate service temperature is set to control fatigue cracking, even though the intermediate temperature is not actually part of the performance grade. However, it can predict how a binder will behave at intermediate temperatures. A limit of 5000 kPa is used for the intermediate service temperature (The Asphalt Institute 2003). The binder/co-product combinations were all tested at three temperatures so each sample's intermediate temperature could be determined. As was with the previous DSR tests, the samples were all tested in triplicate.

The final test in determining the low temperature performance grade of an asphalt binder involves the use of a bending beam rheometer (BBR). A BBR tests an asphalt binder at low service temperatures to determine the asphalts susceptibility to thermal cracking (Roberts *et al*. 1996, The Asphalt Institute 2003). Asphalt binder is very susceptible to thermal cracking at low temperatures due to the fact that asphalt becomes less viscous as it is cooled. Rapid cooling and warming cause the binder to contract and expand, putting large thermal stresses on the material (Roberts *et al*. 1996). The asphalt/lignin blends were tested according to ASTM 6648 (2001). Asphalt is poured into small, rectangular beams and stored below -0.5°C to allow for proper handling. The beams are placed in a fluid bath that maintains low temperatures. After being in the fluid bath for one hour, the beams are individually placed on a loading frame and subjected to a load for 240 seconds. The deflection is measured versus time, which is used to calculate the two key properties of stiffness and change in stiffness (m-value). A sample of asphalt binder can fail at a given temperature by either having a stiffness of greater than 300 MPa or an m-value less than

0.300 (Bahia and Anderson 1994, The Asphalt Institute 2003). The low critical temperatures were also determined using regression analysis from the different test temperatures.

3.4 Physical Property Testing

The binder/co-product combinations were tested for separation effects according to ASTM D 7173 (2005). The blended samples were poured into aluminum cigar tubes and set vertically into an oven at 155°C for at most 48 hours. After heating, the tubes were removed and immediately frozen. The tubes were then cut into three equal-sized portions. The top and bottom portions were tested in a rotational viscometer to determine viscosity differences according to ASTM D 4402 (2006). If there are large viscosity differences, then the lignin must be physically separating out of the asphalt binder. Separation of the lignin from the asphalt could cause potential problems with future binder handling and construction. Along with testing for separation after 48 hours, the blends were also tested at 12 and 24 hours. This will provide a better estimate of when the lignin starts to separate out of solution, if separation occurs.

The solubility of the blended samples was determined immediately after mixing according to ASTM D 2042 (2001). This will show whether or not the lignin-containing co-products are chemically soluble with asphalt. A sample must have less than 1.0 percent be insoluble to pass. Specific gravity was also determined according to ASTM D 70 (2003). Specific gravity testing will show the change in densities of the binders as co-products are added.

3.5 Oxidation Testing

Fourier transform infrared spectroscopy (FTIR) testing was performed to quantify the amount of oxidative aging that occurs with the blended samples. FTIR is an extremely powerful tool for identifying types of chemical bonds (functional groups). When asphalt is oxidized, the chemical structure of asphalt is changed. Functional groups, such as carboxylic acids, ketones, sulfoxides, and anhydrides, are formed from oxidation (Lucena *et al*. 2004). Carboxylic acids and ketones contain one carbonyl group, while anhydrides contain two carbonyl groups. A carbonyl bond is a carbon atom bonded to an oxygen atom with a double covalent bond. Therefore, when FTIR testing is performed, asphalt binders that have oxidized more will have larger amounts of carbonyl groups (Lucena *et al*. 2004). More sulfoxides will also be present. A FTIR test produces a spectrum with peaks that illustrates wave number versus absorbance or transmittance. The larger a peak at a given wave number, the larger the amount of that specific functional group present in the material. Carbonyl groups appear at approximately 1650 cm^{-1}, while sulfoxides appear at 1030 cm^{-1} (Lucena *et al*. 2004). The different asphalt/co-product blends will be analyzed with FTIR to determine the how the blends age with added co-product. A decreased amount of either carboxyl or sulfoxide groups would indicate less oxidative aging.

4. RESULTS

4.1 Performance Grade Testing

4.1.1 Unaged Blends

All binder/co-products combinations were performance graded according to ASTM D 6373 (1999) and AASHTO M 320 (2002). Initially, all samples were tested unaged in a DSR. G*/sin(δ) values were recorded for each combination at three temperatures. Each treatment combination was also tested in triplicate for proper estimation of random error for statistical analysis. To determine the high temperature performance grade on a continuous scale, or high critical temperature, three G*/sin(δ) values were plotted versus temperature on a semi-log scale. Asphalt binder properties have a semi-log relationship with temperature (McGennis *et al*. 1994). The G*/sin(δ) value approximately doubles for every 6°C drop in temperature. Figure 6 illustrates one test for one binder/co-product combination.

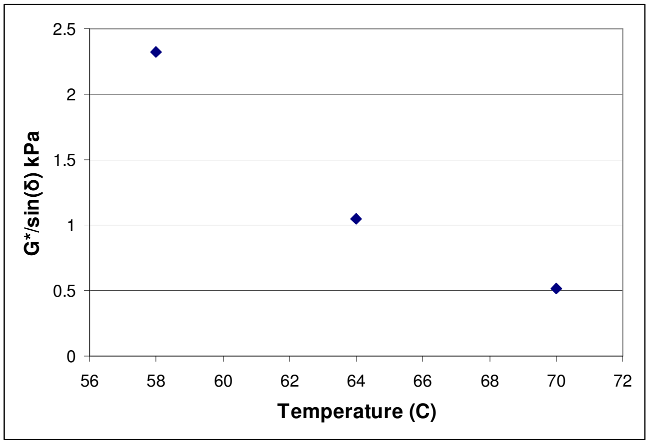

Figure 6. Example of test results for one binder/co-product sample

As mentioned previously, the failure criterion for unaged asphalt is when G*/sin(δ) is less than 1.00 kPa. The high critical temperature was developed for each sample by creating a semi-log regression line using the method of least squares. The temperature when G*/sin(δ) is equal to 1.00 kPa is equal to the high critical temperature. Three high critical temperatures were determined for each binder/co-product combination. The average temperatures for each binder/co-product combination are listed in Table 3. Complete test data including G*/sin(δ) values for each test temperature are the Appendix.

Table 3. Mean values of unaged high critical temperatures

AAD-1			**AAM-1**			**LPMB**			**LB**		
Co-product (type)	**Co-product (%)**	**Temp. (°C)**	**Co-product (type)**	**Co-product (%)**	**Temp. (°C)**	**Co-product (type)**	**Co-product (%)**	**Temp. (°C)**	**Co-product (type)**	**Co-product (%)**	**Temp. (°C)**
A	12	64.8	A	9	68.7	A	12	67.7	A	9	70.4
B	12	67.2	B	9	69.8	B	12	68.7	B	9	72.2
C	12	64.5	C	9	68.8	C	12	66.7	C	9	71.7
A	9	64.8	D	9	71.1	A	9	66.7	A	6	69.9
B	9	66.6	A	6	68.5	B	9	68.6	B	6	71.9
C	9	64.5	B	6	69.3	C	9	67.1	C	6	70.6
D	9	66.7	C	6	68.8	A	6	66.4	A	3	69.3
A	6	64.5	D	6	69.0	B	6	66.7	B	3	71.1
B	6	64.8	A	3	68.5	C	6	65.8	C	3	70.7
C	6	63.4	B	3	68.5	A	3	66.1	--	0	68.9
D	6	65.5	C	3	68.1	B	3	67.2			
A	3	63.7	D	3	69.3	C	3	65.9			
B	3	64.8	--	0	67.8	--	0	62.3			
C	3	62.8									
D	3	64.8									
--	0	62.3									

4.1.2 RTFO Aged Blends

After the blends were individually short-term aged in a RTFO, they were again tested with a DSR. Superpave specifications require that the mass loss after RTFO aging be less than one percent (The Asphalt Institute 2003). The mass loss for all binder/co-product combinations was less than one percent. The RTFO aged samples were tested the same as the unaged samples. The binder/co-product combinations were tested at three temperatures, and each blend was tested in triplicate. However, since RTFO aged samples have been aged by oxidation, the failure criterion is different. The larger the G*/sin(δ) value, the stiffer the asphalt. Following Superpave specifications, 2.20 kPa is used as the failure criterion (The Asphalt Institute 2003). Therefore, the RTFO aged high critical temperature is the temperature when G*/sin(δ) is 2.20 kPa using regression equations for each sample. Table 4 illustrates the critical high temperature for the RTFO aged samples. Also included in Table 4 is the change from RTFO aged high critical temperature to the unaged high critical temperature. The RTFO aged high critical temperatures for AAD-1, LMPB, and LB were predominately increased from the unaged high critical temperatures. With AAM-1, the opposite effect occurred because AAM-1 is known to be resistant to oxidative aging.

Table 4. Mean values of RTFO aged high critical temperatures

AAD-1				AAM-1				LPMB				LB			
Co-prod. (type)	**Co-prod. (%)**	**Temp. (°C)**	**Δ in high T_c**	**Co-prod. (type)**	**Co-prod. (%)**	**Temp. (°C)**	**Δ in high T_c**	**Co-prod. (type)**	**Co-prod. (%)**	**Temp. (°C)**	**Δ in high T_c**	**Co-prod. (type)**	**Co-prod. (%)**	**Temp. (°C)**	**Δ in high T_c**
A	12	67.9	3.1	A	9	67.6	-1.1	A	12	71.8	4.1	A	9	71.9	1.5
B	12	70.1	2.9	B	9	67.2	-2.6	B	12	72.5	3.8	B	9	72.4	0.2
C	12	67.2	2.7	C	9	68.5	-0.3	C	12	71.2	4.5	C	9	72.3	0.6
A	9	67.5	2.7	D	9	69.2	-1.9	A	9	67.7	1.0	A	6	71.4	1.5
B	9	69.3	2.7	A	6	68.2	-0.3	B	9	69.3	0.7	B	6	72.5	0.6
C	9	67.0	2.5	B	6	66.9	-2.4	C	9	69.1	2.0	C	6	71.8	1.2
D	9	67.2	0.5	C	6	67.5	-1.3	A	6	66.8	0.4	A	3	71.6	2.3
A	6	66.9	2.4	D	6	68.1	-0.9	B	6	68.1	1.4	B	3	71.9	0.8
B	6	68.6	3.8	A	3	66.9	-1.6	C	6	68.4	2.6	C	3	71.4	0.7
C	6	66.2	2.8	B	3	66.8	-1.7	A	3	66.4	0.3	--	0	71.1	2.2
D	6	65.3	-0.2	C	3	67.0	-1.1	B	3	67.1	-0.1				
A	3	65.8	2.1	D	3	67.7	-1.6	C	3	67.3	1.4				
B	3	67.5	2.7	--	0	66.7	-1.1	--	0	66.7	4.4				
C	3	66.7	3.9												
D	3	66.8	2.0												
--	0	65.4	3.1												

4.1.3 PAV Aged Blends

After being long-term aged in a PAV, the binder/co-product combinations were again tested with a DSR. PAV aged samples are used to evaluate intermediate critical temperature. The response produced by a DSR for PAV aged samples is G*sin(δ) rather than G*/sin(δ). Using a failure criterion of 5000 kPa and semi-log regression equations, intermediate critical temperatures were created. The means for all asphalt/lignin blends are shown in Table 5. The low critical temperature was determined using a BBR. As previously mentioned, a BBR test produces two responses: stiffness and the m-value. A sample can fail at a given temperature if the stiffness is greater than 300 MPa or if the m-value is less than 0.300. For all samples in this study, the m-value was the limiting value. The stiffness remained under 300 MPa for every test. Linear regression was used to determine the low critical temperatures. The low critical temperature is the value when the m-value is equal to 0.300 minus 10°C. 10°C is subtracted from the low critical temperature due to the theory behind the test methods (The Asphalt Institute 2003). The low critical temperatures for all samples are listed in Table 6.

Table 5. Mean values for PAV aged intermediate critical temperatures

AAD-1			**AAM-1**			**LPMB**			**LB**		
Co-product (type)	**Co-product (%)**	**Temp. (°C)**	**Co-product (type)**	**Co-product (%)**	**Temp. (°C)**	**Co-product (type)**	**Co-product (%)**	**Temp. (°C)**	**Co-product (type)**	**Co-product (%)**	**Temp. (°C)**
A	12	18.5	A	9	22.4	A	12	22.4	A	9	23.9
B	12	19.6	B	9	22.9	B	12	22.7	B	9	24.6
C	12	18.5	C	9	21.4	C	12	22.2	C	9	24.2
A	9	18.2	D	9	23.9	A	9	20.1	A	6	23.9
B	9	19.0	A	6	19.8	B	9	21.8	B	6	24.3
C	9	18.3	B	6	22.0	C	9	20.2	C	6	24.2
D	9	20.3	C	6	22.1	A	6	20.2	A	3	23.7
A	6	18.0	D	6	24.5	B	6	21.9	B	3	24.2
B	6	18.7	A	3	18.6	C	6	20.2	C	3	23.9
C	6	18.1	B	3	20.6	A	3	20.2	--	0	23.5
D	6	19.5	C	3	20.9	B	3	21.9			
A	3	17.3	D	3	23.1	C	3	20.2			
B	3	18.1	--	0	20.3	--	0	20.1			
C	3	17.8									
D	3	19.2									
--	0	17.3									

Table 6. Mean values of PAV aged low critical temperatures

AAD-1			AAM-1			LPMB			LB		
Co-product (type)	Co-product (%)	Temp. (°C)	Co-product (type)	Co-product (%)	Temp. (°C)	Co-product (type)	Co-product (%)	Temp. (°C)	Co-product (type)	Co-product (%)	Temp. (°C)
A	12	-24.1	A	9	-14.9	A	12	-19.6	A	9	-16.7
B	12	-23.2	B	9	-14.8	B	12	-19.1	B	9	-17.5
C	12	-23.4	C	9	-15.2	C	12	-18.8	C	9	-17.4
A	9	-23.8	D	9	-12.7	A	9	-21.2	A	6	-18.3
B	9	-23.5	A	6	-16.1	B	9	-19.6	B	6	-18.7
C	9	-23.8	B	6	-14.9	C	9	-21.5	C	6	-17.9
D	9	-22.1	C	6	-14.7	A	6	-20.9	A	3	-18.4
A	6	-21.9	D	6	-13.1	B	6	-19.8	B	3	-18.1
B	6	-24.1	A	3	-14.0	C	6	-20.3	C	3	-17.6
C	6	-22.7	B	3	-14.9	A	3	-21.5	--	0	-19.5
D	6	-22.5	C	3	-15.2	B	3	-20.1			
A	3	-24.0	D	3	-13.4	C	3	-21.7			
B	3	-22.3	--	0	-15.1	--	0	-22.0			
C	3	-23.8									
D	3	-23.4									
--	0	-23.5									

4.2 Physical Properties

4.2.1 Specific Gravity

The gradation and densities of co-products A, B, and C are listed in Table 7.

Table 7. Gradations and density of lignin-containing co-products

Mesh	Co-product A (% retained)	Co-product B (% retained)	Co-product C (% retained)
60	1.8	0.2	30.2
80	15.1	19.2	53.9
100	13.2	14.8	10.7
200	31.0	40.0	5.0
325	26.0	14.3	0.2
Pan	12.8	12.2	0.0
Density (g/cm^3)	0.88	0.50	0.49

Co-products A, B, and C have bulk densities less than 1.0 g/cm^3. However, when the co-products are blended and dispersed into the binder, the density of the blends increases with the further addition of a co-product. Binders AAM-1 and AAD-1 were each combined with co-products A, B, and C at three, six, and nine percent by weight. As can be seen from Figures 7 and 8, the specific gravity of the blends increases linearly with the amount of co-products added.

4.2.2 Solubility

The solubility of the asphalt/lignin blends was determined according to ASTM D 2042 (2001). The binder/co-product combinations were mixed with trichloroethylene and passed through a fiberglass filter to determine the amount of material that did not solubolize in the binder. A sample fails if greater than 1.0 percent of the total mixture is retained on the filter. AAD-1 was used for testing solubility. Co-products A, B, and C were tested at three percent addition to AAD-1. If the samples failed at three percent, then they would fail at the higher lignin amounts. Table 8 illustrates the amount of material that was not soluble in trichloroethylene. None of the samples passed the 1.0 percent criteria.

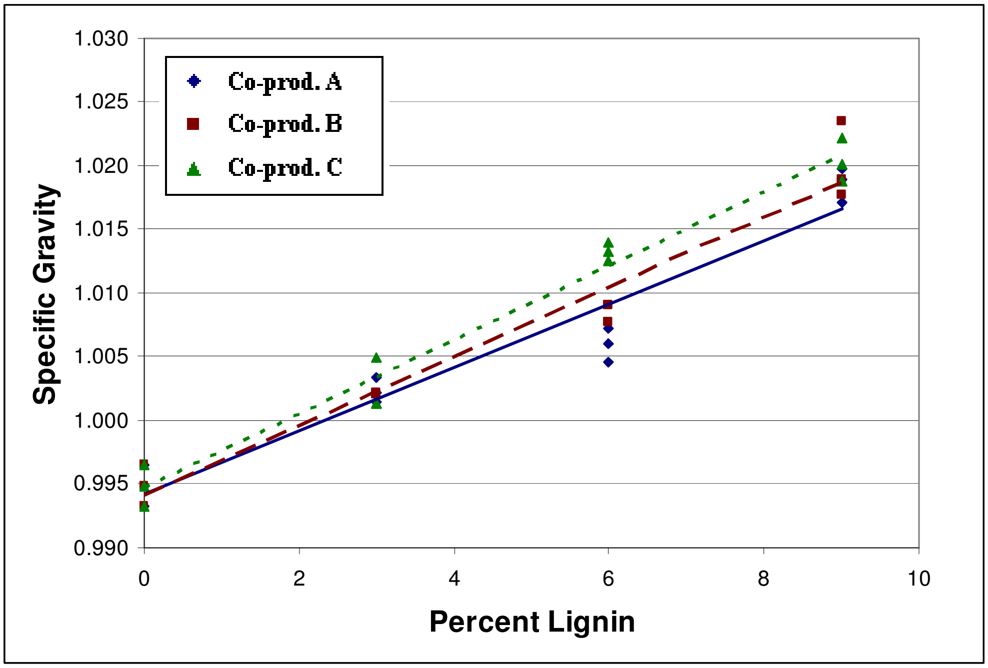

Figure 7. Specific gravity of AAM-1 with co-products A, B, and C

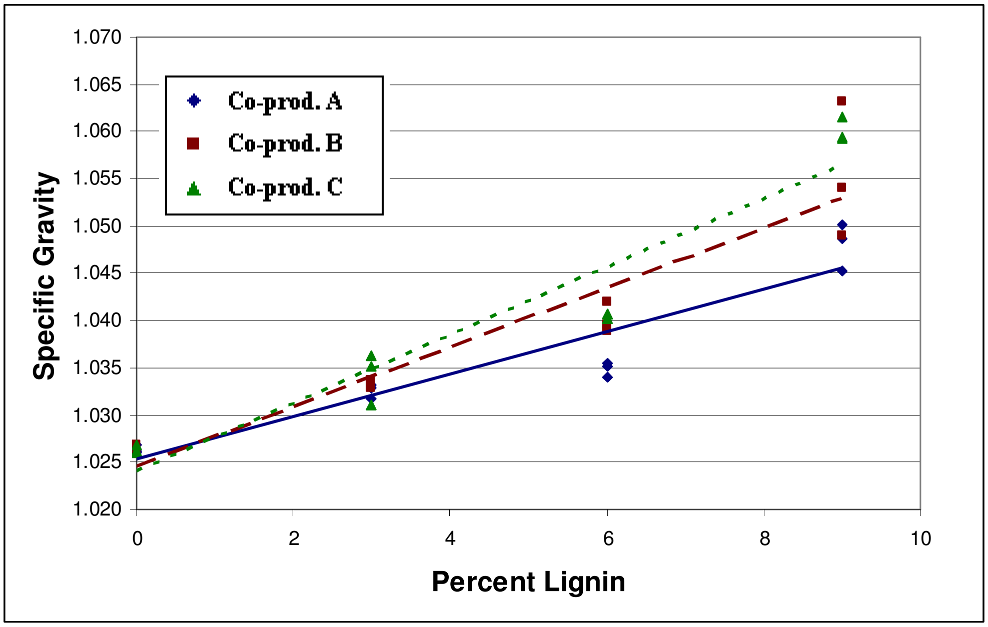

Figure 8. Specific gravity of AAD-1 with co-products A, B, and C

Table 8. Solubility of ADD-1 with three percent of co-products A, B, and C

Co-product (type)	Insoluable (%)	Co-product (type)	Insoluable (%)	Co-product (type)	Insoluable (%)
A	1.9	B	2.5	C	2.5
A	2.1	B	2.1	C	2.3
A	1.3	B	2.1	C	1.9

4.2.3 Separation Testing

Initially, co-products A, B, C, and D were combined with AAD-1 at nine percent to determine which co-product has the greatest and least separation effects. The samples were stored in an oven at 155°C for 6, 24, and 48 hours. The viscosities of the top and bottom portions were measured for each sample according to ASTM D 4402 (2006). Results of the separation testing are listed in Table 9. It can be seen from Table 8 that co-product A has the lowest amount of separation, while co-product B has the greatest separation effects.

Table 9. Separation data of AAD-1 with co-products A, B, C, and D at nine percent

Co-product (type)	Heating time (hours)	Portion (B or T)	Viscosity at 135°C (cP)	Diff. (%)	Co-product (type)	Heating time (hours)	Portion (B or T)	Viscosity at 135°C (cP)	Diff. (%)
A	6	B	427.5	15.2	C	24	B	570.0	33.6
A	6	T	362.5	--	C	24	T	378.3	--
B	6	B	603.3	38.4	D	24	B	525.0	37.1
B	6	T	371.7	--	D	24	T	330.0	--
C	6	B	462.3	21.0	A	48	B	559.3	29.4
C	6	T	365.2	--	A	48	T	395.0	--
D	6	B	455.0	29.2	B	48	B	814.2	50.9
D	6	T	322.0	--	B	48	T	400.0	--
A	24	B	492.5	23.9	C	48	B	665.0	42.9
A	24	T	375.0	--	C	48	T	379.5	--
B	24	B	724.2	46.7	D	48	B	595.5	43.7
B	24	T	385.8	--	D	48	T	335.2	--

Further separation testing was performed with AAD-1 and co-product A at zero, three, six, and nine percent to see the separation effects with differing amounts of co-product. The results are listed in Table 10. It can be seen that the more co-product added, the greater the separation effects. Even with the lowest amount of co-product added (3 percent), significant separation effects are noticed.

Table 10. Separation data of AAD-1 with co-product A at 0, 3, 6 and 9 percent

Co-product (type)	Heating time (hours)	Portion (B or T)	Viscosity at 135°C (cP)	Diff. (%)	Co-product (type)	Heating time (hours)	Portion (B or T)	Viscosity at 135°C (cP)	Diff. (%)
0	6	B	345.0	2.2	6	6	B	401.7	12.2
0	6	T	337.5		6	6	T	352.5	--
0	24	B	365.0	6.4	6	24	B	513.3	22.6
0	24	T	341.5		6	24	T	397.5	--
0	48	B	383.2	5.6	6	48	B	568.3	28.4
0	48	T	361.7		6	48	T	406.7	--
3	6	B	370.0	2.9	9	6	B	427.5	15.2
3	6	T	359.2		9	6	T	362.5	--
3	24	B	413.3	9.1	9	24	B	492.5	23.9
3	24	T	375.8		9	24	T	375.0	--
3	48	B	455.0	15.7	9	48	B	559.3	29.4
3	48	T	383.5		9	48	T	395.0	--

Finally, all binders were tested with the addition of six percent co-product A to determine different separation effects of each binder. Table 11 illustrates the results. AAM-1 has the least separation effects, while the local binder has the greatest effects. The trend of increased separation with longer storage time can also be noticed.

Table 11. Separation data of six percent co-product A with all binders

Binder	Heating time (hours)	Portion (B or T)	Viscosity at 135°C (cP)	Diff. (%)	Binder	Heating time (hours)	Portion (B or T)	Viscosity at 135°C (cP)	Diff. (%)
AAD-1	6	B	401.7	12.2	LB	24	B	788.3	31.2
AAD-1	6	T	352.5	--	LB	24	T	542.5	--
AAM-1	6	B	388.3	3.2	LPMB	24	B	707.5	24.7
AAM-1	6	T	375.8	--	LPMB	24	T	532.5	--
LB	6	B	565.0	7.1	AAD-1	48	B	568.3	28.4
LB	6	T	525.0	--	AAD-1	48	T	406.7	--
LPMB	6	B	602.2	12.7	AAM-1	48	B	740.0	16.8
LPMB	6	T	525.8	--	AAM-1	48	T	615.8	--
AAD-1	24	B	513.3	22.6	LB	48	B	791.7	29.7
AAD-1	24	T	397.5	--	LB	48	T	556.7	--
AAM-1	24	B	704.2	12.8	LPMB	48	B	755.0	28.6
AAM-1	24	T	614.0	--	LPMB	48	T	539.2	--

4.3 Antioxidant Testing

FTIR spectrometry was performed on various combinations to determine the chemical effect the lignin in the co-products has on the binders. Spectrometry testing was only performed on PAV aged blends since they have all undergone long-term oxidative aging. The samples were tested at Western Research Institute in Laramie, Wyoming. The local polymer modified binder was not tested since the polymer interferes with the analysis of the effects of the lignin. Table 12 illustrates the results of the testing. Three and nine percent of co-products A, B, and C were tested with AAD-1, AAM-1, and the local binder (LB). The carbonyl content and sulfoxide contents were reported. Both of these functional groups are products of oxidative aging.

Table 12. FTIR spectrometry analysis

Co-product (Type)	Co-product (%)	AAD-1		AAM-1		LB	
		Carbonyl content (au)	Sulfoxide content (mol/liter)	Carbonyl content (au)	Sulfoxide content (mol/liter)	Carbonyl content (au)	Sulfoxide content (mol/liter)
--	0	0.0776	0.3665	0.1410	0.1440	0.0913	0.3285
A	3	0.0784	0.2840	0.1432	0.1110	0.0866	0.3630
B	3	0.0839	0.3600	0.1502	0.1245	0.0880	0.3430
C	3	0.0902	0.3870	0.1400	0.1310	0.0892	0.3320
A	9	0.0812	0.3985	0.1458	0.1395	0.0964	0.3200
B	9	0.0852	0.4030	0.1380	0.1400	0.0850	0.3590
C	9	0.0884	0.2130	0.1417	0.1260	0.0906	0.2635

The next chapter features the statistical analysis of the previously reported data. Trends will be presented for all data, showing the effects of lignin on the tested physical and chemical properties of asphalt binders.

5. ANALYSIS

5.1 Introduction

Analysis of the binder/co-product treatment combinations took place by analyzing each binder separately. Each binder is divided into four sections: unaged high critical temperature, RTFO aged high critical temperature, PAV aged intermediate critical temperature, and PAV aged low critical temperature. For each section, the lignin-containing co-products (A, B, and C) are analyzed to determine which combination has the most/least beneficial effect on each binder. Each treatment combination is compared to the unmodified binder to determine if the resulting change in performance grade is significant.

For binders AAD-1 and AAM-1, co-product D is included in a separate analysis for all four critical temperature sections. Co-product D is analyzed to determine if its effects are statistically different from the lignin-containing co-products. This comparison is used to determine if the lignin in co-products A, B, and C provides any significant benefit to the binder's high, intermediate, and low temperature properties.

After each binder is separately analyzed, all binder data is combined to determine if different binders behave differently with added co-product. A summary of the high, intermediate, and low temperature performance grades of all binders concludes the performance testing analysis. Subsequent sections analyze separation and binder/co-product chemical antioxidant effects.

5.2 Performance Grades

5.2.1 AAD-1

Binder AAD-1 was blended with three, six, nine, and twelve percent of co-products A, B, and C. Co-products A, B, and C were analyzed to determine which combination has the greatest effect on the binder. AAD-1 was also blended with co-product D (carbohydrate filler with no lignin) at three, six, and nine percent to see if there is chemical activity of the lignin in the other three co-products and the binder. AAD-1 was also tested without the addition of any co-products as a reference. Each blend was tested in triplicate to provide a good measure of random error. Statistical software was used to evaluate the differences between the treatment groups.

5.2.1.1 Unaged high critical temperature

First, the unaged blends were analyzed to see if there were any statistical differences among the treatment groups. An analysis of variance (ANOVA) using the method of least squares was used for examination. An ANOVA was formed evaluating three, six, nine, and twelve percent of co-products A, B, and C to determine the treatment combinations that would have the most beneficial effect on the binder. For the unaged blends, a larger critical high temperature is beneficial. A Type I error (α) of 0.05 was used for all statistical analysis. An α of 0.05 states that there is a five percent chance of rejecting the null hypothesis when it is in fact true. The ANOVA produced an F-statistic with a corresponding p-value less than 0.0001. Since the p-value is less than α, then the null hypothesis of all treatment groups being equal can be rejected. Therefore, there are statistical differences between the treatment groups. Effect tests were then performed to distinguish with what factors the differences lie. An F-statistic and a corresponding p-value were produced for each factor (co-product type and percent) and the interaction between the two factors (co-product type * amount of co-product). Both factors have p-values less than 0.0001 and the interaction effect has a p-value equal to 0.0064. Therefore, all factors and interaction are significant. The model ANOVA and effects test are shown in Table 13.

Table 13. Model ANOVA and effect tests for unaged AAD-1 DSR data

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	49.63	4.51	26.43	<.0001
Error	24	4.10	0.17	--	--
C. Total	35	53.73	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	3	18.67	6.22	36.46	<.0001
Co-prod. type	2	5.78	2.89	16.94	<.0001
Interaction	6	4.11	0.69	4.01	0.0064

Means difference testing was used to evaluate between which treatment groups the differences exist. A student-t least significant difference (LSD) was not used due to the large number of treatment groups. For instance, with twelve treatment groups and an α equal to 0.05, the test would have an overall error rate of 60.0 percent. Tukey honest significant difference (HSD) testing is more accurate with larger amounts of comparisons. α is the error rate for all comparisons, instead of a single comparison as with the LSD procedure. For the amount of co-product factor, the nine and twelve percent treatment groups (levels) have a significantly larger unaged high critical temperature than the three and six percent treatment groups. With the co-product type factor, the high critical temperature for co-product B is significantly larger than co-product s A and C. There are significant interaction effects because three percent co-product A has a larger high critical temperature than six percent co-product C, even though Tukey HSD testing shows that the six percent level is significantly larger than the three percent level.

Next, co-product D was compared with the other co-products using an ANOVA. Three, six, and nine percent of co-products A, B, C, and D were analyzed. Significant differences were found among the treatment groups. The effect tests illustrate differences between both factors, but no significant interaction effect. Tukey HSD testing indicates co-products B and D are significantly larger than co-products A and C, but co-products B and D are not significantly different. Also, the more co-product added, the greater the increase in critical high temperature. These results are very similar to the previous analysis. Co-product D behaves similar to lignin B in unaged AAD-1. The model ANOVA and effect tests are shown in Table 14.

Table 14. Model ANOVA and effect tests for unaged AAD-1 DSR data with co-product D

Analysis of variance					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	11	44.01	4.00	21.05	<.0001
Error	24	4.56	0.19	--	--
C. Total	35	48.57	--	--	--

Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
% Co-prod.	2	16.13	8.07	13.34	<.0001
Co-prod. type	3	8.45	2.82	2.53	<.0001
Interaction	6	1.94	0.32	4.04	0.1644

Using paired t-tests with α equal to 0.05, all combinations of co-products A, B, and C were compared with the neat binder (no co-product added). Each t-test produced a p-value less than α. Therefore, all combinations of co-products A, B, and C with AAD-1 produce a significantly larger high critical temperature than the binder alone. Table 15 illustrates the p-values for all treatment combinations.

Table 15. P-values for paired t-tests for unaged AAD-1 DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0001	9	A	0.0001
3	B	0.0010	9	B	0.0001
3	C	0.0046	9	C	0.0001
6	A	0.0088	12	A	0.0001
6	B	0.0078	12	B	0.0002
6	C	0.0006	12	C	0.0001

The addition of all co-products at all percentages positively benefits the unaged critical high temperature. Generally, the more co-product added, the greater the increase in critical high temperature. The increase in critical high temperature is caused by an increased stiffness of the binder. The more co-product added, the greater the stiffening. Co-product B causes the greatest amount of stiffening of the three lignins.

5.2.1.2 RTFO aged high critical temperature

The RTFO aged blends were analyzed the same as the unaged blends. A larger RTFO aged high critical temperature is beneficial to the asphalt. Significant differences were found with both factors by analyzing three, six, nine, and twelve percent of co-products A, B, and C. Tukey HSD tests show that co-product B produces a significantly larger high critical temperature than co-products A and C. Also, the twelve percent level produces a larger high critical temperature than the other levels. Generally, the more co-product added, the greater the high critical temperature. The model ANOVA and effects tests are shown in Table 16.

Table 16. Model ANOVA and effect tests for RTFO aged AAD-1 DSR data

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	61.36	5.58	54.59	<.0001
Error	24	2.45	0.10	--	--
C. Total	35	63.81	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	3	22.04	7.35	71.92	<.0001
Co-prod. type	2	6.79	3.40	33.24	<.0001
Interaction	6	0.89	0.15	1.44	0.2393

Co-product D was compared with the other co-products by analyzing three, six, and nine percent of co-products A, B, C, and D. Co-product D has a significantly larger high critical temperature than co-product C, but is not significantly different than co-product A or B. Co-product B is not significantly different than co-product D, but is larger than co-products A and C. Also, the more co-product added, the greater the high critical temperature. These results suggest that co-product D behaves no different from the other co-products after the blends have been short-term aged with a RTFO. Table 17 illustrates the model ANOVA and effect tests.

All treatment combinations of co-products A, B, and C were compared with the neat binder. Only three percent of co-products A and C are not significantly different from the binder. All other treatment combinations possess a significantly larger RTFO aged high critical temperature than the neat binder. T-test results are shown in Table 18.

Table 17. Model ANOVA and effect tests for RTFO aged AAD-1 DSR data with co-product D

Analysis of variance						Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	11	47.07	4.28	17.27	<.0001	% Co-prod.	2	11.16	5.58	22.51	<.0001
Error	24	5.95	0.25	--	--	Co-prod. type	3	7.39	2.46	9.95	0.0002
C. Total	35	53.02	--	--	--	Interaction	6	7.33	1.22	4.93	0.0020

Table 18. P-values for paired t-tests for RTFO aged AAD-1 DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0564	9	A	0.0004
3	B	0.0012	9	B	0.0001
3	C	0.4732	9	C	0.0016
6	A	0.0020	12	A	0.0004
6	B	0.0002	12	B	0.0002
6	C	0.0180	12	C	0.0026

The addition of co-products A, B, and C at all amounts caused an increase in the RTFO aged high temperature. The co-product causes the binder to stiffen, thus increasing the RTFO aged critical high temperature. Generally, the more co-product added, the greater the increase in the critical high temperature. Co-product B again provides the greatest stiffening effect.

5.2.1.3 PAV aged intermediate critical temperature

The critical intermediate temperatures were analyzed similar to the critical high temperatures. An ANOVA was produced from analyzing three, six, nine, and twelve percent of co-products A, B, and C. The model ANOVA and effect tests (Table 19) show both factors are significant.

Table 19. Model ANOVA and effect tests for PAV aged AAD-1 DSR data

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	12.26	1.11	12.83	<.0001
Error	24	2.08	0.09	--	--
C. Total	35	14.34	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	3	6.36	2.12	24.41	<.0001
Co-prod. type	2	1.13	0.57	6.51	0.0050
Interaction	6	0.64	0.11	1.23	0.3271

Tukey HSD testing shows that the twelve percent level is significantly larger than the three and six percent levels. Also, the nine percent level is significantly larger than the three percent level. The nine and twelve percent levels and the three and six percent levels were not proven to be different. So generally, the more lignin that is added, the more the intermediate critical temperature increases. Co-products B and C are significantly larger than co-product A, while co-products B and C are not significantly different.

Differences are found with both factors when co-product D is included in the analysis (Table 20). Co-product D has the statistically greatest intermediate critical temperature of all four co-products. The larger the intermediate critical temperature, the stiffer the asphalt, and therefore, the more the asphalt has aged. Co-product D has the statistically largest intermediate critical temperature, so the absence of lignin caused the binder to age and/or stiffen more than the other samples. This relationship suggests chemical antioxidant activity of co-products A, B, and C.

Table 20. Model ANOVA and effect tests for PAV aged AAD-1 DSR data with co-product D

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	24.04	2.19	23.62	<.0001
Error	24	2.22	0.09	--	--
C. Total	35	26.26	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	2	4.62	2.31	24.97	<.0001
Co-prod. type	3	6.16	2.05	22.20	<.0001
Interaction	6	0.51	0.09	0.92	0.5051

All except four treatment combinations proved to be significantly different than the neat binder. Table 21 illustrates the results of the paired t-tests. Three percent of co-products

A, B, and C and nine percent co-product A are not significantly different from the unmodified binder AAD-1.

Table 21. P-values for paired t-tests for PAV aged AAD-1 DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.7824	9	A	0.0528
3	B	0.0536	9	B	0.0008
3	C	0.2420	9	C	0.0106
6	A	0.0434	12	A	0.0108
6	B	0.0056	12	B	0.0006
6	C	0.0182	12	C	0.0036

A larger intermediate critical temperature is beneficial in warmer climates, while it is detrimental in cooler climates. Therefore, the addition of lignin-containing co-products to AAD-1 can cause positive or negative effects. The more co-product added, the greater the increase in intermediate service temperature, which would be more beneficial in warmer climates. In cooler climates, the addition of co-product would have a negative effect by increasing the asphalts susceptibility to fatigue cracking.

5.2.1.4 PAV aged low critical temperature

The final step in analyzing binder AAD-1 is examining the low critical temperature. The treatment combinations were examined to see which produced the best and worst results. As previously mentioned, a smaller low critical temperature is better. Analysis of the low critical temperatures was performed in a similar manner as the high and intermediate critical temperatures. The low critical temperatures exhibited much more variation than the high and intermediate temperatures. BBRs used to examine the asphalt/lignin blends have less precision than DSRs. Therefore, it was more difficult to see statistical differences with BBR test data than DSR test data. An ANOVA was produced by examining treatment combinations of three, six, nine, and twelve percent of co-products A, B, and C. The model

ANOVA (Table 22) indicates no differences among any of the treatment groups. The effects test further supports this with large p-values for all the factors and interactions. However, it can be seen by comparing the treatment means that co-product B produces the highest low critical temperature, which is a negative effect. Also, the more co-product added, generally the larger the low critical temperature. The least negative results can be seen from co-products A and C at low levels.

Table 22. Model ANOVA and effect tests for PAV aged AAD-1 BBR data

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	6.79	0.62	1.50	0.1955
Error	24	9.87	0.41	--	--
C. Total	35	16.66	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	3	0.35	0.12	0.29	0.8354
Co-prod. type	2	1.53	0.77	1.87	0.1766
Interaction	6	2.24	0.37	0.91	0.5058

Co-product D is also not significantly different than the other co-products. The ANOVA produced from analysis of all co-products three, six, and nine percent show no significant differences among the treatment means (Table 23). However, co-product D has the largest average of low critical temperatures than all of the co-products. Co-product D has the worst effect on the binder. This again supports the hypothesis that there are chemical interactions with the lignin in co-products A, B, and C and the binder. Co-product D has the largest low critical temperature, which indicates it has oxidized more than the other treatment combinations. Co-products A, B, and C all have negative effects, but they were less than co-product D. This shows the lignin may have possibly acted as an antioxidant and reduced the amount of oxidative aging on the binders.

The binder alone demonstrated to be significantly different than all treatment combinations except six percent co-product C. Table 24 illustrates the results of the individual paired t-tests of the neat binder with the treatment combinations.

Table 23. Model ANOVA and effect tests for PAV aged AAD-1 BBR data with co-product D

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	13.74	1.25	2.25	0.0470
Error	24	13.33	0.56	--	--
C. Total	35	27.07	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	2	1.36	0.68	1.23	0.3110
Co-prod. type	3	1.76	0.59	1.06	0.3861
Interaction	6	2.84	0.47	0.85	0.5426

Table 24. P-values for paired t-tests for PAV aged AAD-1 BBR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0226	9	A	0.0244
3	B	0.0494	9	B	0.0060
3	C	0.0068	9	C	0.0002
6	A	0.0344	12	A	0.0001
6	B	0.0008	12	B	0.0096
6	C	0.0654	12	C	0.0001

The addition of all co-products to AAD-1 causes an increase in the low critical temperature. The lignins cause the binder to stiffen, thus increasing the low critical temperature. All but one treatment combination is significantly different than the binder alone, while all the binder/co-product combinations are no different. The addition of three percent co-product C had the least negative effect on the binder. In general, the more lignin added, the greater the negative effect.

5.2.2 AAM-1

Binder AAM-1 was blended with three, six, and nine percent of co-products A, B, and C. Co-products A, B, and C were analyzed to determine which combination has the greatest effect on the binder. AAM-1 was also blended with co-product D at three, six, and nine percent to examine the antioxidant ability of the lignin-containing co-products A, B, and C. AAD-1 was also tested without co-product modification.

5.2.2.1 Unaged high critical temperature

Co-products A, B, and C were analyzed at three, six, and nine percent. Table 25 illustrates the results. Significant differences are only found with the amount of co-product factor. Tukey HSD testing concludes that the six and nine percent levels are significantly larger than the three percent level, while the six and nine percent levels are not significantly different. So as was seen with AAD-1, the more co-product added, the greater the stiffening effect, and therefore the greater the increase in critical high temperature.

Table 25. Model ANOVA and effect tests for unaged AAM-1 DSR data

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	8	6.10	0.76	6.32	0.0006
Error	18	2.17	0.12	--	--
C. Total	26	8.28	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	2	2.88	1.44	11.93	0.0005
Co-prod. type	2	0.42	0.21	1.74	0.2031
Interaction	4	0.98	0.24	2.03	0.1338

Co-product D was analyzed with co-products A, B, and C at three, six, and nine percent (Table 26). Significant differences were found with the amount of co-product factor and the interaction term. However, there are no significant differences with the type of co-product factor. Co-product D does not produce a statistically different unaged high critical temperature from the other three lignins.

Table 26. Model ANOVA and effect tests for unaged AAM-1 DSR data with co-product D

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	11	28.42	2.58	7.97	<.0001
Error	24	7.78	0.32	--	--
C. Total	35	36.20	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	2	8.65	4.32	13.34	0.0001
Co-prod. type	3	2.46	0.82	2.53	0.0815
Interaction	6	7.87	1.31	4.04	0.0061

Paired t-tests revealed all treatment combinations, except six percent co-product A, are statistically different than the neat binder. The measures for six percent co-product A have larger than normal variation and accounts for the inability to see significant differences. The results are shown in Table 27.

Table 27. P-values for paired t-tests for unaged AAM-1 DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0239	6	A	0.1232	9	A	0.0002
3	B	0.0189	6	B	<.0001	9	B	<.0001
3	C	0.0310	6	C	0.0007	9	C	0.0144

The addition of co-products A, B, and C at all percentages to AAM-1 increases the unaged high critical temperature. The trends are similar as to those seen with AAD-1. Generally, the more lignin-containing co-product added, the greater the increase in the unaged high critical temperature.

5.2.2.2 RTFO aged high critical temperature

A model ANOVA was formed analyzing co-products A, B, and C at three, six, and nine percent. Significant differences were found among the treatment groups. The effect tests show significant differences only in the amount of co-product factor and interaction. Table 28 illustrates the model ANOVA and effect test.

Table 28. Model ANOVA and effect tests for RTFO aged AAM-1 DSR data

Analysis of variance						Effect tests					
Error	D.F.	S.S.	M.S.	F ratio	Prob > F	Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	8	8.46	1.06	7.11	0.0003	% Co-prod.	2	3.16	1.58	10.61	0.0009
Error	18	2.68	0.15	--	--	Co-prod. type	2	0.08	0.04	0.27	0.7658
C. Total	26	11.14	--	--	--	Interaction	4	2.70	0.67	4.54	0.0104

Tukey HSD tests demonstrate that the six and nine percent levels are significantly larger than the three percent level, but the six and nine percent levels are not significantly different. There is significant interaction as the nine percent level does not always produce the largest high critical temperature. For lignin A, six percent produces a statistically larger high critical temperature than nine percent level.

Co-product D was analyzed by creating an ANOVA with three, six, and nine percent of co-products A, B, C, and D. The model ANOVA (Table 29) confirms significant differences between the treatment means, and the effects test illustrates that those differences are only in the amount of co-product factor. There are no statistical differences between the four co-products. This suggests that the lignin in co-products A, B, and C does not provide substantial benefit to the RTFO aged binder properties since co-product D behaves no differently with respect to the binder's rheological properties.

Table 29. Model ANOVA and effect tests for RTFO aged AAM-1 DSR data with co-product D

Analysis of variance						Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	11	18.04	1.64	9.96	<.0001	% Co-prod.	2	5.99	3.00	18.20	<.0001
Error	24	3.95	0.16	--	--	Co-prod. type	3	1.23	0.41	2.48	0.0853
C. Total	35	21.99	--	--	--	Interaction	6	3.71	0.62	3.76	0.0089

Paired t-tests conclude some treatment combinations are significantly different from the neat binder. Six and nine percent of co-products C and nine percent of co-product A are the two treatment combinations that are significantly different than the binder. Table 30 illustrates the results.

Table 30. P-values for paired t-tests for RTFO aged AAM-1 DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.2651	6	A	0.0993	9	A	0.0007
3	B	0.5806	6	B	0.5451	9	B	0.1805
3	C	0.3440	6	C	0.0046	9	C	0.0094

The addition of all amounts of all co-products increases the RTFO aged high critical temperature. As with AAD-1, the more co-products that are added, the greater the increase is in the high critical temperature due to the stiffening of the unmodified binder.

5.2.2.3 PAV aged intermediate critical temperature

A model ANOVA indicates significant differences within the treatment means. Effect tests confirm all factors and interaction are significant. Table 31 illustrates the results.

Table 31. Model ANOVA and effect tests for PAV aged AAM-1 DSR data

Analysis of variance						**Effect tests**					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	8	96.27	12.03	31.49	<.0001	% Co-prod.	2	27.49	13.74	35.96	<.0001
Error	18	6.88	0.38	--	--	Co-prod. type	2	10.81	5.41	14.14	0.0002
C. Total	26	103.15	--	--	--	Interaction	4	33.98	8.50	22.23	<.0001

Both factors were analyzed using Tukey HSD means testing. For the amount of co-product factor, the nine percent level is significantly larger than both the three and six percent level, while the three and six percent levels are not significantly different. For the type of co-product factor, co-products B and C are statistically larger than co-product A, but co-products B and C are not significantly different. Significant interaction is shown because of six percent lignin A having the lowest intermediate critical temperature of any of the combinations.

Co-product D was examined against the other three lignin-containing co-products using an ANOVA and effect test (Table 32). All factors prove to be significant. Tukey HSD means testing proves that co-product D has a significantly larger low critical temperature than the lignin-containing co-products A, B, and C. This is the same effect as seen with binder AAD-1. Since co-product D is an inert carbohydrate filler, it cannot act as an

Table 32. Model ANOVA and effect tests for PAV aged AAM-1 DSR data with co-product D

Analysis of variance						Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	11	160.41	14.58	32.06	<.0001	% Co-prod.	2	22.89	11.45	25.16	<.0001
Error	24	10.92	0.45	--	--	Co-prod. type	3	33.23	11.08	24.35	<.0001
C. Total	35	171.33	--	--	--	Interaction	6	41.41	6.90	15.17	<.0001

antioxidant by slowing oxidative aging. Lignin-containing co-products A, B, and C have smaller intermediate critical temperatures, therefore they have undergone less oxidative aging. This relationship suggests that the lignin in co-products A, B, and C is interacting with the binder by slowing oxidative aging.

T-tests (Table 33) prove all co-products are significantly different from the binder, except three percent co-products B and C. Also, two of the treatment combinations have a significantly lower intermediate critical temperature than the binder alone (six and three percent lignin A).

Table 33. P-values for paired t-tests for PAV aged AAM-1 DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0054	6	A	0.0161	9	A	0.0053
3	B	0.6309	6	B	0.0013	9	B	0.0007
3	C	0.0761	6	C	0.0032	9	C	0.0004

All treatment combinations, except three and six percent co-product A, cause an increase in the intermediate service temperature. Generally, the more co-product added, the greater the increase in intermediate service temperature. Combinations such as nine percent co-product B would work best in warmer climates, while combinations such as three percent co-product A would work best in cooler climates.

5.2.2.4 PAV aged low critical temperature

Analysis of the critical low temperature of three, six, and nine percent of co-products A, B, and C show significant differences among the treatment means. Effect tests display significant differences with lignin type factor and interaction, but not in the amount of lignin. The model ANOVA and effects test are shown in Table 34.

Table 34. Model ANOVA and effect tests for PAV aged AAM-1 BBR data

Analysis of variance						**Effect tests**					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	8	7.35	0.92	4.02	0.0068	% Co-prod.	2	1.32	0.66	2.89	0.0819
Error	18	4.11	0.23	--	--	Co-prod. type	2	2.46	1.23	5.38	0.0147
C. Total	26	11.46	--	--	--	Interaction	4	5.84	1.46	6.40	0.0022

Tukey HSD testing reveals the only significant difference to be co-product A possessing a significantly larger response than co-product C. Interaction effects can be seen by no specific amount of co-product having the largest low critical temperature. For instance, six percent co-product A has the smallest response for co-product A, while six percent co-product C has the largest response for co-product C.

Co-product D again had the largest average of low critical temperatures than the other three lignin-containing co-products. The model ANOVA and effect tests (Table 35) indicate differences between the type of co-product and interaction effect when including co-product D in the analysis. Again with the low critical temperature, the amount of co-product is not significant. Tukey HSD testing demonstrates co-product D to produce significantly larger low critical temperatures than co-products B and C, but not co-product A. This again suggests that the lignin in lignin-containing co-products A, B, and C provides benefit to the asphalt. A lower critical low temperature indicates less oxidative aging. Since co-products

A, B, and C have lower critical low temperatures than lignin D, they have undergone less oxidative aging.

Table 35. Model ANOVA and effect tests for PAV aged AAM-1 BBR data with co-product D

Analysis of variance					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	11	32.43	2.95	14.47	<.0001
Error	24	4.89	0.20	--	--
C. Total	35	37.32	--	--	--

Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
% Co-prod.	2	0.80	0.40	1.97	0.1611
Co-prod. type	3	6.34	2.11	10.37	0.0001
Interaction	6	7.06	1.18	5.77	0.0008

Only one treatment combination is significantly different from the neat binder AAM-1. Six percent co-product C is significantly larger than AAM-1. Since lower low critical temperatures are generally better for the asphalt, six percent co-product C provides the least desirable response. All other treatment combinations proved to not be statistically different from the binder alone. Results of the paired t-tests are shown in Table 36.

Table 36. P-values for paired t-tests for PAV aged AAM-1 BBR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.1302	6	A	0.0708	9	A	0.3237
3	B	0.2301	6	B	0.2252	9	B	0.5506
3	C	0.5006	6	C	0.0405	9	C	0.4567

Generally, the addition of co-products A, B, and C to AAM-1 causes an increase in the low critical temperature. The co-products cause the binder to stiffen, thus increasing the low critical temperature. All but one treatment combination is not significantly different than the neat binder. The effect of the co-products on the low temperature properties of AAM-1 is negative; however, only one treatment group is significantly different from the neat binder.

5.2.3 Local Binder

LB was blended with three, six, and nine percent of co-products A, B, and C. Co-products A, B, and C were analyzed to determine which combination has the greatest effect on the binder. LB was also tested without the addition of any co-product as a reference.

5.2.3.1 Unaged high critical temperature

An ANOVA was formed analyzing three, six, and nine percent of co-products A, B, and C. Both factors are significant as shown by the model ANOVA and effect tests (Table 37). Tukey HSD testing was performed on the treatment groups of both factors. For the co-product type factor, the high critical temperature for co-products B and C are significantly larger than that of lignin A. However, lignins B and C are not significantly different. For the amount of lignin factor, all treatment groups are statistically different. The nine percent level produces the largest high critical temperature, followed by the six percent level. The three percent level has the statistically lowest high critical temperature.

Table 37. Model ANOVA and effect tests for unaged LB DSR data

Analysis of variance						**Effect tests**					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	8	21.54	2.69	31.06	<.0001	% Co-prod.	2	5.22	2.61	30.13	<.0001
Error	18	1.56	0.09	--	--	Co-prod. type	2	5.13	2.57	29.60	<.0001
C. Total	26	23.10	--	--	--	Interaction	4	0.82	0.20	2.36	0.0921

Paired t-tests were used to evaluate the treatment combinations versus the binder with no co-products added. Table 38 illustrates the p-values for all individual t-tests. All treatment combinations are significantly different from the neat binder.

All treatment combinations were beneficial to the high critical temperature for LB. The trends are similar to the previous two binders. The more co-product added, the greater the increase in the response. The most beneficial combination is the addition of nine percent co-product B. The least beneficial combination was three percent of co-product A.

Table 38. P-values for paired t-tests for unaged LB DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0166	6	A	0.0003	9	A	0.0025
3	B	0.0004	6	B	<.0001	9	B	<.0001
3	C	0.0041	6	C	0.0005	9	C	<.0001

5.2.3.2 RTFO aged high critical temperature

An ANOVA was performed considering all treatment groups. Effect tests show the type of co-product factor and interaction are not significant, while the amount of co-product factor is significant. The model ANOVA and effects tests are shown in Tables 39.

Table 39. Model ANOVA and effect tests for RTFO aged LB DSR data

Analysis of variance

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	8	4.03	0.50	6.62	0.0004
Error	18	1.37	0.08	--	--
C. Total	26	5.39	--	--	--

Effect tests

Error	D.F.	S.S.	M.S.	F ratio	Prob > F
% Co-prod.	2	1.51	0.76	9.95	0.0012
Co-prod. type	2	0.39	0.19	2.55	0.1058
Interaction	4	0.78	0.20	2.58	0.0727

Tukey HSD testing indicates the nine percent level is significantly larger than the three percent level. The six and nine percent levels and the three and six percent levels are not significantly different. In general, the more co-products added, the greater the increase in the RTFO aged high critical temperature.

Paired t-tests were used to evaluate the treatment combinations versus the neat binder. Only six percent co-product A is not significantly different than the neat binder. All other treatment combinations produce significantly larger responses than the neat binder. Table 40 illustrates the t-test results.

Table 40. P-values for paired t-tests for RTFO aged LB DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0096	6	A	0.0709	9	A	0.0234
3	B	0.0443	6	B	0.0010	9	B	0.0001
3	C	0.0187	6	C	0.0007	9	C	0.0084

All combinations of co-product with LB are beneficial for the RTFO aged high critical temperature. As with the previously two analyzed binders, the trend shows that the more co-product that is added, the greater is the stiffening, and therefore the greater is the increase in critical high temperature.

5.2.3.3 PAV aged intermediate critical temperature

An ANOVA and effect tests (Table 41) were done evaluating three, six, and nine percent of co-products A, B, and C. Tukey HSD testing was performed on both factors. Co-product B has a statistically larger response than co-product A, while all other comparisons are not significantly different. The only difference in the amount of co-product is between the three and nine percent levels. The nine percent level is significantly larger than the three percent level.

Table 41. Model ANOVA and effect tests for PAV aged LB DSR data

Analysis of variance						Effect tests					
Error	D.F.	S.S.	M.S.	F ratio	Prob > F	Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	8	1.89	0.24	6.33	0.0006	% Co-prod.	2	0.42	0.21	5.57	0.0131
Error	18	0.67	0.04	--	--	Co-prod. type	2	0.38	0.19	5.02	0.0186
C. Total	26	2.57	--	--	--	Interaction	4	0.05	0.01	0.33	0.8544

Paired t-tests (Table 42) were again used to evaluate the different treatment groups versus the binder with no added co-product. All treatment groups except three and six percent co-product A have a significantly larger intermediate critical temperature than the neat binder. This suggests the addition of co-product significantly affects the intermediate temperature properties of LB.

Table 42. P-values for paired t-tests for PAV aged LB DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0819	6	A	0.1043	9	A	0.0116
3	B	0.0070	6	B	0.0218	9	B	0.0002
3	C	0.0053	6	C	0.0016	9	C	0.0029

The addition of all combinations of co-product to LB creates an increase in the intermediate critical temperature. This trend was also seen with both binders AAD-1 and AAM-1. An increase in the intermediate critical temperature can be beneficial or detrimental, depending upon the climate where the asphalt is used. In general, the more co-product added, the greater the increase in the intermediate critical temperature. Low amounts of co-product A seem to have no significant effect on the intermediate temperature properties.

5.2.3.4 PAV aged low critical temperature

The model ANOVA suggests that there are no significant differences among the treatment groups for low temperature properties of LB. Larger variation between the measurements of the same treatment groups is the cause for the inability to notice statistical differences. However, effect tests show that the amount of co-product factor is significant. Tukey HSD testing illustrates that the nine percent level produces a significantly larger low critical temperature than the three and six percent levels. Table 43 illustrates the low critical temperature analysis.

Table 43. Model ANOVA effect tests for PAV aged LB BBR data

Analysis of variance						Effect tests					
Error	D.F.	S.S.	M.S.	F ratio	Prob > F	Error	D.F.	S.S.	M.S.	F ratio	Prob > F
Model	8	7.74	0.97	2.30	0.0681	% Co-prod.	2	6.01	3.00	7.14	0.0052
Error	18	7.58	0.42	--	--	Co-prod. type	2	0.51	0.25	0.60	0.5581
C. Total	26	15.31	--	--	--	Interaction	4	1.46	0.37	0.87	0.5016

All combinations, except three percent co-product A, are significantly different than the neat binder. Paired t-testing produces p-values smaller than α for all treatment combinations except three percent co-product A. Results are shown in Table 44.

Table 44. P-values for paired t-tests for PAV aged LB BBR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.0909	6	A	0.0302	9	A	0.0002
3	B	0.0141	6	B	0.0011	9	B	0.0011
3	C	0.0004	6	C	0.0243	9	C	0.0159

All treatment combinations have low critical temperatures greater than the neat binder. This effect in and of itself is negative, and all combinations except three percent co-product A prove to be significantly less beneficial than the binder. Statistical analysis shows that the more co-product that is added, the greater the increase in critical low temperature. The different lignin-containing co-products do not produce significantly different low critical temperatures.

5.2.4 Local Polymer Modified Binder

LPMB was blended with three, six, nine, and twelve percent of co-products A, B, and C. Co-products A, B, and C were analyzed to determine which combination has the greatest effect on the binder. LPMB was also tested without the addition of any co-product as a reference.

5.2.4.1. Unaged high critical temperature

An ANOVA was formed analyzing the critical high temperatures of three, six, nine, and twelve percent of co-products A, B, and C. The model ANOVA reveals differences among treatment groups, while the effects test goes shows both factors and the interaction is significant. Table 45 illustrates the results.

Table 45. Model ANOVA effect tests for unaged LPMB DSR data

Analysis of variance						Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	11	27.16	2.47	40.86	<.0001	% Co-prod.	3	13.46	4.49	74.23	<.0001
Error	24	1.45	0.06	--	--	Co-prod. type	2	1.17	0.58	9.64	0.0008
C. Total	35	28.61		--	--	Interaction	6	3.46	0.58	9.55	<.0001

Tukey HSD testing was performed on both factors. Co-product B has significantly larger response than the other two co-products. Co-products A and C are not statistically different. All amounts of co-product are significantly different. The twelve percent level produces the statistically largest high critical temperature, followed by the nine, six, and finally the three percent level. It can be concluded that the further addition of co-product increases the unaged high critical temperature. There is significant interaction between the six and nine percent co-product C high critical temperatures. With co-products A and B, the nine percent level is statistically larger than the six percent level. However, with co-product C, the six percent level is statistically larger than the nine percent level.

Half of the combinations are significantly different than the binder with no co-product. Table 46 exhibits the p-values from the paired t-tests. The combinations shown in bold are not significantly different than the neat binder. These results suggest that in lower amounts, the co-products do affect the high temperature properties of LPMB as much as the previous three binders discussed. This effect could be due to the presence of polymer in the binder, as the other three previously discussed binders do not contain binder.

Table 46. P-values for paired t-tests for unaged LPMB DSR data

Co-prod. (%)	Co-prod. type	Prob>|t|	Co-prod. (%)	Co-prod. type	Prob>|t|
3	A	0.6764	9	A	0.0648
3	B	0.0854	9	B	0.0018
3	C	0.6437	9	C	0.1071
6	A	0.3325	12	A	0.0069
6	B	0.0118	12	B	0.0009
6	C	0.0274	12	C	0.0162

The addition of all types and amounts of lignin-containing co-products increase the high temperature properties of the local polymer-modified binder. However, in lower quantities, the effect is not significant. This effect could be due to the presence of polymer in the binder. The general effect on the unaged high temperatures is the same as the other three previous binders. The polymer and co-products do not seem to interact negatively.

5.2.4.2 RTFO aged high critical temperature

Analysis indicates significant differences between treatment groups. Effect tests show differences (Table 47) between the amount of co-product factor and interaction, but not with the type of co-product factor. Tukey HSD testing shows that all levels for the amount of co-product are significantly different. The twelve percent level has the largest high critical temperature, followed by the nine, six, and the three percent level. The more co-product added, the larger the high critical temperature. As the case with all binders, the co-products stiffen the binder (even with the presence of polymer), which causes the high critical temperature to increase.

Table 47. Model ANOVA effect tests for RTFO aged LPMB DSR data

Analysis of variance						Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	8	4.03	0.50	6.62	0.0004	% Co-prod.	2	1.51	0.76	9.95	0.0012
Error	18	1.37	0.08	--	--	Co-prod. type	2	0.39	0.19	2.55	0.1058
C. Total	26	5.39	--	--	--	Interaction	4	0.78	0.20	2.58	0.0727

T-testing concludes that most treatment groups have a significantly different response than the binder without the addition of co-product. Three percent of co-products A, B, and C and six percent of co-product A are not statistically different from the neat binder. Basically, the lower amounts of co-product to not significantly affect the intermediate temperature properties of the binder. Table 48 illustrates the p-values of the t-tests.

Table 48. P-values for paired t-tests for RTFO aged LPMB DSR data

Co-prod. (%)	**Co-prod. type**	**Prob>|t|**	**Co-prod. (%)**	**Co-prod. type**	**Prob>|t|**
3	A	0.5871	9	A	0.0090
3	B	0.3531	9	B	0.0004
3	C	0.0600	9	C	0.0032
6	A	0.7731	12	A	<.0001
6	B	0.0312	12	B	<.0001
6	C	0.0010	12	C	<.0001

In summary, the addition of co-products to the LPMB causes an increase in stiffness that follows the same trends as the previous three binders. The further addition of co-product causes the binder to stiffen, thus increasing the high critical temperature. The more co-product added, the greater the stiffening. Twelve percent co-product B provides the greatest beneficial response, while three percent co-product A provides the least beneficial response.

5.2.4.3 PAV aged intermediate critical temperature

Significant differences were found between both factors and the interaction. The model ANOVA and effect tests are shown in Table 49. Tukey HSD testing shows that the twelve percent level has a significantly larger response than the other three levels, while the three, six, and nine percent levels are not significantly different. Co-product B has a significantly larger response than co-product A and C, while co-products A and C are not significantly different. The interaction is significant because the different co-products do not produce similar trends with different amount of co-product added.

Table 49. Model ANOVA effect tests for PAV aged LPMB DSR data

Analysis of variance						**Effect tests**					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	8	1.89	0.24	6.33	0.0006	% Co-prod.	2	0.42	0.21	5.57	0.0131
Error	18	0.67	0.04	--	--	Co-prod. type	2	0.38	0.19	5.02	0.0186
C. Total	26	2.57	--	--	--	Interaction	4	0.05	0.01	0.33	0.8544

T-testing identifies that half of the twelve treatment groups are significantly different from the binder alone. Table 50 illustrates the p-values produced from each of the t-tests.

Table 50. P-values from individual t-tests for PAV aged LPMB DSR data

Co-prod. (%)	**Co-prod. type**	**Prob>|t|**	**Co-prod. (%)**	**Co-prod. type**	**Prob>|t|**
3	A	0.4890	9	A	0.7020
3	B	<.0001	9	B	0.0002
3	C	0.5096	9	C	0.4928
6	A	0.6949	12	A	<.0001
6	B	<.0001	12	B	<.0001
6	C	0.9532	12	C	0.0007

In summary, all treatment combinations cause in increase in the intermediate service temperature. Generally, the more co-product added, the greater the increase. As said previously, this effect can be positive or negative depending upon the climate in which the asphalt is used. The trend of increasing intermediate service temperature with the further addition of lignin-containing co-products is present with all four binders tested.

5.2.4.4 PAV aged low critical temperature

The ANOVA and effect tests (Table 51) indicate differences between both factors and interaction. Tukey HSD testing shows that the twelve percent level possesses the statistically largest low critical temperature. The six percent level is significantly larger than the three percent level, but not significantly different from the nine percent level. The three and nine percent levels are not significantly different. Co-product B has a significantly larger low critical temperature than the other two co-products, while co-products A and C are not significantly different. The six and nine percent levels vary with their response trends with the different types of co-products; the cause of significant interaction effects.

Table 51. Model ANOVA and effect tests for PAV aged LPMB BBR data

Analysis of variance						Effect tests					
Error	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**	**Error**	**D.F.**	**S.S.**	**M.S.**	**F ratio**	**Prob > F**
Model	8	7.74	0.97	2.30	0.0681	% Co-prod.	2	6.01	3.00	7.14	0.0052
Error	18	7.58	0.42	--	--	Co-prod. type	2	0.51	0.25	0.60	0.5581
C. Total	26	15.31	--	--	--	Interaction	4	1.46	0.37	0.87	0.5016

Most of the treatment groups have significantly larger responses than the binder. Table 52 illustrates the results. All groups except both three and nine percent co-products A and C are significantly larger than the low critical temperatures produced by the binder.

Table 52. P-values from individual t-tests for PAV aged LPMB BBR data

Amount of lignin (%)	Lignin Type	Prob>|t|	Amount of lignin (%)	Lignin Type	Prob>|t|
3	A	0.2598	9	A	0.3316
3	B	0.0259	9	B	0.0138
3	C	0.4140	9	C	0.1841
6	A	0.0831	12	A	0.0033
6	B	0.0010	12	B	0.0004
6	C	0.0094	12	C	0.0006

In summary, all treatment groups have larger low critical temperatures than the binder. As with the other three binders, the addition of the lignin-containing co-product causes an increase in the low critical temperature of the binder. Generally, the more co-product added, the larger the increase. At lower levels, some of the treatment combinations are not any different from the binder alone.

5.2.5 Effect of Different Binders on Critical Temperatures

Each binder produces significant differences for the four critical temperatures previously analyzed: unaged high critical temperature, RTFO aged high critical temperature, PAV aged intermediate temperature, and PAV aged low critical temperature. This should be expected as each binder possesses very significant different rheological properties. However, it is important to attempt to discover any significant interactions between the different binders and the co-product treatment groups to see if certain co-product combinations perform differently in different binders. Model ANOVAs were formed for each critical temperature by analyzing three, six, and nine percent of co-products A, B, and C with all four binders. The results are shown in Table 53.

Table 53. P-values for binder interaction for critical temperatures (T_c)

Unaged high T_c	**RTFO aged high T_c**	**PAV aged int. T_c**	**PAV aged low T_c**
0.0020	0.0153	0.0010	0.1610

It can be seen that all critical temperatures, except the PAV aged low critical temperature, have p-values less than α. This means that there is significant interaction between the different binders and the different co-product treatment groups. It can be concluded that not all binders behave similar with the addition of lignin-containing co-products (except the low critical temperature). Not only do different combinations of co-product produce significant differences with the critical temperatures, but the critical temperatures are also dependent upon which binder the co-product is being added.

5.2.6 Performance Grade Summary

The following tables (54, 55, 56 and 57) present a summary of the performance grade changes for each binder with the addition of the lignin-containing co-products. Presented in each table is the unaged and RTFO aged high temperatures, and PAV aged intermediate and low temperatures. The changes in temperature from the neat binder are shown next to each temperature column. Finally, the overall change in performance grade is calculated. The overall change is the change in the lowest high critical temperature (unaged or RTFO aged) minus the change in the PAV aged low critical temperature. If the change of the high or low critical temperature is not significant as previously shown, than that difference used for calculation is zero. All significant changes are shown in bold. It can be seen in almost all cases for each binder that the addition of lignin-containing co-products significantly increases the performance grade depending upon the type and amount of co-product added.

Table 54. Critical temperature (T_c) changes for AAD-1

Co-product (%)	**Co-product (type)**	**Unaged high T_c (°C)**	**Δ T_c**	**RTFO aged high T_c (°C)**	**Δ T_c**	**Pav aged int.T_c (°C)**	**Δ T_c**	**PAV aged low T_c (°C)**	**Δ T_c**	**Total sig. Δ PG range**
12	A	**64.8**	**2.5**	**67.9**	**2.5**	**18.5**	**1.2**	**-24.0**	**1.2**	1.3
12	B	**67.2**	**4.9**	**70.1**	**4.7**	**19.6**	**2.3**	**-23.1**	**2.1**	2.8
12	C	**64.5**	**2.2**	**67.2**	**1.8**	**18.5**	**1.2**	**-23.4**	**1.8**	0.4
9	A	**64.8**	**2.5**	**67.5**	**2.1**	18.2	0.9	**-23.6**	**1.6**	0.9
9	B	**66.6**	**4.3**	**69.3**	**3.9**	**19.0**	**1.7**	**-23.4**	**1.8**	2.5
9	C	**64.5**	**2.2**	**67.0**	**1.6**	**18.3**	**1.0**	**-23.7**	**1.5**	0.7
6	A	**64.5**	**2.2**	**66.9**	**1.5**	**18.0**	**0.7**	**-23.9**	**1.3**	0.9
6	B	**64.8**	**2.5**	**68.6**	**3.2**	**18.7**	**1.4**	**-22.6**	**2.6**	-0.1
6	C	**63.4**	**1.1**	**66.2**	**0.8**	**18.1**	**0.8**	-23.9	1.3	1.1
3	A	**63.7**	**1.4**	65.8	0.4	17.3	0.0	**-23.7**	**1.5**	-0.1
3	B	**64.8**	**2.5**	**67.5**	**2.1**	18.1	0.8	**-23.2**	**2.0**	0.5
3	C	**62.8**	**0.5**	66.7	1.3	17.8	0.5	**-24.2**	**1.0**	-0.5
0	--	62.3	0.0	65.4	0.0	17.3	0.0	-25.2	0.0	0.0

Tab1e 55. Critical temperature (T_c) changes for AAM-1

Co-product (%)	Co-product (type)	Unaged high T_c (°C)	Δ T_c	RTFO aged high T_c (°C)	Δ T_c	Pav aged int.T_c (°C)	Δ T_c	PAV aged low T_c (°C)	Δ T_c	Total sig. Δ PG range
9	A	**68.7**	**0.9**	**67.6**	**0.9**	**22.4**	**2.1**	-14.9	0.2	0.9
9	B	**69.8**	**2.0**	67.2	0.5	**22.9**	**2.6**	-14.8	0.3	0.5
9	C	**68.8**	**1.0**	**68.5**	**1.8**	**21.4**	**1.1**	-15.2	-0.1	1.8
6	A	68.5	0.7	68.2	1.5	**19.8**	**-0.5**	-16.1	-1.0	1.5
6	B	**69.3**	**1.5**	66.9	0.2	**22.0**	**1.7**	-14.9	0.2	0.2
6	C	**68.8**	**1.0**	**67.5**	**0.8**	**22.1**	**1.8**	**-14.7**	**0.4**	0.4
3	A	**68.5**	**0.7**	66.9	0.2	**18.6**	**-1.7**	-14.0	1.1	0.2
3	B	**68.5**	**0.7**	66.8	0.1	20.6	0.3	-14.9	0.2	0.1
3	C	**68.1**	**0.3**	67.0	0.3	20.9	0.6	-15.2	-0.1	0.3
0	--	67.8	0.0	66.7	0.0	20.3	0.0	-15.1	0.0	0.0

Table 56. Critical temperature (T_c) changes for LB

Co-product (%)	Co-product (type)	Unaged high T_c (°C)	Δ T_c	RTFO aged high T_c (°C)	Δ T_c	Pav aged int.T_c (°C)	Δ T_c	PAV aged low T_c (°C)	Δ T_c	Total sig. Δ PG range
9	**A**	**70.4**	**1.5**	**71.9**	**0.8**	**23.9**	**0.4**	**-16.7**	**2.8**	-1.3
9	**B**	**72.2**	**3.3**	**72.4**	**1.3**	**24.6**	**1.1**	**-17.5**	**2.0**	1.3
9	**C**	**71.7**	**2.8**	**72.3**	**1.2**	**24.2**	**0.7**	**-17.4**	**2.1**	0.7
6	**A**	**69.9**	**1.0**	71.4	0.3	23.9	0.4	**-18.3**	**1.2**	-0.2
6	**B**	**71.9**	**3.0**	**72.5**	**1.4**	**24.3**	**0.8**	**-18.7**	**0.8**	2.2
6	**C**	**70.6**	**1.7**	**71.8**	**0.7**	**24.2**	**0.7**	**-17.9**	**1.6**	0.1
3	A	**69.3**	**0.4**	**71.6**	**0.5**	23.7	0.2	-18.4	1.1	0.4
3	**B**	**71.1**	**2.2**	**71.9**	**0.8**	**24.2**	**0.7**	**-18.1**	**1.4**	0.8
3	**C**	**70.7**	**1.8**	**71.4**	**0.3**	**23.9**	**0.4**	**-17.6**	**1.9**	-0.1
0	--	68.9	0.0	71.1	0.0	23.5	0.0	-19.5	0.0	0.0

5.3 Separation Effects

When stored at high temperatures, the lignin-containing co-products tend to physically separate from the binder. The results from the solubility testing illustrates that the co-products do not completely form a solution with the binder, but rather a physical mixture. When the blends are stored at high temperature (155°C), the less dense binder physically separates from the denser lignin-containing co-product that has been coated with binder. Generally, the longer the blends are stored, the more separation occurs. Also, the more co-

Table 57. Critical temperature (T_c) changes for LPMB

Co-product (%)	Co-product (type)	Unaged high T_c (°C)	Δ T_c	RTFO aged high T_c (°C)	Δ T_c	Pav aged int.T_c (°C)	Δ T_c	PAV aged low T_c (°C)	Δ T_c	Total sig. Δ PG range
12	A	**67.7**	**5.4**	**71.8**	**5.1**	**22.4**	**2.3**	**-19.6**	**2.4**	3.0
12	B	**68.7**	**6.4**	**72.5**	**5.8**	**22.7**	**2.6**	**-19.1**	**2.9**	3.5
12	C	**66.7**	**4.4**	**71.2**	**4.5**	**22.2**	**2.1**	**-18.8**	**3.2**	1.2
9	A	66.7	4.4	**67.7**	**1.0**	20.1	0.0	-21.2	0.8	3.6
9	B	**68.6**	**6.3**	**69.3**	**2.6**	**21.8**	**1.7**	**-19.6**	**2.4**	3.9
9	C	67.1	4.8	**69.1**	**2.4**	20.2	0.1	-21.5	0.5	4.3
6	A	66.4	4.1	66.8	0.1	20.2	0.1	-20.9	1.1	3.0
6	B	**66.7**	**4.4**	**68.1**	**1.4**	**21.9**	**1.8**	**-19.8**	**2.2**	2.2
6	C	**65.8**	**3.5**	**68.4**	**1.7**	20.2	0.1	-20.3	1.7	3.5
3	A	66.1	3.8	66.4	-0.3	20.2	0.1	-21.5	0.5	0
3	B	67.2	4.9	67.1	0.4	**21.9**	**1.8**	**-20.1**	**1.9**	0
3	C	65.9	3.6	67.3	0.6	20.2	0.1	-21.7	0.3	0
0	--	62.3	0.0	66.7	0.0	20.1	0.0	-22.0	0.0	0

products added, the larger the separation effects of the blends. The separation effects are also co-product type and binder dependent, which can be seen in the following analysis.

5.3.1 Effect of Different Lignins

All co-products (A, B, C, and D) were mixed with AAD-1 to see which co-product had the greatest susceptibility to separation. Figure 9 illustrates the results of the test. It can be seen all co-products separate more as time progresses. Co-product B has the largest separation effects, while co-product A has the smallest. Co-product A is preferred since separation from the binder would cause the least potential problems with handling and transport. Co-product A was selected for further analysis by analyzing different amounts of co-product A.

5.3.2 Effect of Amount of Lignin

Co-product A was added to AAD-1 at zero, three, six, and nine percent. The same testing procedure was used. Figure 10 illustrates the results. Notice how the further addition of co-product causes an increase in the amount of separation. Also, as time progresses, the separation effects increase. The viscosity differences are almost thirty percent with the addition of nine percent co-product A. It can be concluded that the more co-product added, the greater the separation effects.

5.3.3 Effect of Binder Type

All four binders were combined with co-product A at six percent to determine which binder has the greatest separation effects. Figure 9 illustrates the results. The local binder (LB) without polymer has the greatest separation effects, while AAM-1 had the smallest effects. The same trend can be observed that separation increases with time. The different separation effects are likely attributed to binder chemistry and physical properties. The combination with the least separation effects is six percent lignin A with AAM-1.

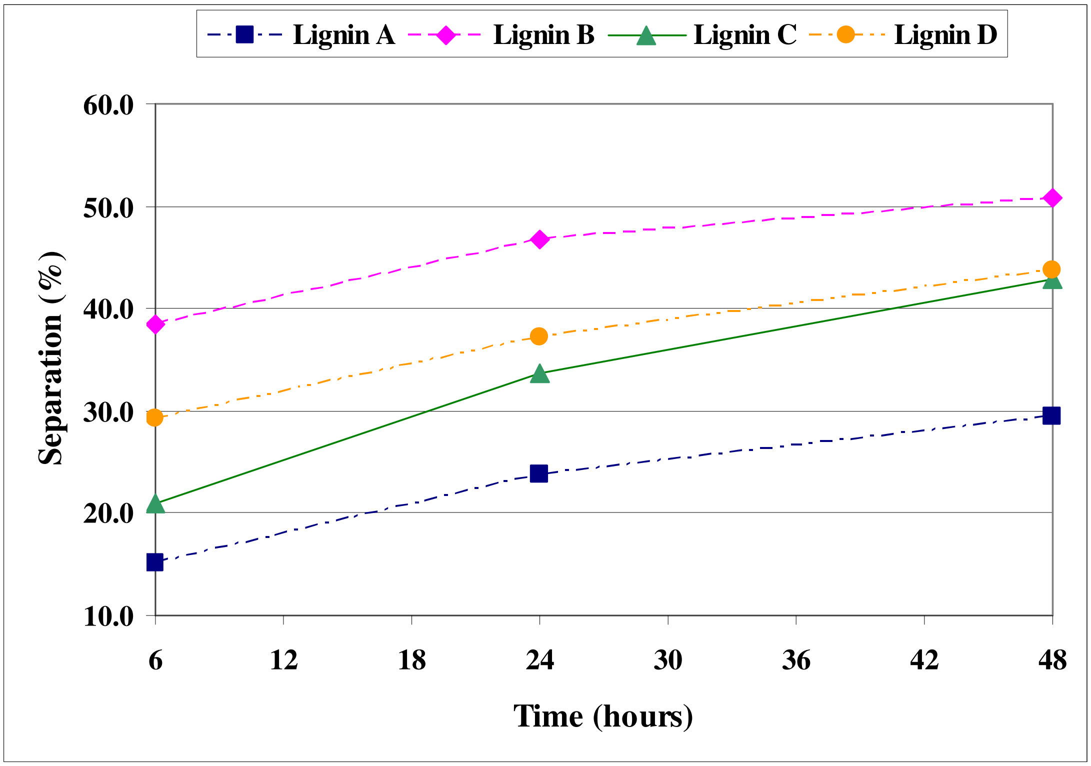

Figure 9. Separation of AAD-1 with all co-products at nine percent

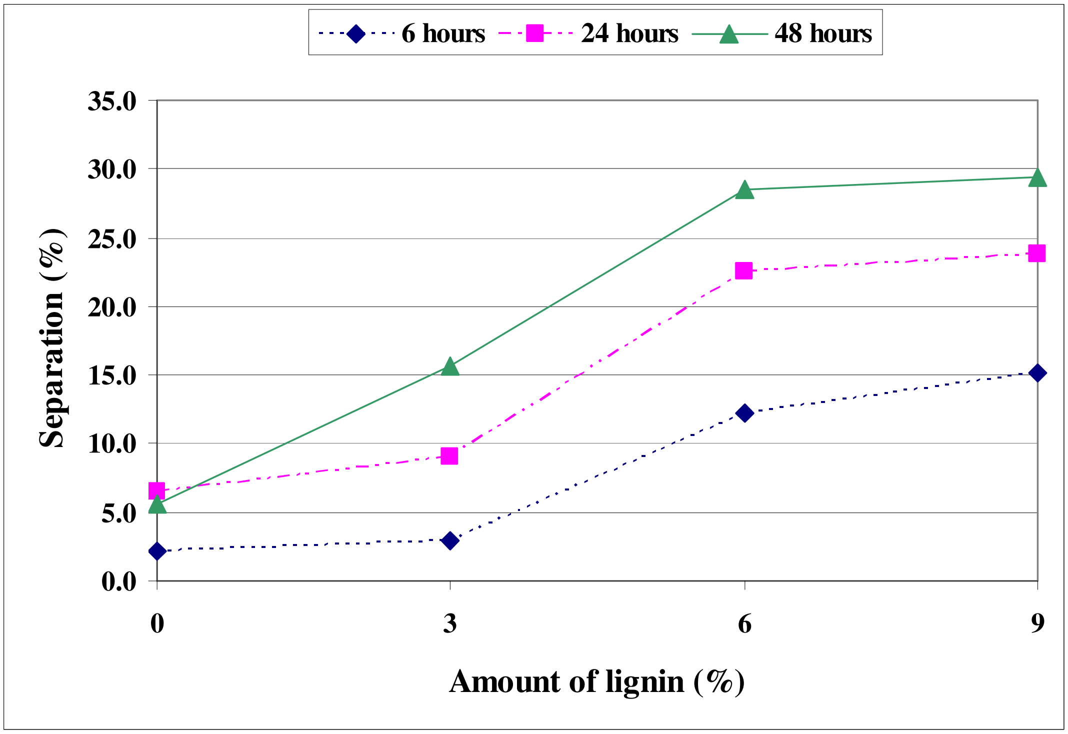

Figure 10. Separation of AAD-1 with co-product A at zero, three, six, and nine percent

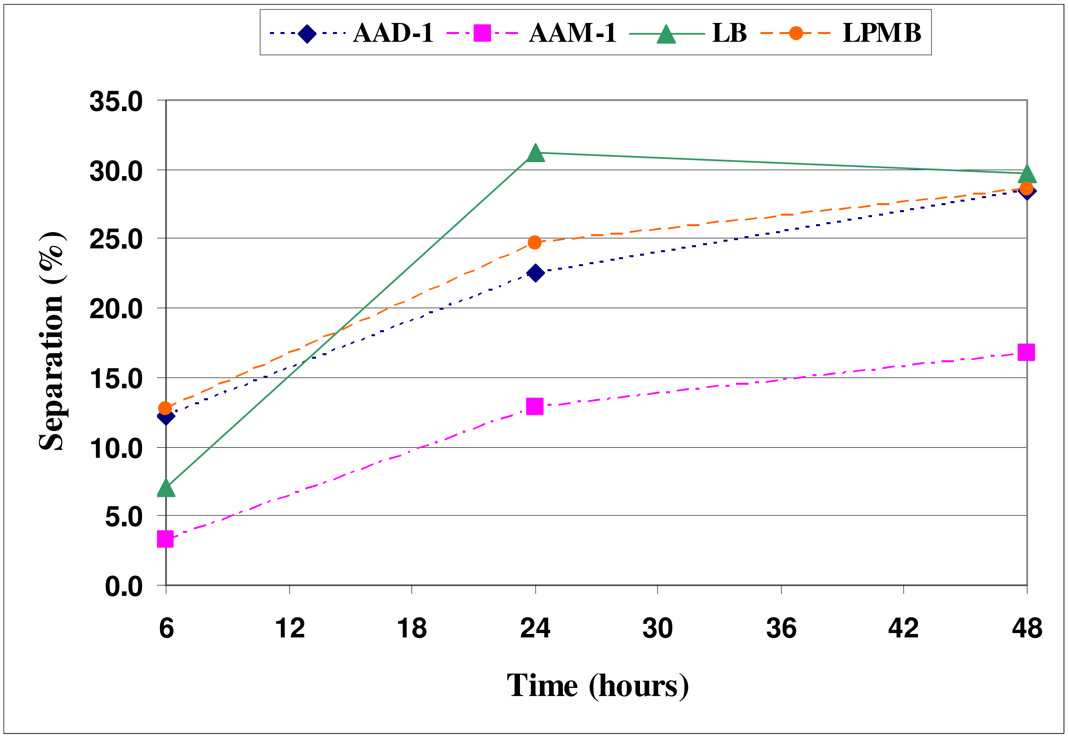

Figure 11. Separation of all binders with nine percent co-product A

5.4 Oxidation Testing

5.4.1 Carbonyl Content

Carbonyl functional groups are present in carboxylic acids, ketones, and anhydrides, all of which are products of asphalt binder oxidation. The results of the oxidation testing show conflicting results (Figure 12). For binder AAD-1, the addition of all three co-products causes an increase in carbonyl groups. Also, the more of each co-product added, generally the greater the increase in the carbonyl content. With binder AAM-1, three percent of co-products A and B and nine percent of A and C cause an increase in the carbonyl content, while three percent co-product C and nine percent co-product B cause a very slight decrease. With binder LB, the addition of three percent of co-products A, B, and C and nine percent co-product B and C actually cause a decrease in the carbonyl content, while only nine percent

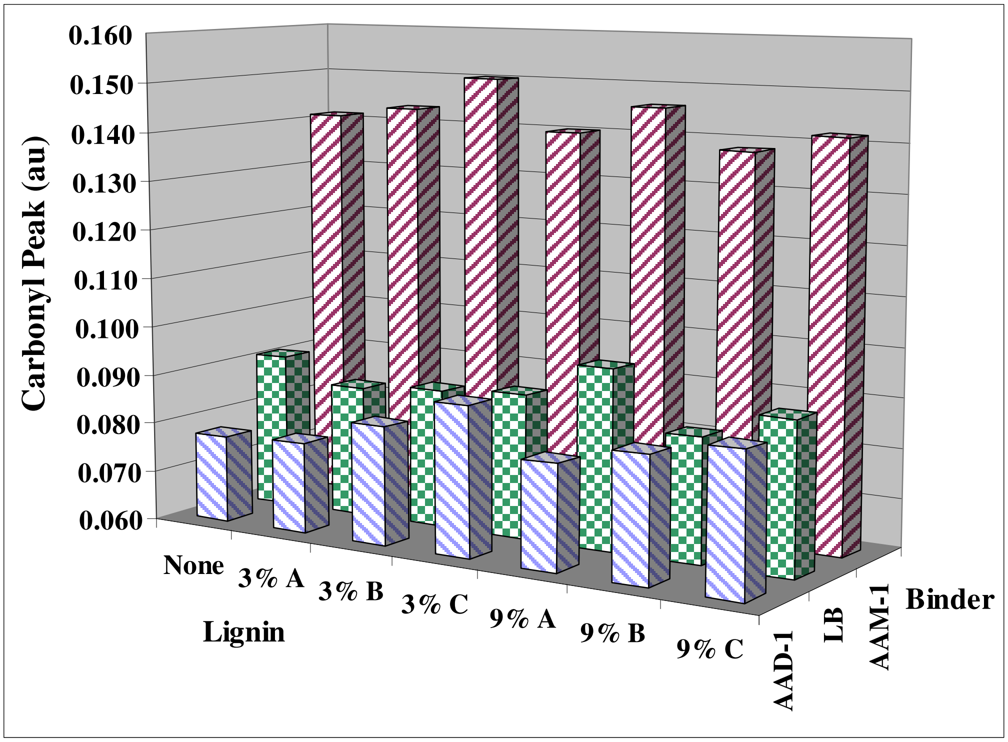

Figure 12. Carbonyl contents of select binder/co-product blends

of A causes a very slight increase. The results of the three binders show no general trends with the carbonyl content. The results are binder specific. Generally, the addition of lignin-containing co-products to binders AAD-1 and AAM-1 increases the carbonyl content. This trend suggests more oxidative aging. The opposite occurs with binder LB. The addition of lignin-containing co-products generally decreases the carbonyl content.

5.4.2 Sulfoxide Content

The sulfoxide testing produced opposite results than the carbonyl testing. Figure 13 illustrates the results from sulfoxide testing with FTIR. With binder AAM-1, the addition of all co-products at all levels caused a decrease in the sulfoxide content. Also, the more of each co-product added, the greater the decrease in sulfoxides. With AAD-1, the addition of co-products A and B at three percent cause a decrease in the sulfoxide content. However,

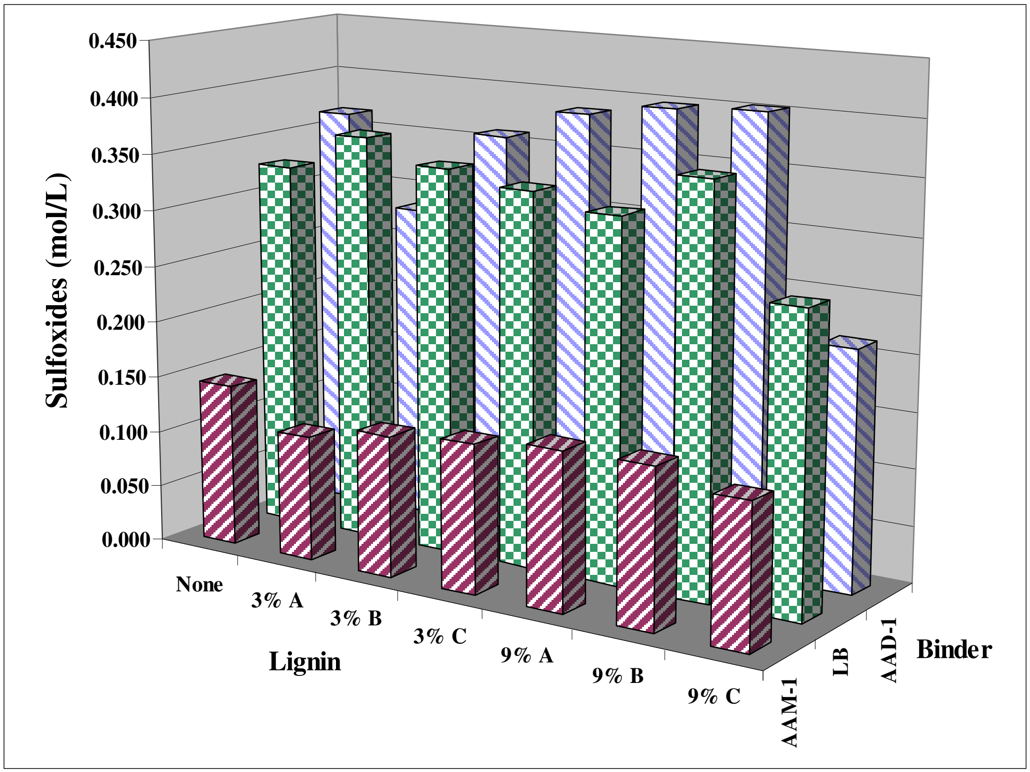

Figure 13. Sulfoxide contents of select binder/co-product blends

when co-products A and B are added at the nine percent level, the sulfoxide content increases. With co-product C, the three percent addition causes a sulfoxide increase, while at the nine percent level, the sulfoxide content decreases. These results suggest different chemical interactions with the different co-products. Finally, with binder LB, the sulfoxide content is increased with all co-products at the three percent level, while at nine percent, the sulfoxide content decreases (except with co-product B).

6. CONCLUSIONS

6.1 Project Summary

Asphalt oxidation is the primary cause of long-term pavement failure. This study investigates the use of a natural antioxidant for use in asphalt binder. Lignin-containing co-products from wet-mill ethanol production were added to asphalt binder to evaluate the co-products' effects on the rheological properties of the binder. The lignin-containing co-products stiffened the binder at all stages of aging. The result of stiffening was an improvement to the high temperature properties, with a worsening to the low temperature properties. However, with many asphalt binder/co-product combinations, the high temperature performance grade was increased significantly, while the decrease in the low temperature performance grade was small and/or not significant. This general widening of the continuous performance grade is beneficial to the binder and suggests some antioxidant activity of the lignin with the binder. The testing of co-product D (no lignin added) supports the idea that there is beneficial antioxidant activity since samples with co-product D aged significantly more than the samples with lignin. FTIR testing also indicates some antioxidant effects by the reduction in some of the various chemical aging products, which are binder and co-product dependent.

6.2 Research Findings

The addition of lignin-containing co-products to asphalt binder causes significant rheological changes depending upon the binder and the type and amount of co-product added. Each binder tested produced differing results, although general trends were noticed for all binders. The overall trend is a stiffening of the binder. The stiffening occurs at all stages of aging and affects the high, intermediate, and low temperature properties of the binders. The stiffening causes improvement to the high temperature properties with the further addition of co-product. However, the addition of co-product slightly worsens the low temperature properties. The overall effect is a general widening of the performance grade scale. The temperature scale widening is co-product type and amount and asphalt binder source specific.

6.2.1 Asphalt High Temperature Properties

The high temperature properties of the binders are improved with the addition of co-product. The data shows that with all binders the high critical temperature is increased with the further addition of co-product. A larger high critical temperature is beneficial because a binder is less susceptible to rutting. The stiffer the asphalt, the greater the increase in the high critical temperature, and the less vulnerable the binder is to rutting. All binders demonstrated similar behaviors. In general, co-product B provided the greatest change in high critical temperature, while co-products A and C provided significantly less of a change in some of the treatment combinations. With all binders, the larger amounts of co-product provided significantly larger changes than the smaller amounts. Most asphalt binder/co-product blends cause significant improvement over the neat binders. Overall, larger amounts of co-product (especially co-product B) significantly improve a binder’s high temperature properties by increasing the high temperature performance grade.

6.2.2 Asphalt Intermediate Temperature Properties

The intermediate temperature properties are significantly changed with the addition of lignin-containing co-products. The intermediate critical temperature predicts how asphalt will behave at normal operating temperatures. A larger intermediate critical temperature suggests an asphalt is more susceptible to fatigue cracking at cooler temperatures. As with the high temperature properties, a larger intermediate critical temperature indicates a stiffer asphalt binder. So a larger intermediate critical temperature is useful in warmer climates, and detrimental in cooler climates. As with the high temperature properties, the more co-product added, the greater the increase in the intermediate critical temperature. Also, co-product B generally provides the greatest stiffening effect, while co-products A and C provide less of an effect. Larger amounts of added co-product produce significantly different intermediate temperatures than the binders with no co-product; while in general, smaller amounts are not significantly different from the neat binders. Overall, larger amounts of co-product (especially co-product B) stiffen an asphalt binder significantly and change the intermediate temperature properties. Larger amounts provide a significant negative effect to the binder, while some smaller combinations are not significantly different than the neat binders.

6.2.3 Asphalt Low Temperature Properties

The low temperature properties of asphalt binder are generally negatively affected by the addition of lignin-containing co-products. The co-products stiffen the binders, thus making them more susceptible to thermal cracking. The stiffening effect can be seen from the increase in the low critical temperatures. In general, the more co-product added, the greater the increase in the low critical temperature. Co-product B provides a significantly greater effect than co-products A and C with all binders. With some of the binders, lower amounts of co-products A and C do not produce a significantly different response than the neat binders. This same effect is seen with the high and intermediate service temperature properties. Overall, larger amounts of co-product (especially co-product B) negatively affect the low temperature properties of asphalt binder, while most combinations with small amounts of co-product do not necessarily produce significantly changes.

6.2.4 Separation Effects

The lignin-containing co-products have the tendency to separate from the binders when stored at high temperatures for long periods of time. The trends developed from the data show that the longer the binders are heated without being stirred, the more the co-products separate. When the lignin-containing co-products are added to the asphalt, the asphalt conglomerates with the co-product, causing an increase in the density of the blends.

Of all the co-products, co-product A showed the least separation, with co-product B separating the most. Also, the more co-product added, the greater the potential for separation. Each binder also demonstrated differing separation effects, with AAM-1 separating the least, and the local binder separating the most. There are many variables which contribute to these differences, most of which being binder/co-product physical and chemical properties. In general, the more co-products added, and the longer the blends are stored in a hot environment, the more the co-products separate from the binder. The results of the solubility testing show that the lignin-containing co-products are not substantially soluble in asphalt binder.

6.2.5 Oxidation Effects

Testing co-product D (carbohydrate filler with no lignin) suggests that the lignin in co-products A, B, and C provides antioxidant benefit to the intermediate and low temperature properties of the asphalts. The results show that blends with co-product D were significantly different than most other treatment combinations. In many cases, the intermediate and low critical temperatures of co-product D blends were significantly higher than with the other three lignin-containing co-products. The critical temperatures were higher because the co-product D blends oxidized and stiffened more than the other three co-products with lignin.

Co-product D blends underwent more oxidative aging than the other three co-products as co-product D contains no antioxidant. Therefore, it is suggested that the lignin in co-products A, B, and C provide some anti-aging effects.

FTIR testing supports the hypothesis that there is some antioxidant activity of the lignin and the asphalt. The chemical reactions were binder/co-product specific. With binders AAM-1 and AAD-1, the addition of lignin-containing co-product caused a general increase in the carbonyl contents, and a general decrease in the sulfoxide contents. Carbonyl groups and sulfoxide groups are both products of oxidative aging in asphalt binder. With binders AAD-1 and AAM-1, the lignin possibly acted as an antioxidant and caused a decrease in the sulfoxide content. However, the other materials in the co-products (cellulose and hemi-cellulose) may have caused the binders to accelerate aging, or they could be acting as fillers. With binder LB, the opposite occurred, with a general increase in sulfoxide content and a decrease in carbonyl content. In general, the results of the FTIR show chemical interactions between the co-products and binders suggesting antioxidant activity of the lignin-containing co-products.

6.3 Recommendations

The addition of lignin-containing co-products to different asphalt binders can benefit the overall properties of the asphalt depending upon the type and amount of co-product added. Generally, the more co-product added, the greater the stiffening effect. The stiffening effect benefits the high temperature properties, but worsens the low temperature properties.

Due to the research findings, three to nine percent of co-product A, B, or C would provide benefit to the asphalt binders studied. Each binder reacted differently with the different co-products, so each binder has a different optimal combination of co-product. Table 58 illustrates the optimal combinations for each binder and the corresponding total increase in the performance grade range. The effects of the co-products are dependent upon the binder used, so using different binders could create different effects.

Table 58. Optimal co-product combinations for different binders

Binder	Optimum co-product combination	Overall increase in performance grade range (°C)
AAD-1	9% B	2.5
AAM-1	9% C	1.8
LB	6% B	2.2
LPMB	9% C	4.3

Due to the separation effects present when the binder/co-products blends are stored at high temperatures, it is recommended to use a recirculation pump system to keep the binder/co-products in a continuously homogenous blend. Other possible scenarios are to mix the co-products in with the binder just before it is mixed with aggregate during construction, or to mix the co-products simultaneously with the aggregate.

6.4 Final Remarks and Future Work

Even though the co-products stiffen the binders and increase the performance grades, they could provide a great benefit to the industry by simply acting as a renewable alternative for asphalt binder. With the price of oil increasing and the world's energy demand increasing, asphalt binder will start to become more of a commodity than a waste product of the oil refining industry. Since over 500 millions tons of hot-mix asphalt is produced annually (Roberts *et al.* 1996), having a product that could extend the asphalt binder supply would be a great advantage. Also, the lignin-containing products would make asphalt more environmentally friendly, making the pavements "green pavements." This research showed co-products from the ethanol industry could provide benefit to the asphalt industry.

Future research needs to be performed with hot-mix asphalt containing lignin modified binder. Mix design and performance testing will evaluate how the binder behaves when combined with aggregate. Moisture sensitivity testing should be performed on binder/co-product blends. Eventual test sections could be constructed and tested with the optimal mix designs. Future research should also be performed on the best possible method to blend the co-product into the asphalt to ensure separation effects are not as much of a concern.

Other sources of lignin could also provide benefit to asphalt binder. There are other biofuels that produce lignin in large quantities as co-products. Bio-oil and cellulosic ethanol production both produce large amounts of lignin that would readily be available for use in asphalt pavements. Similar research could be performed on these sources of lignin to evaluate their ability to be a successful asphalt binder modifier.

APPENDIX

Table 59. AAD-1 unaged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)
58	12	A	2.221	9	B	2.876	6	B	2.360	3	B	2.150
58	12	A	2.122	9	B	2.776	6	B	1.984	3	B	2.338
58	12	A	2.267	9	B	2.591	6	B	2.370	3	B	2.182
64	12	A	1.076	9	B	1.399	6	B	1.156	3	B	1.035
64	12	A	1.042	9	B	1.334	6	B	0.964	3	B	1.144
64	12	A	1.098	9	B	1.264	6	B	1.161	3	B	1.060
70	12	A	0.557	9	B	0.703	6	B	0.580	3	B	0.527
70	12	A	0.541	9	B	0.675	6	B	0.489	3	B	0.578
70	12	A	0.567	9	B	0.649	6	B	0.591	3	B	0.538
58	12	B	2.968	9	C	2.230	6	C	1.872	3	C	1.735
58	12	B	2.880	9	C	2.110	6	C	1.953	3	C	1.748
58	12	B	3.009	9	C	2.134	6	C	1.937	3	C	1.777
64	12	B	1.473	9	C	1.081	6	C	0.905	3	C	0.842
64	12	B	1.412	9	C	1.022	6	C	0.945	3	C	0.853
64	12	B	1.444	9	C	1.029	6	C	0.939	3	C	0.872
70	12	B	0.740	9	C	0.552	6	C	0.442	3	C	0.432
70	12	B	0.711	9	C	0.523	6	C	0.461	3	C	0.451
70	12	B	0.741	9	C	0.521	6	C	0.435	3	C	0.450
58	12	C	2.089	9	D	2.604	6	D	2.291	3	D	2.183
58	12	C	2.086	9	D	2.662	6	D	2.462	3	D	2.219
58	12	C	2.073	9	D	2.612	6	D	2.417	3	D	2.253
64	12	C	1.037	9	D	1.312	6	D	1.102	3	D	1.057
64	12	C	1.058	9	D	1.331	6	D	1.150	3	D	1.084
64	12	C	1.017	9	D	1.295	6	D	1.214	3	D	1.099
70	12	C	0.544	9	D	0.679	6	D	0.587	3	D	0.549
70	12	C	0.543	9	D	0.768	6	D	0.633	3	D	0.561
70	12	C	0.534	9	D	0.668	6	D	0.622	3	D	0.566
58	9	A	2.322	6	A	2.069	3	A	1.911	0	--	1.648
58	9	A	2.342	6	A	2.010	3	A	1.948	0	--	1.700
58	9	A	2.264	6	A	2.405	3	A	2.024	0	--	1.669
64	9	A	1.051	6	A	1.023	3	A	0.949	0	--	0.798
64	9	A	1.012	6	A	0.997	3	A	0.967	0	--	0.814
64	9	A	1.080	6	A	1.168	3	A	0.991	0	--	0.814
70	9	A	0.514	6	A	0.487	3	A	0.478	0	--	0.399
70	9	A	0.553	6	A	0.512	3	A	0.465	0	--	0.402
70	9	A	0.566	6	A	0.600	3	A	0.454	0	--	0.394

Table 60. AAD-1 RTFO aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)
58	12	A	7.388	9	B	8.695	6	B	7.900	3	B	6.944
58	12	A	7.009	9	B	8.556	6	B	7.918	3	B	7.105
58	12	A	7.262	9	B	8.477	6	B	8.350	3	B	6.547
64	12	A	3.555	9	B	4.164	6	B	3.659	3	B	3.322
64	12	A	3.344	9	B	4.097	6	B	3.764	3	B	3.404
64	12	A	3.492	9	B	4.040	6	B	3.974	3	B	3.125
70	12	A	1.765	9	B	2.059	6	B	1.819	3	B	1.671
70	12	A	1.649	9	B	2.059	6	B	1.875	3	B	1.707
70	12	A	1.743	9	B	1.999	6	B	1.950	3	B	1.610
58	12	B	8.405	9	C	6.798	6	C	5.854	3	C	5.620
58	12	B	8.443	9	C	6.304	6	C	5.737	3	C	5.421
58	12	B	9.054	9	C	6.505	6	C	6.128	3	C	5.511
64	12	B	4.420	9	C	3.242	6	C	2.805	3	C	2.712
64	12	B	4.346	9	C	3.013	6	C	2.745	3	C	2.543
64	12	B	4.328	9	C	3.109	6	C	2.921	3	C	2.602
70	12	B	2.149	9	C	1.587	6	C	1.393	3	C	1.421
70	12	B	2.412	9	C	1.495	6	C	1.357	3	C	1.205
70	12	B	2.132	9	C	1.540	6	C	1.436	3	C	1.314
58	12	C	6.027	9	D	6.916	6	D	5.505	3	D	5.340
58	12	C	6.290	9	D	6.678	6	D	4.201	3	D	7.316
58	12	C	6.222	9	D	6.742	6	D	5.330	3	D	7.235
64	12	C	3.341	9	D	3.118	6	D	2.552	3	D	2.479
64	12	C	3.213	9	D	3.169	6	D	1.980	3	D	3.368
64	12	C	2.984	9	D	3.210	6	D	2.485	3	D	3.387
70	12	C	1.648	9	D	1.572	6	D	1.226	3	D	1.207
70	12	C	1.623	9	D	1.584	6	D	0.980	3	D	1.664
70	12	C	1.512	9	D	1.601	6	D	1.262	3	D	1.706
58	9	A	6.542	6	A	6.235	3	A	5.592	0	--	5.475
58	9	A	6.729	6	A	6.240	3	A	5.644	0	--	5.281
58	9	A	6.839	6	A	6.586	3	A	5.658	0	--	5.238
64	9	A	3.216	6	A	3.008	3	A	2.682	0	--	2.635
64	9	A	3.281	6	A	3.013	3	A	2.629	0	--	2.556
64	9	A	3.350	6	A	3.188	3	A	2.741	0	--	2.514
70	9	A	1.608	6	A	1.507	3	A	1.341	0	--	1.330
70	9	A	1.649	6	A	1.515	3	A	1.324	0	--	1.226
70	9	A	1.698	6	A	1.604	3	A	1.370	0	--	1.270

Table 61. AAD-1 PAV aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Co-product (%)	Co-product (type)	G*sin(δ)	Co-product (%)	Co-product (type)	G*sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)
16	12	A	7321	9	B	7340	6	B	7275	3	B	6179
16	12	A	7245	9	B	7414	6	B	6768	3	B	6808
16	12	A	7031	9	B	7199	6	B	6927	3	B	6654
19	12	A	4099	9	B	5044	6	B	4992	3	B	4235
19	12	A	4456	9	B	5123	6	B	4627	3	B	4723
19	12	A	4256	9	B	5021	6	B	4810	3	B	4585
22	12	A	3738	9	B	3401	6	B	3367	3	B	2865
22	12	A	3335	9	B	3502	6	B	3122	3	B	3171
22	12	A	2760	9	B	3359	6	B	3288	3	B	3082
16	12	B	7825	9	C	6854	6	C	6927	3	C	7138
16	12	B	8117	9	C	6873	6	C	6874	3	C	6215
16	12	B	7814	9	C	6541	6	C	6674	3	C	6124
19	12	B	5448	9	C	4948	6	C	4402	3	C	4206
19	12	B	5658	9	C	4751	6	C	4561	3	C	4472
19	12	B	5228	9	C	4561	6	C	4120	3	C	3653
22	12	B	3722	9	C	2849	6	C	2288	3	C	2986
22	12	B	3864	9	C	3029	6	C	2824	3	C	2752
22	12	B	3558	9	C	2811	6	C	3149	3	C	2756
16	12	C	7012	9	D	2713	6	D	2555	3	D	2277
16	12	C	7129	9	D	2823	6	D	2429	3	D	2428
16	12	C	7288	9	D	2802	6	D	2491	3	D	2527
19	12	C	4810	9	D	3958	6	D	3749	3	D	3323
19	12	C	4862	9	D	4136	6	D	3600	3	D	3562
19	12	C	4623	9	D	4146	6	D	3598	3	D	3723
22	12	C	2849	9	D	5743	6	D	5354	3	D	4824
22	12	C	3029	9	D	6001	6	D	5310	3	D	5189
22	12	C	2811	9	D	5983	6	D	5356	3	D	5423
16	9	A	7149	6	A	6600	3	A	5866	0	--	5851
16	9	A	7024	6	A	6855	3	A	5882	0	--	6212
16	9	A	6925	6	A	6466	3	A	5798	0	--	5722
19	9	A	4883	6	A	4479	3	A	3998	0	--	3981
19	9	A	4173	6	A	4125	3	A	4004	0	--	4224
19	9	A	3879	6	A	3964	3	A	4114	0	--	3924
22	9	A	3274	6	A	2981	3	A	2671	0	--	2786
22	9	A	2796	6	A	2787	3	A	2693	0	--	2827
22	9	A	2618	6	A	2920	3	A	2992	0	--	2631

Table 62. AAD-1 PAV aged BBR data

Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value
-12	12	A	131.0	0.309	9	B	113.0	0.321	6	B	126.0	0.301
-12	12	A	142.0	0.323	9	B	118.0	0.307	6	B	136.0	0.309
-12	12	A	135.0	0.315	9	B	145.0	0.321	6	B	124.0	0.305
-18	12	A	267.0	0.282	9	B	292.0	0.244	6	B	266.0	0.245
-18	12	A	234.0	0.253	9	B	252.0	0.253	6	B	291.0	0.255
-18	12	A	256.0	0.271	9	B	324.0	0.252	6	B	272.0	0.264
-12	12	B	145.0	0.321	9	C	243.0	0.237	6	C	272.0	0.228
-12	12	B	135.0	0.312	9	C	266.0	0.253	6	C	216.0	0.260
-12	12	B	141.0	0.302	9	C	249.0	0.254	6	C	243.0	0.246
-18	12	B	294.0	0.248	9	C	138.0	0.325	6	C	123.0	0.319
-18	12	B	289.0	0.255	9	C	133.0	0.320	6	C	127.0	0.337
-18	12	B	262.0	0.247	9	C	134.0	0.318	6	C	113.0	0.321
-18	12	C	208.0	0.250	9	D	73.4	0.358	6	D	69.2	0.374
-18	12	C	241.0	0.270	9	D	78.5	0.390	6	D	65.3	0.375
-18	12	C	223.0	0.256	9	D	79.7	0.374	6	D	66.3	0.388
-12	12	C	148.0	0.315	9	D	161.0	0.301	6	D	174.0	0.313
-12	12	C	135.0	0.309	9	D	181.0	0.298	6	D	161.0	0.304
-12	12	C	142.0	0.312	9	D	176.0	0.304	6	D	140.0	0.298
-12	9	A	141.0	0.318	6	A	118.0	0.327	3	A	120.0	0.307
-12	9	A	144.0	0.330	6	A	134.0	0.311	3	A	118.0	0.319
-12	9	A	124.0	0.307	6	A	114.0	0.308	3	A	125.0	0.320
-18	9	A	222.0	0.249	6	A	242.0	0.266	3	A	231.0	0.259
-18	9	A	255.0	0.255	6	A	235.0	0.272	3	A	237.0	0.262
-18	9	A	243.0	0.260	6	A	268.0	0.271	3	A	234.0	0.265
-12	3	B	125.0	0.299	3	C	240.0	0.261	3	D	60.9	0.386
-12	3	B	118.0	0.319	3	C	240.0	0.270	3	D	63.1	0.368
-12	3	B	125.0	0.320	3	C	231.0	0.250	3	D	51.2	0.368
-18	3	B	235.0	0.255	3	C	115.0	0.329	3	D	147.0	0.328
-18	3	B	240.0	0.251	3	C	117.0	0.315	3	D	146.0	0.304
-18	3	B	237.0	0.264	3	C	125.0	0.328	3	D	132.0	0.309
-12	0	--	102.0	0.335	0	--	106.0	0.338	0	--	108.0	0.334
-18	0	--	214.0	0.272	0	--	200.0	0.265	0	--	196.0	0.268

Table 63. AAM-1 unaged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)
58	9	A	3.773	58	6	A	3.351	58	3	A	3.742
58	9	A	3.779	58	6	A	3.783	58	3	A	3.945
58	9	A	3.842	58	6	A	4.044	58	3	A	3.583
64	9	A	1.770	64	6	A	1.561	64	3	A	1.673
64	9	A	1.729	64	6	A	1.721	64	3	A	1.758
64	9	A	1.720	64	6	A	1.879	64	3	A	1.642
70	9	A	0.861	70	6	A	0.776	70	3	A	0.855
70	9	A	0.853	70	6	A	0.866	70	3	A	0.884
70	9	A	0.892	70	6	A	0.933	70	3	A	0.800
58	9	B	4.323	58	6	B	4.044	58	3	B	3.583
58	9	B	4.368	58	6	B	4.089	58	3	B	3.920
58	9	B	4.348	58	6	B	4.055	58	3	B	3.875
64	9	B	1.986	64	6	B	1.879	64	3	B	1.642
64	9	B	1.989	64	6	B	1.886	64	3	B	1.832
64	9	B	2.017	64	6	B	1.877	64	3	B	1.766
70	9	B	0.987	70	6	B	0.933	70	3	B	0.800
70	9	B	0.980	70	6	B	0.939	70	3	B	0.846
70	9	B	1.000	70	6	B	0.915	70	3	B	0.866
58	9	C	3.708	58	6	C	3.768	58	3	C	3.485
58	9	C	3.532	58	6	C	3.916	58	3	C	3.430
58	9	C	3.930	58	6	C	3.787	58	3	C	3.478
64	9	C	1.710	64	6	C	1.772	64	3	C	1.631
64	9	C	1.650	64	6	C	1.822	64	3	C	1.585
64	9	C	1.838	64	6	C	1.758	64	3	C	1.580
70	9	C	0.860	70	6	C	0.844	70	3	C	0.814
70	9	C	0.867	70	6	C	0.883	70	3	C	0.800
70	9	C	0.942	70	6	C	0.898	70	3	C	0.785
58	9	D	5.406	58	6	D	3.951	58	3	D	3.870
58	9	D	5.394	58	6	D	4.341	58	3	D	3.863
58	9	D	5.379	58	6	D	4.367	58	3	D	3.957
64	9	D	2.456	64	6	D	1.409	64	3	D	1.894
64	9	D	2.405	64	6	D	2.049	64	3	D	1.813
64	9	D	2.462	64	6	D	1.972	64	3	D	1.837
70	9	D	1.262	70	6	D	0.710	70	3	D	0.985
70	9	D	1.243	70	6	D	1.024	70	3	D	0.904
70	9	D	1.258	70	6	D	0.996	70	3	D	0.900
58	0	--	3.376	64	0	--	1.540	70	0	--	0.766
58	0	--	3.405	64	0	--	1.568	70	0	--	0.770
58	0	--	3.418	64	0	--	1.554	70	0	--	0.771

Table 64. AAM-1 RTFO aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Lignin (%)	Lignin (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)
58	9	A	7.911	58	6	A	7.755	58	3	A	7.408
58	9	A	7.662	58	6	A	7.461	58	3	A	7.096
58	9	A	7.745	58	6	A	7.206	58	3	A	7.521
64	9	A	3.419	64	6	A	3.337	64	3	A	3.200
64	9	A	3.462	64	6	A	3.337	64	3	A	3.118
64	9	A	3.376	64	6	A	3.947	64	3	A	3.315
70	9	A	1.639	70	6	A	1.792	70	3	A	1.489
70	9	A	1.632	70	6	A	1.815	70	3	A	1.458
70	9	A	1.599	70	6	A	1.833	70	3	A	1.465
58	9	B	8.438	58	6	B	7.858	58	3	B	7.718
58	9	B	7.797	58	6	B	6.916	58	3	B	7.241
58	9	B	7.792	58	6	B	7.339	58	3	B	7.994
64	9	B	3.563	64	6	B	3.326	64	3	B	3.285
64	9	B	3.112	64	6	B	2.962	64	3	B	3.006
64	9	B	3.303	64	6	B	3.155	64	3	B	3.030
70	9	B	1.649	70	6	B	1.536	70	3	B	1.542
70	9	B	1.436	70	6	B	1.386	70	3	B	1.415
70	9	B	1.499	70	6	B	1.491	70	3	B	1.419
58	9	C	9.054	58	6	C	8.152	58	3	C	8.011
58	9	C	9.032	58	6	C	8.136	58	3	C	7.837
58	9	C	9.196	58	6	C	7.593	58	3	C	7.245
64	9	C	3.938	64	6	C	3.407	64	3	C	3.450
64	9	C	3.994	64	6	C	3.453	64	3	C	2.967
64	9	C	3.947	64	6	C	3.250	64	3	C	3.366
70	9	C	1.772	70	6	C	1.536	70	3	C	1.638
70	9	C	1.815	70	6	C	1.836	70	3	C	1.387
70	9	C	1.833	70	6	C	1.491	70	3	C	1.465
58	9	D	9.987	58	6	D	8.543	58	3	D	7.989
58	9	D	9.727	58	6	D	8.559	58	3	D	7.864
58	9	D	8.833	58	6	D	7.877	58	3	D	7.869
64	9	D	4.538	64	6	D	3.786	64	3	D	3.479
64	9	D	4.237	64	6	D	3.710	64	3	D	3.549
64	9	D	3.764	64	6	D	3.522	64	3	D	3.496
70	9	D	2.191	70	6	D	1.786	70	3	D	1.608
70	9	D	2.071	70	6	D	1.790	70	3	D	1.631
70	9	D	1.829	70	6	D	1.673	70	3	D	1.653
58	0	--	7.408	64	0	--	3.154	70	0	--	1.354
58	0	--	7.127	64	0	--	3.054	70	0	--	1.354
58	0	--	7.328	64	0	--	3.246	70	0	--	1.497

Table 65. AAM-1 PAV aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)
22	9	A	5536	22	6	A	3102	22	3	A	3152
22	9	A	5235	22	6	A	2497	22	3	A	3366
22	9	A	5128	22	6	A	3264	22	3	A	2968
25	9	A	3001	25	6	A	2131	25	3	A	2181
25	9	A	3703	25	6	A	1719	25	3	A	2324
25	9	A	3685	25	6	A	2563	25	3	A	2051
28	9	A	2825	28	6	A	1437	28	3	A	1475
28	9	A	2561	28	6	A	1157	28	3	A	1572
28	9	A	2596	28	6	A	2218	28	3	A	1392
22	9	B	5279	22	6	B	5014	22	3	B	4069
22	9	B	5409	22	6	B	5021	22	3	B	4326
22	9	B	5709	22	6	B	4765	22	3	B	4447
25	9	B	3736	25	6	B	3630	25	3	B	2853
25	9	B	3911	25	6	B	3625	25	3	B	3053
25	9	B	4125	25	6	B	3372	25	3	B	3165
28	9	B	2570	28	6	B	2586	28	3	B	2280
28	9	B	2757	28	6	B	2556	28	3	B	2179
28	9	B	2905	28	6	B	2260	28	3	B	2245
22	9	C	4476	22	6	C	4910	22	3	C	4595
22	9	C	4621	22	6	C	5193	22	3	C	4268
22	9	C	4715	22	6	C	5002	22	3	C	4360
25	9	C	3117	25	6	C	3484	25	3	C	3280
25	9	C	3289	25	6	C	3734	25	3	C	3078
25	9	C	3395	25	6	C	3590	25	3	C	3151
28	9	C	2103	28	6	C	2834	28	3	C	2280
28	9	C	2236	28	6	C	2619	28	3	C	2179
28	9	C	2395	28	6	C	2508	28	3	C	2245
22	9	D	5967	22	6	D	5838	22	3	D	5484
22	9	D	6491	22	6	D	6422	22	3	D	5642
22	9	D	5382	22	6	D	6458	22	3	D	5512
25	9	D	4439	25	6	D	4375	25	3	D	4199
25	9	D	5041	25	6	D	4933	25	3	D	4267
25	9	D	3964	25	6	D	4955	25	3	D	3979
28	9	D	3231	28	6	D	3192	28	3	D	3172
28	9	D	3921	28	6	D	3796	28	3	D	3364
28	9	D	2895	28	6	D	3830	28	3	D	3029
22	0	--	4037	25	0	--	2910	28	0	--	2067
22	0	--	4117	25	0	--	3055	28	0	--	2255
22	0	--	4274	25	0	--	3101	28	0	--	2213

Table 66. AAM-1 PAV aged BBR data

Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value	Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value
-6	9	A	107.0	0.276	-6	6	C	110.0	0.277
-6	9	A	108.0	0.283	-6	6	C	116.0	0.272
-6	9	A	112.0	0.282	-6	6	C	105.0	0.282
-3	9	A	50.4	0.341	-3	6	C	54.1	0.330
-3	9	A	52.9	0.332	-3	6	C	54.0	0.333
-3	9	A	50.5	0.333	-3	6	C	52.0	0.326
-6	9	B	142.0	0.276	-6	6	D	113.0	0.253
-6	9	B	112.0	0.277	-6	6	D	150.0	0.243
-6	9	B	108.0	0.294	-6	6	D	146.0	0.244
-3	9	B	68.0	0.321	-3	6	D	64.2	0.305
-3	9	B	55.6	0.322	-3	6	D	64.9	0.301
-3	9	B	59.5	0.334	-3	6	D	58.9	0.301
-6	9	C	101.0	0.287	-6	3	A	114.0	0.278
-6	9	C	107.0	0.294	-6	3	A	101.0	0.281
-6	9	C	100.0	0.276	-6	3	A	105.0	0.283
-3	9	C	52.1	0.337	-3	3	A	51.7	0.320
-3	9	C	55.0	0.335	-3	3	A	45.8	0.299
-3	9	C	51.2	0.343	-3	3	A	52.9	0.320
-6	9	D	171.0	0.231	-6	3	B	93.1	0.277
-6	9	D	167.0	0.240	-6	3	B	109.0	0.287
-6	9	D	161.0	0.223	-6	3	B	107.0	0.283
-3	9	D	72.3	0.299	-3	3	B	48.6	0.329
-3	9	D	71.5	0.288	-3	3	B	48.7	0.324
-3	9	D	71.3	0.296	-3	3	B	52.4	0.331
-6	6	A	77.4	0.305	-6	3	C	86.5	0.289
-6	6	A	81.6	0.307	-6	3	C	86.6	0.290
-6	6	A	87.2	0.289	-6	3	C	80.6	0.280
-3	6	A	44.0	0.347	-3	3	C	46.3	0.346
-3	6	A	44.0	0.347	-3	3	C	48.3	0.331
-3	6	A	46.4	0.335	-3	3	C	47.2	0.335
-6	6	B	100.0	0.283	-6	3	D	134.0	0.253
-6	6	B	94.3	0.278	-6	3	D	119.0	0.247
-6	6	B	108.0	0.283	-6	3	D	119.0	0.244
-3	6	B	53.0	0.333	-3	3	D	50.8	0.309
-3	6	B	45.9	0.337	-3	3	D	47.6	0.318
-3	6	B	50.7	0.324	-3	3	D	50.3	0.297
-3	0	--	46.0	0.336	-6	0	--	79.8	0.285
-3	0	--	45.8	0.343	-6	0	--	77.0	0.285
-3	0	--	46.2	0.327	-6	0	--	82.6	0.283

Table 67. LB Unaged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)
64	9	A	2.059	6	C	2.215	76	9	C	0.608	3	B	0.555
64	9	A	2.254	6	C	2.312	76	9	C	0.606	3	B	0.587
64	9	A	2.101	6	C	2.461	76	9	C	0.618	3	B	0.543
70	9	A	1.001	6	C	1.064	64	6	A	2.039	3	C	2.055
70	9	A	1.069	6	C	1.102	64	6	A	2.053	3	C	2.250
70	9	A	1.020	6	C	1.201	64	6	A	2.158	3	C	2.359
76	9	A	0.499	6	C	0.486	70	6	A	0.972	3	C	1.000
76	9	A	0.555	6	C	0.486	70	6	A	0.968	3	C	1.078
76	9	A	0.524	6	C	0.490	70	6	A	0.968	3	C	1.126
64	9	B	2.518	3	A	1.837	76	6	A	0.499	3	C	0.514
64	9	B	2.689	3	A	1.893	76	6	A	0.495	3	C	0.546
64	9	B	2.671	3	A	1.982	76	6	A	0.432	3	C	0.579
70	9	B	1.218	3	A	0.893	64	6	B	2.554	0	--	1.628
70	9	B	1.286	3	A	0.913	64	6	B	2.620	0	--	1.639
70	9	B	1.285	3	A	0.954	64	6	B	2.627	0	--	1.681
76	9	B	0.621	3	A	0.452	70	6	B	1.222	0	--	0.987
76	9	B	0.667	3	A	0.453	70	6	B	1.208	0	--	1.023
76	9	B	0.658	3	A	0.435	70	6	B	1.258	0	--	0.965
64	9	C	2.509	3	B	2.291	76	6	B	0.612	0	--	0.405
64	9	C	2.473	3	B	2.430	76	6	B	0.606	0	--	0.409
64	9	C	2.468	3	B	2.280	76	6	B	0.630	0	--	0.421
70	9	C	1.197	3	B	1.099							
70	9	C	1.203	3	B	1.165							
70	9	C	1.205	3	B	1.081							

Table 68. LB RTFO aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Co-product (%)	Co-product (type)	G*/sin(δ)
64	9	A	6.125	6	C	6.023	76	9	C	1.360	3	B	1.372
64	9	A	6.131	6	C	5.997	76	9	C	1.352	3	B	1.221
64	9	A	5.941	6	C	6.321	76	9	C	1.487	3	B	1.372
70	9	A	2.764	6	C	2.643	64	6	A	5.984	3	C	5.994
70	9	A	2.846	6	C	2.791	64	6	A	5.914	3	C	5.784
70	9	A	2.533	6	C	2.794	64	6	A	5.877	3	C	5.987
76	9	A	1.305	6	C	1.318	70	6	A	2.665	3	C	2.612
76	9	A	1.431	6	C	1.305	70	6	A	2.741	3	C	2.546
76	9	A	1.291	6	C	1.215	70	6	A	2.546	3	C	2.564
64	9	B	6.531	3	A	5.867	76	6	A	1.190	3	C	1.221
64	9	B	6.233	3	A	5.867	76	6	A	1.217	3	C	1.231
64	9	B	6.459	3	A	5.608	76	6	A	1.162	3	C	1.195
70	9	B	2.931	3	A	2.632	64	6	B	6.223	0	--	5.897
70	9	B	2.861	3	A	2.632	64	6	B	6.379	0	--	5.713
70	9	B	2.988	3	A	2.587	64	6	B	6.553	0	--	5.994
76	9	B	1.418	3	A	1.306	70	6	B	2.872	0	--	2.665
76	9	B	1.397	3	A	1.306	70	6	B	2.930	0	--	2.541
76	9	B	1.405	3	A	1.256	70	6	B	3.042	0	--	2.549
64	9	C	6.127	3	B	5.995	76	6	B	1.374	0	--	1.115
64	9	C	6.092	3	B	5.865	76	6	B	1.440	0	--	1.096
64	9	C	6.637	3	B	6.263	76	6	B	1.471	0	--	1.121
70	9	C	2.795	3	B	2.815							
70	9	C	2.830	3	B	2.543							
70	9	C	3.091	3	B	2.894							

Table 69. LB PAV aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Co-product (%)	Co-product (type)	G*sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Co-product (%)	Co-product (type)	G*sin(δ)
22	9	A	6354	6	C	6512	28	9	C	3045	3	B	3188
22	9	A	6171	6	C	6439	28	9	C	3267	3	B	3200
22	9	A	6289	6	C	6520	28	9	C	3123	3	B	3047
25	9	A	4496	6	C	4216	22	6	A	6021	3	C	6355
25	9	A	4354	6	C	4635	22	6	A	6001	3	C	6411
25	9	A	4420	6	C	4542	22	6	A	6031	3	C	6287
28	9	A	3073	6	C	3145	25	6	A	4215	3	C	4321
28	9	A	3004	6	C	3211	25	6	A	4536	3	C	4400
28	9	A	3213	6	C	3192	25	6	A	4726	3	C	4296
22	9	B	6801	3	A	6038	28	6	A	2922	3	C	3012
22	9	B	6770	3	A	6158	28	6	A	3106	3	C	3101
22	9	B	6838	3	A	6194	28	6	A	3278	3	C	3192
25	9	B	4773	3	A	4216	22	6	B	6872	0	--	6151
25	9	B	4804	3	A	4345	22	6	B	6872	0	--	6026
25	9	B	4855	3	A	4311	22	6	B	6142	0	--	5948
28	9	B	3307	3	A	2916	25	6	B	4875	0	--	4165
28	9	B	3310	3	A	2993	25	6	B	4536	0	--	4025
28	9	B	3359	3	A	3008	25	6	B	4536	0	--	4116
22	9	C	6521	3	B	6656	28	6	B	3343	0	--	2922
22	9	C	6411	3	B	6614	28	6	B	3106	0	--	2846
22	9	C	6670	3	B	6303	28	6	B	3106	0	--	2640
25	9	C	4366	3	B	4580							
25	9	C	4701	3	B	4651							
25	9	C	4623	3	B	4451							

Table 70. LB PAV aged BBR data

Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value	Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value
-6	9	A	108.0	0.308	-6	6	C	113.0	0.312
-6	9	A	98.8	0.304	-6	6	C	100.0	0.349
-6	9	A	114.0	0.310	-6	6	C	111.0	0.334
-12	9	A	206.0	0.244	-12	6	C	257.0	0.225
-12	9	A	220.0	0.243	-12	6	C	228.0	0.229
-12	9	A	207.0	0.241	-12	6	C	236.0	0.229
-6	9	B	109.0	0.302	-6	3	A	93.7	0.308
-6	9	B	95.0	0.323	-6	3	A	99.7	0.335
-6	9	B	119.0	0.304	-6	3	A	91.0	0.312
-12	9	B	248.0	0.281	-12	3	A	181.0	0.271
-12	9	B	260.0	0.217	-12	3	A	176.0	0.272
-12	9	B	209.0	0.241	-12	3	A	187.0	0.269
-6	9	C	109.0	0.333	-6	3	B	113.0	0.323
-6	9	C	89.8	0.302	-6	3	B	106.0	0.315
-6	9	C	110.0	0.329	-6	3	B	109.0	0.308
-12	9	C	234.0	0.231	-12	3	B	207.0	0.269
-12	9	C	234.0	0.228	-12	3	B	203.0	0.250
-12	9	C	225.0	0.220	-12	3	B	173.0	0.282
-6	6	A	104.0	0.323	-6	3	C	105.0	0.327
-6	6	A	102.0	0.328	-6	3	C	103.0	0.327
-6	6	A	109.0	0.323	-6	3	C	98.9	0.322
-12	6	A	198.0	0.264	-12	3	C	257.0	0.216
-12	6	A	192.0	0.269	-12	3	C	217.0	0.218
-12	6	A	204.0	0.242	-12	3	C	223.0	0.242
-6	6	B	108.0	0.322	-6	0	--	95.0	0.335
-6	6	B	106.0	0.329	-6	0	--	96.1	0.332
-6	6	B	109.0	0.338	-6	0	--	88.0	0.344
-12	6	B	226.0	0.232	-12	0	--	185.0	0.281
-12	6	B	220.0	0.229	-12	0	--	176.0	0.276
-12	6	B	221.0	0.218	-12	0	--	170.0	0.264

Table 71. LPMB unaged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)
58	12	A	4.807	58	9	B	3.256	58	6	C	2.706
58	12	A	4.587	58	9	B	3.334	58	6	C	2.829
58	12	A	4.687	58	9	B	3.412	58	6	C	2.802
64	12	A	1.600	64	9	B	1.703	64	6	C	1.348
64	12	A	1.756	64	9	B	1.610	64	6	C	1.417
64	12	A	1.546	64	9	B	1.638	64	6	C	1.399
70	12	A	0.713	70	9	B	0.902	70	6	C	0.717
70	12	A	0.751	70	9	B	0.836	70	6	C	0.740
70	12	A	0.735	70	9	B	0.872	70	6	C	0.721
58	12	B	4.412	58	9	C	2.500	58	3	A	2.554
58	12	B	4.885	58	9	C	2.584	58	3	A	2.517
58	12	B	4.454	58	9	C	2.390	58	3	A	2.424
64	12	B	1.691	64	9	C	1.402	64	3	A	1.294
64	12	B	1.679	64	9	C	1.426	64	3	A	1.271
64	12	B	1.833	64	9	C	1.410	64	3	A	1.182
70	12	B	0.916	70	9	C	0.680	70	3	A	0.684
70	12	B	0.894	70	9	C	0.707	70	3	A	0.672
70	12	B	0.872	70	9	C	0.655	70	3	A	0.613
58	12	C	4.264	58	6	A	2.408	58	3	B	2.617
58	12	C	4.364	58	6	A	2.550	58	3	B	2.572
58	12	C	4.220	58	6	A	2.712	58	3	B	2.670
64	12	C	1.451	64	6	A	1.231	64	3	B	1.348
64	12	C	1.511	64	6	A	1.284	64	3	B	1.319
64	12	C	1.424	64	6	A	1.350	64	3	B	1.303
70	12	C	0.704	70	6	A	0.646	70	3	B	0.714
70	12	C	0.710	70	6	A	0.677	70	3	B	0.697
70	12	C	0.691	70	6	A	0.709	70	3	B	0.689
58	9	A	2.641	58	6	B	2.875	58	3	C	2.396
58	9	A	2.699	58	6	B	2.726	58	3	C	2.425
58	9	A	2.664	58	6	B	2.881	58	3	C	2.378
64	9	A	1.341	64	6	B	1.442	64	3	C	1.209
64	9	A	1.353	64	6	B	1.420	64	3	C	1.214
64	9	A	1.320	64	6	B	1.409	64	3	C	1.218
70	9	A	0.707	70	6	B	0.752	70	3	C	0.629
70	9	A	0.698	70	6	B	0.752	70	3	C	0.631
70	9	A	0.698	70	6	B	0.743	70	3	C	0.630
58	0	--	2.451	64	0	--	1.215	70	0	--	0.603
58	0	--	2.346	64	0	--	1.194	70	0	--	0.751
58	0	--	2.402	64	0	--	1.115	70	0	--	0.645

Table 72. LPMB RTFO aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*/sin(δ)
58	12	A	12.131	58	9	B	8.368	58	6	C	7.488
58	12	A	11.664	58	9	B	8.608	58	6	C	7.524
58	12	A	11.651	58	9	B	7.987	58	6	C	7.484
64	12	A	6.027	64	9	B	4.032	64	6	C	3.627
64	12	A	5.832	64	9	B	4.168	64	6	C	3.710
64	12	A	5.580	64	9	B	3.951	64	6	C	3.617
70	12	A	2.812	70	9	B	1.993	70	6	C	1.846
70	12	A	2.676	70	9	B	2.102	70	6	C	1.841
70	12	A	2.685	70	9	B	1.993	70	6	C	1.813
58	12	B	12.754	58	9	C	8.451	58	3	A	7.681
58	12	B	12.952	58	9	C	8.541	58	3	A	7.628
58	12	B	12.812	58	9	C	8.230	58	3	A	7.716
64	12	B	6.477	64	9	C	3.985	64	3	A	2.990
64	12	B	6.479	64	9	C	4.231	64	3	A	3.757
64	12	B	6.405	64	9	C	4.021	64	3	A	2.645
70	12	B	2.990	70	9	C	2.003	70	3	A	1.543
70	12	B	2.910	70	9	C	2.159	70	3	A	1.312
70	12	B	2.927	70	9	C	1.834	70	3	A	1.112
58	12	C	11.212	58	6	A	7.657	58	3	B	7.382
58	12	C	10.986	58	6	A	6.120	58	3	B	7.365
58	12	C	10.784	58	6	A	6.149	58	3	B	7.127
64	12	C	5.607	64	6	A	3.180	64	3	B	3.552
64	12	C	5.484	64	6	A	2.955	64	3	B	3.554
64	12	C	5.334	64	6	A	2.985	64	3	B	3.462
70	12	C	2.606	70	6	A	1.611	70	3	B	1.645
70	12	C	2.474	70	6	A	1.451	70	3	B	1.233
70	12	C	2.460	70	6	A	1.494	70	3	B	1.560
58	9	A	7.818	58	6	B	7.571	58	3	C	8.592
58	9	A	6.746	58	6	B	7.856	58	3	C	7.074
58	9	A	6.965	58	6	B	7.700	58	3	C	7.536
64	9	A	3.305	64	6	B	4.126	64	3	C	3.364
64	9	A	3.297	64	6	B	3.978	64	3	C	3.473
64	9	A	3.360	64	6	B	3.717	64	3	C	3.648
70	9	A	1.680	70	6	B	1.816	70	3	C	1.645
70	9	A	1.672	70	6	B	1.451	70	3	C	1.497
70	9	A	1.714	70	6	B	1.883	70	3	C	1.461
58	0	--	7.345	64	0	--	2.951	70	0	--	1.345
58	0	--	7.561	64	0	--	3.125	70	0	--	1.465
58	0	--	7.446	64	0	--	3.215	70	0	--	1.482

Table 73. LPMB PAV aged DSR data

Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)	Temp (°C)	Co-product (%)	Co-product (type)	G*sin(δ)
19	12	A	9502	19	9	B	6382	19	6	C	5453
19	12	A	8523	19	9	B	6691	19	6	C	5528
19	12	A	8751	19	9	B	6611	19	6	C	6007
22	12	A	4912	22	9	B	4951	22	6	C	3940
22	12	A	4866	22	9	B	4994	22	6	C	3947
22	12	A	4938	22	9	B	5211	22	6	C	4290
25	12	A	3650	25	9	B	3541	25	6	C	2754
25	12	A	3450	25	9	B	3464	25	6	C	2742
25	12	A	3386	25	9	B	3661	25	6	C	3009
19	12	B	8954	19	9	C	5767	19	3	A	6198
19	12	B	8512	19	9	C	5606	19	3	A	5581
19	12	B	8233	19	9	C	5788	19	3	A	5876
22	12	B	5332	22	9	C	4067	22	3	A	3725
22	12	B	5267	22	9	C	3971	22	3	A	3884
22	12	B	5009	22	9	C	4199	22	3	A	4132
25	12	B	3929	25	9	C	2802	25	3	A	2876
25	12	B	3860	25	9	C	2744	25	3	A	2644
25	12	B	3656	25	9	C	2848	25	3	A	2834
19	12	C	7512	19	6	A	5720	19	3	B	7233
19	12	C	7964	19	6	A	6072	19	3	B	7122
19	12	C	8245	19	6	A	5484	19	3	B	7304
22	12	C	5058	22	6	A	4039	22	3	B	4822
22	12	C	4315	22	6	A	4315	22	3	B	4791
22	12	C	5153	22	6	A	3844	22	3	B	4912
25	12	C	3650	25	6	A	2771	25	3	B	3451
25	12	C	3449	25	6	A	2981	25	3	B	3352
25	12	C	3768	25	6	A	2638	25	3	B	3516
19	9	A	6051	19	6	B	7153	19	3	C	5547
19	9	A	5348	19	6	B	7031	19	3	C	5695
19	9	A	4818	19	6	B	7125	19	3	C	6102
22	9	A	4215	22	6	B	5027	22	3	C	4125
22	9	A	4561	22	6	B	4890	22	3	C	4025
22	9	A	3964	22	6	B	4988	22	3	C	3745
25	9	A	2891	25	6	B	3385	25	3	C	2850
25	9	A	3151	25	6	B	3352	25	3	C	2741
25	9	A	2971	25	6	B	3414	25	3	C	2994
19	0	--	5751	22	0	--	4098	25	0	--	2934
19	0	--	5760	22	0	--	3733	25	0	--	2946
19	0	--	5706	22	0	--	3824	25	0	--	2901

Table 74. LPMB PAV aged BBR data

Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value	Temp (°C)	Co-product (%)	Co-product (type)	Stiffness (kPa)	m-value
-6	12	A	78.3	0.352	-6	6	A	73.8	0.338
-6	12	A	73.9	0.339	-6	6	A	76.2	0.335
-6	12	A	101.0	0.343	-6	6	A	69.2	0.352
-12	12	A	106.0	0.263	-12	6	A	143.0	0.291
-12	12	A	133.0	0.270	-12	6	A	128.0	0.287
-12	12	A	204.0	0.282	-12	6	A	140.0	0.297
-6	12	B	71.3	0.337	-6	6	B	80.7	0.341
-6	12	B	84.4	0.347	-6	6	B	62.9	0.339
-6	12	B	76.2	0.337	-6	6	B	55.5	0.340
-12	12	B	117.0	0.260	-12	6	B	148.0	0.277
-12	12	B	179.0	0.260	-12	6	B	141.0	0.278
-12	12	B	151.0	0.265	-12	6	B	101.0	0.276
-6	12	C	81.5	0.336	-6	6	C	61.9	0.339
-6	12	C	95.3	0.334	-6	6	C	81.3	0.333
-6	12	C	80.0	0.344	-6	6	C	55.1	0.340
-12	12	C	143.0	0.256	-12	6	C	124.0	0.281
-12	12	C	214.0	0.253	-12	6	C	140.0	0.292
-12	12	C	130.0	0.262	-12	6	C	103.0	0.284
-6	9	A	95.2	0.340	-6	3	A	70.4	0.353
-6	9	A	78.2	0.353	-6	3	A	56.9	0.352
-6	9	A	59.0	0.344	-6	3	A	55.4	0.364
-12	9	A	193.0	0.301	-12	3	A	145.0	0.292
-12	9	A	133.0	0.296	-12	3	A	115.0	0.295
-12	9	A	115.0	0.285	-12	3	A	135.0	0.300
-6	9	B	85.0	0.331	-6	3	B	75.2	0.343
-6	9	B	71.6	0.348	-6	3	B	60.2	0.357
-6	9	B	72.1	0.334	-6	3	B	57.7	0.350
-12	9	B	138.0	0.265	-12	3	B	136.0	0.265
-12	9	B	138.0	0.285	-12	3	B	127.0	0.287
-12	9	B	146.0	0.275	-12	3	B	138.0	0.281
-6	9	C	83.6	0.344	-6	3	C	64.3	0.348
-6	9	C	79.6	0.336	-6	3	C	66.7	0.368
-6	9	C	72.1	0.351	-6	3	C	76.1	0.354
-12	9	C	186.0	0.295	-12	3	C	124.0	0.298
-12	9	C	127.0	0.297	-12	3	C	134.0	0.296
-12	9	C	141.0	0.298	-12	3	C	164.0	0.299
-6	0	--	58.3	0.357	-12	0	--	106.0	0.297
-6	0	--	56.9	0.363	-12	0	--	115.0	0.305
-6	0	--	55.5	0.364	-12	0	--	101.0	0.298

Table 75. AAD-1 critical temperatures

Co-product (%)	Co-product (type)	Unaged High T_c	RTFO Aged High T_c	PAV aged Int T_c	PAV Aged Low T_c	Co-product (%)	Co-product (type)	Unaged High T_c	RTFO Aged High T_c	PAV aged Int T_c	PAV Aged Low T_c
12	A	64.82	68.11	18.73	-24.00	6	B	65.31	68.35	18.94	-22.11
12	A	64.53	67.56	18.62	-23.97	6	B	63.81	68.60	18.36	-23.00
12	A	65.00	67.99	18.12	-24.05	6	B	65.40	68.96	18.65	-22.73
12	B	67.38	69.90	19.64	-23.73	6	C	63.20	66.13	17.96	-23.25
12	B	67.04	70.75	19.94	-23.26	6	C	63.55	65.93	18.23	-24.88
12	B	67.33	69.70	19.38	-22.22	6	C	63.37	66.43	18.05	-23.68
12	C	64.49	67.50	18.42	-23.38	6	D	65.15	65.28	19.59	-23.28
12	C	64.54	67.32	18.59	-23.38	6	D	65.71	65.26	19.47	-22.34
12	C	64.35	66.74	18.42	-23.29	6	D	65.77	65.25	19.50	-21.87
9	A	64.60	67.30	18.77	-23.57	3	A	63.59	65.78	17.24	-22.88
9	A	64.74	67.50	18.09	-24.40	3	A	63.63	65.69	17.26	-24.00
9	A	64.94	67.72	17.83	-22.89	3	A	63.75	65.95	17.31	-24.18
9	B	66.95	69.40	19.02	-23.64	3	B	64.45	67.61	17.67	-21.86
9	B	66.59	69.37	19.17	-22.78	3	B	65.25	67.80	18.47	-23.68
9	B	66.18	69.15	18.92	-23.83	3	B	64.63	67.22	18.26	-24.14
9	C	64.82	67.27	18.43	-23.70	3	C	62.68	66.06	18.25	-24.56
9	C	64.35	66.73	18.43	-23.79	3	C	62.83	65.18	17.81	-24.00
9	C	64.38	66.98	18.06	-23.69	3	C	62.95	65.59	17.28	-24.15
9	D	66.50	67.12	20.12	-22.11	3	D	64.68	65.09	18.72	-24.90
9	D	67.20	67.19	20.46	-21.87	3	D	64.87	67.64	19.30	-22.38
9	D	66.39	67.28	20.45	-22.34	3	D	64.97	67.78	19.65	-22.92
6	A	64.08	66.75	18.12	-24.66	0	--	62.18	65.66	17.23	-25.33
6	A	64.08	66.78	17.98	-23.69	0	--	62.37	65.21	17.67	-25.12
6	A	65.50	67.26	17.73	-23.30	0	--	62.27	65.27	17.07	-25.09

Table 76. AAM-1 critical temperatures

Co-product (%)	Co-product (type)	Unaged High T_c	RTFO Aged High T	PAV aged Int T_c	PAV Aged Low T_c
9	A	68.73	67.62	22.62	-14.89
9	A	68.61	67.63	22.42	-14.96
9	A	68.84	67.46	22.25	-14.94
9	B	69.78	67.76	22.49	-14.40
9	B	69.73	66.80	22.74	-14.47
9	B	69.90	67.12	23.22	-15.55
9	C	68.64	68.37	21.16	-15.22
9	C	68.60	68.53	21.42	-15.56
9	C	69.36	68.54	21.51	-14.93
9	D	71.73	69.88	23.77	-12.96
9	D	71.55	69.37	25.10	-12.25
9	D	71.73	68.42	22.73	-12.84
6	A	67.83	67.99	18.30	-16.36
6	A	68.69	68.06	16.62	-16.53
6	A	69.34	68.62	15.33	-15.28
6	B	69.34	67.25	22.05	-14.98
6	B	69.38	66.44	22.07	-14.88
6	B	69.21	66.95	21.69	-14.76
6	C	68.62	67.32	21.62	-14.70
6	C	68.94	68.21	22.37	-14.62
6	C	68.96	67.05	22.04	-14.77
6	D	67.16	68.32	23.59	-13.29
6	D	70.11	68.27	24.85	-13.05
6	D	69.80	67.80	24.92	-13.05
3	A	68.53	66.99	18.39	-14.43
3	A	68.84	66.80	18.91	-12.83
3	A	68.13	67.01	17.89	-14.62
3	B	68.13	67.23	19.73	-14.67
3	B	68.70	66.60	20.72	-14.95
3	B	68.74	66.70	20.98	-14.94
3	C	68.21	67.64	21.32	-15.42
3	C	68.03	66.55	20.62	-15.27
3	C	67.92	67.03	20.78	-14.91
3	D	69.78	67.58	23.04	-13.48
3	D	69.08	67.69	23.32	-13.76
3	D	69.07	67.72	22.89	-12.83
0	--	67.72	66.56	20.11	-15.12
0	--	67.80	66.45	20.07	-15.22
0	--	67.78	67.04	20.60	-14.84

Table 77. LPMB critical temperatures

Co-product (%)	Co-product (type)	Unaged High T_c	RTFO Aged High T	PAV aged Int T_c	PAV Aged Low T_c
12	A	67.54	72.10	22.62	-19.51
12	A	67.97	71.70	22.29	-19.39
12	A	67.56	71.62	22.33	-20.23
12	B	68.81	72.66	22.96	-18.88
12	B	68.59	72.38	22.81	-19.24
12	B	68.80	72.43	22.45	-19.08
12	C	67.22	71.49	22.29	-18.70
12	C	67.37	71.07	21.88	-18.52
12	C	67.10	71.00	22.62	-19.22
9	A	66.79	67.64	20.57	-22.15
9	A	66.77	67.59	20.25	-21.58
9	A	66.68	67.78	18.77	-20.47
9	B	69.02	69.14	21.63	-18.82
9	B	68.34	69.55	21.77	-20.57
9	B	68.63	69.12	22.03	-19.46
9	C	66.66	69.13	20.22	-21.39
9	C	66.95	69.79	20.00	-21.54
9	C	66.42	68.63	20.34	-21.77
6	A	65.97	67.33	20.15	-20.85
6	A	66.40	66.51	20.68	-20.38
6	A	66.84	66.67	19.79	-21.67
6	B	67.39	68.65	21.93	-19.84
6	B	67.32	67.39	21.78	-19.84
6	B	67.26	68.60	21.92	-19.75
6	C	66.89	68.42	19.81	-20.03
6	C	67.24	68.47	19.90	-20.83
6	C	67.06	68.31	20.62	-20.29
3	A	66.48	66.97	20.41	-21.21
3	A	66.32	66.83	19.91	-21.47
3	A	65.64	65.56	20.36	-22.00
3	B	66.84	67.73	21.90	-19.31
3	B	66.64	66.45	21.76	-20.89
3	B	66.58	67.38	22.02	-20.35
3	C	65.79	67.60	20.05	-21.76
3	C	65.84	67.19	20.12	-21.67
3	C	65.81	67.22	20.38	-21.89
0	--	65.67	66.37	20.24	-21.70
0	--	66.58	66.87	20.03	-22.52
0	--	65.65	66.98	20.01	-21.82

Table 78. LB critical temperatures

Co-product (%)	Co-product (type)	Unaged High T_c	RTFO Aged High T	PAV aged Int T_c	PAV Aged Low T_c
9	A	70.08	71.89	24.03	-16.75
9	A	70.83	72.34	23.79	-16.39
9	A	70.33	71.57	24.00	-16.87
9	B	71.84	72.45	24.58	-16.57
9	B	72.39	72.27	24.58	-17.30
9	B	72.33	72.45	24.68	-16.38
9	C	71.70	72.08	24.04	-17.94
9	C	71.67	72.08	24.29	-16.16
9	C	71.76	72.81	24.31	-17.60
6	A	69.97	71.43	23.56	-18.34
6	A	69.95	71.56	23.82	-18.85
6	A	69.74	71.21	24.06	-17.70
6	B	71.81	72.21	24.70	-17.47
6	B	71.78	72.49	24.36	-17.74
6	B	72.06	72.71	23.93	-17.90
6	C	70.36	71.78	24.00	-16.83
6	C	70.55	71.88	24.24	-18.45
6	C	70.92	71.70	24.22	-17.94
3	A	69.15	71.70	23.57	-17.30
3	A	69.31	71.70	23.77	-19.33
3	A	69.49	71.44	23.78	-17.67
3	B	70.94	72.11	24.32	-18.56
3	B	71.43	71.37	24.34	-17.38
3	B	70.81	72.23	23.96	-17.85
3	C	70.16	71.47	23.89	-17.46
3	C	70.79	71.37	24.02	-17.49
3	C	71.22	71.35	23.91	-17.65
0	--	68.95	71.20	23.62	-19.89
0	--	68.91	70.97	23.42	-19.43
0	--	68.79	71.13	23.38	-19.30

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