DETERMINATION OF THE AMORPHOUS CONTENT OF POLYMERS

BACKGROUND

Xylene Soluble (XS) is an important property for propylene based polymers, widely used to control polypropylene properties. It is related to the atactic, amorphous polymer fraction in polypropylene. The XS is also used to represent the amount of amorphous material in an impact copolymer. The Xylene Soluble weight fraction (or XS%) of an olefin block composite is used to quantify block composite index (BCI) (see, for example, U.S. Patent 8,802,772).

The percent Xylene Soluble (XS%) can be measured by ASTM D 5492-17, through a procedure with several steps. A Flow Injection Polymer Analysis (FIPA) was developed to generate an equivalent XS%; however, for materials with high XS% values and/or with amorphous fractions having a high molecular weight obtaining a reliable XS% for these cases can be challenging due to difficult filtrations.

This is a need for an accurate and fast method to measure the amorphous fraction in a polymer sample, and which can be used to obtain equivalent, or close, values to those obtained by ASTM 5492-17. This need is fulfilled by the following invention.

SUMMARY ~~OF THE INVENTION~~

A method to determine the weight percent of an "amorphous" fraction in an olefin-based polymer composition, comprising one or more olefin-based polymers; said method comprising the following steps:

a) dissolving the olefin-based polymer composition in an organic solvent to form a polymer solution;

b) injecting at least a portion of the polymer solution onto a support material, and wherein the support material has a Co-crystallization Index (CI) value from 0.70 to 1.20;

c) cooling the support material at a rate greater than, or equal to, 0.2°C/min;

d) increasing the temperature of the support material to elute the polymers of the olefin-based polymer composition;

e) generating a chromatogram;

f) determining the peak area of the first elution from its lower integration limit to its upper integration limit;

g) calculating the "amorphous" fraction" based on the following Equation A below:

$$\text{wt\% "amorphous" fraction} = PA_{amorp} / PA_{total} \times 100 \quad \text{(Eqn. A)};$$

wherein PA_{amorp} = peak area of the first elution, and PA_{total} = total peak area of the polymers

of the olefin-based polymer composition.

In one aspect, the present disclosure relates to a method to determine the weight percent of an "amorphous" fraction in an olefin-based polymer composition, comprising one or more olefin-based polymers; said method comprising the following steps:

a) dissolving the olefin-based polymer composition in organic solvent to form a polymer solution;

b) injecting at least a portion of the polymer solution onto a support material;

c) cooling the support material at a rate greater than, or equal to, 0.2°C/min;

d) increasing the temperature of the support material to elute the polymers of the olefin-based polymer composition;

e) generating a chromatogram;

f) determining the peak area of the first elution from its lower integration limit to its upper integration limit;

g) calculating the "amorphous" fraction" based on the following Equation A below:

$$\text{wt\% "amorphous" fraction} = PA_{amorp} / PA_{total} \times 100 \quad \text{(Eqn. A)};$$

wherein PA_{amorp} = peak area of the first elution, and PA_{total} = total peak area of the polymers of the olefin-based polymer composition,

wherein the support material comprises one of the following: a) gold particles, b) gold coated particles, c) particles comprising gold, d) particles comprising a coating comprising gold, e) copper particles, f) copper coated particles, g) particles comprising copper, h) particles comprising a coating comprising copper, i) silver particles, j) silver coated particles, k) particles comprising copper, l) particles comprising a coating comprising silver, or m) a combination thereof.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a TREF chromatogram of DOWLEX 2056A, which is noted for the calculation of A_I. See the discussion of the Co-crystallization Index below.

Figure 2 is a TREF chromatogram of DOWLEX 2056A, which is noted for the calculation of A_0. See the discussion of the Co-crystallization Index below.

Figure 3 is the correlation of elution temperature versus octene content in ethylene-octene copolymer using SM-1 supporting material.

Figure 4 is the chromatogram of BC-2 obtained by using SM-1 supporting material.

Figure 5 is the example of temperature profile using in measuring amorphous fraction.

DETAILED DESCRIPTION

A new “crystallization based chromatography” method has been discovered to accurately determine the amount of amorphous fraction in an olefin-based polymer composition. This method provides very close, or equivalent, results to those obtained by ASTM 5497-17. Moreover, the inventive method require a fraction of the time (for example, less than 18%) of the analysis time needed for ASTM D5497-17. Moreover, the new method can be fully automated.

As discussed above, a method is provided to determine the weight percent of an “amorphous” fraction in a olefin-based polymer composition, comprising one or more olefin-based polymers; said method comprising the following steps:

a) dissolving the olefin-based polymer composition in an organic solvent (for example, dichlorobenzene or trichlorobenzene) to form a polymer solution;

b) injecting at least a portion of the polymer solution onto a support material, and wherein the support material has a Co-crystallization Index (CI) value from 0.70 to 1.20;

c) cooling the support material at a rate greater than, or equal to, 0.2°C/min;

d) increasing the temperature of the support material to elute the polymers of the olefin-based polymer composition;

e) generating a chromatogram (for example, concentration (Intensity) versus temperature);

CLAIMS

1. A method to determine the weight percent of an "amorphous" fraction in an olefin-based polymer composition, comprising one or more olefin-based polymers; said method comprising the following steps:

a) dissolving the olefin-based polymer composition in organic solvent to form a polymer solution;

b) injecting at least a portion of the polymer solution onto a support material~~, and wherein the support material has a Co-crystallization Index (CI) value from 0.70 to 1.20~~;

c) cooling the support material at a rate greater than, or equal to, 0.2°C/min;

d) increasing the temperature of the support material to elute the polymers of the olefin-based polymer composition;

e) generating a chromatogram;

f) determining the peak area of the first elution from its lower integration limit to its upper integration limit;

g) calculating the "amorphous" fraction" based on the following Equation A below:

$$\text{wt\% "amorphous" fraction} = PA_{amorp} / PA_{total} \times 100 \quad \text{(Eqn. A)};$$

wherein PA_{amorp} = peak area of the first elution, and PA_{total} = total peak area of the polymers of the olefin-based polymer composition,

wherein the support material comprises one of the following: a) gold particles, b) gold coated particles, c) particles comprising gold, d) particles comprising a coating comprising gold, e) copper particles, f) copper coated particles, g) particles comprising copper, h) particles comprising a coating comprising copper, i) silver particles, j) silver coated particles, k) particles comprising copper, l) particles comprising a coating comprising silver, or m) a combination thereof.

2. The method of claim 1, wherein the polymer is an olefin-based polymer composition is a "block composite" ("BC") that comprises the following three polymer components:

(i) an ethylene-based polymer (EP) having an ethylene content from 10 mol% to less than 90 mol% (a soft copolymer), based on the total moles of polymerized monomer units in the ethylene-based polymer (EP);

(ii) an alpha-olefin-based polymer (AOP) having an alpha-olefin content of greater than 90 mol% (a hard copolymer), based on the total moles of polymerized monomer units in the alpha-olefin-based polymer (AOP); and

(iii) a block copolymer having an ethylene block (EB) and an alpha-olefin block

(AOB); wherein the ethylene block (soft block/soft segment) contains more polymerized ethylene than the alpha-olefin block (hard block/hard segment), and

wherein the ethylene block has the same or similar Tm as the ethylene-based polymer (EP) of component (i), and

wherein the alpha-olefin block has the same or similar Tm as the alpha-olefin-based polymer (AOP) of component (ii); and

wherein the phrase "same or similar" refers to an absolute Tm differential of ≤ 5°C, further ≤ 4°C, further ≤ 3°C, further ≤ 2°C.

3. The method of claim 1, wherein the polymer is an olefin-based polymer composition is a "crystalline block composite" ("CBC") that comprises the following three polymer components:

(i) a crystalline ethylene based polymer (CEP) having an ethylene content of greater than, or equal to, 90 mol% (also referred to herein as a soft polymer of CBC), based on the total moles of polymerized monomer units in the crystalline ethylene-based polymer (CEP);

(ii) a crystalline alpha-olefin based polymer (CAOP) having an alpha-olefin content of greater than 90 mol% (also referred to herein as a hard polymer of the CBC), based on the total moles of polymerized monomer units in the crystalline alpha-olefin-based polymer (CAOP); and

(iii) a block copolymer comprising a crystalline ethylene block (CEB) and a crystalline alpha-olefin block (CAOB); and

wherein the crystalline ethylene block has the same or similar Tm as the crystalline ethylene-based polymer (CEP) of component (i), and

wherein the crystalline alpha-olefin block has the same or similar Tm as the crystalline alpha-olefin-based polymer (CAOP) of component (ii); and

wherein the phrase "same or similar" refers to an absolute Tm differential of ≤ 5°C, further ≤ 4°C, further ≤ 3°C, further ≤ 2°C.

~~4. The method of any one of the previous claims, wherein the method is selected from the following: i) a crystallization elution fractionation (CEF) chromatography, or ii) a temperature rising elution fractionation (TREF) chromatography.~~

~~5~~4. The method of any one of the previous claims, wherein the support material comprises spherical particles that have D50 value less than, or equal to, 100 microns.

~~6~~5. The method of any one of the previous claims, ~~wherein the support material has a CI from 0.70 to 1.20, and~~ wherein the support material comprises an inert material.

~~7. The method of any one of the previous claims, wherein the support material comprises one of the following: a) gold particles, b) gold coated particles, c) particles comprising gold, d) particles comprising a coating comprising gold, e) copper particles, f) copper coated particles, g) particles comprising copper, h) particles comprising a coating comprising copper, or i) a combination thereof.~~

~~8. The method of any one of the previous claims, wherein the support material comprises one of the following: a) gold particles, b) gold coated particles, c) particles comprising gold, d) particles comprising a coating comprising gold, e) silver particles, f) silver coated particles, g) particles comprising copper, h) particles comprising a coating comprising silver, or i) a combination thereof.~~

~~9~~6. The method of any one of the previous claims, wherein the first elution has a lower integration limit from 10°C to 15°C.

~~10~~7. The method of any one of the previous claims, wherein the first elution has an upper integration limit from 30°C to 45°C, or from 30°C to 40°C.

~~11~~8. The method of any one of the previous claims, wherein the first elution is integrated from 10°C to 40°C, or from 12°C to 40°C, or from 15°C to 40°C.

~~12~~9. The method of any one of the previous claims, wherein the olefin-based polymer composition comprises two olefin-based polymers.

~~13~~10. The method of any one of the previous claims, wherein the olefin-based polymer composition comprises three olefin-based polymers.

~~14~~11. The method of any one of the previous claims, wherein the support material comprises ≥ 90 wt%, or ≥ 95 wt%, or ≥ 98 wt%, or ≥ 99 wt% of the particles, based on the weight of the support material.

~~15~~12. The method of any one of the previous claims, wherein the olefin-based polymer composition comprises 50 wt%, or a majority amount, of polymerized ethylene, based on the weight of the olefin-based polymer composition.