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(71) Applicants: **SK INNOVATION CO., LTD.** [KR/KR]; 26, Jong-ro, Jongno-gu, Seoul 03188 (KR). **SK GEO CENTRIC CO., LTD.** [KR/KR]; 51, Jong-ro, Jongno-gu, Seoul 03161 (KR).

(72) Inventors: **KWON, Ilyoung**; 325 Expo-ro, Yuseong-gu, Daejeon 34124 (KR). **KIM, Taeho**; 325 Expo-ro, Yuseong-gu, Daejeon 34124 (KR). **KIM, Hakbin**; 325 Expo-ro, Yuseong-gu, Daejeon 34124 (KR). **CHEON, Siuk**; 325 Expo-ro, Yuseong-gu, Daejeon 34124 (KR).

(74) Agent: **PLUS INTERNATIONAL IP LAW FIRM**; 10F, 809, Hanbat-daero, Seo-Gu, Daejeon 35209 (KR).

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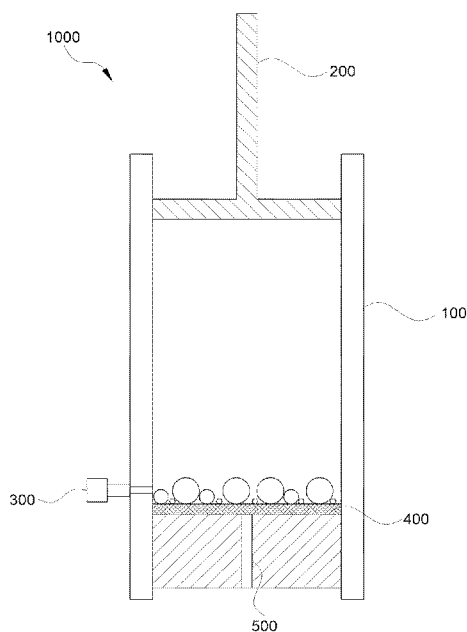
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(54) Title: METHOD FOR MEASURING EASILY EXTRUDABLE PCR RESIN AND PCR RESIN COMPOSITION



(57) Abstract: Provided is a method for measuring an easily extrudable extrusion-moldable PCR resin. Specifically, a method for distinguishing a post-consumer recycled (PCR) resin in a new manner which may distinguish a high grade PCR resin by adding the PCR resin to a capillary rheometer and quantifying a volume value measured when a pressure is increased to a certain level, which is a method for providing determination information of the easily extrudable extrusion-moldable PCR resin.



Description

Title of Invention: METHOD FOR MEASURING EASILY EXTRUDABLE PCR RESIN AND PCR RESIN COMPOSITION

Technical Field

- [1] The present disclosure relates to a method for measuring easily extrudable PCR resin and PCR resin composition

Background Art

- [2] A post-consumer recycled (PCR) resin refers to a recycled resin, and a study for recycling it is currently in progress. The PCR resin is a polymer resin, such as polyethylene (PE), polyethylene terephthalate (PET), polypropylene (PP), poly (vinyl chloride) (PVC), and polystyrene (PS), and the polymer resin is mainly used in clothing, carpets, bottles, plastic lumber, pipes, floor tiles, packaging, fibers, and the like, and thus, the content of a discharged PCR resin is increasing every year.
- [3] Among the PCR resin materials, in the case of stretch wrap used to package large supermarket items of a polyethylene (PE) resin, resin having a large volume and the same properties may be secured in a large amount, and thus, a study for recycling the resin continues.
- [4] However, since a polyethylene PCR resin usually has deteriorated physical properties and processability as compared with a new material polymer resin, it is difficult to use the polyethylene PCR resin again. In particular, the physical properties are greatly deteriorated due to foreign matter inside the resin of the polyethylene PCR resin after processing, and in particular, even when a foreign matter removal filter is separately provided during extrusion, the filter in an extruder is eventually blocked by excessive foreign matter during processing so that continuous processing is very difficult.
- [5] Accordingly, the collected polyethylene PCR resin materials should be differentiated and classified into recyclable grade A resins, non-recyclable C grade resins, and the like, but it is difficult to classify the grade A PCR resin and the grade C PCR resin by visual inspection, ICP elemental analysis, TGA pyrolysis analysis, and the like, and the cost of the analysis is also high.

Disclosure of Invention

Technical Problem

- [6] An embodiment of the present invention is directed to providing a method for determining an easily extrudable PCR resin which enables easy classification of a PCR resin having a foreign matter content at or below a certain level by using a capillary rheometer device of a grade A PCR resin material having a low content of foreign matter.

- [7] Another embodiment of the present invention is directed to providing a PCR resin composition which is easily processed by mixing even a low-grade PCR resin with a new material at an optimal content, since a composition of a mixture of a PCR resin and a new material is measured using a capillary rheometer device.

Solution to Problem

- [8] In one general aspect, a method for measuring an easily extrudable post-consumer recycled (PCR) resin which is a method for providing determination information of the easily extrudable PCR resin is provided, wherein the PCR resin includes a polyolefin resin, a total capacity of a resin at which an initial pressure (P_0) becomes doubled (P_t) when the PCR resin passes through a capillary rheometer satisfies the following Equation 1, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:
- [9] [Equation 1]
- [10] $((V_t - V_a)/V_t) < 0.8$
- [11] wherein V_t is an initial volume (cm^3) of the PCR resin entering the capillary rheometer, and V_a is a volume (cm^3) of a resin filtered by the mesh in the capillary rheometer at which the initial pressure becomes doubled,
- [12] [Equation 2]
- [13] $0.05 \leq M_2/M_1 \leq 0.2$
- [14] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,
- [15] [Equation 3]
- [16] $0.65 \leq WD/SM \leq 0.73$
- [17] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).
- [18] In an exemplary embodiment, the PCR resin may include a polyethylene resin.
- [19] In an exemplary embodiment, the mesh included in the capillary rheometer may be 50 to 200 meshes.
- [20] In an exemplary embodiment, the mesh may have a total permeation area ratio of 30 to 50%.
- [21] In an exemplary embodiment, a pressure applied to the capillary rheometer may be 10 to 1,000 bar.
- [22] In an exemplary embodiment, a cylinder internal temperature of the capillary rheometer may satisfy the following Equation 4:
- [23] [Equation 4]
- [24] $PT_1 + 50^\circ\text{C} < T_1 < PT_1 + 150^\circ\text{C}$
- [25] wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal

temperature of the capillary rheometer.

[26] In an exemplary embodiment, the capillary rheometer may have the piston diameter of 10 to 30 mm and the capillary diameter of 1 to 5 mm.

[27] In another general aspect, a method for measuring an easily extrudable post-consumer recycled (PCR) resin which is a method for providing determination information of an easily extrudable PCR resin composition is provided, wherein the PCR resin includes a polyolefin resin, the following Equation 5 is satisfied when the PCR resin passes through a capillary rheometer, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[28] [Equation 5]

[29] $(\ln(P_L/P_0))/V < 0.16$

[30] [Equation 2]

[31] $0.05 \leq M_2/M_1 \leq 0.2$

[32] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[33] [Equation 3]

[34] $0.65 \leq WD/SM \leq 0.73$

[35] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).

[36] P_0 is an initial internal pressure of the capillary rheometer when the PCR resin is added and a pressure was applied at a volume of 1 cc, and P_L is a pressure finally applied to the capillary rheometer. In addition, V is an internal volume of the capillary rheometer decreased by additional pressure from P_0 , and V refers to a volume until a P_L value becomes twice a P_0 value. When the P_L value is more than twice the P_0 value, V is measured with P_L being limited to twice P_0 . In addition, when P_L is less than twice P_0 , V is set as a final measured volume.

[37] In an exemplary embodiment, the PCR resin may include a new material.

[38] In an exemplary embodiment, the mesh included in the capillary rheometer may be 50 to 200 meshes.

[39] In an exemplary embodiment, the mesh may have a total permeation area ratio of 35 to 50%.

[40] In an exemplary embodiment, a cylinder internal temperature of the capillary rheometer satisfies the following Equation 4:

[41] [Equation 4]

[42] $PT_1+50^\circ\text{C} < T_1 < PT_1+150^\circ\text{C}$

[43] wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal temperature of the capillary rheometer.

[44] In an exemplary embodiment, the capillary rheometer may have the piston diameter

of 10 to 30 mm and the capillary diameter of 1 to 5 mm.

[45] In another general aspect, an excellently melt-extrudable post-consumer recycled (PCR) resin is provided, wherein a total capacity of a resin at which an initial pressure (P_0) becomes doubled (P_L) when the PCR resin passes through a capillary rheometer satisfies the following Equation 1, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[46] [Equation 1]

[47] $((V_t - V_a))/V_t < 0.8$

[48] wherein V_t is an initial volume (cm^3) of the PCR resin entering the capillary rheometer, and V_a is a volume (cm^3) of a resin filtered by the mesh in the capillary rheometer at which the initial pressure becomes doubled,

[49] [Equation 2]

[50] $0.05 \leq M_2/M_1 \leq 0.2$

[51] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[52] [Equation 3]

[53] $0.65 \leq WD/SM \leq 0.73$

[54] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).

[55] In still another general aspect, a post-consumer recycled (PCR) resin composition is provided, wherein the PCR resin includes a polyolefin resin, the following Equation 5 is satisfied when the PCR resin passes through a capillary rheometer, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[56] [Equation 5]

[57] $(\ln(P_L/P_0))/V < 0.16$

[58] [Equation 2]

[59] $0.05 \leq M_2/M_1 \leq 0.2$

[60] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[61] [Equation 3]

[62] $0.65 \leq WD/SM \leq 0.73$

[63] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).

[64] P_0 is an initial internal pressure of the capillary rheometer when the PCR resin is added and a pressure was applied at a volume of 1 cc, and P_L is a pressure finally applied to the capillary rheometer. In addition, V is an internal volume of the capillary rheometer decreased by additional pressure from P_0 , and V refers to a volume until a P_L value becomes twice a P_0 value. When the P_L value is more than twice the P_0 value, V

is measured with P_L being limited to twice P_0 . In addition, when P_L is less than twice P_0 , V is set as a final measured volume.

[65] In an exemplary embodiment, the PCR resin composition may include a new material.

[66] Other features and aspects will be apparent from the following detailed description, the drawings, and the claims.

Advantageous Effects of Invention

[67] The method for measuring a PCR resin of the present disclosure may easily determine a continuously extrudable PCR resin material by deriving a correlation between a foreign matter and an extrusion pressure of the PCR resin using a capillary rheometer device.

[68] In addition, the present disclosure may provide a PCR resin composition which is easily processed by mixing even a low-grade PCR resin with a new material at an optimal content, since a composition of a mixture of a low-grade PCR resin and a new material is measured using a capillary rheometer device.

Brief Description of Drawings

[69] FIG. 1 is a rough schematic diagram of a capillary rheometer device.

[70] FIG. 2 is a graph in which a PCR resin volume to reach an optional pressure using the capillary rheometer device is schematized.

[71] FIG. 3 is a graph of measuring a pressure state occurring when a piston is pressed at a constant speed in the capillary rheometer device.

[72] FIG. 4 is a graph showing a value depending on a PCR resin content in a PCR resin composition using the capillary rheometer device.

Best Mode for Carrying out the Invention

[73] Hereinafter, the present disclosure will be described in more detail with reference to specific examples and exemplary embodiments including the accompanying drawings. However, the following specific examples and exemplary embodiments are only a reference for describing the present disclosure in detail, and the present disclosure is not limited thereto and may be implemented in various forms.

[74] In addition, unless otherwise defined, all technical terms and scientific terms have the same meanings as those commonly understood by one of those skilled in the art to which the present disclosure pertains. The terms used herein are only for effectively describing a certain specific example, and are not intended to limit the present disclosure.

[75] In addition, the singular form used in the specification and claims appended thereto may be intended to include a plural form also, unless otherwise indicated in the context.

- [76] In addition, unless particularly described to the contrary, “any elements will be understood to imply further inclusion of other elements rather than the exclusion of any other elements.
- [77] Conventionally collected polyethylene PCR resin materials should be differentiated and classified into recyclable grade A resins, non-recyclable C grade resins, and the like, but it is difficult to classify the grade A PCR resin and the grade C PCR resin materials by visual inspection, ICP elemental analysis, TGA pyrolysis analysis, and the like, and the cost of the analysis is also high.
- [78] Accordingly, the present disclosure provides a method for measuring an easily extrudable post-consumer recycled (PCR) resin which is a method for providing determination information of the easily extrudable grade A PCR resin is provided, wherein the PCR resin includes a polyolefin resin, a total capacity of a resin at which an initial pressure (P_0) becomes doubled (P_i) when the PCR resin passes through a capillary rheometer satisfies the following Equation 1, a ratio between the piston diameter and the capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3.
- [79] In addition, the polyolefin resin may be any one or more polyolefin resins selected from polyethylene, polypropylene, polyketone, polypropylene copolymer, polymethylpentene, polystyrene, and polyvinyl chloride, specifically, may be any one or more polyolefin resins selected from polyethylene, polypropylene, and polystyrene, and specifically, may be a polyethylene resin, but is not limited thereto.
- [80] According to an exemplary embodiment of the present disclosure, the polyethylene resin may be any one or more polyethylene resins selected from low density polyethylene (LDPE), linear low-density polyethylene (LLDPE), crosslinked high-density polyethylene (XLPE), and high-density polyethylene, but is not limited thereto:
- [81] [Equation 1]
- [82] $((V_i - V_a))/V_i < 0.8$
- [83] wherein V_i is an initial volume (cm^3) of the PCR resin entering the capillary rheometer, V_a is a volume (cm^3) of a resin filtered by the mesh from the capillary rheometer at which the initial pressure becomes doubled, and when all resin inside the capillary rheometer is discharged without reaching twice the pressure, a V_a value is set as a V_i ,
- [84] [Equation 2]
- [85] $0.05 \leq M_2/M_1 \leq 0.2$
- [86] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,
- [87] [Equation 3]
- [88] $0.65 \leq WD/SM \leq 0.73$

- [89] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).
- [90] A high-quality continuously extrudable PCR resin material may be easily determined without particular analysis and device in the collected PCR resin, by the method for measuring an easily extrudable PCR resin.
- [91] Now, the method of measuring an easily extrudable PCR resin will be described using FIG. 1 as an example. However, FIG. 1 is only an example for describing the capillary rheometer of the present disclosure in detail, and the present disclosure is not limited to FIG. 1.
- [92] The capillary rheometer device 1,000 includes a cylinder body 100, a piston 200, a pressure gauge 300, a mesh 400, and a capillary die 500, but the constituents may be further added, if necessary.
- [93] A PCR resin material is added to the capillary rheometer device 1000, the cylinder body 100 is heated until the PCR resin material is melted, and then the piston 200 is pressed at a certain speed to discharge a melted PCR resin through the capillary of the capillary die 500.
- [94] The certain speed of the piston may be 0.01 to 0.5 mmsec^{-1} , 0.05 to 0.3 mmsec^{-1} , or 0.08 to 0.2 mmsec^{-1} but is not limited thereto.
- [95] Since the mesh 400 is installed in a lower portion of the cylinder body 100, when the melted PCR resin is discharged, foreign matter inside the PCR resin blocks the hole of the mesh and the internal pressure of the cylinder body 100 is increased. Herein, as the foreign matter content is high, the pressure is rapidly increased even when the piston 200 is pressed a little, and when the pressure is excessively applied, the mesh may burst.
- [96] That is, in the present disclosure, a continuously extrudable PCR resin may be distinguished by calculating a time taken to reach a certain pressure or a PCR resin volume inside the cylinder body, depending on the content of foreign matter.
- [97] When the PCR resin satisfying the following Equation 1 of the present disclosure is used, it is continuously extrudable and a PCR resin having excellent physical properties may be distinguished. Specifically, in the case of a PCR resin having a value depending on a pressure in the following Equation 1 of 0.8 or more, a mesh for filtering foreign matter in extrusion in an extruder may be broken due to excessive pressure, and even when a film is manufactured by extrusion, the physical properties of the film may be greatly deteriorated due to excessive foreign matter. However, in the case of a PCR resin having a value depending on the pressure in the following Equation 1 of less than 0.8, extrusion is well performed, and when a film is manufactured, film manufacture by excessive foreign matter due to an excessive amount is suppressed, and thus, mechanical properties such as tensile strength are excellent. The following Equation 1 may be less than 0.8, specifically, the upper limit may be 0.7 or

less, 0.6 or less, 0.5 or less, 0.4 or less, 0.3 or less, 0.2 or less, or 0.1 or less and the lower limit may be 0.0 or more, 0.001 or more, and specifically, 0.0 or more and less than 0.8, specifically 0.001 or more and less than 0.8, and specifically 0.01 to 0.7, but the present disclosure is not limited thereto.

[98] As the following Equation 1 is close to 0, the foreign matter content is almost 0, and when the following V_t value and the V_a value are the same, it may have equivalent quality to a new material having almost no foreign matter:

[99] [Equation 1]

[100] $((V_t - V_a))/V_t < 0.8$

[101] wherein V_t is an initial volume (cm^3) of the PCR resin entering the capillary rheometer, V_a is a volume (cm^3) of a resin filtered by the mesh from the capillary rheometer at which the initial pressure becomes doubled, and when all resin inside the capillary rheometer is filtered without reaching twice the pressure, a V_a value is set as a V_t .

[102] A standard of the capillary rheometer device 1000 from which Equation 1 is derived may be a device satisfying the following Equations 2 and 3:

[103] [Equation 2]

[104] $0.05 \leq M_2/M_1 \leq 0.2$

[105] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[106] [Equation 3]

[107] $0.65 \leq WD/SM \leq 0.73$

[108] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).

[109] A PCR resin satisfying Equation 1 using the capillary rheometer device 1000 in which a piston diameter and a capillary diameter of the capillary rheometer device 1000 satisfy Equation 2 and a wire diameter of the mesh 400 and a sieve mesh size are based on Equation 3 has excellent continuous extrudability and when it is processed into a film, its physical properties are also excellent.

[110] According to an exemplary embodiment of the present disclosure, the PCR resin may include a polyethylene resin, and since the polyethylene resin PCR resin which is distinguished as a PCR resin satisfying Equation 1 using the capillary rheometer device 1000 satisfying Equations 2 and 3 is extrudable, a polyethylene PCR resin product which has excellent physical properties and is continuously extrudable without blockage may be provided.

[111] According to an exemplary embodiment of the present disclosure, the mesh included in the capillary rheometer may have an upper limit of 200 meshes or less and a lower limit of 50 meshes or more, specifically 50 to 200 meshes, but is not limited thereto.

[112] According to an exemplary embodiment of the present disclosure, the mesh may have a total permeation area ratio of 30 to 50%, specifically 50% or less, 48% or less,

45% or less, 43% or less, 42% or less, 41% or less, or 40% or less as an upper limit and 30% or more, 33% or more, 36% or more, 37% or more, 38% or more, or 39% or more as a lower limit, specifically 37 to 48%, specifically 38 to 40%, but is not limited thereto.

[113] According to an exemplary embodiment of the present disclosure, a pressure applied to the capillary rheometer may be 10 to 1,000 bar, specifically 10 to 500 bar, and specifically 30 to 100 bar, but is not limited thereto.

[114] According to an exemplary embodiment of the present disclosure, a cylinder internal temperature of the capillary rheometer may satisfy the following Equation 4, but it is not limited thereto as long as it is in a melting temperature range of a polymer, and as an example, may be 300°C or lower, 250°C or lower, or 200°C or lower as an upper limit and may be 50°C or higher, 100°C or higher, or 150°C or higher as a lower limit, and specifically, 50 to 300°C, specifically 100 to 300°C, specifically 150 to 300°C, specifically 150 to 250°C, and specifically 150 to 230°C, but is not limited thereto:

[115] [Equation 4]

[116] $PT_1 + 50^\circ\text{C} < T_1 < PT_1 + 150^\circ\text{C}$

[117] wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal temperature of the capillary rheometer.

[118] According to an exemplary embodiment of the present disclosure, the piston diameter of the capillary rheometer may be 10 to 30 mm, specifically 12 to 25 mm and the capillary diameter may be 1 to 5 mm, specifically 1.5 to 3 mm, but the present disclosure is not limited thereto.

[119] The present disclosure may separate and provide an excellently melt-extrudable post-consumer recycled (PCR) resin is provided, wherein a total capacity of a resin at which an initial pressure (P_0) becomes doubled (P_t) when the PCR resin passes through a capillary rheometer satisfies the following Equation 1, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3. Since the PCR resin having excellent melt extrudability satisfies the following Equation 1, a recycled film having excellent physical properties and also excellent processability may be provided. Since the capillary rheometer is described in detail above, the description thereof will be omitted here:

[120] [Equation 1]

[121] $((V_t - V_a))/V_t < 0.8$

[122] wherein V_t is an initial volume (cm^3) of the PCR resin entering the capillary rheometer, and V_a is a volume (cm^3) of a resin filtered by the mesh in the capillary rheometer at which the initial pressure becomes doubled,

- [123] [Equation 2]
- [124] $0.05 \leq M_2/M_1 \leq 0.2$
- [125] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,
- [126] [Equation 3]
- [127] $0.65 \leq WD/SM \leq 0.73$
- [128] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).
- [129] Since Equations 1 to 3 are described in detail above, the description thereof will be omitted here.
- [130] According to an exemplary embodiment of the present disclosure, the PCR resin may include a polyethylene resin, but since it is also described above, the description thereof will be omitted here.
- [131] According to an exemplary embodiment of the present disclosure, the mesh included in the capillary rheometer may be 50 to 200 meshes, but since it is also described above, the description thereof will be omitted here.
- [132] According to an exemplary embodiment of the present disclosure, the mesh may have a total permeation area ratio of 35 to 50%, but since it is also described above, the description thereof will be omitted here.
- [133] According to an exemplary embodiment of the present disclosure, a pressure applied to the capillary rheometer may be 10 to 1,000 bar, but since it is also described above, the description thereof will be omitted here.
- [134] According to an exemplary embodiment of the present disclosure, a cylinder internal temperature of the capillary rheometer may satisfy the following Equation 4, but since it is also described above, the description thereof will be omitted here:
- [135] [Equation 4]
- [136] $PT_1 + 50^\circ\text{C} < T_1 < PT_1 + 150^\circ\text{C}$
- [137] wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal temperature of the capillary rheometer.
- [138] According to an exemplary embodiment of the present disclosure, the capillary rheometer may have the piston diameter of 10 to 30 mm and the capillary diameter of 1 to 5 mm, but since it is also described above, the description thereof will be omitted here.
- [139] The PCR resin having excellent melt extrudability may be used alone by melt extrusion, or may be used with a new material further mixed with the PCR resin.
- [140] The new material may be the polyolefin-based resin described above, but is not limited thereto.
- [141] According to another exemplary embodiment of the present disclosure, an easily extrudable post-consumer recycled (PCR) resin composition may be provided, wherein the PCR resin includes a polyolefin resin, the following Equation 5 is satisfied when

the PCR resin passes through a capillary rheometer, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[142] [Equation 5]

[143] $(\ln(P_L/P_0))/V < 0.16$

[144] [Equation 2]

[145] $0.05 \leq M_2/M_1 \leq 0.2$

[146] wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[147] [Equation 3]

[148] $0.65 \leq WD/SM \leq 0.73$

[149] wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).

[150] P_0 is an initial internal pressure of the capillary rheometer when the PCR resin is added and a pressure was applied at a volume of 1 cc, and P_L is a pressure finally applied to the capillary rheometer. In addition, V is an internal volume of the capillary rheometer decreased by additional pressure from P_0 , and V refers to a volume until a P_L value becomes twice a P_0 value. When the P_L value is more than twice the P_0 value, V is measured with P_L being limited to twice P_0 . In addition, when P_L is less than twice P_0 , V is set as a final measured volume.

[151] Since Equations 2, 3, and 5 are described in detail above, the description thereof will be omitted here.

[152] According to an exemplary embodiment of the present disclosure, the PCR resin may include a polyethylene resin, but since it is also described above, the description thereof will be omitted here.

[153] According to an exemplary embodiment of the present disclosure, the mesh included in the capillary rheometer may be 50 to 200 meshes, but since it is also described above, the description thereof will be omitted here.

[154] According to an exemplary embodiment of the present disclosure, the mesh may have a total permeation area ratio of 35 to 50%, but since it is also described above, the description thereof will be omitted here.

[155] According to an exemplary embodiment of the present disclosure, a pressure applied to the capillary rheometer may be 10 to 1,000 bar, but since it is also described above, the description thereof will be omitted here.

[156] According to an exemplary embodiment of the present disclosure, a cylinder internal temperature of the capillary rheometer may satisfy the following Equation 4, but since it is also described above, the description thereof will be omitted here:

[157] [Equation 4]

[158] $PT_1+50^\circ\text{C} < T_1 < PT_1+150^\circ\text{C}$

[159] wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal temperature of the capillary rheometer.

[160] According to an exemplary embodiment of the present disclosure, the capillary rheometer may have the piston diameter of 10 to 30 mm and the capillary diameter of 1 to 5 mm, but since it is also described above, the description thereof will be omitted here.

[161] Hereinafter, the examples of the present disclosure will be further described with reference to the specific experimental examples. It is apparent to those skilled in the art that the examples and the comparative examples included in the experimental examples only illustrate the present disclosure and do not limit the appended claims, and various modifications and alterations of the examples may be made within the range of the scope and spirit of the present invention, and these modifications and alterations will fall within the appended claims.

[162]

[163] [Examples and Comparative Examples]

[164] An increased pressure occurring in a sample and a flow rate of a sample which passed through a capillary rheometer die were measured using a capillary rheometer (RHEOGRAPH 25, Gottfert).

[165] The measurement was performed using a capillary die having a diameter of 2.0 mm and a length of 25 mm. In addition, the diameter of the piston of the rheometer device was 15 mm and a length of 250 mm, and a 120 mesh filter having a sieve size of 0.13 mm, a wire diameter of 0.08 mm, and an opening ratio of 38.3% was mounted on the upper portion of the capillary die.

[166] A capillary die on which a 120 mesh filter is attached was mounted on the lower end of the barrel of the rheometer device and the temperature was maintained at a die test temperature of 190°C. When a PCR resin sample was loaded in a barrel area of the rheometer, air trapped in the sample was all removed so that a volume of 40 cm³ or more was loaded, and then the sample was extruded at a selected piston descending speed of 0.1 mmsec⁻¹ through a capillary die in a barrel. When the sample was processed by passing through a capillary die in the barrel, the pressure applied to the sample and the amount of the sample filtered through the capillary die on which the mesh was mounted were recorded over time to confirm a pressure increase tendency by foreign matter. The results are listed in the following Table 1:

[167] [Table 1]

	Exempl e 1	Exempl e 2	Exempl e 3	Exempl e 4	Examp le 5	Comparativ e Example 1

Type of resin		PCR A-1	PCR A-2	PCR A-3	PCR A-4	PCR A-5	PCR C
Volume (cm ³)	1.5 times the initial pressure	13.13	19.20	12.26	13.40	≥ 40	2.50
	Later volume/initial volume	0.672	0.52	0.694	0.665	0	0.938
	2.0 times the initial pressure	18.88	35.43	24.62	20.57	≥ 40	4.48
	Later volume/initial volume	0.528	0.114	0.385	0.486	0	0.888

[168] The resins in Table 1 and FIG. 1 are those collected by each company, and as a result of extruding the PCR resin under the capillary rheometer conditions of the present disclosure, all except Comparative Example 1 satisfied the conditions of Equation 1. In addition, in FIG. 2 also, it was confirmed that in Comparative Example 1, when the PCR resin was pushed out at a certain speed, the pressure was rapidly increased.

[169] In addition, for the PCR resin composition, a mixed composition of new material polyethylene resin with the PCR resin of the comparative example was added to a capillary rheometer and then the measurement was performed in the same manner as in the comparative example. Content ranges of the new material in the PCR resin are shown in Table 1 and FIG. 4:

[170] [Table 2]

	PCR resin: PCR C					
PCR resin content	10 wt%	15 wt%	25 wt%	50 wt%	75 wt%	100 wt%
a value	0.01295	0.02365	0.04355	0.07203	0.11485	0.1598

[171] Each measurement was performed with the capillary rheometer device in a range of 10 to 100 wt% of the PCR resin content in the PCR resin composition, and measurements were substituted into Equation 5 to derive the value. As shown in Table 2 and FIG. 4, it is confirmed that the value is linearly related to the PCR resin content ratio, and extrusion processing is performed well from the PCR resin content of 75 wt% or less. Thus, it is shown that when a resin is processed, specifically a thin film and the like are manufactured, a PCR resin mixing ratio may be identified in advance through Equation 5.

[172] As a result, in the present disclosure, it was identified that extrusion processing is not performed well due to the foreign matter in a PCR resin and a method for separating a

PCR resin which is extrusion-processed well, which may identify it in advance, is described, and in particular, even for a low grade PCR resin or a composition mixed with a new material, a method for separating a PCR resin composition which may identify a critical point of a PCR resin content at which extrusion processing is well performed in advance is described.

[173] Hereinabove, although the present disclosure has been described by specific matters, limited implementations, and drawings, they have been provided only for assisting the entire understanding of the present disclosure, and the present disclosure is not limited to the implementations, and various modifications and changes may be made by those skilled in the art to which the present disclosure pertains from the description.

[174] Therefore, the spirit of the present disclosure should not be limited to the above-described exemplary embodiments, and the following claims as well as all modified equally or equivalently to the claims are intended to fall within the scope and spirit of the disclosure.

[175]

[176] [Detailed Description of Main Elements]

[177] 1000: Capillary rheometer device

[178] 100: Cylinder body

[179] 200: Piston

[180] 300: Pressure gauge

[181] 400: Mesh

[182] 500: Capillary die

Claims

- [Claim 1] A method for measuring an easily extrudable post-consumer recycled (PCR) resin which is a method for providing determination information of the easily extrudable PCR resin,
wherein the PCR resin includes a polyolefin resin,
a total capacity of a resin at which an initial pressure (P0) becomes doubled (Pt) when the PCR resin passes through a capillary rheometer satisfies the following Equation 1,
a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:
[Equation 1]
$$((V_t - V_a)) / V_t < 0.8$$

wherein V_t is an initial volume (cm³) of the PCR resin entering the capillary rheometer, and V_a is a volume (cm³) of a resin filtered by the mesh in the capillary rheometer at which the initial pressure becomes doubled,
[Equation 2]
$$0.05 \leq M_2 / M_1 \leq 0.2$$

wherein M_1 is a piston diameter, and M_2 is a capillary diameter,
[Equation 3]
$$0.65 \leq WD / SM \leq 0.73$$

wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).
- [Claim 2] The method for measuring an easily extrudable PCR resin of claim 1, wherein the PCR resin includes a polyethylene resin.
- [Claim 3] The method for measuring an easily extrudable PCR resin of claim 1, wherein the mesh included in the capillary rheometer is 50 to 200 meshes.
- [Claim 4] The method for measuring an easily extrudable PCR resin of claim 3, wherein the mesh has a total permeation area ratio of 30 to 50%.
- [Claim 5] The method for measuring an easily extrudable PCR resin of claim 1, wherein a pressure applied to the capillary rheometer is 10 to 1,000 bar.
- [Claim 6] The method for measuring an easily extrudable PCR resin of claim 1, wherein a cylinder internal temperature of the capillary rheometer satisfies the following Equation 4:

[Equation 4]

$$PT_1 + 50^{\circ}\text{C} < T_1 < PT_1 + 150^{\circ}\text{C}$$

wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal temperature of the capillary rheometer.

[Claim 7] The method for measuring an easily extrudable PCR resin of claim 1, wherein the capillary rheometer has the piston diameter of 10 to 30 mm and the capillary diameter of 1 to 5 mm.

[Claim 8] A method for measuring an easily extrudable post-consumer recycled (PCR) resin which is a method for providing determination information of an easily extrusion-moldable PCR resin composition, wherein the PCR resin includes a polyolefin resin, the following Equation 5 is satisfied when the PCR resin passes through a capillary rheometer, a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[Equation 2]

$$0.05 \leq M_2/M_1 \leq 0.2$$

wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[Equation 3]

$$0.65 \leq WD/SM \leq 0.73$$

wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm),

[Equation 5]

$$(\ln(P_L/P_0))/V < 0.16$$

wherein P_0 is an initial internal pressure of the capillary rheometer when the PCR resin is added and a pressure was applied at a volume of 1 cc, P_L is a pressure finally applied to the capillary rheometer, and V is an internal volume of the capillary rheometer decreased by additional pressure from P_0 , refers to a volume until a P_L value becomes twice a P_0 value, is measured with P_L being limited to twice P_0 when the P_L value is more than twice the P_0 value, and is set as a final measured volume when P_L is less than twice P_0 .

[Claim 9] The method for measuring an easily extrudable PCR resin of claim 8, wherein the PCR resin includes a new material.

[Claim 10] The method for measuring an easily extrudable PCR resin of claim 8, wherein the mesh included in the capillary rheometer is 50 to 200

meshes.

[Claim 11] The method for measuring an easily extrudable PCR resin of claim 8, wherein the mesh has a total permeation area ratio of 35 to 50%.

[Claim 12] The method for measuring an easily extrudable PCR resin of claim 8, wherein a cylinder internal temperature of the capillary rheometer satisfies the following Equation 4:

[Equation 4]

$$PT_1 + 50^{\circ}\text{C} < T_1 < PT_1 + 150^{\circ}\text{C}$$

wherein PT_1 is a melting temperature of the PCR resin, and T_1 is the cylinder internal temperature of the capillary rheometer.

[Claim 13] The method for measuring an easily extrudable PCR resin of claim 8, wherein the capillary rheometer has the piston diameter of 10 to 30 mm and the capillary diameter of 1 to 5 mm.

[Claim 14] An excellently melt-extrudable post-consumer recycled (PCR) resin, wherein a total capacity of a resin at which an initial pressure (P_0) becomes doubled (P_i) when the PCR resin passes through a capillary rheometer satisfies the following Equation 1,

a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[Equation 1]

$$((V_t - V_a))/V_t < 0.8$$

wherein V_t is an initial volume (cm^3) of the PCR resin entering the capillary rheometer, and V_a is a volume (cm^3) of a resin filtered by the mesh in the capillary rheometer at which the initial pressure becomes doubled,

[Equation 2]

$$0.05 \leq M_2/M_1 \leq 0.2$$

wherein M_1 is a piston diameter, and M_2 is a capillary diameter,

[Equation 3]

$$0.65 \leq WD/SM \leq 0.73$$

wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm).

[Claim 15] An easily extrudable post-consumer recycled (PCR) resin composition, wherein the PCR resin includes a polyolefin resin, the following Equation 5 is satisfied when the PCR resin passes through a capillary rheometer,

a ratio between a piston diameter and a capillary diameter in the capillary rheometer satisfies the following Equation 2, and a sieve mesh size and a wire diameter of a mesh included in the capillary rheometer satisfy the following Equation 3:

[Equation 2]

$$0.05 \leq M_2/M_1 \leq 0.2$$

wherein M_1 is a barrel diameter, and M_2 is a capillary diameter,

[Equation 3]

$$0.65 \leq WD/SM \leq 0.73$$

wherein WD is a wire diameter (mm), and SM is a sieve mesh size (mm),

[Equation 5]

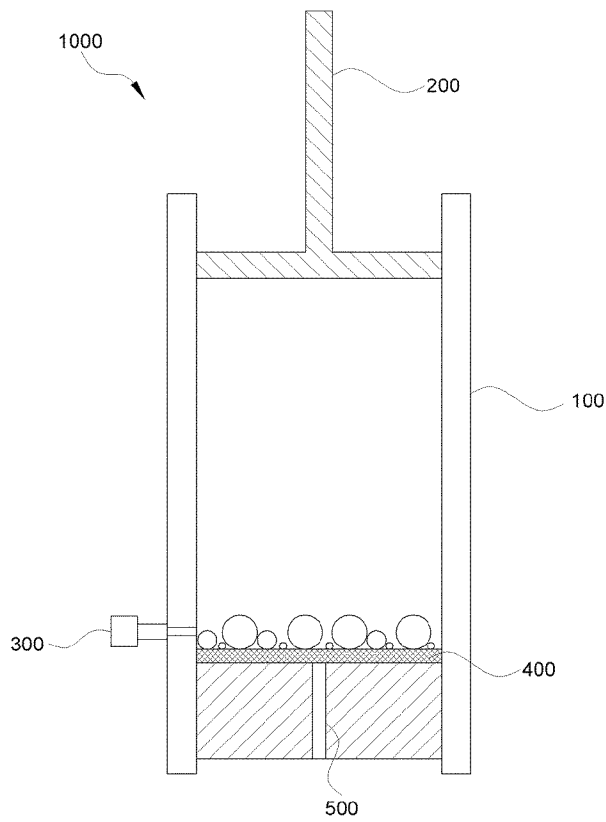
$$(\ln(P_L/P_0))/V < 0.16$$

wherein P_0 is an initial internal pressure of the capillary rheometer when the PCR resin is added and a pressure was applied at a volume of 1 cc, P_L is a pressure finally applied to the capillary rheometer, and V is an internal volume of the capillary rheometer decreased by additional pressure from P_0 , refers to a volume until a P_L value becomes twice a P_0 value, is measured with P_L being limited to twice P_0 when the P_L value is more than twice the P_0 value, and is set as a final measured volume when P_L is less than twice P_0 .

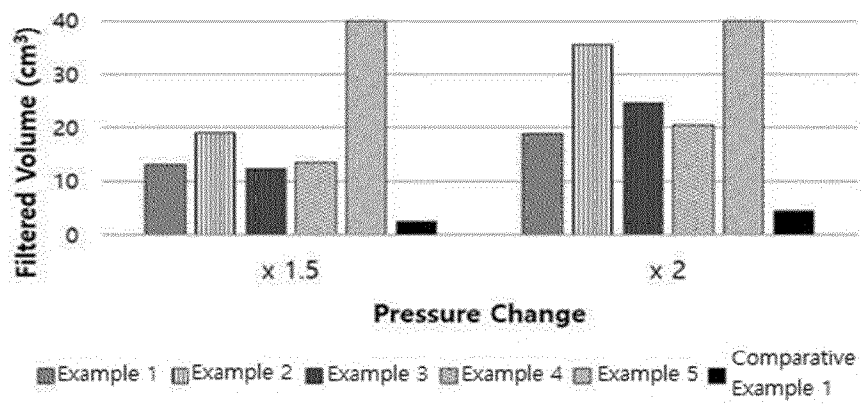
[Claim 16]

The easily extrudable PCR resin composition of claim 15, wherein the PCR resin composition includes a new material.

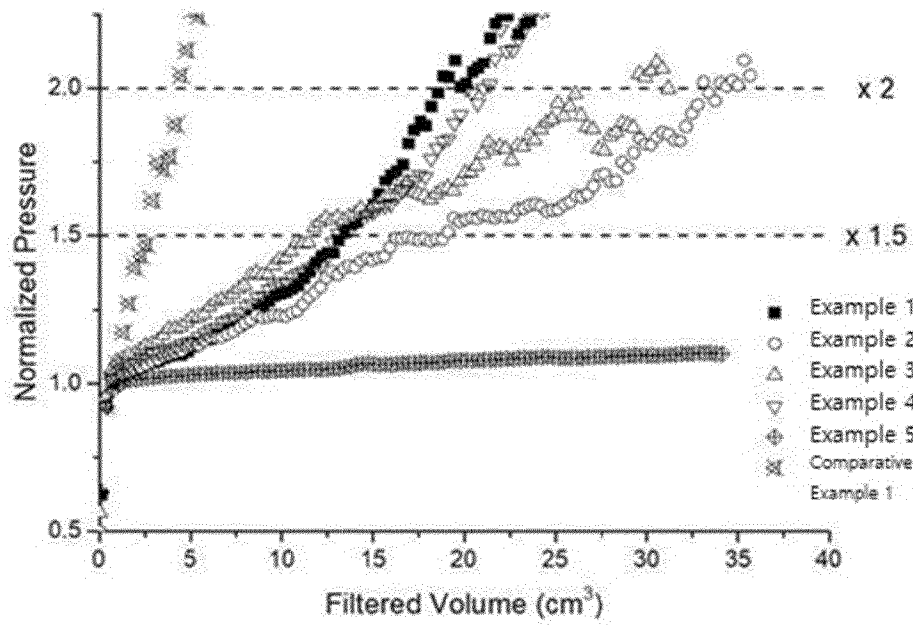
[Fig. 1]



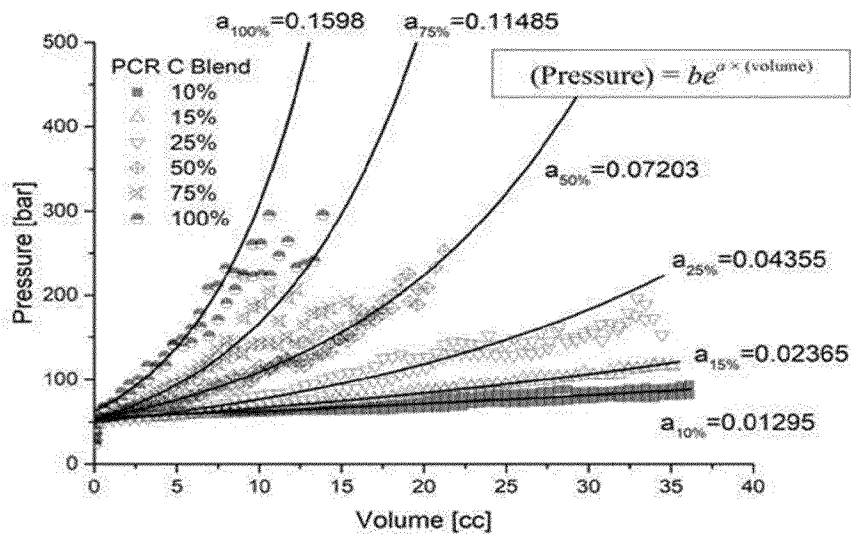
[Fig. 2]



[Fig. 3]



[Fig. 4]



INTERNATIONAL SEARCH REPORT

International application No.

PCT/KR2024/095673

A. CLASSIFICATION OF SUBJECT MATTER G01N 33/44(2006.01)i 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) G01N 33/44(2006.01); B29C 45/14(2006.01); B29C 48/16(2019.01); B29C 48/92(2019.01); B32B 27/36(2006.01); C08F 110/02(2006.01); C08G 75/02(2006.01); C08L 23/06(2006.01); C08L 23/12(2006.01); C08L 53/00(2006.01) Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Korean utility models and applications for utility models Japanese utility models and applications for utility models Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) eKOMPASS(KIPO internal) & Keywords: polyolefine, recycling, capillary rheometer, sieve mesh size, wire diameter		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	KR 10-2022-0124413 A (LG CHEMICAL LTD.) 14 September 2022 (2022-09-14) claims 1-10	1-16
A	KR 10-1781607 B1 (SK CHEMICALS CO., LTD.) 26 September 2017 (2017-09-26) whole document	1-16
A	KR 10-0338843 B1 (SUMITOMO CHEMICAL CO.) 23 November 2002 (2002-11-23) whole document	1-16
A	KR 10-2395347 B1 (HANWHA TOTAL PETROCHEMICAL CO., LTD.) 09 May 2022 (2022-05-09) whole document	1-16
A	KR 10-1728926 B1 (TEIJIN LTD.) 20 April 2017 (2017-04-20) whole document	1-16
☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents: “A” document defining the general state of the art which is not considered to be of particular relevance “D” document cited by the applicant in the international application “E” earlier application or patent but published on or after the international filing date “L” document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) “O” document referring to an oral disclosure, use, exhibition or other means “P” document published prior to the international filing date but later than the priority date claimed “T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention “X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone “Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art “&” document member of the same patent family		
Date of the actual completion of the international search 16 July 2024		Date of mailing of the international search report 17 July 2024
Name and mailing address of the ISA/KR Korean Intellectual Property Office 189 Cheongsa-ro, Seo-gu, Daejeon 35208, Republic of Korea Facsimile No. +82-42-481-8578		Authorized officer HEO, Joo Hyung Telephone No. +82-42-481-5373

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/KR2024/095673

Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
KR	10-2022-0124413	A	14 September 2022	None			
KR	10-1781607	B1	26 September 2017	EP	2546279	A2	16 January 2013
				EP	2546279	A4	22 July 2015
				EP	2546279	B1	23 June 2021
				JP	2013-522385	A	13 June 2013
				JP	2016-156025	A	01 September 2016
				JP	5969925	B2	17 August 2016
				JP	6108579	B2	05 April 2017
				KR	10-2011-0102185	A	16 September 2011
				US	2012-0322971	A1	20 December 2012
				US	2015-0152227	A1	04 June 2015
				US	9249263	B2	02 February 2016
				US	9422401	B2	23 August 2016
				WO	2011-111940	A2	15 September 2011
				WO	2011-111940	A3	23 February 2012
KR	10-0338843	B1	23 November 2002	CN	100069329	C	08 August 2001
				CN	100122346	A	15 May 1996
				JP	08-231816	A	10 September 1996
				JP	3548632	B2	28 July 2004
				KR	10-1996-0004439	A	23 February 1996
				US	5616627	A	01 April 1997
KR	10-2395347	B1	09 May 2022	CN	114381061	A	22 April 2022
				CN	114381061	B	12 September 2023
				KR	10-2022-0052133	A	27 April 2022
KR	10-1728926	B1	20 April 2017	EP	2468502	A1	27 June 2012
				JP	2013-021721	A1	24 January 2013
				JP	5798922	B2	21 October 2015
				KR	10-2012-0056823	A	04 June 2012
				US	2012-0148847	A1	14 June 2012
				WO	2011-021721	A1	24 February 2011