Title: METHODS AND PROCESSES FOR PRODUCING PROCESS OILS WITH A LOW POLYAROMATIC HYDROCARBON CONTENT

Abstract: A method or process for producing, manufacturing, or extracting a process oil with a low polyaromatic hydrocarbon (PAH) or polycyclic aromatic (PCA) content of less than approximately 3% by weight. The process includes providing a feed oil, a solvent, and an anti-solvent (e.g., water) to a counter-current extraction column. The extraction column is heated to an extraction temperature. Nitrogen gas is introduced to facilitate removal of PCA from the feed oil and thereby facilitate or effectuate production of process oils with a PCA content of less than approximately 3% by weight.
METHODS AND PROCESSES FOR PRODUCING PROCESS OILS WITH A LOW POLYAROMATIC HYDROCARBON CONTENT

Technical Field

The present disclosure relates generally to process oils and methods for the manufacture, production, and/or extraction of such process oils. More specifically, the present disclosure relates to process oils that include less than 3% by weight of polyaromatic hydrocarbons or polycyclic aromatics and methods for the manufacture, production, and/or extraction of such process oils.

Introduction

Process oils, for example, rubber process oils or rubber extender oil compositions, are petroleum derivatives that are typically used for processing rubber compounds (e.g., a natural rubber or a synthetic rubber such as styrene butadiene rubber (SBR)). The use of process oils facilitates modification(s) or enhancement(s) to the properties or characteristics of the rubber compound. For instance, the addition of a process oil to a rubber compound can reduce a temperature required during rubber compound processing in order to prevent scorching of the rubber compound during a grinding procedure, or decrease the rubber compound's viscosity, or improve the rubber compound's general workability. Process oils, for example rubber extender oil compositions or rubber process oils, can be used for the processing of rubber compounds in a variety of industrial applications, such as the manufacture of automobile rubber tires.

Different types of process oils typically have specific physical and chemical properties. For example, process oils typically have a specific miscibility, kinematic viscosity, solvency, density, aniline point, flash point, and/or polyaromatic hydrocarbon (PAH) or polycyclic aromatic (PCA) content. Typically the properties of a particular process oil determine its compatibility for use in the processing of a particular type of rubber compound. In addition, the properties of a particular process oil are generally tailored
and/or selected depending on the intended or desired use(s) of a processed rubber compound (also known as a rubber product).

A highly aromatic process oil (e.g., rubber extender oil composition) is typically more compatible for use with rubber compounds that have a substantial amount or concentration of aromatic groups, for example SBRs. Such highly aromatic rubber extender oil compositions are also known as distillate aromatic extracts (DAEs). DAEs tend to have an aromatic hydrocarbon content of at least 70% by weight. The term "aromatic hydrocarbon" typically refers to a molecule or compound that is composed primarily of carbon and hydrogen atoms, and that includes at least one ring structure composed of conjugated unsaturated carbon bonds (e.g., a benzene moiety). In addition, the term "aromatic hydrocarbons" also includes PCAs or PAHs. PCAs or PAHs are compounds having two or more ring structures (i.e., aromatic rings) that are fused together.

The toxicity of PCAs is of increasing health and environmental concern. The use of process oils having a high PCA content for the processing of rubber compounds, for example in the manufacture of automobile tires, has been said to contribute to environmental pollution and health problems in the form of tire dust produced during the use of said automobile tires.

Rubber extender oil compositions or rubber process oils conventionally have a high polyaromatic hydrocarbon (PAH) or polycyclic aromatic compound (PCA) content. PCAs or PAHs have been identified as a potentially carcinogenic compound. Accordingly, rubber extender oil compositions having greater than 3% by weight of PCAs or PAHs are classified as "carcinogenic" according to the European legislation (EU Substance Directive 67/548/EEC), and must be labeled with the risk phrase "R45" (may cause cancer) and the label "T" (toxic, skull and crossbones) in Europe.

Process oils (e.g., rubber extender oil compositions) having less than 3% by weight of PCAs or PAHs are increasingly desired for health, safety, and environmental protection
purposes. Rubber extender oil compositions having greater than 3% by weight of PCAs or PAHs are classified as "carcinogenic" according to the European legislation (EU Substance Directive 67/548/EEC) and must be labeled with the risk phrase "R45" (may cause cancer) and the label "T" (toxic, skull and crossbones) in Europe. Accordingly, rubber extender oil compositions of a PCA content by weight of less than 3% is of an increasing commercial value.

Treated distillate aromatic extracts (TDAEs) have been proposed as replacement compositions for rubber extender oil compositions having greater than 3% by weight of PCAs or PAHs. TDAEs can be manufactured from DAEs by further severe processing, for example by using solvent extraction processes, which lowers the concentration of PCAs or PAHs to below the threshold of 3% by weight.

The production or extraction of rubber extender oil compositions (e.g., TDAEs) with a PCA or PAH content of less than 3% by weight typically requires performance of multiple processing steps as well as the use of specialized processing equipment or systems. Generally, a large capital investment is required for the production or extraction of process oils, such as rubber extender oil compositions, with a PCA content by of less than 3% by weight. The increasing demand in the lubricants market for process oils (e.g., rubber extender oil compositions such as TDAEs) with a PCA content of less than 3% by weight for automotive and industrial applications, is likely to affect future availability of DAEs, which are commonly used as a feedstock or feed oil in the manufacture of TDAEs.

Accordingly, it is generally desirable to develop enhanced processes for producing process oils (e.g., rubber extender oil compositions such as TDAEs) that have a PCA or PAH content of less than 3% by weight.

Summary

In accordance with a first aspect of the present disclosure, there is disclosed a process for producing a process oil. The process includes introducing a feed oil into an extraction
column and introducing a solvent into the extraction column at a percentage volume of solvent to feed oil of between approximately 80% and approximately 1000%. The process further includes bubbling one of an inert gas and a substantially inert gas through the extraction column, and extracting polycyclic aromatic compounds from the feed oil to obtain a process oil with a polycyclic aromatic compound content of less than approximately 3% by weight.

In accordance with a second aspect of the present disclosure, there is disclosed a process oil with a polycyclic aromatic content of less than approximately 3% by weight. The process oil has a kinematic viscosity at 100°C of between approximately 20 mm²/second and approximately 32 mm²/second, a density of at least approximately 1.010 kg/liter, a flash point of at least approximately 220°C, an aniline point of at least approximately 40.0°C, and a glass transition temperature of at most approximately -49°C.

In accordance with a third aspect of the present disclosure, there is disclosed a rubber composition with a polycyclic aromatic content of less than approximately 3% by weight, the rubber composition being manufactured by processing of a rubber compound with a process oil that is produced using a process of particular aspects of the present disclosure.

In accordance with a fourth aspect of the present disclosure, there is disclosed a system for extracting polycyclic aromatic compounds. The system includes a countercurrent extraction column, a first inlet carried by the extraction column for mediating introduction of a feed oil, and a second inlet carried by the extraction column for mediating introduction of at least one of a solvent and an anti-solvent. In addition, the system includes a plurality of rotatable plates disposed within the extraction column, each of the plurality of rotatable plates including a plurality of openings through which each of the feed oil, solvent, and anti-solvent can be communicated, and a gas bubbling module coupled to the extraction column for introducing a substantially inert gas into the extraction column.
Brief Description of the Figures Incorporated Herewith

Embodiments, and aspects, of the present disclosure are described hereinafter with reference to the figures, in which:

Figure 1 is a flowchart of a process for producing, manufacturing, or extracting a process oil according to an embodiment the present disclosure;

Figure 2 shows a partial schematic view of a system for producing, manufacturing, or extracting a process oil according to an embodiment of the present disclosure;

Figure 3 to 16 show representative experimental data relating to a number of different properties or characteristics of eight rubber compositions (i.e., rubber compositions A to H) that are produced using various process oils.

Detailed Description

Many embodiments of the present disclosure relate to process oils with a polycyclic aromatic (PCA) or polyaromatic hydrocarbon (PAH) (PCA and PAH are hereinafter collectively referred to as PCA) content of less than 3% by weight. In addition, many embodiments of the present disclosure relate to methods and processes for manufacturing, producing, and/or extracting process oils with a PCA content of less than 3% by weight.

For purposes of the present disclosure, references to process oils relate to process oils for use in processing rubber compounds, including natural rubber and synthetic rubber (e.g., styrene butadiene rubber (SBR)). For instance, in some embodiments, the process oil is known as a rubber extender oil composition or a rubber process oil. It will, however, be understood by a person of ordinary skill in the art that embodiments of the present disclosure may also relate to other types of process oils, for example, process oils that are used in the processing of other materials (e.g., various polymers) for automotive and/or industrial applications.
Selected Process Oil Properties and Characteristics

As previously mentioned, embodiments of the present disclosure relate to process oils that can be used in association with the processing of rubber compounds. In most embodiments, a process oil exhibits one or more properties and/or characteristics (e.g., physical and/or chemical properties) that facilitate or enhance a compatibility of the process oil for the processing of a selected rubber compound. For example, one or more properties or characteristics of the process oil may be selected in view of processibility and/or bleed resistance of a selected rubber compound.

In some embodiments, the properties and/or characteristics of the process oil are specifically chosen for facilitating or ensuring one or more desired properties or characteristics exist in a processed rubber compound (i.e., a rubber product obtained from the processing of the rubber compound by the process oil). Particular properties and/or characteristics of process oils extracted or produced in accordance with the present disclosure are described as follows:

(a) **PCA Content**

In many embodiments of the present disclosure, the process oil has a PCA content of less than approximately 3% by weight.

According to the European legislation (EU Substance Directive 67/548/EEC), process oils having a PCA content of less than 3% by weight need not be classified as "carcinogenic", and need not be labeled with the risk phrase "R45" (may cause cancer) and the label "T" (toxic, skull and crossbones) in Europe. Therefore, process oils according to many embodiments of the present disclosure need not be classified as "carcinogenic", and need not be labeled with the risk phrase "R45" (may cause cancer) and the label "T" (toxic, skull and crossbones) in Europe.

(b) **Polar Compound Content**

In many embodiments, the process oil includes less than approximately 25% by weight of a polar compound. In several embodiments, the process oil includes between
approximately 15% and approximately 20% by weight of a polar compound. In some embodiments, the process oil includes between approximately 16% and approximately 19% by weight of a polar compound. In selected embodiments, the process oil includes between approximately 17% and approximately 18% by weight of a polar compound.

In many embodiments, the content of a polar compound in the process oil can be varied to produce process oils of particular characteristics, for example for increasing compatibility of use of the process oil with a selected rubber compound. For instance, the content of a polar compound in the process oil can be varied for improving compatibility of blending of the process oil with a selected rubber compound. In some embodiments, the content of a polar compound in the process oil is selected for reducing or minimizing occurrence of bleeding in a processed rubber compound (i.e., a rubber product produced by processing of a rubber compound with the process oil). In some embodiments, the rubber compound is a vulcanized rubber compound. The content of the polar compound can be measured according to ASTM D 2007.

(c) **Kinematic Viscosity**

In many embodiments, the process oil has a kinematic viscosity at 100°C of between approximately 10 mm²/second and approximately 40 mm²/second. In numerous embodiments, the process oil has a kinematic viscosity at 100°C of between approximately 20 mm²/second and approximately 35 mm²/second. In some embodiments, the process oil has a kinematic viscosity at 100°C of between approximately 20 mm²/second and approximately 32 mm²/second. In selected embodiments, the process oil has a kinematic viscosity at 100°C of between approximately 25 mm²/second and approximately 31 mm²/second.

In many embodiments, the kinematic viscosity of the process oil is selected for enhancing processibility and operability in blending with a rubber compound. Particularly when used with an aromatic vulcanized rubber compound, the kinematic viscosity of the process oil provided by the present disclosure is selected for reducing or minimizing
occurrence of bleeding in said aromatic vulcanized rubber compound. The kinematic viscosity can be measured according to ASTM D 445.

(d) **Aromatic Hydrocarbon Content**

In many embodiments of the present disclosure, the process oil has an aromatic hydrocarbon content of at least approximately 15% by weight. In some embodiments, the process oil has an aromatic hydrocarbon content of at least approximately 20% by weight. In selected embodiments, the process oil has an aromatic hydrocarbon content of at least approximately 25% by weight.

The aromatic hydrocarbon content of a process oil typically affects an affinity for, and a compatibility with, a rubber compound. When a process oil is blended with a rubber compound, the aromatic hydrocarbon present in the process oil is effective for enhancing processibility and providing an extending property of the rubber compound. In many embodiments of the present disclosure, the aromatic hydrocarbon content of the process oil is selected for enhancing at least one of processibility and providing an extending property of the rubber compound. The content of the aromatic hydrocarbon is measured according to ASTM D 2007.

(e) **Flashpoint**

In many embodiments of the present disclosure, the flash point of the process oil is at least approximately 200°C. In some embodiments, the flash point of the process oil is at least approximately 210°C. In selected embodiments, the flash point of the process oil is at least approximately 220°C.

In many embodiments of the present disclosure, the flash point of the process oil is selected for reducing the flammability of the process oil to thereby enhance ease of handling the process oil. The flash point of the process oil can be measured according to ASTM D 92 (COC°C).
Methods and Processes for Producing, Manufacturing, or Extracting a Process Oil

As above-mentioned, the present disclosure provides methods and/or processes for producing, manufacturing, and/or extracting process oils having particular characteristics, including a PCA content of less than approximately 3% by weight.

In various embodiments of the present disclosure, process oil extraction or production involves the use of at least one feed oil (also known as a feedstock or base oil). In some embodiments, a process oil can be extracted or produced using two or more different feed oils. In many embodiments, the feed oil is a petroleum derivative. In some embodiments, the feed oil can include atmospheric residue, vacuum residual oil, and/or deasphalting oil that is obtained from deasphalting oils with hydrocarbons.

(a) Selected Feed Oil Properties

In many embodiments, a feed oil has a PCA content of at least approximately 20% by weight, a density of at least approximately 0.950 kg/liter, a kinematic viscosity at 100°C of between approximately 20 mm²/s and approximately 80 mm²/s, a flash point of at least approximately 200°C, an aniline point of at least approximately 40°C, a glass transition temperature (T_g) of between approximately -66°C and -32°C, a aromatic hydrocarbon content of at least approximately 70% by weight, and a N-methylpyrrolidone purity of at least approximately 99%. In some embodiments, the feed oil can have at least one different property from that listed above, for example, the feed oil can have a PCA content of at least approximately 22% by weight, a density of at least approximately 1.010 kg/liter, a kinematic viscosity at 100°C of between approximately 20 mm²/s and 80 mm²/s, a flash point of at least approximately 220°C, an aniline point of at least approximately 40°C, a glass transition temperature (T_g) of at most approximately -37°C, a aromatic hydrocarbon content of at least approximately 85% by weight, and a N-methylpyrrolidone purity of at least approximately 99.5%.

In selected embodiments, a feed oil (e.g., feed oil A and feed oil B) can have one or more properties or characteristics as listed in Table 1 below.
### Table 1: Properties/Characteristics of Feed Oil

<table>
<thead>
<tr>
<th>CHARACTERISTIC</th>
<th>UNIT</th>
<th>TEST METHOD</th>
<th>TYPICAL QUALITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density @ 15 °C</td>
<td>g/cm³</td>
<td>ASTM D 4052</td>
<td>Feed A 1.0279</td>
</tr>
<tr>
<td>Flash Point, COC</td>
<td>°C</td>
<td>ASTM D 92</td>
<td>Feed B 0.9956</td>
</tr>
<tr>
<td>Kinematic Viscosity @ 100 °C</td>
<td>cSt</td>
<td>ASTM D 445</td>
<td>79.85</td>
</tr>
<tr>
<td>Pour Point</td>
<td>°C</td>
<td>ASTM D 97</td>
<td>+ 30</td>
</tr>
<tr>
<td>Aniline Point</td>
<td>°C</td>
<td>ASTM D 611</td>
<td>+ 30</td>
</tr>
<tr>
<td>Sulphur Content</td>
<td>% wt</td>
<td>ASTM D 4294</td>
<td>Nil</td>
</tr>
<tr>
<td>Water Content</td>
<td>% vol</td>
<td>ASTM D 95</td>
<td>Nil</td>
</tr>
<tr>
<td>PCA Content</td>
<td>% wt</td>
<td>IP 346</td>
<td>Feed A 21.2</td>
</tr>
<tr>
<td>Total Acid Number</td>
<td>mg KOH/g</td>
<td>ASTM D 664</td>
<td>Feed B 6.0</td>
</tr>
<tr>
<td>Refractive Index @ 75 °C</td>
<td></td>
<td>ASTM D 1747</td>
<td>1.56800</td>
</tr>
<tr>
<td>Refractive Index @ 20 °C</td>
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<td>Exxon</td>
<td>1.5623</td>
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<tr>
<td>Refractivity Intercept</td>
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<td>Exxon</td>
<td>1.0740</td>
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<tr>
<td>Viscosity Gravity Constant</td>
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<td>Carbon Type Analysis</td>
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<tr>
<td>- Aromatic Carbon (CA)</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>- Paraffinic Carbon (CP)</td>
<td></td>
<td></td>
<td>46</td>
</tr>
<tr>
<td>- Naphthenic Carbon (CN)</td>
<td></td>
<td></td>
<td>28</td>
</tr>
<tr>
<td>Benzo (a) Pyrene</td>
<td>ppm</td>
<td>Grimmer</td>
<td>0.177 - 0.704</td>
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<tr>
<td>Total 8 PAHs</td>
<td>ppm</td>
<td>Grimmer</td>
<td>3.899 - 5.592</td>
</tr>
<tr>
<td>Total Nitrogen</td>
<td>ppm</td>
<td></td>
<td>999</td>
</tr>
<tr>
<td>CCR</td>
<td></td>
<td></td>
<td>5.00</td>
</tr>
<tr>
<td>Distillation</td>
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<td>ASTM D 2887</td>
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</tr>
<tr>
<td>IBP</td>
<td></td>
<td></td>
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</tr>
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<td>5%</td>
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</tr>
<tr>
<td>90%</td>
<td></td>
<td></td>
<td>638.5</td>
</tr>
<tr>
<td>95%</td>
<td></td>
<td></td>
<td>663.5</td>
</tr>
<tr>
<td>FBP</td>
<td></td>
<td>ASTM D 2783</td>
<td>698.5</td>
</tr>
<tr>
<td>Aromatic, SMS</td>
<td>% wt</td>
<td></td>
<td>5.87</td>
</tr>
<tr>
<td>Mono</td>
<td></td>
<td></td>
<td>4.58</td>
</tr>
<tr>
<td>Di</td>
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<td>6.50</td>
</tr>
<tr>
<td>Tri</td>
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<td></td>
<td>9.20</td>
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<tr>
<td>Tetra</td>
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</tr>
<tr>
<td>Total</td>
<td></td>
<td></td>
<td>26.15</td>
</tr>
</tbody>
</table>

Preparation or extraction of the feed oil can be performed using methods, processes, and/or techniques known to a person of ordinary skill in the art. In addition, the properties and/or characteristics of feed oils used in the preparation, manufacture, and/or extraction of particular process oils of the present disclosure can be varied in a manner that improves manufacturing yield or a likelihood of producing a process oil having...
particular properties or property ranges. As non-limiting examples for enhancing understanding of the present disclosure, methods, processes, or techniques for determining and/or verifying certain properties of particular feed oils are provided as follows:

(b) Feed Oil Property Measurement, Characterization, or Verification

(i) Determination of PCA content
The PCA (or PAH) content or concentration in a feed oil can be performed using the dimethylsulphoxide-refractive index method (IP 346/80) (Institute of Petroleum, 1985) and/or the Grimmer method (Grimmer et al., 1981a; Grimmer et al., 1981b) by HRGC/MS. The PCA content in the feed oil can be expressed as a percentage by weight thereof (i.e., w/w %).

In many embodiments, the PCA content of a feed oil is at least approximately 20% by weight. In some embodiments, the PCA content of the feed oil is at least approximately 25% by weight. In selected embodiments, the PCA content of the feed oil is at least approximately 30%, or more, by weight.

(ii) Determination of glass transition temperature
Thermal analysis can be performed using a differential scanning calorimeter (TA, Q2000 DSC) under helium gas. Sample(s) of the feed oil can be obtained and used for analyzing or determining the glass transition temperature (Tg) of the feed oil. Sample(s) of the feed oil are run on a Q-series DSC that is attached to a refrigerated cooling system. The sample(s) are run from -90°C to 50°C at an average rate of temperature increase of approximately 20°C/minute in aluminum Tzero hermetic pans.

Modulated DSC (MDSC) is typically used to make an accurate measurement of Tg. Modulated parameters used can be: period = 60s; amplitude = 1°C; and ramp rate = 2°C/minute. MDSC is a technique that takes the total heat flow signal and splits the total heat flow signal into two additional signals, the two additional signals known as a
reversing heat flow and a non-reversing heat flow. A temperature modulation is applied to the sample(s) at a specific frequency and amplitude in order to obtain these additional signals. The reversing heat flow provides information on Tg and heat capacity. The non-reversing heat flow provides information on kinetic events. The total heat flow is the same as a signal that would be obtained from a standard DSC experiment.

In many embodiments, the Tg temperature of the feed oil is at most approximately -37°C. Alternatively, the Tg temperature of the feed oil can be altered, the alteration of the Tg temperature of the feed oil being, for example, depending on one or more conditions during production of the process oil.

(Hi) Determination of aniline point

To determine the aniline point of a feed oil, a volume of an aniline (also known as phenylamine or aminobenzene) sample and a volume of a feed oil sample are placed in a tube and mixed mechanically. The mixture is then heated at a controlled rate until the aniline sample (aniline phase) and the feed oil sample (feed oil phase) become miscible. The miscible mixture of the aniline and feed oil samples is then cooled at a controlled rate. The temperature at which the two phases in the miscible mixture separate is known as the aniline point (or mixed aniline point).

The aniline point is most often used to provide an estimate of the aromatic hydrocarbon content of a sample (e.g., mixture or compound). The aniline point (or mixed aniline point) is useful as an aid in the characterization of pure hydrocarbons and in the analysis of hydrocarbon mixtures. Aromatic hydrocarbons typically exhibit the lowest aniline points and paraffins typically exhibit the highest aniline points. In a homologous series, the aniline point typically increases with increasing molecular weight.

In many embodiments, the aniline point of the feed oil is at least approximately 35°C. In some embodiments, the aniline point of the feed oil is at least approximately 40°C, approximately 50°C, approximately 60°C, or approximately 70°C. In most embodiments,
the aniline point of the feed oil can be altered, for example, depending on one or more conditions during production of the process oil.

(iv) **Determination of density**

To determine the density of a feed oil, a volume (e.g., approximately 0.7mL) of a sample of feed oil is introduced into an oscillating tube and the change in oscillating frequency caused by the change in mass of the oscillating tube when the sample of feed oil is introduced thereinto is used in conjunction with calibration data to determine the density of the sample. Alternatively, the density of the feed oil can be measured according to ASTM D 4052.

As previously mentioned, in many embodiments, the density of the feed oil is at least approximately 0.950 kg/liter. In some embodiments, the density of the feed oil is between at approximately 0.900g/cm³ and approximately 1.00g/cm³. In other embodiments, the density of the feed oil is between at approximately 1.00g/cm³ and approximately 1.30g/cm³. In many embodiments, the density of the feed oil can be altered. For example, the density of the feed oil can be altered depending on one or more conditions during production of the process oil.

Although exemplary methods, processes, and/or techniques for determining PCA content, glass transition temperature, aniline point, and density of the feed oil are described above, a person of ordinary skill in the art will understand that alternative methods, processes, and/or techniques for determining PCA content, glass transition temperature, aniline point, and density of the feed oil can also be used. In addition, a person of ordinary skill in the art will understand that other properties (e.g., physical and/or chemical properties) of the feed oil can also be selected, determined, and/or varied as-desired.

(c) **Representative Process Oil Extraction or Manufacturing Processes**

Figure 1 is a flowchart of a process 100 for producing, manufacturing, or extracting a process oil according to an embodiment the present disclosure. Figure 2 is a system 10.
for producing, manufacturing, or extracting a process oil according to an embodiment of the present disclosure. In general, a process 100 for producing, manufacturing, or extracting a process oil according to the present disclosure includes a number of process portions, including process portions that can be performed by way of the system 10 of Figure 2.

In a first process portion 110 of the process 100, at least one feed oil is introduced into a counter-current extraction column 20 such as that in the system 10 shown in Figure 2. In most embodiments, the feed oil is introduced into the counter-current extraction column 20 at a first inlet 25 that is carried by the extraction column 20 and positioned at a bottom portion of the extraction column 20.

Portions of various types of counter-current extraction columns can be used in association with a process 100 according to the present disclosure. Counter-current extraction columns and apparatuses are known in the art, and can be used for liquid-liquid extraction (also known as solvent extraction and partitioning). Two countercurrent extraction apparatuses are described in United States Patent Nos. US 4101408 and US 4151089, which are incorporated herein by reference in their entirety.

In many embodiments, the extraction column 20 includes a number of plates 50 disposed therewithin. In some embodiments, the plates 50 are positioned along a length of the extraction column 20. In most embodiments, the plates 50 include a number of holes or openings 55 formed therein for allowing material (e.g., fluid) communication therethrough. In some embodiments, different plates 50 have holes 55 of different sizes. For instance, the size of the holes 55 can differ from one plate 50 to another, and/or the sizes of the holes 55 on any given plate 50 can be varied, for-example, depending on a type, density, or size of material that is to be communicated therethrough.

In a second process portion 120, a solvent is introduced into the extraction column 20. In many embodiments, the solvent is introduced at a second inlet 30 positioned at a top portion of the extraction column 20. In multiple embodiments of the present disclosure,
the solvent is a polar solvent. In some embodiments, the solvent is N-methylpyrrolidone. In other embodiments, the solvent can be a furfural, phenol, or other type of compound.

In various embodiments, the system 10 includes a controller (not shown) coupled to the extraction column 20. The controller can be configured for setting, controlling, and/or adjusting a percentage volume of solvent to feed oil (i.e., volume ratio of the solvent to feed oil). In many embodiments, percentage volume of solvent to feed oil introduced within the extraction column 20 is between approximately 80% and approximately 1000%. In some embodiments, the percentage volume of solvent to feed oil within the extraction column 20 is between approximately 100% and approximately 500%. In specific embodiments, the percentage volume of solvent to feed oil within the extraction column 20 is between approximately 150% and approximately 300%.

In some embodiments, the percentage volume of solvent to feed oil within the extraction column 20 can be selected, controlled, and adjusted in order to produce a process oil of particular characteristics in accordance with the present disclosure. For instance, the percentage volume of solvent to feed oil within the extraction column 20 can be controlled and adjusted depending on one or more conditions (e.g., physical conditions such as temperature) during the process 100.

In a third process portion 130, the extraction column 20 is heated to an extraction temperature. In most embodiments, the extraction temperature is between approximately 80°C and approximately 120°C. In some embodiments, the extraction temperature is between approximately 90°C and approximately 110°C. In various embodiments, the system 10 includes a temperature controller (not shown) for setting, controlling, and/or adjusting the extraction temperature. The temperature controller can establish or control the extraction temperature in order to facilitate the production of a process oil having particular characteristics in accordance with the present disclosure. For instance, the extraction temperature can be set, controlled, and adjusted for facilitating the production of a process oil with a PCA content of less than 3% by weight.
In a fourth process portion 140, an anti-solvent such as demineralized water (hereinafter referred to as water) is added into the extraction column 20. With respect to its activity as an anti-solvent, this means that the water mitigates at least one action or function that is performed by the solvent. In many embodiments, water is added or introduced into the extraction column 20 via the second inlet 30. In other embodiments, water is added into the extraction column 20 via a different inlet (not shown) that is positioned at the top portion of the extraction column 20.

In several embodiments, water reduces the solvent's ability to extract or remove PCA from the feed oil. Accordingly, increasing the concentration or amount of water introduced into the extraction column 20 results in a reduced rate and/or quantity of PCA extraction or removal from the feed oil, and hence an increased PCA content in the process oil being produced. In some embodiments, increasing the concentration or amount of water introduced into the extraction column 20 increases yield of process oil produced.

In a number of embodiments, water is mixed with the solvent prior to their introduction into the extraction column 20. In most embodiments, the volume ratio of water to solvent (or percentage volume of water per unit volume of solvent) can be varied in order to facilitate the production of a process oil having particular characteristics as provided by the present disclosure. For instance, the volume ratio of water to solvent (or percentage volume of water per unit volume of solvent) can be varied for at least one of increasing yield of process oil produced and enhancing removal of PCA from the feed oil (i.e., reducing PCA content of process oil produced).

In some embodiments, the system 10 includes an additional controller (not shown) that is couplable to the extraction column 20 for determining, controlling, and/or adjusting the percentage volume of water to solvent (i.e., volume ratio of water to solvent) introduced into the extraction column 20.
In a fifth process portion 150, at least one inert or substantially inert gas such as dry
nitrogen gas is bubbled or introduced into the extraction column 20. Depending upon
eembodiment details, nitrogen gas may be substituted with a different gas, for instance,
argon or helium; or a partially inert or substantially non-reactive gas such as carbon
dioxide.

In most embodiments, the inert gas is introduced at a bottom portion or region of the
extraction column 20. The inert gas can be introduced into the extraction column 20
using a gas bubbler, a gas bubbling module, or a gas introduction machine, device,
apparatus, system, or means (collectively identified as reference numeral of 35). In many
embodiments, the gas introduction device 35 is coupled to or disposed at the bottom of
the extraction column 20. Typically, the inert gas is introduced or bubbled into the
extraction column 20 at a controlled rate. The inert gas can be introduced into the
extraction column 20 at a predetermined and/or controlled pressure. For instance,
nitrogen gas can be introduced into the extraction column 20 at a pressure of between
approximately 0.1 Psi and approximately 25 Psi (e.g., 0.1 Psi, 1 Psi, 10 Psi, 15 Psi, 20
Psi, and 25 Psi).

In some embodiments, the system 10 includes a gas controller (not shown) that is
couplable to the extraction column 20 for setting, controlling, and/or adjusting the
pressure of the inert gas that is introduced into the extraction column 20. In selected
embodiments the gas controller further controls and/or adjusts the size of the inert gas
bubbles entering, or introduced, into the extraction column 20. In many embodiments, the
size of the inert gas bubbles is between approximately 0.01cm and approximately 0.5cm.
In some embodiments, the size of the inert gas bubbles is between approximately 0.05cm
and 0.1cm.

The use of an inert gas, more specifically the introduction of an inert gas such as nitrogen
gas into the extraction column 20, helps to increase the rate, and/or the amount, of PCA
being removed from the feed oil. This can be because the introduction of the inert gas
into the extraction column 20 enhances the interaction, and reaction, between feed oil and
solvent molecules. More specifically, this can be because the introduction of the inert gas into the extraction column 20 increases the contact area (i.e., surface area for contact) between the feed oil and solvent molecules.

As previously mentioned, in some embodiments, the size of nitrogen gas bubbles introduced into the extraction column 20 can be selected, controlled, or adjusted. For example, the size of nitrogen gas bubbles introduced into the extraction column 20 can be selected, controlled, and adjusted, for increasing the contact area between said nitrogen gas bubbles and the feed oil molecules, as well as between said nitrogen gas bubbles and the solvent molecules. In some embodiments, decreasing the size of nitrogen gas bubbles increases the overall contact surface area between the nitrogen gas bubbles and each of the feed oil and solvent molecules to thereby increase the rate, and/or amount, of PCA being removed from the feed oil. For example, in selected embodiments, decreasing the size of nitrogen air bubbles from a diameter of approximately 1cm to a diameter of approximately 0.1mm can cause an approximately 37% decrease in the PCA content of the feed oil (e.g., decrease in PCA content of the feed oil from approximately 3.8% to 2.4%).

In addition, in many embodiments, the natural buoyancy that exists with the nitrogen gas bubbles promotes phase separation between the feed oil and the solvent to thereby facilitate or effectuate the extraction or removal of PCA from the feed oil.

Process portions 110 to 150 facilitate or effectuate an extraction or removal of PCA from the feed oil(s), where such extraction or removal occurs in a sixth process portion 160. PCA that is extracted from the feed oil is removed from the extraction column 20 using methods or techniques known to a person of ordinary skill in the art. In many embodiments, PCA is removed from the extraction column 20 at a first outlet 40 that is disposed or positioned at the bottom portion of the extraction column 20. In some embodiments, other impurities (e.g., asphaltene) are removed from the extraction column 20 together with the PCA.
The extraction or removal of PCA from the feed oil enables the production of process oils that exhibit particular properties or characteristics according to the present disclosure. For example, the extraction of PCA from the feed oil facilitates or effectuates the production of process oils that have a PCA content of less than 3% by weight.

Depending upon embodiment details, the sequence of one or more of process portions 110 to 150 can vary or be interchangeable. In addition, the duration of each of process portions 110 to 150 can be determined and varied in order to produce process oils having particular properties or characteristics. For example, the sequence and/or duration of one or more of process portions 110 to 150 can be determined, and varied, for increasing the yield of the process oil produced and/or facilitating the removal of PCA from the feed oil (i.e., decreasing the PCA content of the process oil produced).

In a seventh process portion 170, the process oil produced (i.e., process oil product) is collected from the extraction column 20. In many embodiments, the process oil is collected via a second outlet 45 that is disposed or positioned at a top portion of the extraction column 20. The collection of the process oil can be performed using methods, processes, or techniques known to a person of ordinary skill in the art. The process oil produced can be referred to as the raffinate of the process 100.

In an optional eighth process portion 180, a secondary processing treatment of the process oil can be performed. In many embodiments, the second processing treatment enhances performance (e.g., improves one or more properties or characteristics) of the process oil. The secondary processing treatment can include, for example, a distillation treatment or a de-waxing treatment.

In many embodiments of the present disclosure, parameters, for example quantity of reagent, relative quantities of reagents, duration, temperature, and rate, in association with each of the process portions 110 to 180 may be varied in order to produce a process oil having particular characteristics or properties. For example, parameters corresponding to one or more of the process portions 110 to 180 can be varied for increasing a rate of
extraction of PCA from the feed oil, increasing a total quantity of PCA extraction from the feed oil (and hence decreasing the PCA content of the process oil produced), and/or increasing the yield of the process oil produced.

In some embodiments, an increase in the total quantity of PCA extracted from the feed oil (and hence a decrease in the PCA content in the process oil being produced) corresponds, or occurs simultaneously, with a decrease in the yield of the process oil being produced. Therefore, to enhance or maximize the yield of process oil produced, while at the same time maintaining the PCA content of the process oil produced at less than 3% by weight to meet a set of regulations (e.g., regulations set by the above-referenced EU directive), parameters of one or more of process portions 110 to 150 can be controlled and varied accordingly.

The particular effect(s) of varying a set of parameters in association with selected process portions 110 to 180, as according to several embodiments of the present disclosure, are described and illustrated in examples one to four provided below.

**EXAMPLE ONE**

As previously described, the solvent is added into the extraction column 20 in the process portion 120. Experiments were performed in association with the present disclosure to study the effects of varying quantities of solvent introduced into the extraction column 20. More specifically, experiments were conducted to study the effects of different percentages of volume of solvent to volume of feed oil within the extraction column 20.

In the experiments of example one, solvent and feed oil were added into the extraction column 20 at varying percentages by volume. Different percentages of volume of solvent per unit volume of feed oil were simulated within the extraction column 20. The percentages of volume of solvent per unit volume of feed oil simulated in the experiments were approximately 200%, approximately 150%, approximately 140%, approximately 120%, approximately 100%, approximately 95%, and approximately 90%.
For the experiments performed in association with example one, each of percentage volume of water per unit volume of solvent, and temperature, within the extraction column 20 was maintained at a constant value. More specifically, the percentage volume of water per unit volume of solvent in the extraction column 20 was maintained at between approximately 7.6% and approximately 7.8%, and the temperature within the extraction column 20 was maintained at between approximately 95°C and approximately 115°C.

**Results**

The experiments showed that increasing the percentage of volume of solvent per unit volume of feed oil (i.e., increasing the volume ratio of solvent to feed oil) within the extraction column 20 results in an increased amount of PCA being removed or extracted from the feed oil. In other words, increasing the percentage of volume of solvent per unit volume of feed oil within the extraction column 20 results in production of process oil with a lower content (by weight) of PCA.

More specifically, results as shown in Table 2 below indicate that increasing the percentage of volume of solvent per unit volume of feed oil within the extraction column 20 from approximately 90% to approximately 200% results in a decrease in the content (by weight) of PCA of the process oil produced from approximately 3.3% to approximately 1.8%.

Results of the experiments of example one also showed that increasing the percentage volume of solvent per unit volume of feed oil (i.e., increasing the volume ratio of solvent to feed oil) within the extraction column 20 results in a lower yield of process oil being produced. More specifically, the increase in percentage of volume of solvent per unit volume of feed oil within the extraction column 20 from approximately 90% to approximately 200% results in a decrease in yield of process oil produced from approximately 70% to approximately 35%.
Results obtained from experiments of example one also showed that increasing the percentage of volume of solvent per unit volume of feed oil within the extraction column 20 results in production of process oils with a higher glass transition (Tg) temperature, a higher aniline point, and a lower kinematic viscosity.

Conclusion

Results obtained from the experiments of example one show that the increase in the percentage of volume of solvent per unit volume of feed oil (i.e., increasing the volume ratio of solvent to feed oil) introduced within the extraction column 20 facilitates or effectuates the production of a process oil with a lower content by weight of PCA. However, increasing the percentage volume of solvent per unit volume of feed oil introduced within the extraction column 20 simultaneously results in a lower yield of the process oil produced. To balance, enhance, or maximize the yield of process oil produced, while simultaneously ensuring that PCA content remains less then 3% by weight (e.g., to comply with national or international regulations), the percentage of quantity of solvent to quantity of feed oil introduced into the extraction column 20 can be selected and adjusted.

Results indicate that a percentage volume of solvent per unit volume of feed oil greater than approximately 90%, and approaching or exceeding approximately 95% (for instance, a volume ratio of solvent to feed oil greater than approximately 0.90, (e.g., at least approximately 0.92 or 0.93, or approaching or exceeding approximately 0.95), facilitates or effectuates the production of a process oil having a PCA content of less than 3%. Results further indicate that as the percentage volume of solvent per unit volume of feed oil is increased from approximately 90% to approximately 200%, the resulting process oil PCA content decreases from approximately 3.3% to approximately 1.8%. Thus, the PCA content of a process oil extracted or produced in accordance with the present disclosure can be selectively decreased by employing an appropriate increased percentage volume of solvent per unit volume of feed oil. For instance, the PCA content of a process oil extracted or produced in accordance with the present disclosure can be selectively further decreased by increasing the percentage volume of solvent to feed oil above
approximately 200%, for example approximately 300%, 400%, 500%, 600%, 700%, 800%, 900%, and 1000%. However, increasing the percentage volume of solvent per unit volume of feed oil decreases the percentage yield. Therefore, to increase percentage yield of a process oil, the percentage volume of solvent to feed oil can be decreased below approximately 90%, for example approximately 85%, 80%, or 75%.

More particularly, results indicate that in order to enhance or maximize the percentage yield of the process oil, while simultaneously ensuring that the PCA content of the process oil is less than approximately 3%, the percentage volume of solvent per unit volume of feed oil should be at least somewhat above approximately 90% (e.g., the volume ratio of solvent to feed oil should be at least between approximately 0.91 or 0.92 to 0.95).

Table 2: Effect of varying percentages of quantity of solvent to quantity of feed oil

(With constant percentage of water between 7.6% - 7.8 % and temperature between 95°C - U5°C)

<table>
<thead>
<tr>
<th>Percentage of solvent : feed oil (%)</th>
<th>Refractive index</th>
<th>PCA content (% by weight)</th>
<th>Tg (°C)</th>
<th>Flash Point (°C)</th>
<th>Aniline Point (°C)</th>
<th>Pour Point</th>
<th>Kv at 100°C</th>
<th>Percentage Yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>1.4980</td>
<td>1.8</td>
<td>-52</td>
<td>289</td>
<td>75</td>
<td>+30</td>
<td>24</td>
<td>35</td>
</tr>
<tr>
<td>150</td>
<td>1.5032</td>
<td>1.85</td>
<td>-51</td>
<td>289</td>
<td>74</td>
<td>+30</td>
<td>24.5</td>
<td>38</td>
</tr>
<tr>
<td>140</td>
<td>1.5047</td>
<td>2.0</td>
<td>-50</td>
<td>289</td>
<td>73</td>
<td>+30</td>
<td>24.8</td>
<td>45</td>
</tr>
<tr>
<td>120</td>
<td>1.5067</td>
<td>2.1</td>
<td>-48</td>
<td>289</td>
<td>72</td>
<td>+30</td>
<td>25</td>
<td>50</td>
</tr>
<tr>
<td>100</td>
<td>1.5088</td>
<td>2.3</td>
<td>-47</td>
<td>289</td>
<td>71</td>
<td>+30</td>
<td>25.3</td>
<td>60</td>
</tr>
<tr>
<td>95</td>
<td>1.5135</td>
<td>2.7</td>
<td>-45</td>
<td>289</td>
<td>69</td>
<td>+30</td>
<td>25.5</td>
<td>65</td>
</tr>
<tr>
<td>90</td>
<td>1.5255</td>
<td>3.3</td>
<td>-44</td>
<td>289</td>
<td>68</td>
<td>+30</td>
<td>26</td>
<td>70</td>
</tr>
</tbody>
</table>
EXAMPLE TWO

Demineralized water (or water) is introduced into the extraction column 20 in the fourth process portion 140 according to several embodiments of the present disclosure. Water mixes with the solvent that is introduced in the extraction column 20. Experiments were conducted in association with the present disclosure to study the effects of varying quantities (volumes) of water introduced into the extraction column 20.

In the experiments of example two, the content of water in the solvent (e.g., N-methylpyrrolidone (NMP)) was varied. More specifically, different percentages of volume of water, namely approximately 2%, 4%, 6%, 8%, 10%, and 12%, per unit volume of solvent were mixed with the solvent in the experiments of example two.

For the experiments of example two, each of percentage volume of solvent per unit volume of feed oil and the temperature within the extraction column 20 was maintained at a constant value. More specifically, the percentage volume of solvent per unit volume of feed oil within the extraction column 20 was maintained at approximately 95% and the temperature within the extraction column 20 was maintained at between approximately 95°C and approximately 115°C.

Results

The results obtained from the experiments showed that increasing the percentage of volume of water per unit volume of the solvent (e.g., NMP) results in a decrease in quantity of PCA being removed or extracted from the feed oil. That is, increasing the percentage volume of water to solvent results in production of process oil having a higher content by weight of PCA. More specifically, an increase in percentage of volume of water to solvent from approximately 2% to approximately 12%-results an increasing PCA content from approximately 1.8% to approximately 2.9% by weight within the process oil produced.

In addition, the experiments of example two also showed that an increase in percentage volume of water to solvent results in an increase in yield of process oil. More
particularly, an increase in the percentage volume of water to solvent from approximately 2% to approximately 12% results in an increase in yield of the process oil from approximately 25% to approximately 75%.

Experiments of example two further indicate that increasing the percentage volume of water to solvent results in the production of process oil with higher glass transition temperatures and lower aniline points.

Conclusion

The results obtained from experiments of example two indicate that an increase in the percentage volume of water to solvent facilitates or effectuates a higher yield of process oil being produced. However, by increasing the percentage volume of water to solvent, the PCA content by weight of the produced process oil increases. The results indicate that water mitigates against the removal or extraction of PCA from the feed oil as effected by the solvent. Hence, water can be considered an anti-solvent insofar as water mitigates at least one function or action (e.g., PCA removing or extracting function) of the solvent.

Accordingly, the results indicate that in order to maximize yield of process oil being produced while simultaneously ensuring that PCA content of the process oil remains below 3% by weight (e.g., to comply with national or international regulations), the percentage volume of water to solvent, and hence the quantity of water being introduced into the extraction column 20 in process portion 140, can be selected or adjusted to produce a process oil having a target PCA content by way of a process having a target or acceptable yield.

Results further indicate that in order to maximize process oil yield while simultaneously ensuring that PCA content of the process oil remains below 3% by weight, the percentage volume of water to solvent should be approximately 12%. In addition, results indicate that the PCA content of the process oil decreased by a surprisingly significant amount (e.g., approximately 22% reduction in PCA content) when the percentage of water in
solvent decreased from approximately 8% to approximately 6%. Furthermore, results indicate that this decrease in percentage of water in solvent from approximately 8% to approximately 6% does not cause an atypical level of decrease in the percentage yield.

Table 3: Effect of varying percentages of quantity of water in the solvent
(With constant percentage of quantity of solvent to quantity of feed oil and temperature of between 95°C - 105°C)

<table>
<thead>
<tr>
<th>Percentage of water in solvent (%)</th>
<th>Properties of process oil produced</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Refractive index</td>
</tr>
<tr>
<td>2</td>
<td>1.4816</td>
</tr>
<tr>
<td>4</td>
<td>1.4965</td>
</tr>
<tr>
<td>6</td>
<td>1.5032</td>
</tr>
<tr>
<td>8</td>
<td>1.5132</td>
</tr>
<tr>
<td>10</td>
<td>1.5175</td>
</tr>
<tr>
<td>12</td>
<td>1.5210</td>
</tr>
</tbody>
</table>

EXAMPLE THREE

As indicated above, in the third process portion 130, the extraction column 20 is heated to an extraction temperature. Experiments were conducted in association with the present disclosure to study the effects of different extraction temperatures on the production of the process oil.

In the experiments of example three, the extraction temperature was varied. More specifically, production of the process oil was carried out at extraction temperatures of approximately 40°C, approximately 50°C, approximately 60°C, approximately 70°C, approximately 80°C, approximately 90°C, and approximately 100°C.
For the experiments of example three, each of percentage quantity of solvent to quantity of feed oil and the percentage of water in the solvent was maintained at a constant value. More specifically, the percentage quantity of solvent to quantity of feed oil within the extraction column 20 was maintained at approximately 150% and the percentage of quantity of water to solvent was maintained at between approximately 6.5% and approximately 7.5%.

Results
Results of the experiments of example three show that an increase in temperature causes a corresponding increase in the amount of PCA being removed or extracted from the feed oil. Thus, the use of a higher extraction temperature results in the production of a process oil having a lower PCA content by weight. More specifically, results show that increasing the extraction temperature from approximately 40°C to approximately 100°C results in a decrease in PCA content from approximately 3.21% to approximately 2.46% by weight in the process oil produced. Additionally, results demonstrate that an extraction temperature equal to or exceeding approximately 80°C results in a process oil having less than approximately 2.53% PCA content by weight. That is, at or above an extraction temperature of approximately 80°C, the PCA content of a process oil produced in accordance with the present disclosure can be at least approximately 15% less than a 3% target maximum or upper threshold PCA content.

Conclusion
Results indicate that an increase in extraction temperature facilitates or effectuates production of a process oil of lower PCA content by weight. Accordingly, the extraction temperature can be selected and/or controlled during the process 100 for producing a process oil that has less or significantly less than approximately 3% by weight of PCA. The results of this set of experiments indicate that in order to produce a process oil having a PCA content of less than approximately 3% by weight, the extraction temperature should be greater than approximately 40°C, and approaching, equaling, or exceeding at least approximately 50°C.
Results also indicate having an extraction temperature of between approximately 80°C and 100°C enables the greatest decrease in the PCA content of the process oil. In some embodiments, when the extraction temperature is at approximately 100°C, the column must be pressurized for maintaining water in the solvent at a liquid phase. In addition, results indicate a significant decrease in PCA content from approximately 2.8% to 2.53% (i.e., an approximately 10% reduction in PCA content) when the extraction temperature is increased from approximately 70°C to 80°C. This significant decrease in PCA content when the extraction temperature is increased from approximately 70°C to 80°C as provided by some embodiments of the present disclosure is unexpected.

Table 4: Effects of varying the extraction temperature
(With constant percentage of quantity of solvent to quantity of feed oil and percentage quantity of water in the solvent)

<table>
<thead>
<tr>
<th>Extraction temperature (°C)</th>
<th>Properties of process oil produced</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Refractive index</td>
</tr>
<tr>
<td>40</td>
<td>1.5217</td>
</tr>
<tr>
<td>50</td>
<td>1.5145</td>
</tr>
<tr>
<td>60</td>
<td>1.5123</td>
</tr>
<tr>
<td>70</td>
<td>1.5109</td>
</tr>
<tr>
<td>80</td>
<td>1.4985</td>
</tr>
<tr>
<td>90</td>
<td>1.4980</td>
</tr>
<tr>
<td>100</td>
<td>1.4988</td>
</tr>
</tbody>
</table>

EXAMPLE FOUR

In the fifth process portion 150 according to several embodiments of the present disclosure, an inert gas is introduced or bubbled into the extraction column 20.
Experiments were conducted in association with the present disclosure to study the effects caused by the introduction of nitrogen gas at different pressures.

Nitrogen gas was introduced into the extraction column 20 at different pressures, more specifically, at approximately 5 psi, approximately 10 psi, approximately 15 psi, and approximately 20 psi. Each of percentage quantity of solvent to quantity of feed oil, percentage of water in the solvent, and extraction temperature was maintained at a constant value. More specifically, the percentage quantity of solvent to quantity of feed oil within the extraction column 20 was maintained at approximately 150% and the percentage of quantity of water to solvent was maintained at between approximately 6.5% and approximately 7.5%, and the extraction temperature was maintained at between approximately 95°C and approximately 115°C.

Results
Experiments show that increasing the pressure of nitrogen gas bubbled or introduced into the extraction column 20 results in increased removal or extraction of PCA from the feed oil. In other words, increasing the pressure of nitrogen gas bubbled or introduced into the extraction column results in the production of a process oil with a decreasing content by weight of PCA. More specifically, an increase in the pressure of nitrogen gas from approximately 5 psi to approximately 20 psi results in a decrease in PCA content by weight in the process oil produced from approximately 3.3% to approximately 1.8%.

Conclusion
Results indicate that an increase in the pressure of nitrogen gas introduced into the extraction column facilitates or effectuates a production of a process oil of lower PCA content by weight. Accordingly, the pressure of nitrogen gas introduced into the extraction column can be selected prior to or during the process oil production in accordance with the present disclosure in order to obtain a process oil having less than approximately 3% by weight of PCA.
Results also indicate that in order to facilitate or effectuate the production of a process oil having a PCA content of less than approximately 3%, the pressure of nitrogen gas introduced into the extraction column 20 should exceed approximately 5 psi, and should further approach at least between approximately 7 psi to 10 psi (e.g., at least about 7 - 8 psi). Nitrogen gas pressures greater than approximately 10 psi, for example 15 psi, 20 psi, and 25 psi, can result in the production of a process oil having a PCA content that is significantly less than 3% (e.g., less than approximately 2.6%). However, depending upon embodiment details, other parameters or conditions associated with other process portions (e.g., process portions 110 to 180) can be selected to produce a process oil having a PCA content of less than 3% when the nitrogen gas pressure is less than approximately 5%, for example approximately 4%, 3%, 2%, or 1%.

In some embodiments of the present disclosure, the pressure of the nitrogen gas can be varied depending on the diameter of the extraction column and/or the operation of an apparatus configured for introducing nitrogen into the extraction column, for instance, a gas distribution coil system. Having a gas distribution coil system with smaller holes (coil openings or holes) through which nitrogen gas is introduced into the extraction column facilitates the introduction of smaller nitrogen gas bubbles at a higher nitrogen gas pressure.

Table 5: Effects of varying pressure of nitrogen gas

(With constant percentage of quantity of solvent to quantity of feed oil, percentage quantity of water in the solvent, and temperature)

<table>
<thead>
<tr>
<th>Nitrogen gas pressure (psi)</th>
<th>Properties of process oil produced</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Refractive index</td>
</tr>
<tr>
<td>-----------------------------</td>
<td>------------------</td>
</tr>
<tr>
<td>5</td>
<td>1.5220</td>
</tr>
<tr>
<td>10</td>
<td>1.5159</td>
</tr>
</tbody>
</table>
Particular Process Oil Compatibility for Rubber Compound Processing

It is a typical requirement that a new, enhanced, and/or modified process oil should be at least as compatible as an existing process oil for processing a material (e.g., a rubber compound). In most embodiments, the process oil of the present disclosure is compatible or highly compatible for use in the processing of rubber compounds (e.g., vulcanizing SBR). In many embodiments, the process oil of the present disclosure is at least as compatible as other existing or ordinary process oils for use in the processing of rubber compounds. In some embodiments, the process oil may be used as a plasticizer of a thermoplastic resin, an ingredient of printing ink or a softening agent of regenerated asphalt.

To demonstrate and/or to evaluate the compatibility, or suitability of use, of particular process oils provided by the present disclosure for processing rubber compounds to obtain processed rubber compositions (which are rubber products obtained from the processing of the rubber compounds with the process oil), various properties of the processed rubber compositions obtained in association with particular process oils of the present disclosure are evaluated together with existing rubber compositions that are obtained in association with existing or ordinary process oils.

Preparation or manufacture of rubber compositions

The rubber compound used for preparing or manufacturing rubber compositions in association with embodiments of the present disclosure is based on styrene-butadiene rubber (SBR) as per ASTM D 3185-99.

In most embodiments, mixing of raw materials used for the preparation of the rubber composition was performed using a 2-roll mill (Lab Tech model KRM 150) at a set temperature of approximately 50°C with a two-step mixing technique that is known to a person of ordinary skill in the art. Additionally, the time taken for mixing and preparing a
masterbatch (hereinafter referred to as compound A) was approximately 10 minutes, and the time taken for mixing compound A with sulfur and cure accelerator was approximately 5 minutes. The mixture obtained (hereinafter referred to as compound B) was sheeted in order to cool the mixture (or batch) for avoiding a premature vulcanization phenomenon (typically known as scorching).

To prepare vulcanized specimens, uncured compounds were compression molded using a hydraulic hot-press under pressure of 15 tons at 145°C. Cure time used for vulcanizing the specimens was determined from the optimum cure time with 90% complete cure \( (t_{90}) \) as measured from the moving die rheometer (MDR; Tech Pro, USA) at 145°C as per ASTM D 5289.

Rheological properties of rubber compositions were measured using Mooney viscosimeter (as per ASTM D 1646), capillary rheometer, and rubber process analyzer (RPA) at 100°C. In the case of vulcanizates (e.g., rubber compounds or rubber compositions that have been vulcanized), hardness, tensile, heat build-up, and abrasion resistance tests were performed according to ASTM D 2240, ASTM D 412, ASTM D 623 and DIN 53516 respectively.

The following examples illustrate several properties of a number of rubber compositions, each of which was produced using a different process oil. The results, discussion, and figure(s) provided with each of the examples below illustrate the compatibility of particular process oils provided by the present disclosure for rubber compound processing. More specifically, the examples below illustrate that particular process oils of the present disclosure are at least as compatible as existing or ordinary process oils for the processing of rubber compounds.

Examples five to eighteen describe, and provide, representative experiments, and results obtained with said experiments, in association with a number of rubber compositions (i.e., rubber compositions A to H as listed below).
List of Rubber Compositions Tested in Each of Examples Five to Nineteen

A: Rubber composition (A) produced using a process oil provided by an embodiment of the present disclosure.
B: Rubber composition (B) produced using the process oil of A, but obtained at a different processing phase.
C: Rubber composition (C) produced using a process oil provided by an embodiment of the present disclosure.
D: Rubber composition (D) produced using an existing or ordinary process oil with a PCA content of more than 3% by weight.
E: Rubber composition (E) produced using a process oil provided by an embodiment of the present disclosure.
F: Rubber composition (F) produced using a process oil provided by an embodiment of the present disclosure.
G: Rubber composition (G) produced using a process oil provided by an embodiment of the present disclosure.
H: Rubber composition (H) produced using a process oil provided by an embodiment of the present disclosure.

EXAMPLE FIVE

Mooney Viscosity

Mooney viscosity is typically defined as the shearing torque resisting rotation of a cylindrical metal disk (or rotor) embedded in rubber within a cylindrical cavity.

In the experiments of example five, the Mooney viscosity of each of rubber compositions A to H was determined. In addition, the Mooney viscosities of rubber compositions A to H at different oil loadings (i.e., having varying concentrations of corresponding process oils) were determined. The oil loading or varying concentrations of the process oil were approximately 5 phr (i.e., based on hundred parts by weight of the rubber composition), approximately 10 phr, and approximately 20 phr.
Results and Discussion
As shown in Figure 3, there is no significant difference in the Mooney viscosities of rubber compositions that are produced with particular process oils of the present disclosure (i.e., rubber compositions A and B) as compared to rubber compositions produced with existing process oils (i.e., rubber compositions C to H).

Results indicate that Mooney viscosity decreases with an increasing concentration of process oil in the rubber composition (i.e., when the concentration of process oil in the rubber composition increases from approximately 5 phr to approximately 20 phr). This decrease in Mooney viscosity observed with the increasing concentration of process oil in a rubber composition is similarly replicated with each of rubber compositions A to H.

Accordingly, the results obtained from the experiments of example five indicate that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

Figure 3: Influence of Type of Process Oil, and Oil Loading, on Mooney Viscosity of Rubber Compositions A to H
EXAMPLE SIX

Viscosity-Shear Rate Correlation

Viscosity is a measure of the flow resistance of a fluid (i.e., a process oil or liquid rubber composition), which is being deformed by either shear stress or extensional stress.

In the experiments of example six, the viscosity-shear rate correlation of each of rubber compositions A to H was measured. More specifically, the viscosity-shear rate correlation of each of rubber compositions A to H for each of process oil loadings (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 15 phr, was measured.

Results and Discussion

Figure 4a shows the viscosity-shear rate correlation for each of rubber compositions A to H at a process oil loading of approximately 5 phr. Figure 4b shows the viscosity-shear rate correlation for each of rubber compositions A to H at a process oil loading of approximately 10 phr. Figure 4c shows the viscosity-shear rate correlation for each of rubber compositions A to H at a process oil loading of approximately 15 phr.

As shown in Figure 4a to 4c, there is no significant statistical difference in the viscosity-shear rate correlations for each of rubber compositions A to H. More specifically, at each of process oil loadings of approximately 5 phr, approximately 10 phr, and approximately 15 phr, there is no significant difference in the viscosity-shear rate correlation between rubber compositions A to H. In addition, results indicate there is no significant difference in the viscosity-shear rate correlation for each of rubber compositions A to H, even when the process oil loadings of each of rubber compositions A to H are increased from approximately 5 phr to approximately 15 phr.

Results indicate that the viscosity of each of rubber compositions A to H decreases with increasing shear rate from approximately 5 s\(^{-1}\) to approximately 1000 s\(^{-1}\). Results show that this decrease in viscosity observed with an increasing shear rate is similarly found
with each of rubber compositions A to H, and at each of process oil loadings of approximately 5 phr, approximately 10 phr, and approximately 15 phr.

Accordingly, the results obtained from the experiments of example six indicate that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

**Figure 4a: Influence of Type of Process Oil, at a Process Oil Loading of 5phr, on Viscosity-Shear Rate of Rubber Compositions A to H**

![Viscosity-Shear Rate Graph](image-url)
Figure 4b: Influence of Type of Process Oil, at a Process Oil Loading of 10 phr, on Viscosity-Shear Rate of Rubber Compositions A to H

Figure 4c: Influence of Type of Process Oil, at a Process Oil Loading of 15 phr, on Viscosity-Shear Rate of Rubber Compositions A to H
EXAMPLE SEVEN

Complex Viscosity - Frequency Correlation

Complex viscosity is a frequency dependent viscosity function that is determined during forced harmonic oscillation of shear stress. The complex viscosity of a material (e.g., rubber composition) is related to the complex shear modulus and represents the angle between the viscous stress and the shear stress. The complex viscosity function is also defined as the difference between the dynamic viscosity and the out-of-phase viscosity.

In the experiments of example seven, the complex viscosity of each of rubber compositions A to H was measured at different frequencies (rad s⁻¹). More specifically, the complex viscosity of each of rubber compositions A to H was measured at frequencies from approximately 1.0 rad s⁻¹ to approximately 100 rad s⁻¹. In addition, the complex viscosity-frequency correlation of each of rubber compositions A to H, at each of process oil loadings (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, was measured.

Results and Discussion

Figure 5a shows the complex viscosity-frequency correlation for each of rubber compositions A to H at a process oil loading of approximately 5 phr. Figure 5b shows the complex viscosity-frequency correlation for each of rubber compositions A to H at a process oil loading of approximately 10 phr. Figure 5c shows the complex viscosity-frequency correlation for each of rubber compositions A to H at a process oil loading of approximately 20 phr.

Results indicate is no significant statistical difference in the complex viscosity-frequency correlation for each of rubber compositions A to H at each-of process oil loading of approximately 5 phr, 10 phr, and 20phr. In addition, there is no significant statistical difference in the complex viscosity-frequency correlation for rubber compositions A to H, even when the process oil loadings were increased from approximately 5 phr to approximately 20 phr.
Results indicate that the complex viscosity of each of rubber compositions A to H decreases with increasing frequency (i.e., oscillatory flow) from approximately 1.0 rad s\(^{-1}\) to approximately 100 rad s\(^{-1}\). Results show that this decrease in complex viscosity observed with an increasing frequency (i.e., oscillatory flow) is similarly found with each of rubber compositions A to H.

Accordingly, the results obtained from the experiments of example seven indicate that particular process oils of the present disclosure can be considered to be at least as compatible as existing process oils for the processing of rubber compounds.

**Figure 5a: Influence of Type of Process Oil, at a Process Oil Loading of 5phr, on Complex Viscosity-Frequency Correlation of Rubber Compositions A to H**
Figure 5b: Influence of Type of Process Oil, at a Process Oil Loading of 10 phr, on Complex Viscosity-Frequency Correlation of Rubber Compositions A to H

Figure 5c: Influence of Type of Process Oil, at a Process Oil Loading of 20 phr, on Complex Viscosity-Frequency Correlation of Rubber Compositions A to H
EXAMPLE EIGHT

Storage Modulus

The storage modulus in a viscoelastic solid (e.g., a rubber composition) measures a stored energy, representing the elastic portion, of the viscoelastic solid.

In experiments of example eight, the storage modulus of each of rubber compositions A to H was measured. In addition, the storage modulus of each of rubber compositions A to H, at various frequencies, and at each of process oil loadings (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr was measured.

Results and Discussion

Figure 6a shows the storage modulus-frequency correlation for each of rubber compositions A to H at a process oil loading of approximately 5 phr. Figure 6b shows the storage modulus-frequency correlation for each of rubber compositions A to H at a process oil loading of approximately 10 phr. Figure 6c shows the storage modulus-frequency correlation for each of rubber compositions A to H at a process oil loading of approximately 20 phr.

Results show that the storage modulus of the rubber compositions A to H are statistically similar at varying frequencies from approximately 0.5 rad/s to approximately 500 rad/s, and at the process oil loadings of approximately 5 phr, 10 phr, and 20 phr. Results also indicate that the storage modulus of the of rubber compositions A to H increases with increasing frequencies from approximately 0.5 rad/s to approximately 500 rad/s. This increase is similar for each of rubber compositions A to H.

The results of the experiments of example eight show that particular process oils provided by the present disclosure provide similar storage-modulus-frequency correlation compared to existing process oils. The results of the experiments of example eight indicate that the use of particular process oils provided by the present disclosure are as compatible as existing process oils for the processing of rubber compounds.
Figure 6a: Influence of Type of Process Oil, at a Process Oil Loading of 5phr, on Storage Modulus-Frequency Correlation of Rubber Compositions A to H

Figure 6b: Influence of Type of Process Oil, at a Process Oil Loading of 10phr, on Storage Modulus-Frequency Correlation of Rubber Compositions A to H
Figure 6c: Influence of Type of Process Oil, at a Process Oil Loading of 20 phr, on Storage Modulus-Frequency Correlation of Rubber Compositions A to H

EXAMPLE NINE

Final Torque

Torque refers to a moment of force or a measure of a force's tendency to produce torsion and rotation about an axis.

In the experiments of example nine, the torque of each of rubber compositions A to H was measured. In addition, the torque of each of rubber compositions A to H, at each of process oil loadings (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr was measured.

Results and Discussion

Figure 7a to 7c show the variation of torque with time of each of rubber compositions A to H at the process oil loadings of approximately 5 phr, approximately 10 phr, and approximately 20 phr.
Results show that the variation in torque over time statistically similar for each of rubber compositions A to H. The results of the experiments of example nine indicate that the process oil loading may influence torque of the rubber composition produced. Increasing the process oil loadings from approximately 5 phr to approximately 20 phr can produce a decrease in torque of each of rubber compositions A to H. This decrease in torque is similar for each of rubber compositions A to H.

Accordingly, results of experiments of example nine suggests that particular process oils of the present disclosure can still be as compatible as existing process oils for the processing of rubber compounds.

**Figure 7a: Influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H**
Figure 7b: Influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H

Figure 7c: Influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H
EXAMPLE TEN

Scorch time
In the field of rubber processing or rubber manufacturing, scorch time refers to a time during which a rubber composition or rubber compound can be worked at a given temperature before curing begins.

In the experiments of example ten, the scorch time for each of rubber compositions A to H was measured. In addition, the scorch time of each of the rubber compositions A to H, at each of process oil loading (i.e., process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr was measured.

Results and Discussion
Results show statistical similarity with respect to the scorch time of each of rubber compositions A to H. In addition, results indicate that the scorch time of each of rubber compositions A to H increases with an increase in process oil loadings from approximately 5 phr to approximately 20 phr. Results show that this increase in scorch time that is observed with an increasing process oil loading of the rubber composition is statistically similar for each of rubber compositions A to H.

Accordingly, the results of the experiments of example ten indicate that the use of particular process oils provided by the present disclosure does not have a significant effect on the scorch time of rubber compositions. Therefore, the results obtained from the experiments of example ten show that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.
Figure 8: Influence of Type of Process Oil, and Oil Loading, on Scorch Time of Rubber Compositions A to H

EXAMPLE ELEVEN

_Cure Time_

Curing, also known as vulcanizing, is a chemical process for converting rubber, rubber compositions, or related polymers into more durable materials via an addition of curatives. The process of curing involves the formation of crosslinks or bridges between individual polymer chains.

In the experiments of example eleven, time taken for curing each of rubber compositions A to H was measured. In addition, the time taken for curing each of rubber compositions A to H, having each of process oil loadings (i.e., process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, was measured.

_Results and Discussion_

Results show that the cure time of each of rubber compositions A to H is statistically similar to each other. In addition, results indicate that the cure time of each of rubber compositions A to H increases with an increase in process oil loadings from
approximately 5 phr to approximately 20 phr. Results show that this increase in cure time that is observed with increasing the process oil loading of the rubber composition is statistically similar for each of rubber compositions A to H.

Accordingly, the results of the experiments of example eleven indicate that the use of particular process oils provided by the present disclosure does not have a significant effect on the cure time of rubber compositions. Therefore, the results obtained from the experiments of example eleven show that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

**Figure 9: Influence of Type of Process Oil, and Oil Loading, on Cure Time of Rubber Compositions A to H**

**EXAMPLE TWELVE**

*Shore Hardness*

Shore hardness is a measure of the resistance of a material (e.g., a rubber composition) to indentation by 3 spring-loaded indenter. An increase in value of shore hardness reflects an increase in resistance of the material. Shore hardness is measured by the Shore
(Durometer) test. Shore hardness can be measured using either the Shore A or Shore D scale. The Shore A scale is typically used for 'softer' materials.

In the experiments of example twelve, the hardness of each of rubber compositions A to H was measured. In addition, the hardness of each of rubber compositions A to H, having each of process oil loadings (i.e., process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, was measured.

Results and Discussion

Results show that the hardness of each of rubber compositions A to H is statistically similar to each other. In addition, results indicate that the hardness of each of rubber compositions A to H decreases with an increase in process oil loadings from approximately 5 phr to approximately 20 phr. Results show that this decrease in hardness that is observed with increasing process oil loading of the rubber composition is statistically similar for each of rubber compositions A to H.

Accordingly, the results of the experiments of example twelve indicate that the use of particular process oils provided by the present disclosure does not have a significant effect on the hardness of rubber compositions. Therefore, the results obtained from the experiments of example twelve show that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.
Figure 10: Influence of Type of Process Oil, and Oil Loading, on Harness of Rubber Compositions A to H

EXAMPLE THIRTEEN

100% Modulus

100% modulus refers to an amount of tensile stress sustained by a material (e.g., a rubber composition) at 100% elongation. In other words, 100% modulus refers to an amount of force required to produce a 100% elongation of the material.

In the experiments of example thirteen, the 100% modulus of each of rubber compositions A to H was measured. In addition, the 100% modulus of each of rubber compositions A to H, at each of process oil loadings (i.e., process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, was measured.

Results and Discussion

Results show that the 100% modulus of each of rubber compositions A to H is statistically similar. In addition, results indicate that the 100% modulus of each of rubber compositions A to H decreases with an increase in process oil loadings from approximately 5 phr to approximately 20 phr. Results show that this decrease in 100%
modulus observed with increasing process oil loading of the rubber composition is statistically similar for each of rubber compositions A to H.

Accordingly, the results of the experiments of example thirteen indicate that the use of particular process oils provided by the present disclosure does not have a significant effect on the 100% modulus of rubber compositions. Therefore, the results obtained from the experiments of example thirteen show that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

Figure 11: Influence of Type of Process Oil, and Oil Loading, on Modulus at 100% elongation of Rubber Compositions A to H

EXAMPLE FOURTEEN

300% Modulus

300% modulus refers to an amount of tensile stress sustained by a material (e.g., a rubber composition) at 300% elongation. In other words, 300% modulus refers to an amount of force required to produced a 300% elongation of the material.
In the experiments of example fourteen, the 300% modulus of each of rubber compositions A to H is measured. In addition, the 300% module of each of rubber compositions A to H, having each of process oil loading (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, is measured.

Results and Discussion

Results show that the 300% modulus of each of rubber compositions A to H is statistically similar. In addition, results indicate that the 300% modulus of each of rubber compositions A to H decreases with an increase in process oil loadings from approximately 5 phr to approximately 20 phr. Results show that this decrease in 300% observed with increasing process oil loading in the rubber composition is statistically similar for each of rubber compositions A to H.

Accordingly, the results of the experiments of example fourteen indicate that the use of particular process oils provided by the present disclosure does not have a significant effect on the 300% modulus of rubber compositions. Therefore, the results obtained from the experiments of example fourteen show that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

Figure 12: Influence of Type of Process Oil, and Oil Loading, on Modulus at 300% elongation of Rubber Compositions A to H
EXAMPLE FIFTEEN

Tensile Strength
Tensile strength is typically reflected by a maxima of a stress-strain curve. In general, the tensile strength of a material (e.g., a rubber composition) reflects a resistance of the material to a force tending to tear it apart, or a maximum load that the material can carry without fracturing.

In the experiments of example fifteen, the tensile strength of each of rubber compositions A to H is measured. In addition, the tensile strength of each of rubber compositions A to H, at different process oil loadings (or process oils concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, is measured.

Results and Discussion
Results show that the tensile strength of each of rubber compositions A to H is statistically similar. In addition, results indicate that the tensile strength of each of rubber compositions A to H decreases with an increase in process oil loadings from approximately 5 phr to approximately 20 phr. Results show that the decrease in the tensile strengths with increased process oil loading was statistically similar for each of rubber compositions A to H.

The results demonstrate that the use of particular process oils provided by the present disclosure does not have a significant effect on the tensile strength of rubber compositions. Accordingly, the results obtained from the experiments of example fifteen indicate that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.
Elongation at Break

The elongation at break (i.e., elongation-to-break) is the strain on a sample (e.g., rubber composition) when the sample breaks. The elongation at break corresponds to a breaking or maximum load of the sample, and is often expressed as a percentage of the original length of the sample.

In the experiments of example sixteen, the elongation at break of each of rubber compositions A to H (expressed as a percentage of the original length of the rubber composition) is measured. In addition, the elongation at break of each of rubber compositions A to H, at each of process oil loadings (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr is measured.

Results and Discussion

Results show that elongation at break (expressed as a percentage of the original length of the rubber composition) was statistically similar for each of rubber compositions A to H. Results also show that the elongation at break was increases with increasing process oil loadings from approximately 5 phr to approximately 20 phr. The increase in elongation at

Figure 13: Influence of Type of Process Oil, and Oil Loading, on Tensile Strength of Rubber Compositions A to H
break with increasing oil loadings is statistically similar for each of rubber compositions A to H.

The results show that the use of particular process oils provided by the present disclosure does not have a significant effect on the elongation at break of rubber compositions. Accordingly, the results obtained from the experiments of example sixteen indicate that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

Figure 14: Influence of Type of Process Oil, and Oil Loading, on Elongation at Breaking Point of Rubber Compositions A to H

Heat Build-Up (Temperature Increment)

The degree or amount of heat build-up, measured in terms of temperature increment, can be reflective of volatility of a rubber composition.

The heat build-up in each of rubber compositions A to H was measured in the experiments of example seventeen. In addition, the heat build-up in each of rubber
compositions A to H, having each of process oil loadings (or process oil concentrations) of approximately 5 phr, approximately 10 phr, and approximately 20 phr, was measured.

Results and Discussion

Results show that the heat build-up, measured in terms of temperature increment, was statistically similar for each of rubber compositions A to H. Results also show that the heat build-up was statically similar at each of the process oil loadings of approximately 5 phr, approximately 10 phr, and approximately 20 phr for each of rubber compositions A to H.

The results show that the use of particular process oils provided by the present disclosure does not have a significant effect on the heat build-up of rubber compositions that are produced using said process oils (e.g., rubber compositions A and B). Accordingly, the results obtained from the experiments of example seventeen indicate that particular process oils of the present disclosure can be considered to be as compatible as existing process oils for the processing of rubber compounds.

Figure 15: Influence of Type of Process Oil, and Oil Loading, on Temperature Increment under DIN Standard Test of Rubber Compositions A to H
EXAMPLE EIGHTEEN

Volume Loss under Standard Abrasion Test

The degree of abrasive wear in a rubber composition depends on abrasive particle size, shape, and hardness, the magnitude of stress imposed by the abrasive particle, and the frequency of contact of the abrasive particle. These conditions can be standardized, in a standard abrasion test, to evaluate relative susceptibility of different rubber compositions to abrasive wear.

In the experiments of example eighteen, the degree of abrasive wear sustained by each of the rubber compositions A to H is determined or measured using the standard abrasion test. A volume loss measured using the standard abrasion test indicates degree of abrasive wear sustained by a rubber composition. In addition, in the experiments of example eighteen, the degree of abrasive wear sustained by each of rubber compositions A to H at different process oil loadings (i.e., different process oil concentrations) is measured. The different process oil loadings are approximately 5 phr, approximately 10 phr, and approximately 20 phr.

Results and Discussion

Results show that the volume loss measured using the standard abrasion test is statistically similar for each of rubber compositions A to H. Results also show that the volume loss measured using the standard abrasion test increases with increased process oil loading (i.e., increased concentration of process oil), from approximately 5 phr to approximately 20 phr, within the rubber composition. Results indicate that the increase in volume loss due to increased process oil loading is similarly found for each of rubber compositions A to H.

The results show that the use of particular process oils provided by the present disclosure does not have a significant effect on the degree of abrasive wear of rubber compositions produced using said process oils. Accordingly, the results obtained from the experiments of example eighteen indicate that particular process oils of the present disclosure can be
considered to be as compatible as existing process oils for the processing of rubber compounds.

**Figure 16: Influence of Type of Process Oil, and Oil Loading, on Volume Loss under Standard Abrasion Test of Rubber Compositions A to H**

Conclusion of Discussions of Examples Five to Eighteen

As described in examples five to eighteen above, the processibility and properties (e.g., mechanical and/or physical properties) of a number of rubber compositions produced using different process oils (including both process oils provided by the present disclosure as well as existing or ordinary process oils known in the art) were evaluated or determined.

The results obtained by the experiments of examples five to eighteen demonstrate that the processibility and properties of rubber compositions produced using particular process oils provided by the present disclosure is at least similar, generally equivalent, or equivalent to the processibility and properties of rubber compositions produced using existing process oils known in the art. Results also indicate that there is no adverse effect or impact to the processibility and/or properties of a rubber composition caused by a
processing of rubber compounds (e.g., rubber compounds used as starting material) by a
particular process oil of the present disclosure.

In summary, the process oils provided by the present disclosure are at least as compatible
as existing process oils known in the art for use in the processing of rubber compounds.

Particular process oils of the present disclosure can replace existing or ordinary process
oils that are currently used for rubber processing, rubber manufacturing, automotive,
and/or industrial applications. Process oils produced in accordance with the present
disclosure can have a PCA content of less than or significantly less than 3% by weight.

Such process oils therefore do not need to be labeled with the risk phrase "R45" (may
cause cancer) and the label "T" (toxic, skull and crossbones) in Europe under European
legislation (EU Substance Directive 67/548/EEC). Accordingly, potential markets for the
process oils provided by the present disclosure include Europe.

In addition, in many embodiments, a process for producing a process oil in accordance
with the disclosure is at least one of more efficient, more cost-effective, and safer as
compared to existing or conventional processes or methods for producing process oils. In
many embodiments, at least one of increased efficiency, increased cost-effectiveness, and
enhanced safety associated with a process provided by the present disclosure is due to the
introduction of nitrogen gas into the extraction column. In addition, in multiple
embodiments, at least one of increased efficiency, increased cost-effectiveness, and
enhanced safety associated with the process is due to a percentage volume of solvent to
feed oil that is provided by the present disclosure. Furthermore, in some embodiments, at
least one of increased efficiency, increased cost-effectiveness, and enhanced safety
associated with the process is due to a percentage volume of anti-solvent (e.g., water) to
solvent that is provided by the present disclosure.

Although embodiments and particular examples of the present disclosure have been
described above, a person of ordinary skill in the art will understand that various
modifications, alterations, and variations may be made to the process oils described
above, as well as to the processes or methods for producing, manufacturing, and/or
extracting the process oils, without departing from the scope and spirit of the present disclosure. In addition, while advantages associated with particular process oils, and the processes of manufacture thereof, have been described in the disclosure, other embodiments may also exhibit such advantages, and not all embodiments need necessarily exhibit such advantages to fall within the scope of the disclosure.
Claims

1. A process for producing a process oil comprising:
   introducing a feed oil into an extraction column;
   introducing a solvent into the extraction column at a percentage volume of solvent to feed oil of between approximately 80% and approximately 1000%;
   bubbling one of an inert gas and a substantially inert gas through the extraction column; and
   extracting polycyclic aromatic compounds from the feed oil to obtain a process oil with a polycyclic aromatic compound content of less than approximately 3% by weight.

2. The process as in claim 1, wherein the bubbling of one of the inert gas and substantially inert gas through the extraction column facilitates the extraction of polycyclic aromatic compounds from the feed oil.

3. The process as in claim 2, wherein the substantially inert gas facilitates interaction between the feed oil and the solvent to thereby facilitate extraction of polycyclic aromatic compounds from the feed oil.

4. The process as in claim 1, wherein the extraction column is a countercurrent extraction column.

5. The process as in claim 1, wherein the solvent comprises N-methyl-2-pyrrolidone.

6. The process as in claim 1, further comprising controlling the percentage volume of feed oil to solvent for at least one of increasing yield -and decreasing polycyclic aromatic content of the process oil being produced.

7. The process as in claim 6, wherein the percentage volume of solvent to feed oil in the extraction column is between 100% and 400%.
8. The process as in claim 1, wherein the substantially inert gas is nitrogen gas.

9. The process as in claim 8, the nitrogen gas introduced into the extraction column at a controlled rate.

10. The process as in claim 9, wherein the nitrogen gas is bubbled through the extraction column at a pressure of at least approximately 1.0 psi.

11. The process as in claim 10, wherein the nitrogen gas is bubbled through the extraction column at a pressure of at least approximately 7.0 psi.

12. The process as in claim 9, wherein the nitrogen gas has a gas bubble diameter of between approximately 0.01cm and approximately 0.5cm.

13. The process as in claim 1, further comprising:

   introducing an anti-solvent into the extraction column, wherein the anti-solvent mixes with the solvent within the extraction column to facilitate a control of the polarity of the solvent for thereby facilitating at least one of increased yield and decreased polycyclic aromatic content of the process oil being produced.

14. The process as in claim 13, further comprising increasing a percentage volume of anti-solvent to solvent within the extraction column to thereby facilitate an increased yield of the process oil being produced.

15. The process as in claim 14, wherein percentage volume of anti-solvent to solvent within the extraction column is up to approximately 12%.

16. The process as in claim 15, wherein percentage volume of anti-solvent to solvent within the extraction column is at least approximately 10%.
17. The process as in claim 14, further comprising decreasing a percentage volume of anti-solvent to solvent within the extraction column to thereby facilitate a decreased polycyclic aromatic content of the process oil being produced.

18. The process as in claim 17, wherein the percentage volume of anti-solvent to solvent within the extraction column less than approximately 5%.

19. The process as in claim 13, wherein the anti-solvent is demineralized water.

20. The process as in claim 1, wherein a temperature within the extraction column is more than approximately 50°C.

21. The process as in claim 1, wherein a temperature within the extraction column is between approximately 80°C and approximately 120°C.

22. The process as in claim 1, wherein the process oil has a kinematic viscosity at 100°C of between approximately 20 mm²/second and 32 mm²/second.

23. The process as in claim 22, wherein the process oil has a density of at least approximately 1.0 kg/liter, a flash point of at least approximately 220°C, an aniline point of at least approximately 40.0°C, and a glass transition temperature of at most approximately -49°C.

24. A process oil with a polycyclic aromatic content of less than approximately 3% by weight, the process oil having a kinematic viscosity at 100°C of between approximately 20 mm²/second and approximately 32 mm²/second, a density of at least approximately 1.010 kg/liter, a flash point of at least approximately 220°C, an aniline point of at least approximately 40.0°C, and a glass transition temperature of at most approximately -49°C.
25. The process oil as in claim 24, wherein the process oil is produced using the process of one of claims 1 to 23.

26. A rubber composition with a polycyclic aromatic content of less than approximately 3% by weight, the rubber composition being manufactured by processing of a rubber compound with a process oil that is produced using the process of one of claims 1 to 23.

27. A system for extracting polycyclic aromatic compounds comprising:

   a countercurrent extraction column;
   a first inlet carried by the extraction column for mediating introduction of a feed oil;
   a second inlet carried by the extraction column for mediating introduction of at least one of a solvent and an anti-solvent;
   a plurality of rotatable plates disposed within the extraction column, each of the plurality of rotatable plates including a plurality of openings through which each of the feed oil, solvent, and anti-solvent can be communicated; and
   a gas bubbling module coupled to the extraction column for introducing a substantially inert gas into the extraction column.

28. The system as in claim 27, the gas bubbling module comprising a controller for controlling at least one of pressure and gas bubble size of the substantially inert gas being introduced into the extraction column.

29. The system as in claim 27, further comprising a controller for controlling a percentage volume of solvent to feed oil introduced within the extraction column.

30. The system as in claim 27, further comprising a controller couplable to the second inlet for controlling a percentage volume of anti-solvent to solvent being introduced into the extraction column.
Introducing feed oil into extraction column

Introducing solvent into the extraction column

Heating the extraction column

Introducing an anti-solvent (e.g., water) into the extraction column

Introducing nitrogen gas into the extraction column

Extraction of PCA from the feed oil

Collection of process oil from the extraction column

Secondary processing of the process oil

FIGURE 1
FIGURE 2
Figure 3: Influence of Type of Process Oil, and Oil Loading, on Mooney Viscosity of Rubber Compositions A to H

Figure 4a: Influence of Type of Process Oil, at a Process Oil Loading of 5phr, on Viscosity-Shear Rate of Rubber Compositions A to H
Figure 4b: Influence of Type of Process Oil, at a Process Oil Loading of 10 phr, on Viscosity-Shear Rate of Rubber Compositions A to H

Figure 4c: Influence of Type of Process Oil, at a Process Oil Loading of 15 phr, on Viscosity-Shear Rate of Rubber Compositions A to H
Figure 5a: Influence of Type of Process Oil, at a Process Oil Loading of 5 phr, on Complex Viscosity-Frequency Correlation of Rubber Compositions A to H

Figure 5b: Influence of Type of Process Oil, at a Process Oil Loading of 10 phr, on Complex Viscosity-Frequency Correlation of Rubber Compositions A to H
Figure 5c: Influence of Type of Process Oil, at a Process Oil Loading of 20phr, on Complex Viscosity-Frequency Correlation of Rubber Compositions A to H

Figure 6a: Influence of Type of Process Oil, at a Process Oil Loading of 5phr, on Storage Modulus-Frequency Correlation of Rubber Compositions A to H
Figure 6b: Influence of Type of Process Oil, at a Process Oil Loading of 10phr, on Storage Modulus-Frequency Correlation of Rubber Compositions A to H

Figure 6c: Influence of Type of Process Oil, at a Process Oil Loading of 20phr, on Storage Modulus-Frequency Correlation of Rubber Compositions A to H
Figure 7a: Influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H

![Graph showing the relationship between time and torque for Figure 7a]

Figure 7b: Influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H

![Graph showing the relationship between time and torque for Figure 7b]
Figure 7c: Influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H

![Graph showing influence of Type of Process Oil, and Oil Loading, on Final Torque of Rubber Compositions A to H.]

Figure 8: Influence of Type of Process Oil, and Oil Loading, on Scorch Time of Rubber Compositions A to H

![Graph showing influence of Type of Process Oil, and Oil Loading, on Scorch Time of Rubber Compositions A to H.]

SUBSTITUTE SHEET (RULE 26)
Figure 9: Influence of Type of Process Oil, and Oil Loading, on Cure Time of Rubber Compositions A to H

Figure 10: Influence of Type of Process Oil, and Oil Loading, on Harness of Rubber Compositions A to H
Figure 11: Influence of Type of Process Oil, and Oil Loading, on Modulus at 100% elongation of Rubber Compositions A to H

Figure 12: Influence of Type of Process Oil, and Oil Loading, on Modulus at 300% elongation of Rubber Compositions A to H
Figure 13: Influence of Type of Process Oil, and Oil Loading, on Tensile Strength of Rubber Compositions A to H

![Graph showing tensile strength (MPa) for different compositions and oil loadings.]

Figure 14: Influence of Type of Process Oil, and Oil Loading, on Elongation at Breaking Point of Rubber Compositions A to H

![Graph showing elongation at break (%) for different compositions and oil loadings.]

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Figure 15: Influence of Type of Process Oil, and Oil Loading, on Temperature Increment under DIN Standard Test of Rubber Compositions A to H

Figure 16: Influence of Type of Process Oil, and Oil Loading, on Volume Loss under Standard Abrasion Test of Rubber Compositions A to H
# INTERNATIONAL SEARCH REPORT

**International application No**
PCT/SG2009/000497

**A. CLASSIFICATION OF SUBJECT MATTER**

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According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

CIOG B01D B01J B01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, BIOSIS, COMPENDEX, INSPEC, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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* Further documents are listed in the continuation of Box C.  
* See patent family annex.

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**Date of the actual completion of the international search**
15 December 2010

**Date of mailing of the international search report**
22/12/2010

**Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-3040, Fax: (+31-70) 340-3016**

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**Nazari o, Luis**
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