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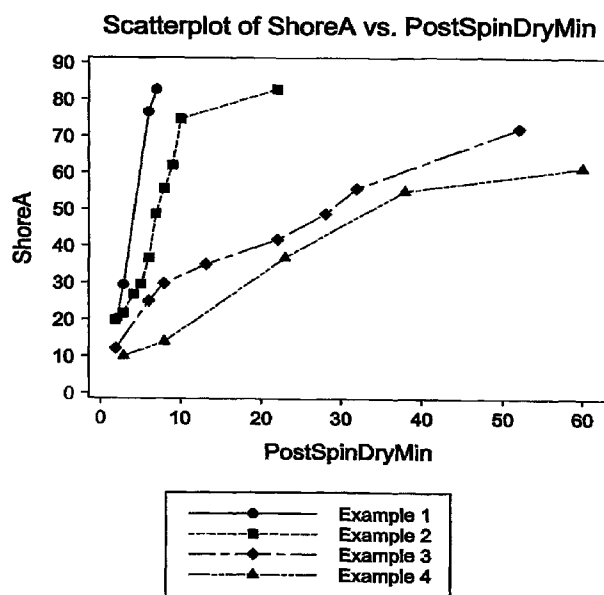
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(54) Title: PROCESS TO PRODUCE POLYMERS WITH IMPROVED HANDLING PROPERTIES



(57) Abstract: The instant disclosure is directed to a process to produce polymer pellets having improved handling characteristics comprises the steps of contacting one or more monomers in the presence of a catalyst within a reactor, under suitable polymerization conditions to produce a polymer having a melt flow rate of about 1 to about 500 dg/min at 230 °C, 2.16 kg, extruding the polymer into an aqueous bath to produce polymer pellets, separating at least a portion of the aqueous bath from the pellets, and aging the pellets for a post-spin time of less than or equal to about 10 minutes, to produce a polymer pellet having a Shore A hardness of greater than or equal to 45, wherein the melt flow rate of the polymer is controlled by inclusion of hydrogen gas within the reactor.

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PROCESS TO PRODUCE POLYMERS WITH IMPROVED HANDLING PROPERTIES

Field of the Invention

[0001] The instant disclosure is directed to a process to produce polymer pellets having improved handling properties. In particular, this disclosure is directed to a process to produce ethylene-propylene copolymer pellets having improved handling characteristics.

Background of the Invention

[0002] Commercially-available polymers are generally supplied as discrete particles, often referred to as pellets, beads, or the like. Polymer pellets, however, may be difficult to process and/or manufacture due to poor handling characteristics. In particular, pellets of various polymers tend to agglomerate upon packaging, and/or during other final processing steps. It is very difficult to break these agglomerates back into free-flowing pellets during downstream processing.

[0003] Various dusting agents, such as low density polyethylene and EVA can be used to minimize the extent of agglomeration. However, addition of dusting agents requires addition of these extraneous materials, which may also impact certain applications of a polymer. For example, certain dusting agents may not be acceptable when the polymers are to be utilized in applications requiring direct food contact. Another tool available for the prevention of agglomerates is to blend a problematic polymer with polymers which do not tend to form agglomerates, to produce blends which remain free flowing. However, this approach not only adds cost to the product, but may also restrict the range of potential applications for a particular product.

[0004] A simple, cost-effective process to produce stable polymer pellets remains an elusive goal in the art.

Summary of the Invention

[0005] In an embodiment, a process to produce polymer pellets having improved handling characteristics comprises the steps of:

contacting one or more monomers in the presence of a catalyst within a reactor, under suitable polymerization conditions to produce a polymer having a melt flow rate of about 1 to about 500 dg/min at 230°C, 2.16 kg;

extruding the polymer into an aqueous bath to produce polymer pellets;

separating at least a portion of the aqueous bath from the pellets;

aging the pellets for a post-spin time of less than or equal to about 10 minutes, to produce a polymer pellet having a Shore A hardness of greater than or equal to 45, wherein the melt flow rate of the polymer is controlled by inclusion of hydrogen gas within the reactor.

[0006] In some embodiments, calcium stearate and/or emulsifiers may be added to the water that is circulated through the pelletizer to assist in the pelletization by keeping the pellets from forming chains or strands. The pelletizer water supply is often cooled which also assists with the pelletization.

Brief Description of the Figures

[0007] Figure 1 is a graphical representation of the Shore A hardness of exemplary and comparative polymers verses the post-spin time.

Detailed Description of the Invention

[0008] Various specific embodiments, versions and examples of the present disclosure will now be described, including preferred embodiments and definitions that are adopted herein for purposes of understanding the claimed invention.

[0009] As used herein, the phrase "suitable polymerization conditions" relates to the selection of polymerization conditions and components, which are necessary to obtain the production of a desired polymer in light of process parameters and component properties. There are numerous permutations of polymerization processes to produce the polymers disclosed herein, as well as

numerous variations in the polymerization components available to produce such polymers having one or more of the desired attributes.

[0010] As used herein, the term “reactor”, is defined to include any container(s) in which a chemical reaction occurs. The term polymer may refer to a homopolymer, a copolymer, an interpolymer, a terpolymer, or the like. When a polymer is referred to as comprising a monomer, the monomer is present in the polymer in the polymerized form of the monomer, or in a derivative form of the monomer.

[0011] The term “alkyl” refers to a hydrocarbon group having from 1 to 20 carbon atoms, which may be derived from the corresponding alkane, alkene, or alkyne, by removing one or more hydrogens from the formula. Examples include a methyl group (CH_3), which is derived from methane (CH_4), and an ethyl group (CH_3CH_2), which is derived from ethane (CH_3CH_3).

[0012] The term “aryl” refers to a hydrocarbon group comprising 5 to 20 carbon atoms that form a conjugated ring structure characteristic of aromatic compounds. Examples of aryl groups or substituents include benzene, naphthalene, phenanthrene, anthracene, and the like, which possess alternating double bonding (“unsaturation”) within a cyclic structure. An aryl group is derived from an aromatic compound by dropping one or more hydrogens from the formula.

[0013] The term “substituted alkyl group(s)” refers to replacement of at least one hydrogen atom on an alkyl, alkene, alkyne, or aryl group having 1 to 20 carbon atoms, by at least one substituent. Examples of substituents include halogen (chlorine, bromine, fluorine, or iodine), amino, nitro, sulfoxy (sulfonate or alkyl sulfonate), thiol, alkylthiol, hydroxy, alkoxy, and straight, branched, or cyclic alkyls, alkenes, or alkynes having 1 to 20 carbon atoms. Examples of alkyl substituents include methyl, ethyl, propyl, tert-butyl, isopropyl, isobutyl, and the like. Examples of alkoxy substituents include methoxy, ethoxy, propoxy, isopropoxy, butoxy, isobutoxy, secondary butoxy, tertiary butoxy, pentyloxy, isopentyloxy, hexyloxy, heptyloxy, octyloxy, nonyloxy, and decyloxy. The term haloalkyl refers to straight or branched chain alkyl groups having 1 to 20 carbon atoms in which at least one hydrogen atom is substituted by at least one halogen.

[0014] The term “stable pellets” refers to pellets that do not exhibit tendencies to agglomerate or otherwise render their physical shape unsuitable for downstream processing. Stable pellets maintain a free flowing form, and can be handled in typical bulk-handling equipment.

[0015] A definition of Ziegler-Natta catalysts may be found in the chapter “Definitions, stereochemistry, experimental methods and commercial polymers”, from the book by John Boor, Jr., “Ziegler-Natta Catalysts and Polymerizations”, p. 32-35, Academic Press.

[0016] The chromium catalyst may be one of the well-known Phillips catalysts or either belongs to another classification. A definition of the Phillips catalysts may be found in the chapter “Metal alkyl-free Catalysts” from the book by John Boor Jr., “Ziegler-Natta Catalysts and Polymerizations”, p.279-324, Academic Press.

[0017] Melt flow rate (MFR) is determined consistent with ASTM D1238 at 190°C using a 2.16 kg mass, unless otherwise specified.

Polymerization Process

[0018] Polymers are produced by contacting one or more monomers in the presence of a catalyst within a reactor, under suitable polymerization conditions. The polymer produced by any one process will have various properties. The polymer is then extruded, which includes the polymer being pelletized (e.g., chopped), or otherwise converted into particles upon exiting the extruder. Often, the pellets are dispersed into an aqueous bath to both cool the pellets and quench the polymerization reaction. Polymers may also be extruded into a fast-flowing stream of water in an underwater pelletizer wherein the extrudate is cut into pellets of the desired size and shape. In some embodiments, calcium stearate and/or emulsifiers may be added to the water that is circulated through the pelletizer to assist in the pelletization by keeping the pellets from forming chains or strands. The pelletizer water supply is often cooled which also assists with the pelletization. The pellets are then “dried”, in that at least a portion of the aqueous bath is removed from the pellets. This drying step is typically accomplished utilizing a centrifuge or other similar apparatus (a spin dryer) wherein the aqueous

bath is preferably centrifugally separated from the pellets. Next, the pellets may be further processed including being cooled, dried, packaged, and/or the like.

[0019] The delay between exiting the spin dryer and further processing is referred to herein as the “post-spin time”, typically expressed in terms of minutes. During this post-spin time, it has been found that various polymers become harder in terms of Shore A hardness with respect to time. Stable pellets preferably have a Shore A hardness of at least 40. Pellets having a Shore A hardness of at least 40 tend to be free flowing, and do not typically form agglomerates or otherwise deform so as not to be processable in bulk handling equipment. However, pellets with a Shore A hardness of less than 40 tend to be “sticky” in that they readily adhere to various surfaces, and/or to one another, forming agglomerates. Pellets having a Shore A hardness of less than 40 may tend to form a negative angle of repose, and may tend to bridge or otherwise not flow through bulk handling equipment. Pellets having a Shore A hardness of less than 40 may tend to deform under the weight of adjoining pellets in bags and storage containers. Deformed pellets tend to agglomerate into interlocked cakes that are difficult to break apart.

[0020] The post-spin time of a process, wherein the polymer pellet produced is allowed to harden to produce a pellet having an acceptable Shore A hardness (i.e., greater than or equal to about 40 Shore A) involves introducing a residence time within a process. The longer the post-spin time required to produce an acceptably hard particle, the longer is the residence time which must be incorporated into a particular process. Accordingly, the post-spin time of the instant process is preferably minimized to less than about 10 minutes, more preferably less than about 9 minutes, more preferably less than about 8 minutes, more preferably less than about 7 minutes, more preferably less than about 6 minutes, more preferably less than about 5 minutes, more preferably less than about 4 minutes, more preferably less than about 3 minutes, more preferably less than about 2 minutes, more preferably less than about 1 minute. Polymers in general (the term polymers as used here includes copolymers, terpolymers, etc.,) that exhibit higher levels of crystallinity are less likely to agglomerate than those that are less crystalline.

[0021] In an embodiment, the Shore A hardness of a polymer pellet immediately after the post-spin time is preferably greater than about 40, more preferably greater than about 45, more preferably greater than about 50, more preferably greater than about 55, more preferably greater than about 60, more preferably greater than about 65 Shore A.

Melt Flow Rate Control

[0022] Numerous conditions and parameters of polymerization affect the properties of the polymer produced. These properties may be manipulated/controlled, to produce polymers having particular properties. More than one approach may be utilized to control a particular property. Polymerization parameters known to affect the crystallinity and molecular weight of a polymer, and thus the melt flow rate of a polymer, include monomer concentration, comonomer concentration, hydrogen concentration and reactor temperature. These parameters may be controlled in order to achieve optimum production economics by high production rate and low catalyst consumption.

[0023] Melt flow rate is typically controlled by temperature, referred to herein as "temperature MFR control", wherein the temperature of the polymerization reactor is controlled around various set-points to produce a polymer having a particular range of melt flow rate. The temperatures utilized to produce a particular melt flow rate depend on the type of polymer being produced, the catalyst used, the process being used, the presence and type of solvent being used, along with a myriad of other factors. However, in general, the higher the temperature, the higher the melt flow rate of a given polymer.

[0024] Melt flow rate may also be controlled by addition of hydrogen to the polymerization reactor, referred to herein as "hydrogen MFR control." Addition of hydrogen avoids higher molecular weights and thus, results in a higher melt flow rate at lower operating temperatures than can be achieved using temperature MFR control alone. The amount of hydrogen added to the reactor to control the melt flow rate is typically referred to in terms of a weight to weight ratio of hydrogen feed rate to a monomer feed rate. For example, in polymers comprising ethylene, the melt flow rate of the final polymer may be controlled utilizing a

hydrogen feed rate to ethylene feed rate on the order of about 1 to 10,000 ppm hydrogen to ethylene. In practice, the control of the flow rates of ethylene and /or comonomer(s) is adjusted so as to keep constant the ratio between the concentrations of the various monomers.

[0025] It has been unexpectedly discovered that polymers produced having a melt flow rate between about 1 and about 500 dg/min at 230°C, 2.16 kg, can be made with considerable ease when small amounts of hydrogen are used to control the MFR of the product (i.e., hydrogen MFR control). The polymers made using hydrogen MFR control have been found to harden rapidly during the post-spin time. Furthermore, polymers made using hydrogen MFR control have been found not to agglomerate in the package or packaging equipment, as compared to essentially identical polymers wherein the melt flow rate is controlled using temperature alone (i.e., temperature MFR control.)

Polymers

[0026] In an embodiment, the polymers include any polymer formed from the polymerization of one or more olefin monomers (e.g., mono-olefins, poly-olefins, alpha-olefins, and the like, comprising 2 to 20 carbon atoms, preferably comprising 2 to 10 carbon atoms) by a high pressure, low pressure, or intermediate pressure process: or by Ziegler-Natta catalysts, chromium catalysts, metallocene catalysts, and/or the like.

[0027] Exemplary polymers include the family of polyolefin resins, polyesters (such as polyethylene terephthalate, polybutylene terephthalate), polyamides (such as nylons), polycarbonates, styrene-acrylonitrile copolymers, polystyrene, polystyrene derivatives, polyphenylene oxide, polyoxymethylene, and fluorine-containing thermoplastics. The preferred thermoplastic resins are crystallizable polyolefins that are formed by polymerizing C₂ to C₂₀ olefins such as, but not limited to, ethylene, propylene and C₄ to C₁₂ α-olefins, such as 1-butene, 1-hexene, 1-octene, 2-methyl-1-propene, 3-methyl-1-pentene, 4-methyl-1-pentene, 5-methyl-1-hexene, and mixtures thereof. Copolymers of ethylene and propylene or ethylene or propylene with another α-olefin, such as 1-butene-1; pentene-1,2-methylpentene-1,3-methylbutene-1; hexene-1,3-methylpentene-1,4-

methylpentene-1,3,3-dimethylbutene-1; heptene-1; hexene-1; methylhexene-1; dimethylpentene-1 trimethylbutene-1; ethylpentene-1; octene-1; methylpentene-1; dimethylhexene-1; trimethylpentene-1; ethylhexene-1; methylethylpentene-1; diethylbutene-1; propylpentane-1; decene-1; methylnonene-1; nonene-1; dimethyloctene-1; trimethylheptene-1; ethyloctene-1; methylethylbutene-1; diethylhexene-1 and dodecene-1, may also be used.

[0028] The polymer may further comprise one or more additives including a stabilizer, an inhibitor of oxidative, thermal, and/or ultraviolet light degradation; lubricants, mold release agents, colorants including dyes and pigments, fibrous and particulate fillers and reinforcements, nucleating agents, plasticizers, and the like, utilized in quantities known to those skilled in the art.

[0029] In a preferred embodiment, the polymer comprises a random propylene polymer comprising propylene, and at least one other α -olefin. The random propylene polymer is preferably a copolymer of ethylene and propylene, produced using a metallocene catalyst. The ethylene content is preferably in the range of about 4 to 25 %, more preferably 8 to 20 percent by weight. The preferred copolymers also exhibit MFR's in the range of 1 to 500 dg/min at 230°C, 2.16 kg, more preferably 5 to 200 dg/min at 230°C, 2.16 kg. It is well known that such products can be made at various reactor operating temperatures, where higher temperatures are often used when polymers with higher MFR's are desired. However, hydrogen MFR control has been unexpectedly found to produce pellets having a Shore A hardness of greater than 40 immediately after less than or equal to about 10 minutes of post-spin time.

[0030] In an embodiment, the polymer is a random copolymer of propylene and at least one comonomer selected from ethylene, C_4 - C_{12} α -olefins, and combinations thereof. In a particular aspect of this embodiment, the copolymer includes ethylene-derived units in an amount ranging from a lower limit of 2%, 5%, 6%, 8%, or 10% by weight to an upper limit of 20%, 25%, or 28% by weight. This embodiment will also include propylene-derived units present in the copolymer in an amount ranging from a lower limit of 72%, 75%, or 80% by weight to an upper limit of 98%, 95%, 94%, 92%, or 90% by weight. These percentages by weight are based on the total weight of the propylene and

ethylene-derived units; i.e., based on the sum of weight percent propylene-derived units and weight percent ethylene-derived units being 100%.

[0031] Comonomer content of discrete molecular weight ranges can be measured by Fourier Transform Infrared Spectroscopy (FTIR) in conjunction with samples collected by GPC. One such method is described in Wheeler and Willis, *Applied Spectroscopy*, 1993, vol. 47, pp. 1128-1130. Different but similar methods are equally functional for this purpose and well known to those skilled in the art.

[0032] Comonomer content and sequence distribution of the polymers can be measured by ^{13}C nuclear magnetic resonance (^{13}C NMR), and such methods are well known to those skilled in the art.

[0033] In one embodiment, the polymer is a random propylene copolymer having a narrow compositional distribution. In another embodiment, the polymer is a random propylene copolymer having a narrow compositional distribution and a melting point as determined by DSC of from 10°C to 105°C. The copolymer is described as random because for a polymer comprising propylene, comonomer, and optionally diene, the number and distribution of comonomer residues is consistent with the random statistical polymerization of the monomers. In stereoblock structures, the number of block monomer residues of any one kind adjacent to one another is greater than predicted from a statistical distribution in random copolymers with a similar composition. Historical ethylene-propylene copolymers with stereoblock structure have a distribution of ethylene residues consistent with these blocky structures rather than a random statistical distribution of the monomer residues in the polymer. The intramolecular composition distribution (i.e., randomness) of the copolymer may be determined by ^{13}C NMR, which locates the comonomer residues in relation to the neighbouring propylene residues. The intermolecular composition distribution of the copolymer is determined by thermal fractionation in a solvent, by methods known to those skilled in the art. For purposes herein, a copolymer is considered to have a narrow compositional distribution if approximately 75% by weight, preferably 85% by weight, of the copolymer is isolated as one or two adjacent, soluble fractions with the balance of the copolymer in immediately preceding or succeeding fractions.

Each of these fractions has a composition (wt% comonomer such as ethylene or other α -olefin) with a difference of no greater than 20% (relative), preferably 10% (relative), of the average weight % comonomer of the copolymer.

[0034] To produce a copolymer having the desired randomness and narrow composition, it is beneficial if (1) a single sited metallocene catalyst is used which allows only a single statistical mode of addition of the first and second monomer sequences and (2) the copolymer is well-mixed in a continuous flow stirred tank polymerization reactor which allows only a single polymerization environment for substantially all of the polymer chains of the copolymer.

[0035] The crystallinity of the polymers may be expressed in terms of heat of fusion. Embodiments of the instant disclosure include polymers having a heat of fusion, as determined by differential scanning calorimetry (DSC), ranging from a lower limit of 1.0 J/g, or 3.0 J/g, to an upper limit of 50 J/g, or 10 J/g. Without wishing to be bound by theory, it is believed that the polymers of embodiments of the instant disclosure have generally isotactic crystallizable propylene sequences, and the above heats of fusion are believed to be due to the melting of these crystalline segments.

[0036] The crystallinity of the polymer may also be expressed in terms of crystallinity percent. The thermal energy for the highest order of polypropylene is estimated at 189 J/g. That is, 100% crystallinity is equal to 189 J/g. Therefore, according to the aforementioned heats of fusion, the polymer has a polypropylene crystallinity within the range having an upper limit of 65%, 40%, 30%, 25%, or 20%, and a lower limit of 1%, 3%, 5%, 7%, or 8%.

[0037] The level of crystallinity is also reflected in the melting point. The term "melting point," as used herein, is the highest peak among principal and secondary melting peaks as determined by DSC. In an embodiment of the instant disclosure, the polymer has a single melting point. Typically, a sample of the random propylene copolymer will show secondary melting peaks adjacent to the principal peak, which are considered together as a single melting point. The highest of these peaks is considered the melting point. The polymer preferably has a melting point by DSC ranging from an upper limit of 105°C, 90°C, 80°C, or 70°C, to a lower limit of 20°C, 25°C, 30°C, 35°C, 40°C, or 45°C.

[0038] The polymers used in the instant disclosure have a weight average molecular weight (Mw) within the range having an upper limit of 5,000,000 g/mol, 1,000,000 g/mol, or 500,000 g/mol, and a lower limit of 10,000 g/mol, 20,000 g/mol, or 80,000 g/mol, and a molecular weight distribution Mw/Mn (MWD), sometimes referred to as a "polydispersity index" (PDI), ranging from a lower limit of 1.5, 1.8, or 2.0 to an upper limit of 40, 20, 10, 5, or 4.5. The Mw and MWD, as used herein, can be determined by a variety of methods, including those in U.S. Patent No. 4,540,753 to Cozewith, et al., and references cited therein, or those methods found in Verstrate et al., *Macromolecules*, v. 21, p. 3360 (1988), the descriptions of which are incorporated by reference herein for purposes of U.S. practice.

[0039] In one embodiment, the polymer has a Mooney viscosity, ML(1+4) @ 125°C, of 100 or less, 75 or less, 60 or less, or 30 or less. Mooney viscosity, as used herein, can be measured as ML(1+4) @ 125°C according to ASTM D1646, unless otherwise specified.

[0040] The polymers used in embodiments of the instant disclosure can have a tacticity index (m/r) ranging from a lower limit of 4 or 6 to an upper limit of 8, 10, or 12. The tacticity index, expressed herein as "m/r", is determined by ¹³C NMR. The tacticity index m/r is calculated as defined in H.N. Cheng, *Macromolecules*, 17, 1950 (1984). The designation "m" or "r" describes the stereochemistry of pairs of contiguous propylene groups, "m" referring to meso and "r" to racemic. An m/r ratio of 0 to less than 1.0 generally describes a syndiotactic polymer and an m/r ratio of 1.0 an atactic material. An isotactic material theoretically may have a ratio approaching infinity, and many by-product atactic polymers have sufficient isotactic content to result in ratios of greater than 50. In one embodiment, the polymer has isotactic stereoregular propylene crystallinity. The term "stereoregular" as used herein means that the predominant number, i.e. greater than 80%, of the propylene residues have the same 1,2 insertion, and the stereochemical orientation of the pendant methyl groups are the same, either meso or racemic.

[0041] The propylene-based polymers can have a triad tacticity of three propylene units, as measured by ¹³C NMR of 75% or greater, 80% or greater,

82% or greater, 85% or greater, or 90% or greater. Preferred ranges include from about 50 to about 99 %, more preferably from about 60 to about 99%, more preferably from about 75 to about 99% and more preferably from about 80 to about 99%; and in other embodiments from about 60 to about 97%. Triad tacticity is determined by the methods described in U.S. Patent Application Publication 20040236042.

[0042] The polymer may be produced by any process that provides the desired polymer properties, in heterogeneous polymerization on a support, such as slurry or gas phase polymerization, or in homogeneous conditions in bulk polymerization in a medium comprising largely monomer or in solution with a solvent as diluent for the monomers. For industrial uses, continuous polymerization processes are preferred. Homogeneous polymers are often preferred in the instant disclosure. For these polymers, preferably the polymerization process is a single stage, steady state, polymerization conducted in a well-mixed continuous feed polymerization reactor. The polymerization can be conducted in solution, although other polymerization procedures such as gas phase or slurry polymerization, which fulfil the requirements of single stage polymerization and continuous feed reactors, are contemplated.

[0043] The polymer may be made advantageously by the continuous solution polymerization process described in WO 02/34795, advantageously in a single reactor and separated by liquid phase separation from the alkane solvent.

[0044] Preferred methods for producing the polymers are found in U.S. Patent Application Publication 20040236042 and U.S. Patent 6,881,800, which are incorporated by reference herein. In another embodiment, the polymers can include copolymers prepared according the procedures in WO 02/36651 which is incorporated by reference here. Likewise, the polymer can include polymers consistent with those described in WO 03/040201, WO 03/040202, WO 03/040095, WO 03/040201, WO 03/040233, and/or WO 03/040442. Pyridine amine complexes, such as those described in WO03/040201 are also useful to produce the polymers useful herein. The catalyst can involve a fluxional complex, which undergoes periodic intra-molecular re-arrangement so as to provide the desired interruption of stereoregularity as in U.S. 6,559,262. The catalyst can be a

stereorigid complex with mixed influence on propylene insertion, see Rieger EP1070087. The catalyst described in EP1614699 could also be used.

[0045] The process of the instant disclosure provides an inexpensive route to manufacture stable polymeric pellets after a post-spin time of less than or equal to about 10 minutes. The instant process is now exemplified including the use of hydrogen MFR control and comparative temperature MFR control, for the manufacture of polymers with the appropriate MFR and ethylene content, along with free flowing and storage-stable pellet characteristics.

Examples

[0046] In Example 1, a copolymer of ethylene and propylene was produced according to the process of the instant disclosure using hydrogen MFR control, wherein the reactor temperature was maintained at 92.5°C. A hafnium-metallocene catalyst was used in conjunction with a fluoro-borate activator. The reactor operating conditions are given in Table 1a.

Table 1a	
R1 C ₂ Conversion	78.77
R1 C ₃ Conversion	32.36
R1 Feed °C	10.96
R1 Temp °C	92.08
R1 Agitation RPM	700
Ambient Temp °C	22.16
R1C ₂ Kg/hr	2.03
R1 C ₃ Kg/hr	29.14
R1 C ₆ Kg/hr	89.91
R1 H ₂ mg/hr	2690
R1 CatEff g/g	185,315

[0047] The pellets produced were easily processed and did not exhibit any tendencies to agglomerate. The composition of the product was 14.22 % ethylene and the MFR was 99.14. The hardness of the pellet was measured using a Durometer (Type Shore A). The pellet characteristics are shown below in Table 1b. The term PostSpinDryMin' refers to the time (measured here in minutes) after the pellet has left the spin dryer. The time of interest is the time lapsed after the

pellet has been cut and the cooling process has begun. It is estimated that pellets spend about 5 to 10 seconds in transit from the pelletizer to the spin dryer.

Table 1b	
Pellet C ₂ wt%	14.22
Pellet MFR dg/min @ 190°C, 2.16 kg	99.14
Pellet Diameter (mm)	4.00
Pellet Length (mm)	6.00
Pellet Shape	egg
Grams/100Pellets	2.95
PostSpinDryMin	ShoreA
2	20
3	30
6	77
7	83

[0048] In Example 2, a copolymer of ethylene and propylene was made utilizing hydrogen MFR control, wherein the reactor temperature was maintained at 92.5 C. A hafnium-metallocene catalyst was used in conjunction with a fluoro-borate activator.

Table 2a	
R1 C ₂ Conversion	76.44
R1 C ₃ Conversion	32.42
R1 Feed °C	11.95
R1 Temp °C	92.67
R1 Agitation RPM	700
Ambient Temp °C	27.97
R1C ₂ Kg/hr	1.99
R1 C ₃ Kg/hr	29.14
R1 C ₆ Kg/hr	89.96
R1 H ₂ mg/hr	336
R1 CatEff g/g	215,412

Table 2b	
Pellet C ₂ wt%	13.84
Pellet MFR dg/min @ 190°C, 2.16 kg	62
Pellet Diameter (mm)	4.50
Pellet Length (mm)	7.00
Pellet Shape	egg
Grams/100Pellets	6.10
PostSpinDryMin	ShoreA
2	20
3	22
4	27
5	30
6	37
7	49
8	56
9	62
10	75
22	83

[0049] The pellets made were easily processed and did not exhibit any tendencies to agglomerate.

[0050] In Comparative Example 3, the polymer was produced utilizing temperature MFR control. A copolymer of ethylene and propylene was made wherein the reactor temperature was maintained at 92.5 C. A hafnium-metallocene catalyst was used in conjunction with a fluoro-borate activator.

Table 3a	
R1 C ₂ Conversion	72.30
R1 C ₃ Conversion	33.24
R1 Feed °C	8.74
R1 Temp °C	92.53
R1 Agitation RPM	750
Ambient Temp °C	27.59
R1C ₂ Kg/hr	2.25
R1 C ₃ Kg/hr	29.14
R1 C ₆ Kg/hr	90.03
R1 H ₂ mg/hr	0
R1 CatEff g/g	512,622

Table 3b	
Pellet C ₂ wt%	14.26
Pellet MFR dg/min @ 190°C, 2.16 kg	18.11
Pellet Diameter (mm)	3.50
Pellet Length (mm)	6.00
Pellet Shape	Egg
Grams/100Pellets	3.95
PostSpinDryMin	ShoreA
2	12
6	25
8	30
13	35
22	42
28	49
32	56
52	72

[0051] The polymer produced had poor handling characteristics and agglomerated upon packaging.

[0052] Comparative Example 4 was also produced using temperature MFR control. A copolymer of ethylene and propylene was made wherein the reactor temperature was maintained at 105 °C. A hafnium-metallocene catalyst was used in conjunction with a fluoro-borate activator.

Table 4a	
R1 C ₂ Conversion	76.37
R1 C ₃ Conversion	39.07
R1 Feed °C	12.79
R1 Feed Temp °C	10.60
R1 Temp °C	104.81
R1 Agitation RPM	750
Ambient Temp °C	31.29
R1C ₂ Kg/hr	2.37
R1 C ₃ Kg/hr	28.11
R1 C ₆ Kg/hr	90.11
R1 H ₂ mg/hr	0
R1 CatEff g/g	397,092

Table 4b	
Pellet C ₂ wt%	14.4
Pellet MFR dg/min @ 190°C, 2.16 kg	37.65
Pellet Diameter (mm)	5
Pellet Length (mm)	7
Pellet Shape	Egg
Grams/100Pellets	6.20
PostSpinDryMin	ShoreA
3	10
8	14
23	37
38	55
60	61

[0053] The polymer produced had poor handling characteristics and agglomerated upon packaging.

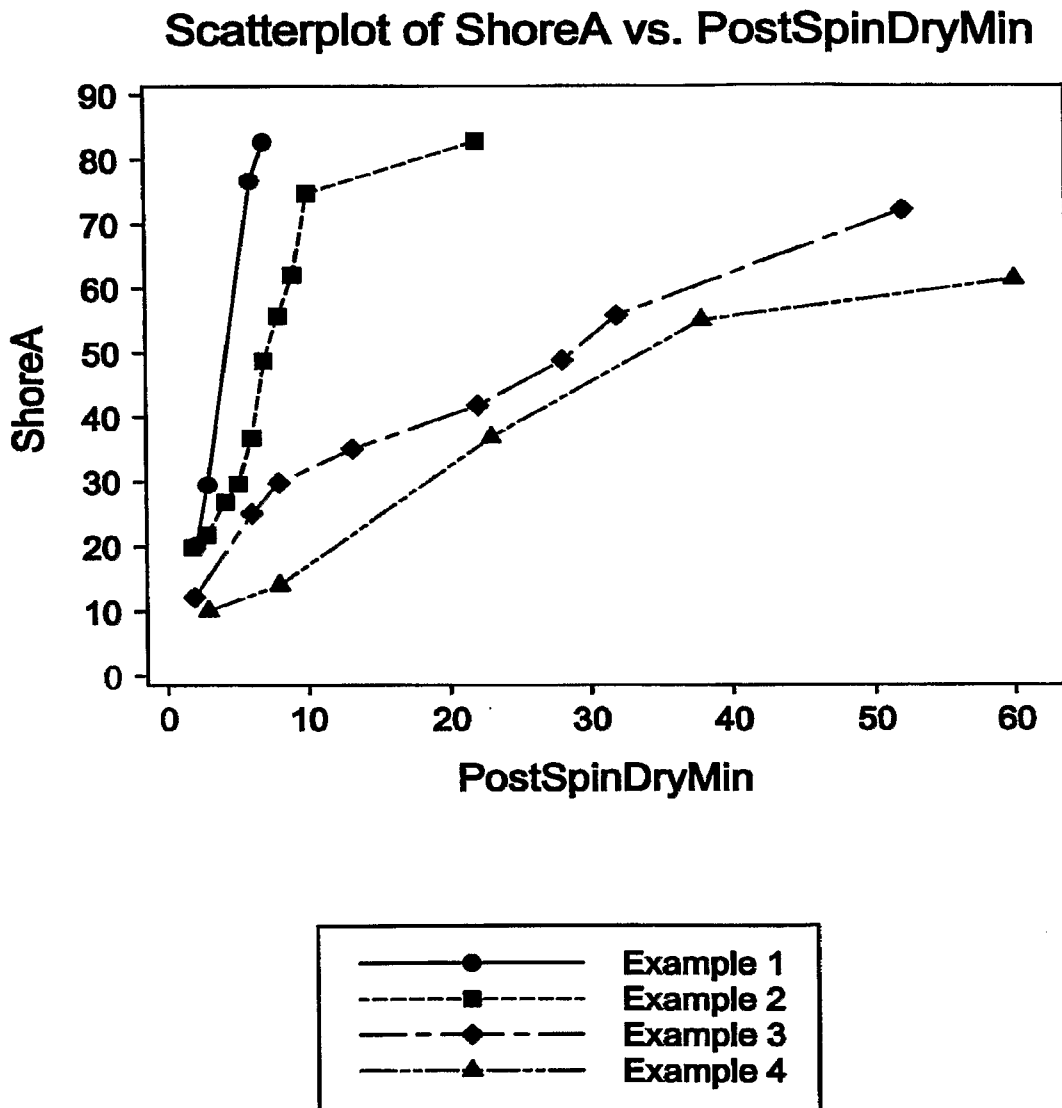
[0054] Figure 1 is a graphical representation of Shore A hardness vs. post-spin time of Examples 1 and 2 and Comparative Examples 3 and 4.

[0055] All documents described herein are incorporated by reference herein, including any priority documents and/or testing procedures. As is apparent from the foregoing general description and the specific embodiments, while forms of the invention have been illustrated and described, various modifications can be made without departing from the spirit and scope of the invention. Accordingly, it is not intended that the invention be limited thereby.

Claims

We claim:

1. A process to produce polymer pellets having improved handling characteristics, comprising:
contacting one or more monomers comprising propylene in the presence of a catalyst within a reactor, under suitable polymerization conditions to produce a polymer having a propylene content greater than or equal to 50 wt% and a melt flow rate of about 1 to about 500 dg/min at 230°C, 2.16 kg;
extruding the polymer into an aqueous bath to produce polymer pellets;
separating at least a portion of the aqueous bath from the pellets;
aging the pellets for a post-spin time of less than or equal to about 10 minutes, to produce a polymer pellet having a Shore A hardness of greater than or equal to 45, wherein the melt flow rate of the polymer is controlled by inclusion of hydrogen gas within the reactor.



INTERNATIONAL SEARCH REPORT

International application No

PCT/US2007/007998

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08F10/06

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)
C08F

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, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2005/192416 A1 (FRIEDERSDORF CHRIS B [US]) 1 September 2005 (2005-09-01) the whole document	1
X	US 2004/024146 A1 (FRIEDERSDORF CHRIS B [US]) 5 February 2004 (2004-02-05) the whole document	1
P,X	WO 2006/138052 A (EXXONMOBIL CHEM PATENTS INC [US]; DATTA SUDHIN [US]; WILLIAMS MICHAEL) 28 December 2006 (2006-12-28) the whole document	1
P,X	WO 2006/083515 A (EXXONMOBIL CHEM PATENTS INC [US]; HARRINGTON BRUCE A [US]; WILLIAMS MI) 10 August 2006 (2006-08-10) the whole document	1



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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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.

G document member of the same patent family

Date of the actual completion of the international search

18 September 2007

Date of mailing of the international search report

24/09/2007

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2007/007998

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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WO 2006138052 A	28-12-2006	NONE	
WO 2006083515 A	10-08-2006	NONE	