



(51) International Patent Classification:

C08F 2/34 (2006.01) G05B 19/00 (2006.01)

C08F 2/01 (2006.01) C08F 10/00 (2006.01)

G05B 17/00 (2006.01)

(21) International Application Number:

PCT/US2024/057269

(22) International Filing Date:

25 November 2024 (25.11.2024)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

63/611,333 18 December 2023 (18.12.2023) US

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CV, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IQ, IR, IS, IT, JM, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, MG, MK, MN, MU, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, CV, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SC, SD, SL, ST,

SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, ME, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))
- as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))

Published:

- with international search report (Art. 21(3))

(54) Title: METHODS FOR IMPROVING GAS-PHASE POLYMERIZATION

(57) Abstract: Disclosed are a method and control system for controlling a gas-phase polymerization process comprising a. gas-phase fluidized bed reactor, a production loop, and a recovery' loop. The process is controlled using a process simulation model of steady state operation of the process to produce a. simulated output comprising predicted values of the set of control parameters based on selected values of the set of manipulated parameters in parallel with a process optimizer to select values for a set of manipulated parameters that satisfies an objective function within the constraints of a. set of control parameters, which are functions of one or more manipulated parameters.



METHODS FOR IMPROVING GAS-PHASE POLYMERIZATION

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of U.S. Provisional Application Number 63/611333, filed on December 18, 2023, entitled "Methods For Improving Gas-Phase Polymerization", the entirety of which is incorporated by reference herein.

FIELD OF THE INVENTION

[0002] The present disclosure relates to systems and methods for operating a polyolefin polymerization reactor with improved production rates.

BACKGROUND OF THE INVENTION

[0003] Polyolefins may be produced using gas-phase polymerization processes. In a gas-phase fluidized bed polymerization process, the process may include a production loop and a recovery loop. In the production loop, a gas-phase material is withdrawn from the top of the gas-phase reactor. The gas-phase material comprises an inert gas, such as nitrogen, an induced condensing agent (ICA), and unreacted monomer. This overhead gas stream flows to a recycle gas compressor where it is pressured to flow through a heat exchanger before reentering the reactor. The gas stream enters the bottom of the reactor where it passes through a distributor plate at the bottom of the fluidized bed (which includes solid polymer particles) to maintain fluidization of the bed. Monomer (e.g., ethylene), optional comonomer (e.g., C₃-C₂ alpha olefin), and catalyst are added to the fluidized bed under conditions suitable to promote polymerization of the monomer and optional comonomer. Such polymerization conditions can include, but are not limited to, temperature, pressure, ICA composition, ICA partial pressure, and ethylene partial pressure in the reaction zone of the reactor. These parameters can be manipulated to control other conditions related to operation of the reactor such as, but not limited to, catalyst activity, reactor cooling, stickiness of polymer particles, and solids carryover in the overhead gas stream.

[0004] A portion of the fluidized bed is withdrawn into a recovery loop, which includes a product discharge system where polymer particles are concentrated by separating the polymer particles from at least a portion of reactor gas, which is returned to the reactor. At this stage, the polymer particles comprise an amount of absorbed ICA resulting from the equilibrium gas conditions in the reaction zone of the reactor. The polymer particles are then conveyed to a stripper vessel by a mixture of reactor gas and inert convey gas (e.g., nitrogen). The polymer particles enter the top of the stripper vessel while inert gas (e.g., nitrogen) is added to the bottom of the

stripper vessel. Countercurrent contact of the inert gas and the polymer particles results in at least a portion of the absorbed ICA being removed from the polymer particles to exit the stripper vessel in an overhead gas stream. The stripped polymer product is sent to further finishing processes and storage. The stripper overhead gas stream is compressed and cooled to condense at least a portion of the ICA to form a liquid ICA stream and a vent stream. A portion of the vent stream is routed to the product discharge system to assist in conveying polymer particles to the stripper vessel and the remainder is removed from the gas-phase polymerization facility. The liquid ICA stream is returned to the gas-phase reactor.

[0005] Some methods have been developed to increase polymer production rates in a production loop. US 2022/009832 discloses a method for increasing polymer production rates by increasing heat removal from a gas-phase reactor. Heat removal is increased by increasing the concentration of an ICA composition, comprising two induced condensing agents, in the reactor gas to a stickiness limit of the polymer particles. The stickiness limit is determined by the relative amounts of the two induced condensing agents in the ICA composition and solubility of each of the two or more induced condensing agents in the polymer particles relative to isopentane.

[0006] The foregoing disclosures focus on optimization of a limited number of process parameters in the production loop. It would be desirable to optimize a greater number of process parameters in the production loop simultaneously by more advanced process control such as real-time optimization (RTO) of the production loop.

[0007] Some methods have been developed to increase polymer production rates through improved control of the recovery loop. U.S. Pat. No. 11,492,422 discloses use of a product discharge system comprising first and second pairs of upstream and downstream lock hoppers having a crosstie between upstream lock hoppers, a crosstie between downstream lock hoppers, and valve means to enable control of reaction zone pressure and/or nitrogen partial pressure in the gas-phase fluidized bed reactor.

[0008] U.S. Pat. No. 11,578,147 discloses use of a photoionization detector in a recovery loop to control inert gas flow to the stripper vessel, wherein the photoionization detector configured to ionize C₄ to C₈ hydrocarbons to measure a hydrocarbon content of stripped polymer product.

[0009] The foregoing disclosures focus on optimization of a limited number of process parameters in the recovery loop. It would be desirable to optimize a greater number of process parameters in the recovery loop simultaneously by more advanced process control such as real-time optimization (RTO) of the recovery loop. It would further be desirable to optimize process

parameters in the production loop and the recovery loop simultaneously by more advanced process control such as real-time optimization (RTO) of the recovery loop.

[0010] There are some RTO applications for gas-phase polyethylene (GPPE) reactors in academic literature. For example, RTO is applied to GPPE reactors for the purpose of optimizing grade transitions in Selwa BenAmor, Francis J. Doyle, Randall McFarlane, "Polymer grade transition control using advanced real-time optimization software," *Journal of Process Control*, Vol. 14, Issue 4, 2004, Pages 349-364, ISSN 0959-1524, <https://doi.org/10.1016/j.jprocont.2003.06.001>. However, these publications are usually focused on polymer properties and do not address process parameters such as induced condensing agent composition and/or concentration in reactor gas in the production loop or any process parameters in the recovery loop.

[0011] Even within the constraints of safe operation, real-time control of gas-phase polymerization reactors is complex. The complexity of reactor control adds further to the difficulty and uncertainty of experimentation if one wishes to alter operating conditions to achieve higher production rates. Large-scale gas-phase plants are expensive and highly productive. Risks associated with experimentation in such plants are high because downtime (such as that caused by passing the melt initiation temperature) is costly. Therefore, exploring design and operating boundaries experimentally is difficult in view of the costs and risks.

[0012] There is a remaining need for methods of determining stable operating conditions for gas fluidized bed polymerization with condensing agents, to facilitate design of the plant and the determination of suitable process conditions for suitable or maximum production rates in a given plant design. Furthermore, because reactor conditions vary with time there is a need for real-time calculation and control of process parameters to maximize production rates. Additionally, process improvements have been made related to operation of both the production loop and the recovery loop. However, operation of the production loop and the recovery loop are interdependent based on their mutual connections to the gas-phase reactor. Therefore, there is a need for real-time optimization of a gas-phase polymerization process including both the production loop and the recovery loop.

SUMMARY OF THE INVENTION

[0013] This disclosure provides processes for polymerizing olefins at increased production rates by real-time optimization of gas-phase polymerization process. In one embodiment, a method for optimizing a gas-phase polymerization process relates to a process comprising a gas-

phase fluidized bed reactor, a production loop, and a recovery loop. The gas-phase fluidized bed reactor has operating conditions comprising a set of manipulated parameters. The combination of the production loop and the recovery loop has operating conditions comprising a set of control parameters. Each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process.

[0014] In one embodiment, a method for optimizing a gas phase polymerization process comprises simulating the operation of the process using a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of the set of control parameters based on selected values of the set of manipulated parameters. The method further comprises measuring one or more inputs from the process to confirm or correct output from the process simulation model. Process optimization is performed using an optimizer by selecting values for each parameter in the set of manipulated parameters to satisfy an objective function within the constraints of the set of control parameters, and selected values for each parameter in the set of manipulated parameters are used to control the process.

[0015] In some embodiments, the production loop implements: withdrawing a gas phase stream from the gas phase fluidized bed reactor, wherein the gas phase stream comprises an induced condensing agent (ICA) component; pressurizing the gas phase stream in a recycle gas compressor to produce a pressurized recycle stream; cooling the pressurized recycle stream to condense at least a portion of the ICA component to produce a condensed recycle stream; and recycling the condensed recycle stream to the gas phase fluidized bed reactor.

[0016] In some embodiments, the recovery loop implements: withdrawing a portion of first polymer particles from a fluidized bed of the gas phase fluidized bed reactor; separating the first polymer particles from entrained reactor gas in a product discharge system to produce a first polymer product, wherein the first polymer product comprises a first amount of an induced condensing agent (ICA) component; contacting the first polymer product with an inert gas in a stripper vessel to form a second polymer product and second gas stream, wherein the second polymer product comprises a reduced amount of the ICA component, and the second gas stream comprises a gas phase of the ICA component removed from the first polymer product; cooling the second gas stream to condense at least a portion of the gas phase of the ICA component to form a liquid stream comprising the condensed ICA and a third gas stream; and recycling the hydrocarbon

liquid stream to the gas phase fluidized bed reactor.

[0017] In another aspect, a process control system for controlling a gas phase polymerization process is disclosed. The process control system, comprises a process simulation model and an optimizer. The process simulation model of steady state operation of the process produces a simulated output comprising predicted values of a set of control parameters based on selected values of a set of manipulated parameters. The optimizer selects values for each parameter in the set of manipulated parameters to satisfy an objective function within the constraints of the set of control parameters. The process control system further comprises a multiple-input/multiple-output controller adapted to produce, during each operational cycle of the process control system, multiple control outputs configured to control the process based on the selected values of each parameter in a set of manipulated parameters provided to the multiple-input/multiple output controller from the process simulation model and/or the optimizer during each operational cycle of the process control system.

[0018] The foregoing has outlined rather broadly the features and technical advantages of the present invention in order that the detailed description of the invention that follows may be better understood. Additional features and advantages of the invention will be described hereinafter, which form the subject matter of the claims of the invention. It should be appreciated by those skilled in the art that the conception and specific embodiments disclosed may be readily utilized as a basis for modifying or designing other catalyst compositions and/or processes for carrying out the same purposes of the present invention. It should also be realized by those skilled in the art that such equivalent constructions do not depart from the spirit and scope of the invention as set forth in the appended claims. The novel features which are believed to be characteristic of the invention, both as to its compositions and methods, together with further objects and advantages will be better understood from the following description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0019] The claimed subject matter may be understood by reference to the following description taken in conjunction with the accompanying drawings, in which like reference numerals identify like elements, and in which:

[0020] FIG. 1 is a schematic diagram of a gas-phase polymerization system (emphasis on production loop), according to one embodiment;

[0021] FIG. 2 is a schematic diagram of a gas-phase polymerization system (emphasis on recovery loop), according to one embodiment;

[0022] FIG. 3 is a diagram of a real-time optimization control system for controlling a gas-phase polymerization process, according to one embodiment; and

[0023] FIG. 4 is a graph showing a nonlinear relationship between ICA added to the gas-phase polymerization reactor and ICA recovered in the recovery loop over a range of optimized manipulated parameters, according to one embodiment.

[0024] While the disclosed process and system are susceptible to various modifications and alternative forms, the drawings illustrate specific embodiments and/or aspects herein described in detail by way of example. It should be understood, however, that the description herein of a specific embodiment is not intended to limit the invention to the particular forms disclosed, but on the contrary, the intention is to cover all modifications, equivalents, and alternatives falling within the spirit and scope of the invention as defined by the appended claims.

DETAILED DESCRIPTION OF THE INVENTION

[0025] Illustrative embodiments of the subject matter claimed below will now be disclosed. In the interest of clarity, some features of some actual implementations may not be described in this specification. It will be appreciated that in the development of any such actual embodiments, numerous implementation-specific decisions must be made to achieve the developer's specific goals, such as compliance with system-related and business-related constraints, which will vary from one implementation to another. Moreover, it will be appreciated that such a development effort, even if complex and time-consuming, would be a routine undertaking for those of ordinary skill in the art having the benefit of this disclosure.

[0026] The words and phrases used herein should be understood and interpreted to have a meaning consistent with the understanding of those words and phrases by those skilled in the relevant art. No special definition of a term or phrase, i.e., a definition that is different from the ordinary and customary meaning as understood by those skilled in the art, is intended to be implied by consistent usage of the term or phrase herein. To the extent that a term or phrase is intended to have a special meaning, i.e., a meaning other than the broadest meaning understood by skilled artisans, such a special or clarifying definition will be expressly set forth in the specification in a definitional manner that provides the special or clarifying definition for the term or phrase.

[0027] For example, the following discussion contains a non-exhaustive list of definitions of several specific terms used in this disclosure (other terms may be defined or clarified in a definitional manner elsewhere herein). These definitions are intended to clarify the meanings of

the terms used herein. It is believed that the terms are used in a manner consistent with their ordinary meaning, but the definitions are nonetheless specified here for clarity.

Definitions

[0028] As used herein, “inert” means that the referenced composition (gas or otherwise) is inert to reaction conditions within a reaction vessel in a reactor system (e.g., does not react in a polymerization reactor in a gas phase polymerization system).

[0029] As used herein, “control parameter” is a dependent parameter or variable in operating conditions in a process that is directly or indirectly dependent upon one or more manipulated parameters. A control parameter is further a constrained parameter or variable in the operating conditions in that it is subject to an equality constraint (e.g., hydrocarbon solubility in polymer particles in the fluidized bed) or an inequality constraint (e.g., dMIT, or delta-MIT, i.e., delta from Melt Initiation Temperature, a measure of nearness of operating temperature to melt initiation temperature, and hence an indicator of resin stickiness; see U.S. Pat. No. 7,774,178). An equality constraint produces a control parameter having a certain value in response to selecting specific values of one or more manipulated parameters upon which the controlled parameter depends directly or indirectly (alternatively, this could be phrased as having both an upper limit and a lower limit of a manipulated parameter immediately surrounding the target value, such that the target value of such manipulated parameter is maintained). An inequality constraint produces a limit on a control parameter in response to selecting specific values of one or more manipulated parameters upon which the controlled parameter depends directly or indirectly—i.e., the control parameter is either greater than or less than a certain value in response to selecting specific values of one or more manipulated parameters upon which the controlled parameter depends directly or indirectly (e.g., upper limits and/or lower limits selected for each of the manipulated parameters). In some instances, a control parameter, subject to either an equality or inequality constraint, may be further constrained to a limited range of values related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process. All of the foregoing are considered examples of constraints for a control parameter.

[0030] As used herein, “iC₄” and “isobutane” refer to 2-methylpropane.

[0031] As used herein, “iC₅” and “isopentane” refer to 2-methylbutane.

[0032] As used herein, “ICA” refers to a condensing agent. “ICAs” refers to condensing agents. “ICA composition” refers to the total condensing agent in the reactor and encompasses

compositions with two or more condensing agents. ICAs suitable for use in methods of the present disclosure may include C₃-C₆ hydrocarbons or combinations thereof. For example, ICAs suitable for use may include n-butane, isobutane, n-pentane, isopentane, neo-pentane, hexane, isohexane, and other hydrocarbon compounds that are similarly non-reactive in the polymerization process.

5 A “binary ICA composition” is an ICA composition that includes two ICAs, and a “ternary ICA composition” is an ICA composition that includes three ICAs.

[0033] As used herein, “instantaneous,” with reference to a material property in a continuous reaction, denotes the value of the material property of the most recently value. The most recently produced quantity typically undergoes mixing with previously produced quantities of the material
10 before a mixture of the recently and previously produced material exits the reactor. In contrast, with reference to a product being produced by a continuous reaction, “average” (or “bed average”) value (at a time “T”) of a property denotes the value of the property of the material, such as polymer product that exits the reactor at time T.

[0034] As used herein, “linear low density polyethylene” (LLDPE) refers to polyethylene
15 having a density in the range of from 0.890 to 0.970 g/cm³, from 0.905 to 0.960 g/cm³, 0.910 to 0.950 g/cm³ or from 0.910 to 0.940 g/cm³, and a melt index (2.16 kg at 190°C) in the range of from 0.1 to 50 dg/min. or from 0.5 to 20 dg/min., that is linear and is substantially free of long chain branching. LLDPE can be produced with single-site transition metal catalysts, such as, but not limited to, metallocene catalysts in gas-phase reactors and/or in slurry reactors and/or in
20 solution reactors

[0035] As used herein, “manipulated parameter” is an independent parameter or variable in operating conditions in a process that is intentionally maintained or changed to determine a response in one or more control parameters of the process.

[0036] As used herein, “melt index” refers to a measure of the use of flow of the melt of the
25 thermoplastic polymer. Melt index may be measured according to ASTM D1238-13 at suitable weight and temperature. Generally, the melt index of polyolefins is measured at 2.16 kg at 190°C, 5 kg at 190°C, or 21.6 kg at 190°C.

[0037] As used herein, “M_n” is number average molecular weight, “M_w” is weight average molecular weight, and “M_z” is z average molecular weight, wt % is weight percent, and mol % is
30 mole percent. Molecular weight distribution (MWD), also referred to as polydispersity index (PDI), is defined to be M_w divided by M_n. Unless otherwise noted, all molecular weights (e.g., M_w, M_n, M_z) are reported in units of g/mol.

[0038] As used herein, “nC₄” and “n-butane” refer to normal-butane.

[0039] As used herein, “nC₅” and “n-pentane” refer to normal-pentane.

[0040] As used herein, “nC₆” and “n-hexane” refer to normal-hexane.

[0041] As used herein, “neoC₅” and “neo-pentane” refer to 2,2-dimethylpropane.

5 [0042] As used herein, “objective parameter” means a value of a quantifiable aspect corresponding to an input to or an output from the operation of the gas phase polymerization process, wherein the objective relates to a quantifiable aspect of operating the gas phase polymerization process. For example, one aspect of the operation of a gas phase polymerization process could be profitability. Inputs of raw materials and energy would have a corresponding
10 cost associated with each unit of raw materials and energy. Outputs from the process would have a corresponding value per unit of measure, such as a sales value of polymer product or a cost of managing a vent stream for the polymer product stripper. In another example, an aspect of the operation of a gas phase polymerization process could be energy consumed per mass unit of polymer product. Inputs of raw materials and energy would have a corresponding amount of
15 energy associated with each unit of polymer product produced.

[0043] As used herein, “polyethylene” denotes a polymer of ethylene and optionally one or more C₃-C₁₈ alpha-olefins, while the term “polyolefin” denotes a polymer of one or more C₂-C₁₈ alpha-olefins and optionally one or more comonomers. An “olefin” is an unsaturated hydrocarbon that contains at least one carbon-carbon double bond. An alpha-olefin is a hydrocarbon that
20 contains at least one carbon-carbon double bond at one end of a carbon chain (e.g., 1-butene, vinyl-cyclohexane). For the purposes of this disclosure, ethylene shall be considered an α -olefin. Further, when a polymer is referred to as having “ethylene” content, or content of any other monomer, it should be understood that the polymer has content derived from such monomer (in the polymerized form of the monomer). Thus, for example, an “ethylene content” of 90mol% in a
25 polymer is equivalent to “ethylene-derived content” of 90mol% in such polymer.

[0044] As used herein, “real-time” means data processed, and systems adjusted, without intentional delay, given the processing limitations of the system and the time to accurately measure the data.

[0045] The term “gas-phase polymerization” refers to the production of polymer in a gas-phase
30 reactor (referred to herein simply as a “reactor”). It should also be noted that when a “gas-phase” polymerization or reactor is referenced, it is contemplated that monomers are typically reacted in a gas phase in a reaction zone; however, the monomers need not necessarily be supplied to the

reactor in a gas phase. Rather, the monomers may be supplied in a gas phase, liquid phase (condensed phase), or a hybrid gas-liquid phase. Accordingly, when gaseous monomeric streams or cycle gas streams are referenced herein as part of a gas-phase polymerization reactor system or process, it should be understood that such gas streams can in fact be at least partially condensed (that is, in a gas-liquid hybrid phase). In other words, any stream referenced as a gas stream, recirculated gas, or the like, in the context of a gas-phase reaction system as described herein, can be considered to optionally be at least partially liquefied, as is known in the art. See discussion of so-called “condensed mode” of operating certain gas-phase polymerization reactors, e.g., in Namkajorn et al., *Condensed Mode Cooling for Ethylene Polymerization: Part III. The Impact of Induced Condensing Agents on Particle Morphology and Polymer Properties*, J. MACROMOL. CHEM. AND PHYS. 217, 1521-1528 (Wiley 2016), where it is noted that in some fluidized bed gas-phase polymerization reactors, recycle stream or cycle gas can be cooled to a temperature below its dew point so that it is partially liquified, and then fed into the bottom of the fluidized bed reactor, where latent heat of vaporization of the liquid in the feed absorbs the heat of polymerization and thereby offers increased cooling and the potential for increased reaction rates.

Production loop

[0046] The methods described may be used in pilot plant or commercial size reactors including a variety of designs. For example, the model can be used in commercial-scale reactions, such as gas-phase fluidized-bed polymerization reactions, that can be monitored and optionally also controlled. Some such reactions can occur in a reactor having the geometry of the fluidized bed reactor 101 discussed with respect to FIG. 1. In other embodiments, a reactor is monitored and optionally also controlled while operating to perform polymerization using any of a variety of different processes (e.g., slurry or gas-phase processes).

[0047] Generally, in a gas-phase fluidized-bed process used for producing polymers, a gaseous stream containing one or more monomers is continuously cycled through a fluidized bed in the presence of a catalyst under reactive conditions. The gaseous stream is withdrawn from the fluidized bed and recycled back into the reactor. Simultaneously, polymer product is withdrawn from the reactor and fresh monomer is added to replace the polymerized monomer. (See, for example, U.S. Pat. Nos. 4,543,399; 4,588,790; 5,028,670; 5,317,036; 5,352,749; 5,405,922; 5,436,304; 5,453,471; 5,462,999; 5,616,661; and 5,668,228; the substance of which are fully incorporated herein by reference for purposes of U.S. patent practice.)

[0048] FIG. 1 is a schematic diagram of a production loop 100 that can be monitored and controlled in accordance with embodiments described. Production loop 100 comprises a fluidized bed reactor 101 and production loop peripheral equipment 102. The polymerization system 100 includes a fluidized bed reactor 101. The fluidized bed reactor 101 has a bottom end 103, a straight section 105, a top expanded section 107, and a distributor plate 109 within the straight section 105. A fluidized bed 111 of granular polymer (once formed) and catalyst particles is contained within the straight section 105 and may optionally extend slightly into the top expanded section 107. The bed is fluidized by the steady flow of recycle gas through the distributor plate 109. The recycle gas enters fluidized bed reactor through line 113, additional reaction and inert gases (including ICAs) may be added in adjustable ratios through line 115. Aluminum alkyl (optional) may be added through line 117. The flow rate of the recycle gas is regulated to maintain circulation of fluidized bed 111. In some embodiments, a recycle gas velocity of from about 1 ft/sec to about 3 ft/sec, such as from about 2 ft/sec to about 3 ft/sec, or from about 2.4 ft/sec to about 2.8 ft/sec is used to maintain a fluidized bed 111 in the reactor 101 while operating the reactor 101 at a total pressure of about 4200 kPa or less, about 700 kPa to about 4200 kPa, about 1300 kPa to about 2800 kPa, or about 1700 kPa to about 2500 kPa.

[0049] The polymerization system 100 has one or more catalyst lines 119 for controlling the addition of polymerization catalyst to a reaction zone (not shown) within fluidized bed 111, and generally within straight section 105. Within the reaction zone, the catalyst particles react with reaction gases including an olefin monomer (e.g., ethylene) and optionally a comonomer and other reaction gases (e.g., hydrogen) to produce the granular polymer particles. As new polymer particles are produced, other polymer particles are continually withdrawn from the fluidized bed 111 through a product discharge line 121 to product recovery system comprising recovery loop peripheral equipment 202. An exemplary product recovery system 200 and recovery loop peripheral equipment 202 are shown and described in more detail in connection with FIG. 2 below. The fluidized bed 111 may be maintained at a constant height by withdrawing a portion of the fluidized bed 111 at a rate equal to the rate of formation of particulate product. The product may be removed continuously or nearly continuously via a series of valves (not shown) into the recovery loop peripheral equipment 202, and the recovery loop peripheral equipment also includes means for returning recovered condensed liquid back to the production loop peripheral equipment via recycle line 133, where it joins cycle gas returning to the reactor 101 via line 113. Optionally, the system could further include a fixed-volume chamber to which the product discharge line 121

delivers the discharged product prior to entering the product recovery system 200 shown in FIG. 2, and such chamber could also include a line for venting gas back to the reactor 101; in this way, product discharge including solid product and unreacted gas can enter the fixed-volume chamber while unreacted gas is simultaneously vented back to the reactor 101, allowing for highly efficient removal of the product, while recycling a large portion of the unreacted gases back to the reactor.

[0050] The polymerization system 100 also has a cooling loop which includes a first recycle gas line 139, compressor 141, a second recycle gas line 143, and cooling system 145 (such as a circulating gas cooler), coupled with the fluidized bed reactor 101. Cooling system 145 may accept cooling water via line 147 and expel heated water via line 149. Cooling of the recycle gas is a method used to cool the polymerization system 100 to reduce or eliminate issues that may arise from exothermic polyolefin production. During operation, the cooled circulating gas from cooling system 145 flows via line 113 through inlet 151 into the bottom of the fluidized bed reactor 101, then propagates upward through the distributor plate 109, into and through the fluidized bed 111, and out from the fluidized bed reactor 101 via outlet 153 back into the first recycle gas line 139.

[0051] The top expanded section 107 may also be known as a “velocity reduction zone,” and is designed to reduce the quantities of particle entrainment in the recycle gas line 139 from the fluidized bed. The diameter of the top expanded section 107 generally increases with the distance from straight section 105. The increased diameter causes a reduction in the speed of the gas stream, which allows most of the entrained particles to settle back into the fluidized bed 111, thereby minimizing the quantities of solid particles that are “carried over” from the fluidized bed 111 through the recycle gas line 139. In some instances, a screen (not shown) may be included upstream of the compressor 141 to remove larger material from the recycle gas line 139.

[0052] To maintain a reactor temperature, the temperature of the recycle gas may be continuously adjusted up or down to accommodate changes in the rate of heat generation due to the polymerization reaction. One or more temperature sensors 155 may be located in the fluidized bed and used with a control system and the cooling loop to control the temperature of the fluidized bed 111 near the process set-point. Heated reactor gas, which carries heat energy from the fluidized bed reactor 101, is withdrawn from the outlet 153 and is pumped by the compressor 141 via line 143 to cooling system 145 where the temperature of the heated reactor gas is reduced and at least a portion of the ICA composition present is condensed to a liquid. The recycle gas from the cooling system 145, including condensed liquids, flows via line 113 to the reactor inlet 151 to cool the fluidized bed 111. Temperature sensors (not shown) near the inlet and outlet of the cooling

system 145 may provide feedback to a control system (not shown) to regulate the amount by which cooling system 145 reduces the temperature of the recycle gas entering the fluidized bed reactor 101.

[0053] The fluidized bed reactor 101 may also include one or more skin temperature sensors (one such skin temperature sensor 157 is shown for illustrative purposes in FIG. 1), mounted in positions along a wall of the straight section 105 of the fluidized bed reactor 101 so as to protrude into the bed from the reactor wall by a small amount (e.g., about one eighth to one quarter of an inch (3-6 mm)). The skin temperature sensors 157 may be configured and positioned to sense the temperature of the resin near the wall of the fluidized bed reactor 101 during operation.

[0054] The temperature sensors in the fluidized bed 111 can also or instead include a resistance temperature sensor 155 positioned and configured to sense bed temperature during reactor operation at a location within the fluidized bed reactor 101 away from the reactor wall. The resistance temperature sensor can be mounted so as to protrude into the bed more deeply than the skin temperature sensors 157 (e.g., about 8 to 18 inches (20-46 cm) away from the reactor wall). Although multiple resistance temperature sensors 155 can be deployed along the reactor wall, only one is shown in FIG. 1 for illustrative purposes.

[0055] Other sensors and other apparatuses may be employed to measure other reaction parameters during a polymerization reaction. The reaction parameters may include instantaneous and bed-averaged resin product properties (e.g., melt index and density of the polymer resin product being produced by the polymerization system 100 during a polymerization reaction). Resin product properties may be measured by periodically sampling the resin when exiting the reactor (e.g., about once per hour or at any other desired frequency), and performing the appropriate tests in a quality control laboratory.

[0056] Other measured reaction parameters may include reactor gas composition (e.g., concentrations and partial pressures of reactant gases, ICA, and other inert gases, such as nitrogen, inert hydrocarbon, and the like). The reactor gas composition may be measured by removal of gas from upper portion 107 via line 159 to a gas chromatograph ("GC") system 161. GC system 161 may also be connected by lines (not shown) other than line 159 to other parts of polymerization system 100, such as recycle gas line 139, compressor 141, line 143 or any combination thereof.

[0057] The process control variables may be controlled to obtain the desired productivity for the polymerization system 100 and properties for the resin. For example, the parameters used to control gas-phase composition within the fluidized bed reactor 101 can include the concentration

(partial pressure) and composition of the ICA composition and comonomer, the partial pressure of monomer, the type and properties of catalysts, and the temperature of the reaction process. For example, a polymerization reaction during a transition from production of a certain grade of polyolefin to a different grade may be controlled by controlling process control variables to ensure
5 that the product (e.g., the granular resin) has properties compliant with an initial specification set at the start of the transition, the product produced during the transition ceases to comply with the initial specification set at a first time, and the product has properties compliant with a final specification set at the end of the transition. In the processes described herein, stickiness of the resin during the reaction may be controlled by a control system adjusting (or regulating) the
10 temperature in the reaction zone and/or the concentration (partial pressure) of the ICA composition used in the reaction.

[0058] Polyolefin polymerization may be performed by contacting in a reactor (such as the fluidized bed reactor 101 of FIG. 1) an olefin monomer (optionally with a comonomer) with one or more catalysts (supported or not) in the presence of ICA composition and optionally hydrogen.
15 The individual flow rates of olefin monomer, optional comonomer, optional hydrogen, and ICA composition (or individual components thereof) may be controlled to maintain fixed gas composition targets. The concentration of all gases may be measured with a chromatograph. A solid catalyst, a catalyst slurry, or liquid solution of the catalyst(s) may be injected directly into the reactor using a carrier gas (e.g., purified nitrogen), where the feed rate of catalyst(s) may be
20 adjusted to change or maintain the catalyst inventory in the reactor.

[0059] In some embodiments, the polymerization reaction may be performed at a reactor pressure of about 4,200 kPa or less, about 700 kPa to about 4,200 kPa, about 1,300 kPa to about 2,800 kPa, or about 1,700 kPa to about 2,500 kPa.

[0060] Generally, the olefin monomer concentration is controlled and monitored by the olefin
25 monomer partial pressure. In some embodiments, the olefin partial pressure may be at about 4200 kPa or less, such as about 500 kPa to about 2,000 kPa, about 1,000 kPa to about 1,800 kPa, about 1,200 kPa to about 1,700 kPa, or about 1,400 kPa to about 1,600 kPa.

[0061] The comonomer concentration may be controlled and monitored by a comonomer to
30 olefin monomer mole ratio (or alternatively, the flow rates of comonomer and olefin monomer are held at a fixed ratio). When present, the comonomer may be at a relative concentration to the olefin monomer that will achieve the desired weight percent incorporation of the comonomer into the finished polyolefin. In some embodiments, the comonomer may be present with the olefin

monomer in a mole ratio in the gas-phase of from about 0.0001 to about 50 (comonomer to olefin monomer), from about 0.0001 to about 5, from about 0.0005 to about 1.0, or from about 0.001 to about 0.5.

[0062] The olefin monomer or comonomers, for example, may be a C₂-C₁₈ alpha-olefin. In some embodiments, the olefin monomer is ethylene, and a comonomer is a C₃-C₁₂ alpha olefin. In some embodiments, the olefin monomer may be ethylene or propylene, and a comonomer may include C₄-C₁₀ alpha-olefins. For example, C₂-C₁₈ alpha-olefins that may be utilized as a comonomer in embodiments described may include: ethylene, propylene, 1-butene, 1-pentene, 1-hexene, 1-heptene, 1-octene, 4-methylpent-1-ene, 1-decene, 1-dodecene, 1-hexadecene, and the like, and a combination thereof. Additionally, a polyene may be used as a comonomer according to some embodiments described. For example, polyenes may include: 1,3-hexadiene, 1,4-hexadiene, cyclopentadiene, dicyclopentadiene, 4-vinylcyclohex-1-ene, methyloctadiene, 1-methyl-1,6-octadiene, 7-methyl-1,6-octadiene, 1,5-cyclooctadiene, norbornadiene, ethylidene norbornene, 5-vinylidene-2-norbornene, 5-vinyl-2-norbornene, and olefins formed in situ in the polymerization medium. When olefins are formed in situ in the polymerization medium, the formation of polyolefins containing long chain branching may occur. Additional examples of comonomers may include isoprene, styrene, butadiene, isobutylene, chloroprene, acrylonitrile, and cyclic olefins. Combinations of the foregoing may be utilized in the methods described.

[0063] Examples of polymers that can be produced in accordance with the method described may include the following: homopolymers and copolymers of C₂-C₁₈ alpha olefins; polyvinyl chlorides, ethylene propylene rubbers (EPRs), ethylene-propylene diene rubbers (EPDMs); polyisoprene; polystyrene; polybutadiene; polymers of butadiene copolymerized with styrene; polymers of butadiene copolymerized with isoprene; polymers of butadiene with acrylonitrile; polymers of isobutylene copolymerized with isoprene; ethylene butene rubbers and ethylene butene diene rubbers; polychloroprene; norbornene homopolymers and copolymers with one or more C₂-C₁₈ alpha olefin; and terpolymers of one or more C₂-C₁₈ alpha olefins with a diene. In some embodiments, the polyolefin produced by the method described may include olefin homopolymers (e.g., homopolymers of ethylene or propylene). In some instances, the polyolefin produced may be copolymers, terpolymers, and the like of the olefin monomer and the comonomer.

[0064] In some embodiments, the polyolefin produced may be a polyethylene or a polypropylene. Exemplary polyethylenes produced by the methods described may be

homopolymers of ethylene or copolymers of ethylene (or terpolymers of ethylene) having at least one alpha-olefin (comonomer) where the ethylene-derived content may be at least about 50% by weight of the total monomers involved. Such copolymers of ethylene can include comonomer-derived content, and in particular content derived from one or more $C_3 - C_{20}$ alpha-olefins, such as one or more of 1-butene, 1-hexene, or 1-octene. Exemplary polypropylenes produced by the methods described may be homopolymers of propylene or interpolymers of propylene and at least one alpha-olefin (comonomer) where the propylene content may be at least about 50% by weight of the total monomers involved.

[0065] Hydrogen gas is often used in olefin polymerization to control the final properties of the polyolefin. For some types of catalyst systems, increasing concentrations (or partial pressures) of hydrogen may alter the molecular weight or melt index of the polyolefin generated. The melt index can thus be influenced by the hydrogen concentration. Generally, the amount of hydrogen in the polymerization is expressed as a mole ratio relative to the total polymerizable monomer (e.g., relative to ethylene or relative to a blend of ethylene and hexene or propylene). The amount of hydrogen used in some polymerization processes is an amount necessary to achieve the desired melt index (or molecular weight) of the final polyolefin resin. In some embodiments, the mole ratio in the gas-phase of hydrogen to total polymerizable monomer (H_2 to monomer) may be about 0.00001 or greater, about 0.0005 or greater, about 0.001 or greater, about 10 or less, about 5 or less, about 3 or less, or about 0.10 or less, where a range may include a combination of an upper mole ratio with a lower mole ratio described.

Recovery loop

[0066] Recovery loop 200 comprises a fluidized bed reactor 101 and recovery loop peripheral equipment 202, as shown in FIG. 2. The polymerization system 100 includes a fluidized bed reactor 101. Particulate polymer product is removed intermittently from the reactor 101 via stream 121 typically from a region just above the distributor plate and passed to a polymer product recovery system 200 as shown in FIG. 1 and FIG. 2 (optionally, with intervening delivery to a fixed volume vessel with gas vent return to the reactor 101, not shown in FIGs. 1 and 2 but discussed previously in connection with the product discharge line 121 of FIG. 1). Product withdrawal is usually initiated when the product inventory in the reactor exceeds a desired value. The inventory in the reactor is generally determined by fluidized bed level and/or weight measurements, or from secondary measurements used to infer fluidized bed level and/or weight. Upon detection of a high reactor inventory, the product discharge system is initiated to pass the

polymer product to the product discharge system 210, as shown in FIG. 2 and further explained below.

[0067] The polymer product as it leaves the reactor is saturated with unreacted monomer as well as other hydrocarbons added to, or produced in, the polymerization process. The polymer product is discharged from the reactor along with reactor gas, comprising unreacted monomer (e.g., ethylene) optionally comonomer (e.g., hexene), inert carrier gas used to fluidize the catalyst and transport it into the reactor in the first place. The inert carrier gas can be nitrogen, though it can be any inert gas that would not react or otherwise alter the polymerization reaction. Carrier gases for gas-phase fluidized bed polymerization reactors are well known in the art, and their identity is not the focus of the present disclosure. The reactor gas may further comprise hydrogen (used, e.g., as a chain transfer agent in polymerization) as well as reaction and other process byproducts such as water, ammonia, methane, higher alkanes (e.g., an ICA composition), carbon dioxide, and/or other compounds of oxygen, carbon, and/or hydrogen. To recover hydrocarbons entrained with the polymer product and initiate purification of the product, the particulate polymer collected by the product discharge system 210 is transferred by a conveying gas, such as nitrogen or a mixture of nitrogen and ethylene, via line 212 to a monomer stripping vessel 220. The polymer product enters the top of the stripping vessel 220 and, as it flows downwardly through the stripping vessel 220, is contacted with fresh and/or recycled stripping gas supplied to the bottom of the vessel via line 235. The countercurrent contact between the polymer product and the stripping gas flushes out reactor gas entrained in the polymer product and strips and desorbs hydrocarbons, including ICA and unreacted monomer, dissolved in the polymer product powder. The stripping gas can be a gas inert to the polymerization process, such as nitrogen. In some instances, the polymer granules may also be treated with a small stream of humidified nitrogen to deactivate trace quantities of residual catalyst.

[0068] The stripped polymer product exits the bottom of the vessel 220 and is fed via line 236 to a finishing section (not shown), whereas the hydrocarbon-containing stripping gas effluent exits the top of the vessel 220 and is fed via line 222 to the low-pressure side of a recovery compressor 230. The high-pressure side of the recovery compressor 230 is connected to a condenser 240 where at least a portion of the hydrocarbons contained in the compressed stripping gas effluent are cooled and condensed. The condensed liquid is then recovered in a condensed liquids drum 250 before being sent via line 133 to line 113 to be recycled to the fluidized bed reactor 101.

[0069] The gaseous component of the stripping gas effluent remaining after passage through the condenser 240 is fed by line 252 to a split where a portion is removed and, after passage through a surge tank 270, is recycled to line 272 to assist in conveying the polymer product in the product discharge system 210 to the stripping vessel 220. The remainder of the stripping gas effluent in line 252 is fed by line 254 to a membrane separation system 260 where entrained hydrocarbons are removed from the effluent before the remainder of the effluent is fed via line 237 to a flare (not shown).

[0070] The product discharge system 210 shown in FIG. 2 comprises a first pair of lock hoppers and a second pair of lock hoppers connected in parallel between line 121 from the reactor and line 212 to the stripper vessel 220. Such lock hopper configuration and operation are described in U.S. Pat. No. 11,492,422, the substance of which is fully incorporated herein. Each pair of lock hoppers comprises an upstream lock hopper connected by valve means to the reactor and to a corresponding downstream lock hopper. Each downstream lock hopper is connected by valve means to the corresponding upstream lock hopper and by further valve means to line 212. Each downstream lock hopper is connected by a valve means to a source of conveying gas to facilitate transfer of polymer product from the downstream lock hopper to line 212. In addition, a first valved connection, also referred to as the upstream crosstie, is provided between the upstream lock hoppers, and a second valved connection, also referred to as the downstream crosstie, is provided between the downstream lock hoppers. The timing and frequency of opening and closing the upstream and downstream crossties in relation to the other lock hopper valves provides additional control of the rate of polymer product withdrawal from the reactor, the reactor pressure, and/or the amount of nitrogen in the reactor gas in the reactor. In other embodiments, a product discharge system can comprise other configurations of one or more pairs of upstream and downstream lock hoppers with or without crossties, or can comprise other equipment configured to separate polymer particles from reactor gas and reduce pressure to a level compatible with the stripper vessel 220. The product discharge system 210 can be utilized to provide alternate control of the rate of polymer product withdrawal from the reactor, the reactor pressure, and/or the amount of nitrogen in the reactor gas in the reactor.

[0071] Since the monomer stripping vessel 220 operates at a lower pressure, often a significantly lower pressure, than the reactor 101, the construction and operation of the product discharge system 210 is designed not only to provide efficient removal of the solid polymer

product but also to allow reduction in effluent pressure without excessive loss of the reactor contents.

Polymerization Process Control System

[0072] FIG. 3 is a block diagram of a control system 300 that can be used to control the polymerization system comprising a production loop 100 (comprising production loop peripheral equipment 102 and reactor 101) and/or a recovery loop 200 (comprising the recovery loop peripheral equipment 202). The control system 300 may be a distributed control system (DCS), a direct digital controller (DDC), a programmable logic controller (PLC), or other suitable systems or combination of systems. The control system has a processor that implements machine readable instructions from a storage system to provide a process simulation model 310 and an optimizer 320. Illustrative processors may include a single core processor, a multiple core processor, a virtual processor, a virtual processor in a cloud implementation, an application specific integrated circuit (ASIC), or a combination of these systems. Illustrative storage systems can include random access memory (RAM), read only memory (ROM), hard drives, virtual hard drives, RAM drives, cloud storage systems, optical storage systems, physically encoded instructions (for example, in an ASIC), or a combination of these systems.

[0073] In some embodiments, a method of optimizing a gas-phase polymerization process comprises simulating the operation of the gas phase polymerization process using a process simulation model 310 of steady state operation of the process to produce a simulated output comprising predicted values of the set of control parameters 315 based on selected values of the set of manipulated parameters 305. The gas phase polymerization process comprises a gas-phase fluidized bed reactor 101, production loop peripheral equipment 102 and recovery loop peripheral equipment 202.

[0074] The gas-phase fluidized bed reactor 101 has operating conditions comprising a set of manipulated parameters 305. The combination of the production loop 100 and the recovery loop 200 have operating conditions comprising a set of control parameters 315. Each control parameter in the set of control parameters 315 is a function of one or more of the manipulated parameters 305 and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process.

[0075] In some embodiments, a method for optimizing a gas-phase polymerization process further comprises measuring one or more inputs 325 from the process to confirm or correct output from the process simulation model.

[0076] In some embodiments, a method for optimizing a gas-phase polymerization process further comprises performing process optimization using an optimizer by selecting values for each parameter in the set of manipulated parameters 305 based on one or more measured inputs 325 from the process and the simulated output from the process simulation model 310 to minimize or maximize an objective function 330 within the constraints of the set of control parameters 315. Objective parameters 335 (e.g., costs of inputs to the process and value of outputs from the process) are provided to the objective function 330 for correlation to applicable aspects of the process.

[0077] In some embodiments, a method for optimizing a gas-phase polymerization process further comprises using the selected values for each parameter in the set of manipulated parameters 305 to control the process.

[0078] In some embodiments, operation of the production loop 100 comprises: withdrawing a gas-phase stream from the gas-phase fluidized bed reactor 101, wherein the gas-phase stream comprises an induced condensing agent (ICA) component; pressurizing the gas-phase stream in a recycle gas compressor to produce a pressurized recycle stream; cooling the pressurized recycle stream to condense at least a portion of the ICA component to produce a condensed recycle stream; and recycling the condensed recycle stream to the gas-phase fluidized bed reactor 101.

[0079] In some embodiments, the production loop 100 comprises the following: a fluidized bed in the gas-phase fluidized bed reactor 101 comprising polymer particles; the ICA component is a mixture of two or more induced condensing agents; and the amount of the ICA component is maximized to a stickiness limit of the polymer particles, wherein the stickiness limit is determined by the relative amounts of the two or more induced condensing agents in the ICA component and solubility of each of the two or more induced condensing agents in the polymer particles relative to isopentane.

[0080] In some embodiments, the production loop 100 comprises the following: a fluidized bed in the gas-phase fluidized bed reactor 101 comprising polymer particles; and the amount of the ICA component is maximized by reducing reactor temperature and adding additional ICA to a stickiness limit of the polymer particles.

[0081] In some embodiments, operation of the recovery loop 200 comprises: withdrawing a portion of first polymer particles from a fluidized bed of the gas-phase fluidized bed reactor 101;

separating the first polymer particles from entrained reactor gas in a product discharge system to produce a first polymer product, wherein the first polymer product comprises a first amount of an induced condensing agent (ICA) component; contacting the first polymer product with an inert gas in a stripper vessel to form a second polymer product and second gas stream, wherein the second
5 polymer product comprises a reduced amount of the ICA component, and the second gas stream comprises a gas-phase of the ICA component removed from the first polymer product; cooling the second gas stream to condense at least a portion of the gas-phase of the ICA component to form a liquid stream comprising the condensed ICA and a third gas stream; and recycling the hydrocarbon liquid stream to the gas-phase fluidized bed reactor 101.

10 [0082] In some embodiments, the set of manipulated parameters 305 comprises make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor.

[0083] The set of control parameters 315 can comprise reactor cooling capacity, cycle gas
15 condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof.

20 [0084] Simulating the operation of the process can comprise simulating the operation of the process concurrently with the operation of the process.

[0085] Also or instead, simulating the operation of the process can comprise measuring feedback inputs 325 from the process, and correcting simulation results based on the measured feedback inputs 325 from the process.

25 [0086] Optionally, process optimization can be performed independently of simulating the operation of the process, or it can be performed as part of simulating the operation of the process.

[0087] The objective(s) can relate to quantifiable aspect(s) of operating the gas phase polymerization process. An objective parameter in a set of objective parameters is a value of the quantifiable aspect corresponding to an input to or an output from the operation of the gas phase
30 polymerization process. The objective function is satisfied by maximizing or minimizing the sum of selected objective parameters.

[0088] In various embodiments, the objective function is maximizing a sum of values of selected products from and the costs of selected inputs to the gas phase polymerization process.

[0089] Also or instead, the objective function can minimize an amount of energy consumed in the process to produce a polymer product, wherein energy is measured as energy per mass unit of the polymer product.

[0090] In another aspect, a process control system for controlling a gas-phase polymerization process is disclosed herein. The process control system comprises a process simulation model, an optimizer, and a multiple-input/multiple-output controller.

[0091] The process simulation model simulates steady state operation of the process to produce a simulated output comprising predicted values of a set of control parameters based on selected values of a set of manipulated parameters.

[0092] The optimizer selects values for each parameter in the set of manipulated parameters based on one or more measured inputs from the process and the simulated output from the process simulation model to satisfy an objective function within the constraints of the set of control parameters.

[0093] The multiple-input/multiple-output controller is adapted to produce, during each operational cycle of the process control system, multiple control outputs configured to control the process based on the selected values of each parameter in a set of manipulated parameters provided to the multiple-input/multiple output controller from the optimizer during each operational cycle of the process control system. "Operational cycle" as used in this context refers to a single execution of a control process (e.g., any of the control processes described herein, such as any of the control methods described in the "Certain Embodiments" section below; and/or, e.g., an execution of the simulation of steady state operation of the process to produce a simulated output comprising predicted values of a set of control parameters, based on selected values of a set of manipulated parameters). It is envisioned that the controller could control the gas-phase polymerization process using a continuous series of operational cycles. In this way, the process controller is continually, serially, intaking inputs and producing outputs (e.g., the aforementioned simulated outputs) that can be adapted for ongoing continuous control of the polymerization process.

[0094] The process control system can be adapted to control a gas phase polymerization process. The gas-phase polymerization process comprises a gas-phase fluidized bed reactor, a production loop, and a recovery loop. The gas-phase fluidized bed reactor has operating conditions

comprising the set of manipulated parameters. The combination of the production loop and the recovery loop has operating conditions comprising the set of control parameters. Each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process.

[0095] In various embodiments, the production loop can implement: withdrawing a gas-phase stream from a gas-phase fluidized bed reactor, wherein the gas-phase stream comprises an induced condensing agent (ICA) component; pressurizing the gas-phase stream in a recycle gas compressor to produce a pressurized recycle stream; cooling the pressurized recycle stream to condense at least a portion of the ICA component to produce a condensed recycle stream; and recycling the condensed recycle stream to the gas-phase fluidized bed reactor.

[0096] The recovery loop can implement: withdrawing a portion of first polymer particles from a fluidized bed of a gas-phase fluidized bed reactor and separating the first polymer particles from entrained reactor gas in a product discharge system to produce a first polymer product, wherein the first polymer product comprises a first amount of an induced condensing agent (ICA) component. The first polymer product is contacted with an inert gas in a stripper vessel to form a second polymer product and second gas stream. The second polymer product comprises a reduced amount of the ICA component, and the second gas stream comprises a gas-phase of the ICA component removed from the first polymer product. The second gas stream is cooled to condense at least a portion of the gas-phase of the ICA component to form a liquid stream comprising the condensed ICA and a third gas stream. The ICA is recycled to the gas-phase fluidized bed reactor.

[0097] The set of manipulated parameters can include one or more of: make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor.

[0098] The set of control parameters can include one or more of: reactor cooling capacity, cycle gas condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof.

Optimization of gas phase polymerization process

[0099] Disclosed are methods for optimizing a gas phase polymerization process, wherein the process comprises a gas phase fluidized bed reactor, a production loop, and a recovery loop. The gas phase fluidized bed reactor has operating conditions comprising a set of manipulated parameters. The combination of the production loop and the recovery loop has operating conditions comprising a set of control parameters, and each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process.

[0100] A method for optimizing the gas phase polymerization process comprises simulating the operation of the process using a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of the set of control parameters based on selected values of the set of manipulated parameters. Selected measured values from the operating process modeled by the process simulation model are obtained to confirm or correct output from the process simulation model. The process is optimized using a process optimizer to select values for each parameter in the set of manipulated parameters based on one or more measured inputs from the process and the simulated output from the process simulation model to satisfy an objective function within the constraints of the set of control parameters. The selected values for each parameter in the set of manipulated parameters to control the process.

[0101] In various embodiments, the production loop can implement withdrawing a gas phase stream from the gas phase fluidized bed reactor, wherein the gas phase stream comprises an induced condensing agent (ICA) component. The withdrawn gas stream is pressurized in a recycle gas compressor to produce a pressurized recycle stream. The pressurized recycle stream is cooled in a recycle gas cooler to condense at least a portion of the ICA component to produce a condensed recycle stream. The condensed recycle stream is then recycled to the gas phase fluidized bed reactor.

[0102] A fluidized bed in the gas phase fluidized bed reactor can comprise polymer particles, and the reactor gas in the reaction zone can comprise an ICA component which is a mixture of two or more induced condensing agents. The amount of the ICA component is maximized to a stickiness limit of the polymer particles, wherein the stickiness limit is determined by the relative amounts of the two or more induced condensing agents in the ICA component and solubility of each of the two or more induced condensing agents in the polymer particles relative to isopentane.

A method for maximizing the partial pressure of the ICA component, and thereby maximizing the polymer production rate, in this manner is described in US 2022/0098332, the substance of which is fully incorporated herein by reference. In some embodiments, the ratio of two induced condensing agents in the ICA component is a manipulated parameter and the stickiness limit of polymer particles is a control parameter which is an inequality constraint.

[0103] Also or instead, a fluidized bed in the gas phase fluidized bed reactor can comprise polymer particles, and the reactor gas in the reaction zone can comprise an ICA component. The amount of the ICA component is maximized by reducing reactor temperature and adding additional ICA to a stickiness limit of the polymer particles. In some embodiments, the reactor temperature and the partial pressure of the ICA component are manipulated parameters and the stickiness limit of polymer particles is a control parameter which is an inequality constraint.

[0104] In accordance with various embodiments, the recovery loop implements withdrawing a portion of first polymer particles from a fluidized bed of the gas phase fluidized bed reactor. Entrained reactor gas is separated from the withdrawn first polymer particles in a product discharge system and returned to the reactor. The product discharge system further functions to consolidate the withdrawn first polymer particles and reduce pressure to level compatible with the downstream stripper vessel. The first polymer particles are then transported by convey gas and/or reactor gas to the stripper vessel. The polymer particles enter the upper portion of the stripper vessel while inert gas (e.g., nitrogen) is added to the bottom of the stripper vessel. Countercurrent contact of the inert gas and the polymer particles results in at least a portion of the absorbed ICA being removed from the polymer particles to exit the stripper vessel in an overhead gas stream. The stripped polymer product is sent to further finishing processes and storage. The stripper overhead gas stream is compressed and cooled to condense at least a portion of the ICA to form a liquid ICA stream and a vent stream. A portion of the vent stream is routed to the product discharge system to assist in conveying polymer particles to the stripper vessel and the remainder is removed from the gas-phase polymerization facility. The liquid ICA stream is recycled to the gas-phase reactor.

[0105] The product discharge system can comprise first and second pairs of upstream and downstream lock hoppers having a crosstie between upstream lock hoppers, a crosstie between downstream lock hoppers, and valve means to enable control of reaction zone pressure and/or nitrogen partial pressure in the gas phase fluidized bed reactor. Such lock hopper operation is disclosed in U.S. Pat. No. 11,492,422, the substance of which is fully incorporated herein by

reference. The reaction zone pressure can in various embodiments be a manipulated parameter correlating to nitrogen partial pressure in the gas phase fluidized bed reactor as a control parameter.

[0106] Optionally, the recovery loop can further comprise a photoionization detector configured to ionize C₄ to C₈ hydrocarbons to measure a hydrocarbon content of the polymer product discharged from the stripper vessel. The use of such photoionization detector is described in U.S. Pat. No. 11,578,147, the substance of which is fully incorporated herein by reference. Moreover, the partial pressure of the ICA composition in the reaction zone can be a manipulated parameter correlating to nitrogen concentration in the reactor, the production loop, and the recovery loop as control parameters.

[0107] Also or instead, the set of manipulated parameters can comprise any one of more of the following: make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas phase fluidized bed reactor, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor.

[0108] Further, the set of control parameters can comprise any one or more of the following in accordance with various embodiments: reactor cooling capacity, cycle gas condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof.

[0109] A non-exhaustive list of control parameters and manipulated parameters upon which these control parameters depend are shown in Table 1, below. This illustrates the interdependence of the listed control parameters, but in some instances it will be appreciated that some control parameters are functions of other control parameters in addition to certain manipulated parameters. Traditional linear controls are unable to optimize the process when it is desired to optimize so many variables simultaneously. Furthermore, traditional control models do not include control parameters related to the recovery loop.

TABLE 1

Control parameters	Manipulated parameters (reaction zone of the gas-phase fluidized bed reactor)				
	ICA component composition	Partial pressure of the ICA component	Partial pressure of ethylene	Temperature	Pressure
Reactor cooling capacity	X	X		X	X
Cycle gas condensation thermodynamics	X	X			
Hydrocarbon solubility in polymer particles	X	X		X	X
Thermodynamic closed-loop control relationships	X	X	X	X	X
Polymer particle stickiness	X	X		X	X
Catalyst productivity			X	X	
Discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance*	X	X			
Purging nitrogen requirements*	X	X	X	X	X
VLE flashes*	X	X		X	
Vent to flare flow rates and compositions*	X	X			
Production and recovery loops and reactor nitrogen balance*	X	X		X	

* related to recovery loop

5 [0110] Simulating the operation of the process optionally can comprise simulating the operation of the process concurrently with the operation of the process, thereby creating a simulated process “shadow.” Various simulation techniques may be used with this approach, from high Fidelity first principal models to the “black box” generic modeling. For example, it is known to implement process simulation using HYSYS (a high-fidelity simulation program) when
 10 designing a plant and to later use that same simulation to assist in performing plant operations. Concurrent simulation, on the other hand, allows continuous feedback updates of the modeling results from the process measurements, resulting in superior performance and fidelity to be achieved, particularly compared to simple modeling techniques, such as step responses when concurrent simulation is not applied. As a result, the cost of developing and running simulation is
 15 dramatically reduced, and is acceptable for common implementation process control systems. A process control system with concurrent simulation features would improve performance and extend functionality, including for intensive processing system diagnostics and optimization.

[0111] Simulating the operation of the process can comprise measuring feedback inputs from the process, and correcting simulation results based on the measured feedback inputs from the
 20 process.

[0112] In some example operations in accordance with the present disclosure, gas-phase reactor production rates can be maximized by first increasing reactor temperature until limited by

cooling water capacity. Then ICA concentration is increased to a selected threshold value less than the stickiness limit for the selected polymerization system. Next, reactor temperature is decreased, and ICA concentration is further increased while maintaining the aforementioned threshold value.

5 [0113] In some instances, the extent to which reduction of reactor temperature is taken is limited by a minimum reactor temperature (that is, a lower limit of reactor temperature). A minimum or lower limit reactor temperature is determined by the dew point of the gas in the reactor. As the reaction zone temperature approaches the dew point of the reactor gas (cycle gas) in a selected polymerization system, the driving force to assure that all of the ICA composition is vaporized is reduced. That is to say, if less than all of the ICA composition is vaporized in the reactor, then less than all of the latent heat of vaporization in the ICA composition is available for cooling the reactor.

[0114] In some instances, the extent to which reactor temperature is reduced is limited by a maximum density of the reactor gas (cycle gas). Increasing the density of the recycle gas with a higher concentration of an ICA composition reduces the sweep velocity, or velocity of recycle gas through the holes in the distributor plate. If the sweep velocity is too low, polymer may begin to build up to eventually cause plugging of the holes in the distributor plate. Sweep velocity can be increased by opening the discharge vanes on the cycle loop compressor. However, this can have the follow-on effect of increasing superficial velocity in the reactor due to higher density of the recycle gas.

[0115] Also or instead, the extent to which the last step is taken may be limited by a the particular properties of a polymer grade being produced, such as, but not limited to, density, I_2 , I_{21} , and/or MIR (I_{21}/I_2).

[0116] Also or instead, the extent to which the last step is taken can be limited by losses of the ICA composition from the reactor. A higher concentration of the ICA composition in the recycle gas results in a higher amount of the ICA composition being absorbed by the polymer product. Processes and/or equipment in the product recovery portion of a polymer production facility may have limitations in the amount of the ICA composition in the polymer product that can be recovered for recycling or disposal.

30 Catalyst

[0117] Exemplary catalysts suitable for use in the embodiments described may include: Ziegler Natta catalysts, chromium based catalysts, vanadium based catalysts (e.g., vanadium

oxychloride and vanadium acetylacetonate), metallocene catalysts and other single-site or single-site-like catalysts, cationic forms of metal halides (e.g., aluminum trihalides), anionic initiators (e.g., butyl lithiums), cobalt catalysts and mixtures thereof, nickel catalysts and mixtures thereof, rare earth metal catalysts (i.e., those containing a metal having an atomic number in the Periodic Table of 57 to 103), such as compounds of cerium, lanthanum, praseodymium, gadolinium and neodymium. A single catalyst may be used, or a mixture of catalysts may be employed, if desired. The catalyst may be soluble or insoluble, supported or unsupported. Further, the catalyst may be a prepolymer, spray dried with or without a filler, a liquid, or a solution, slurry/suspension, or dispersion.

10 [0118] Traditional Ziegler-Natta catalysts are transitional metal catalysts that are well known in the art. Examples of transition metal catalysts are discussed in U.S. Pat. Nos. 4,115,639, 4,077,904, 4,482,687, 4,564,605, 4,721,763, 4,879,359 and 4,960,741, the disclosures of which are hereby fully incorporated herein by reference. The transition metal catalyst compounds that may be used in the present invention include transition metal compounds from Groups 3 to 17, 15 preferably 4 to 12, more preferably 4 to 6 of the Periodic Table of Elements.

[0119] Transition metal catalyst compounds based on magnesium/titanium electron-donor complexes that are useful in the invention are described in, for example, U.S. Pat. Nos. 4,302,565 and 4,302,566, the disclosures of which are hereby fully incorporated herein by reference.

20 [0120] British Patent Application No. 2,105,355 and U.S. Pat. No. 5,317,036, the disclosures of which are hereby incorporated herein by reference, describe various vanadium catalyst compounds.

[0121] Still other transition metal catalyst compounds and catalyst systems suitable for use in the present invention are disclosed in U.S. Pat. Nos. 4,124,532, 4,302,565, 4,302,566, 4,376,062, 4,379,758, 5,066,737, 5,763,723, 5,849,655, 5,852,144, 5,854,164 and 5,869,585 and published 25 EP-A2 0 416 815 A2 and EP-A1 0 420 436, the disclosures of which are hereby fully incorporated herein by reference.

[0122] Other catalysts may include cationic catalysts such as $AlCl_3$, and other cobalt, iron, nickel and palladium catalysts well known in the art. See for example U.S. Pat. Nos. 3,487,112, 4,472,559, 4,182,814 and 4,689,437, the disclosures of which are hereby fully incorporated herein 30 by reference.

[0123] For more details on Ziegler-Natta catalysts, see for example, U.S. Pat. Nos. 3,687,920, 4,086,408, 4,376,191, 5,019,633, 4,482,687, 4,101,445, 4,560,671, 4,719,193, 4,755,495, 5,070,055, the disclosures of which are hereby incorporated herein by reference.

[0124] Also or instead, a catalyst compound of the present disclosure can be a chromium or chromium-based catalyst. Such catalysts and polymerization processes have been described, for example, in U.S. Patent Application Publication No. 2011/0010938 and U.S. Pat. Nos. 7,915,357, 8,129,484, 7,202,313, 6,833,417, 6,841,630, 6,989,344, 7,504,463, 7,563,851, 8,420,754, and 8,101,691.

[0125] In some embodiments processes disclosed herein utilize single-site transition metal catalysts. Exemplary -site transition metal catalysts suitable for use in the embodiments described may include metallocene catalysts and other single-site or single-site-like catalysts. A single catalyst may be used, or a mixture of catalysts may be employed, if desired. The catalyst may be soluble or insoluble, supported or unsupported. Further, the catalyst may be a prepolymer, spray dried with or without a filler, a liquid, or a solution, slurry/suspension, or dispersion.

[0126] Metallocenes may include “half sandwich” and “full sandwich” compounds having one or more pi-bonded ligands (e.g., cyclopentadienyl and ligands isolobal to cyclopentadienyl) bound to at least one Group 3 to Group 12 metal atom (Including the Lanthanide series and Actinide series elements), and one or more leaving groups bound to the at least one metal atom. The metallocene may be supported on a support material, and may be supported with or without another catalyst component.

[0127] The structure of the metallocene catalyst compound may take on many forms, such as those disclosed in, for example, U.S. Pat. Nos. 5,026,798, 5,703,187, and 5,747,406, including a dimer or oligomeric structure, such as disclosed in, for example, U.S. Pat. Nos. 5,026,798 and 6,069,213. Others include those catalysts described in U.S. Patent Application Publication Nos. US2005/0124487A1, US2005/0164875A1, and US2005/0148744. In some embodiments, the metallocene may be formed with a hafnium metal atom (e.g., bis(n-propylcyclopentadienyl) hafnium X_n, bis(n-butylcyclopentadienyl) hafnium X_n, or bis(n-pentylcyclopentadienyl) hafnium X_n, where X is one of chloride or fluoride and n is 2), such as is described in U.S. Pat. Nos. 6,242,545 and 7,157,531.

[0128] A catalyst compound of the present disclosure can be a chromium or chromium-based catalyst. Chromium-based catalysts include chromium oxide (CrO₃) and silylchromate catalysts. Chromium catalysts have been the subject of much development in the area of continuous

fluidized-bed gas-phase polymerization for the production of polyethylene polymers. Such catalysts and polymerization processes have been described, for example, in U.S. Patent Application Publication No. 2011/0010938 and U.S. Pat. Nos. 7,915,357, 8,129,484, 7,202,313, 6,833,417, 6,841,630, 6,989,344, 7,504,463, 7,563,851, 8,420,754, and 8,101,691.

- 5 [0129] Other catalysts for use in processes of the present disclosure include “non-metallocene complexes” that are defined to be transition metal complexes that do not feature a cyclopentadienyl anion or substituted cyclopentadienyl anion donors (e.g., cyclopentadienyl, fluorenyl, indenyl, methylcyclopentadienyl). Examples of families of non-metallocene complexes that may be suitable can include late transition metal pyridylbisimines (e.g., U.S. Pat. No. 7,087,686), group 4
10 pyridyldiamidos (e.g., U.S. Pat. No. 7,973,116), quinolinyldiamidos (e.g., U.S. Pub. No. 2018/0002352 A1), pyridylamididos (e.g., U.S. Pat. No. 7,087,690), phenoxyimines (e.g., Accounts of Chemical Research 2009, 42, 1532-1544), and bridged bi-aromatic complexes (e.g., U.S. Pat. No. 7,091,292), the substance of which are fully incorporated herein by reference.

Polyolefin Products

- 15 [0130] This disclosure also relates to compositions of matter produced by the methods described.

- [0131] In some embodiments, the methods described produce ethylene homopolymers or ethylene copolymers, such as any of those previously described herein, e.g., ethylene- α -olefin (e.g., C₃ to C₂₀) copolymers (such as ethylene-butene copolymers, ethylene-hexene and/or
20 ethylene-octene copolymers) having: a M_w/M_n of greater than 1 to 4, or greater than 1 to 3.

- [0132] Likewise, the processes of this disclosure produce ethylene copolymers. In some embodiments, the polyolefin copolymers produced have from about 0 mol % to about 25 mol %, from about 0.5 mol % to about 20 mol %, from about 1 mol % to about 15 mol %, or from about 3 mol % to about 10 mol % of one or more C₃ to C₂₀ olefin comonomer. The one or more C₃ to
25 C₂₀ olefin comonomer may include C₃ to C₁₂ alpha-olefin, such as propylene, butene, hexene, octene, decene, or dodecene, and preferably the comonomer is an alpha-olefin (e.g., 1-butene, 1-hexene, 1-octene, 1-decene, or 1-dodecene).

- [0133] In some embodiments, the monomer is ethylene and the comonomer is hexene (especially 1-hexene), for example, from about 1 mol % to about 15 mol % hexene, such as about
30 1 mol % to about 10 mol %.

- [0134] In at least one embodiment, the ethylene polymer composition is produced having: i) at least 50 mol % ethylene-derived content; ii) a density of 0.89 g/cc or more, such as 0.918 g/cc or

more, or 0.935 g/cc or more; and a g'_{vis} of about 0.97 or greater. The composition optionally has comonomer-derived content, such as content derived from one or more of 1-butene, 1-hexene- and 1-octene.

[0135] The polymers produced can have an M_w of 5,000 g/mol to 1,000,000 g/mol, such as 25,000 g/mol to 750,000 g/mol, or 50,000 to 500,000 g/mol, and/or an M_w/M_n of greater than 1 to about 40, such as about 1.2 to about 20, about 1.3 to about 10, about 1.4 to about 5, about 1.5 to about 4, or about 1.5 to about 3, with ranges from any foregoing low end to any foregoing high end contemplated.

[0136] The polymer produced can have either a unimodal or multimodal molecular weight distribution as determined by Gel Permeation Chromatography (GPC). By “unimodal” is meant that the GPC trace has one peak or inflection point. By “multimodal” is meant that the GPC trace has at least two peaks or inflection points. An inflection point is that point where the second derivative of the curve changes in from negative to positive or vice versa. Thus, in certain embodiments, the polymer produced can have a unimodal molecular weight distribution. Alternatively, in yet further embodiments, the polymer produced has a bimodal molecular weight distribution.

[0137] Unless otherwise indicated modality, M_w , M_n , M_z , MWD, g value and g'_{vis} are determined by using a High Temperature Size Exclusion Chromatograph (either from Waters Corporation or Polymer Laboratories), which may be equipped with a differential refractive index detector (DRI), a light scattering (LS) detector, and a viscometer. Experimental details, including detector calibration, are described in: T. Sun, P. Brant, R.R. Chance, and W.W. Graessley, *Macromolecules*, Volume 34, Number 19, 6812-6820, (2001).

Certain Embodiments

[0138] Disclosed herein is a method optimizing a gas-phase polymerization process, wherein:

- the process comprises a gas-phase fluidized bed reactor, a production loop, and a recovery loop;
- the gas-phase fluidized bed reactor has operating conditions comprising a set of manipulated parameters;
- the combination of the production loop and the recovery loop has operating conditions comprising a set of control parameters; and
- each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a

physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process; and

the method comprises:

- 5 simulating the operation of the process using a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of the set of control parameters based on selected values of the set of manipulated parameters;
- measuring one or more inputs from the process to confirm or correct output from the process simulation model;
- 10 performing process optimization using an optimizer by selecting values for each parameter in the set of manipulated parameters based on one or more measured inputs from the process and the simulated output from the process simulation model to minimize or maximize an objective function within the constraints of the set of control parameters; and
- 15 using the selected values for each parameter in the set of manipulated parameters to control the process.

[0139] In some embodiments, the method is further characterized by one or more of the following:

- a) the production loop implements:
 - 20 withdrawing a gas-phase stream from a gas-phase fluidized bed reactor, wherein the gas-phase stream comprises an induced condensing agent (ICA) component;
 - pressurizing the gas-phase stream in a recycle gas compressor to produce a pressurized recycle stream;
 - cooling the pressurized recycle stream to condense at least a portion of the ICA
 - 25 component to produce a condensed recycle stream; and
 - recycling the condensed recycle stream to the gas-phase fluidized bed reactor;
- b) the recovery loop implements:
 - withdrawing a portion of first polymer particles from a fluidized bed of a gas-phase
 - fluidized bed reactor;
 - 30 separating the first polymer particles from entrained reactor gas in a product discharge system to produce a first polymer product, wherein the first polymer

- product comprises a first amount of an induced condensing agent (ICA) component;
- contacting the first polymer product with an inert gas in a stripper vessel to form a second polymer product and second gas stream, wherein the second polymer product comprises a reduced amount of the ICA component, and the second gas stream comprises a gas-phase of the ICA component removed from the first polymer product;
- cooling the second gas stream to condense at least a portion of the gas-phase of the ICA component to form a liquid stream comprising the condensed ICA and a third gas stream; and
- recycling the hydrocarbon liquid stream to the gas-phase fluidized bed reactor; wherein in further embodiments, the recovery loop further comprises comprising recovering the second polymer product for finishing and storage;
- c) the set of manipulated parameters comprises make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor;
- d) the set of control parameters comprises reactor cooling capacity, cycle gas condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof;
- e) simulating the operation of the process comprises simulating the operation of the process concurrently with the operation of the process;
- f) simulating the operation of the process comprises: measuring feedback outputs from the process, and correcting simulation results based on the measured feedback outputs from the process;
- g) performing process optimization comprises performing process optimization independent of simulating the operation of the process;

- h) the objective function is maximizing a difference between value of a polymer product produced in the process and a cost of inputs to produce the polymer product; and
- i) the objective function is minimizing an amount of energy per mass unit of a polymer product consumed in the process to produce the polymer product.

5 **[0140]** In some embodiments of the method related to the production loop:
a fluidized bed in the gas-phase fluidized bed reactor comprises polymer particles;
the ICA component is a mixture of two or more induced condensing agents;
the set of manipulated parameters comprises an amount of the ICA component and the
relative amounts of the two or more induced condensing agents in the ICA component;
10 the set of control parameters comprises a stickiness of the polymer particles; and
the gas-phase polymerization process is optimized by maximizing the amount of the ICA
component while maintaining the stickiness of the polymer particles below a constraint
of a stickiness limit of the polymer particles, wherein the stickiness limit is determined
by (i) the relative amounts of the two or more induced condensing agents in the ICA
15 component and (ii) solubility of each of the two or more induced condensing agents in
the polymer particles relative to isopentane.

[0141] In some embodiments of the method related to the production loop:
a fluidized bed in the gas-phase fluidized bed reactor comprises polymer particles;
the set of manipulated parameters comprises an amount of the ICA component and a reactor
20 temperature;
the set of control parameters comprises a stickiness of the polymer particles; and
the gas-phase polymerization process is optimized by maximizing the amount of the ICA
component by reducing the reactor temperature and adding additional ICA, while
maintaining the stickiness of the polymer particles below a constraint of a stickiness
25 limit of the polymer particles.

[0142] In some embodiments of the method related to the recovery loop:
the product discharge system comprises first and second pairs of upstream and downstream
lock hoppers having a crosstie between upstream lock hoppers, a crosstie between
downstream lock hoppers, and valve means to enable control of reaction zone pressure
30 base on the amount of reactor gas withdrawn in each product discharge cycle, wherein
increasing withdrawal rates decreases reaction zone pressure and decreasing
withdrawal rates increases reaction zone pressure;

the set of manipulated parameters comprises reaction zone pressure in the gas-phase fluidized bed reactor;

the set of control parameters comprises direct reactor venting; and

the gas-phase polymerization process is optimized by controlling reaction zone pressure through reactor gas withdrawal rates to maintain the amount of direct reactor venting below a constraint of a threshold amount of direct reactor venting.

[0143] In some embodiments of the method related to the recovery loop:

the recovery loop comprises a photoionization detector configured to ionize C₄ to C₈ hydrocarbons to measure a hydrocarbon content of the second polymer product;

the set of manipulated parameters comprises a reaction zone pressure, a reaction zone temperature, and an amount of the ICA component in the gas-phase fluidized bed reactor;

the set of control parameters comprises the hydrocarbon content of the second polymer product and the amount of effluent to flare; and

the gas-phase polymerization process is optimized by controlling the reaction zone pressure, the reaction zone temperature, and the amount of the ICA component in the gas-phase fluidized bed reactor to maintain the amount of effluent to flare below a constraint of a threshold amount of effluent to flare, and further to maintain the amount of hydrocarbon in the second polymer product below a threshold amount of hydrocarbon in the second polymer product.

[0144] Disclosed herein is a process control system for controlling a gas-phase polymerization process, the process control system comprising:

a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of a set of control parameters based on selected values of a set of manipulated parameters;

an optimizer to select values for each parameter in the set of manipulated parameters based on one or more measured inputs from the process and the simulated output from the process simulation model to minimize or maximize an objective function within the constraints of the set of control parameters; and

a multiple-input/multiple-output controller adapted to produce, during each operational cycle of the process control system, multiple control outputs configured to control the process based on the selected values of each parameter in a set of manipulated

parameters provided to the multiple-input/multiple output controller from the process simulation model and/or the optimizer during each operational cycle of the process control system.

[0145] In some embodiments of the process control system:

- 5 a) the process comprises a gas-phase fluidized bed reactor, a production loop, and a recovery loop;
- b) the gas-phase fluidized bed reactor has operating conditions comprising a set of manipulated parameters;
- c) the combination of the production loop and the recovery loop has operating
10 conditions comprising a set of control parameters; and
- d) each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process.

15 [0146] In some embodiments of the process control system, the process is further characterized by one or more of:

- a) production loop implements:
 - withdrawing a gas-phase stream from a gas-phase fluidized bed reactor, wherein the
 gas-phase stream comprises an induced condensing agent (ICA) component;
 - 20 pressurizing the gas-phase stream in a recycle gas compressor to produce a
 pressurized recycle stream;
 - cooling the pressurized recycle stream to condense at least a portion of the ICA
 component to produce a condensed recycle stream; and
 - recycling the condensed recycle stream to the gas-phase fluidized bed reactor;
- 25 b) the recovery loop implements:
 - withdrawing a portion of first polymer particles from a fluidized bed of a gas-phase
 fluidized bed reactor;
 - separating the first polymer particles from entrained reactor gas in a product
 discharge system to produce a first polymer product, wherein the first polymer
30 product comprises a first amount of an induced condensing agent (ICA)
 component;

- 5 contacting the first polymer product with an inert gas in a stripper vessel to form a second polymer product and second gas stream, wherein the second polymer product comprises a reduced amount of the ICA component, and the second gas stream comprises a gas-phase of the ICA component removed from the first polymer product;
- cooling the second gas stream to condense at least a portion of the gas-phase of the ICA component to form a liquid stream comprising the condensed ICA and a third gas stream; and
- recycling the hydrocarbon liquid stream to the gas-phase fluidized bed reactor;
- 10 wherein in some embodiments the recovery loop further comprises recovering the second polymer product for finishing and storage.
- c) the set of manipulated parameters comprises make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor; and
- 15 d) the set of control parameters comprises reactor cooling capacity, cycle gas condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities,
- 20 VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof.

[0147] Disclosed herein is a second method optimizing a gas-phase polymerization process, wherein:

- 25 the process comprises a gas-phase fluidized bed reactor and a production loop;
- the gas-phase fluidized bed reactor has operating conditions comprising a set of manipulated parameters, comprising make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in a reaction zone of the gas-phase fluidized bed reactor;
- 30 the production loop has operating conditions comprising a set of control parameters; and

each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process; and

5 the method comprises:

simulating the operation of the process using a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of the set of control parameters based on selected values of the set of manipulated parameters;

10 measuring one or more inputs from the process to confirm or correct output from the process simulation model;

performing process optimization using an optimizer by selecting values for each parameter in the set of manipulated parameters based on one or more measured inputs from the process and the simulated output from the process simulation model to minimize or maximize an objective function within the constraints of the set of control parameters; and

15 using the selected values for each parameter in the set of manipulated parameters to control the process.

[0148] In some embodiments, the second method is further characterized by one or more of the following:

- 20 a) the production loop implements:
- withdrawing a gas-phase stream from a gas-phase fluidized bed reactor, wherein the gas-phase stream comprises an induced condensing agent (ICA) component; pressurizing the gas-phase stream in a recycle gas compressor to produce a
- 25 pressurized recycle stream;
- cooling the pressurized recycle stream to condense at least a portion of the ICA component to produce a condensed recycle stream; and
- recycling the condensed recycle stream to the gas-phase fluidized bed reactor;
- b) the set of control parameters comprises reactor cooling capacity, cycle gas
- 30 condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and

reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof;

- c) simulating the operation of the process comprises simulating the operation of the process concurrently with the operation of the process;
- d) simulating the operation of the process comprises:
measuring feedback outputs from the process, and
correcting simulation results based on the measured feedback outputs from the process;
- e) performing process optimization comprises performing process optimization independent of simulating the operation of the process;
- f) the objective function is maximizing a difference between value of a polymer product produced in the process and a cost of inputs to produce the polymer product; and
- g) the objective function is minimizing an amount of energy per mass unit of a polymer product consumed in the process to produce the polymer product.

[0149] In some embodiments of the second method related to the production loop:

a fluidized bed in the gas-phase fluidized bed reactor comprises polymer particles;
the ICA component is a mixture of two or more induced condensing agents;
the set of manipulated parameters comprises an amount of the ICA component and the relative amounts of the two or more induced condensing agents in the ICA component;
the set of control parameters comprises a stickiness of the polymer particles; and
the gas-phase polymerization process is optimized by maximizing the amount of the ICA component while maintaining the stickiness of the polymer particles below a constraint of a stickiness limit of the polymer particles, wherein the stickiness limit is determined by (i) the relative amounts of the two or more induced condensing agents in the ICA component and (ii) solubility of each of the two or more induced condensing agents in the polymer particles relative to isopentane.

[0150] In some embodiments of the method related to the production loop:

a fluidized bed in the gas-phase fluidized bed reactor comprises polymer particles;
the set of manipulated parameters comprises an amount of the ICA component and a reactor temperature;
the set of control parameters comprises a stickiness of the polymer particles; and

the gas-phase polymerization process is optimized by maximizing the amount of the ICA component by reducing the reactor temperature and adding additional ICA, while maintaining the stickiness of the polymer particles below a constraint of a stickiness limit of the polymer particles.

5 **[0151]** In some embodiments of the second method related to the recovery loop:

the product discharge system comprises first and second pairs of upstream and downstream lock hoppers having a crosstie between upstream lock hoppers, a crosstie between downstream lock hoppers, and valve means to enable control of reaction zone pressure base on the amount of reactor gas withdrawn in each product discharge cycle, wherein
10 increasing withdrawal rates decreases reaction zone pressure and decreasing withdrawal rates increases reaction zone pressure;

the set of manipulated parameters comprises reaction zone pressure in the gas-phase fluidized bed reactor;

the set of control parameters comprises direct reactor venting; and

15 the gas-phase polymerization process is optimized by controlling reaction zone pressure through reactor gas withdrawal rates to maintain the amount of direct reactor venting below a constraint of a threshold amount of direct reactor venting.

[0152] In some embodiments of the second method related to the recovery loop:

the recovery loop comprises a photoionization detector configured to ionize C₄ to C₈
20 hydrocarbons to measure a hydrocarbon content of the second polymer product;

the set of manipulated parameters comprises a reaction zone pressure, a reaction zone temperature, and an amount of the ICA component in the gas-phase fluidized bed reactor;

the set of control parameters comprises the hydrocarbon content of the second polymer
25 product and the amount of effluent to flare; and

the gas-phase polymerization process is optimized by controlling the reaction zone pressure, the reaction zone temperature, and the amount of the ICA component in the gas-phase fluidized bed reactor to maintain the amount of effluent to flare below a constraint of a threshold amount of effluent to flare, and further to maintain the amount of
30 hydrocarbon in the second polymer product below a threshold amount of hydrocarbon in the second polymer product.

Test Methods/Polymer Characterization

[0153] Density (g/cm³): Density measurements were made following ASTM D-1505.

[0154] Gel permeation chromatography ("GPC") 4D Methodology:

- a) Unless otherwise indicated, the distribution and the moments of molecular weight (M_w , M_n , M_z , M_w/M_n , etc.), the comonomer content (C_2 , C_3 , C_6 , etc.), the branching index (g'), and CCDI (M_w -specific, 5-95, and M_n - M_z) are determined by using a high temperature Gel Permeation Chromatography (Polymer Char GPC-IR) equipped with a multiple-channel band-filter based Infrared detector IR5, an 18-angle light scattering detector and a viscometer. Three Agilent PLgel 10- μ m Mixed-B LS columns are used to provide polymer separation. Aldrich reagent grade 1,2,4- trichlorobenzene ("TCB") with 300 ppm antioxidant butylated hydroxytoluene ("BHT") is used as the mobile phase. The TCB mixture is filtered through a 0.1- μ m Teflon filter and degassed with an online degasser before entering the GPC instrument. The nominal flow rate is 1.0 ml/min. and the nominal injection volume is 200 μ l. The whole system including transfer lines, columns, and detectors are contained in an oven maintained at 145°C. Given amount of polymer sample is weighed and sealed in a standard vial with 80- μ l flow marker (heptane) added to it. After loading the vial in the autosampler, polymer is automatically dissolved in the instrument with 8 ml added TCB solvent. The polymer is dissolved at 160°C with continuous shaking for about 1 hour for most polyethylene samples or 2 hours for polypropylene samples. The TCB densities used in concentration calculation are 1.463 g/ml at room temperature and 1.284 g/ml at 145°C. The sample solution concentration is from 0.2 to 2.0 mg/ml, with lower concentrations being used for higher molecular weight samples. The concentration (c), at each point in the chromatogram is calculated from the baseline-subtracted IR5 broadband signal intensity (I), using the following equation: $c = \beta I$, where β is the mass constant. The mass recovery is calculated from the ratio of the integrated area of the concentration chromatography over elution volume and the injection mass which is equal to the pre-determined concentration multiplied by injection loop volume. The conventional molecular weight (IR MW) is determined by combining universal calibration relationship with the column calibration which is performed with a series of monodispersed polystyrene (PS) standards ranging from 700 to 10M g/mole. The MW at each elution volume is calculated with following equation:

$$\log M = \frac{\log(K_{PS}/K)}{\alpha+1} + \frac{\alpha_{PS}+1}{\alpha+1} \log M_{PS},$$

where the variables with subscript "PS" stand for polystyrene while those without a subscript are for the test samples. In this method, $\alpha_{PS}=0.67$ and $K_{PS}=0.000175$, while α and K for other materials are as calculated and published in literature (Sun, T. et al. *Macromolecules* 2001, 34, 6812), except that for purposes of this invention and claims thereto, $\alpha=0.695$ and $K=0.000579$ for linear ethylene polymers, $\alpha=0.705$ and $K=0.0002288$ for linear propylene polymers, $\alpha=0.695$ and $K=0.000181$ for linear butene polymers, α is 0.695 and K is $0.000579 \times (1 - 0.0087 \times w_{2b} + 0.000018 \times (w_{2b})^2)$ for ethylene-butene copolymer where w_{2b} is a bulk weight percent of butene comonomer, α is 0.695 and K is $0.000579 \times (1 - 0.0075 \times w_{2b})$ for ethylene-hexene copolymer where w_{2b} is a bulk weight percent of hexene comonomer, and α is 0.695 and K is $0.000579 \times (1 - 0.0077 \times w_{2b})$ for ethylene-octene copolymer where w_{2b} is a bulk weight percent of octene comonomer. Concentrations are expressed in g/cm³, molecular weight is expressed in g/mole, and intrinsic viscosity (hence K in the Mark-Houwink equation) is expressed in dl/g unless otherwise noted.

b) The comonomer composition is determined by the ratio of the IR5 detector intensity corresponding to CH₂ and CH₃ channel calibrated with a series of PE and PP homo/copolymer standards whose nominal value are predetermined by NMR or FTIR. In particular, this provides the methyls per 1000 total carbons ("CH₃/1000TC") as a function of molecular weight. The short-chain branch ("SCB") content per 1000TC ("SCB/1000TC") is then computed as a function of molecular weight by applying a chain-end correction to the CH₃/1000TC function, assuming each chain to be linear and terminated by a methyl group at each end. The weight % comonomer is then obtained from the following expression in which f is 0.3, 0.4, 0.6, 0.8, and so on for C₃, C₄, C₆, C₈, and so on co-monomers, respectively:

$$w_2 = f * SCB/1000TC.$$

c) The bulk composition of the polymer from the GPC-IR and GPC-4D analyses is obtained by considering the entire signals of the CH₃ and CH₂ channels between the integration limits of the concentration chromatogram. First, the following ratio is obtained

$$\text{Bulk IR ratio} = \frac{\text{Area of CH}_3 \text{ signal within integration limits}}{\text{Area of CH}_2 \text{ signal within integration limits}}$$

d) Then the same calibration of the CH₂ and CH₃ signal ratio, as mentioned previously in obtaining the CH₃/1000TC as a function of molecular weight, is applied to obtain the bulk CH₃/1000TC. A bulk methyl chain ends per 1000TC ("bulk CH₃end/1000TC") is obtained by weight- averaging the chain-end correction over the molecular-weight range. Then

$$w2b = f * \text{bulk CH3}/1000\text{TC},$$

$$\text{bulk SCB}/1000\text{TC} = \text{bulk CH3}/1000\text{TC} - \text{bulk CH3end}/1000\text{TC},$$

and bulk SCB/1000TC is converted to bulk w2 in the same manner as described above.

- e) The LS detector is the 18-angle Wyatt Technology High Temperature DAWN HELEOSII. The LS molecular weight (M) at each point in the chromatogram is determined by analyzing the LS output using the Zimm model for static light scattering (*Light Scattering from Polymer Solutions*, Huglin, M. B., Ed.; Academic Press, 1972.):

$$\frac{K_0 c}{\Delta R(\theta)} = \frac{1}{MP(\theta)} + 2A_2 c.$$

- Here, $\Delta R(\theta)$ is the measured excess Rayleigh scattering intensity at scattering angle θ , c is the polymer concentration determined from the IR5 analysis, A_2 is the second virial coefficient, $P(\theta)$ is the form factor for a monodisperse random coil, and K_0 is the optical constant for the system:

$$K_0 = \frac{4\pi^2 n^2 (dn/dc)^2}{\lambda^4 N_A},$$

- where N_A is Avogadro's number, and (dn/dc) is the refractive index increment for the system. The refractive index, $n=1.500$ for TCB at 145°C and $\lambda = 665 \text{ nm}$. For analyzing polyethylene homopolymers, ethylene-hexene copolymers, and ethylene-octene copolymers, $dn/dc=0.1048 \text{ ml/mg}$ and $A_2=0.0015$; for analyzing ethylene-butene copolymers, $dn/dc=0.1048*(1-0.00126*w2) \text{ ml/mg}$ and $A_2= 0.0015$ where $w2$ is weight percent butene comonomer.

- f) A high temperature Agilent (or Viscotek Corporation) viscometer, which has four capillaries arranged in a Wheatstone bridge configuration with two pressure transducers, is used to determine specific viscosity. One transducer measures the total pressure drop across the detector, and the other, positioned between the two sides of the bridge, measures a differential pressure. The specific viscosity, η_s , for the solution flowing through the viscometer is calculated from their outputs. The intrinsic viscosity, η_s , at each point in the chromatogram is calculated from the equation $[\eta]=\eta_s/c$, where c is concentration and is determined from the IR5 broadband channel output. The viscosity MW at each point is calculated as $M = K_{ps}M^{aps+1}/[\eta]$, where α_{ps} is 0.67 and K_{ps} is 0.000175.

- g) The branching index (g'_{vis}) is calculated using the output of the GPC-IR5-LS-VIS method as follows. The average intrinsic viscosity, $[\eta]_{avg}$, of the sample is calculated by:

$$[\eta]_{i,j} = \frac{\sum c_i [\eta]_i}{\sum c_i},$$

where the summations are over the chromatographic slices, i , between the integration limits.

h) The branching index g'_{vjs} is defined as $g'_{vjs} = ([\eta]_{avg})/(KM_v^\alpha)$, where M_v is the viscosity-average molecular weight based on molecular weights determined by LS analysis and the K and α are for the reference linear polymer, which are, for purposes of this invention and claims thereto, $\alpha = 0.695$ and $K = 0.000579$ for linear ethylene polymers, $\alpha = 0.705$ and $K = 0.0002288$ for linear propylene polymers, $\alpha = 0.695$ and $K = 0.000181$ for linear butene polymers, $\alpha = 0.695$ and K is $0.000579 \times (1 - 0.0087 w_{2b} + 0.000018 \times (w_{2b})^2)$ for ethylene-butene copolymer where w_{2b} is a bulk weight percent of butene comonomer, α is 0.695 and K is $0.000579 \times (1 - 0.0075 \times w_{2b})$ for ethylene-hexene copolymer where w_{2b} is a bulk weight percent of hexene comonomer, and α is 0.695 and K is $0.000579 \times (1 - 0.0077 \times w_{2b})$ for ethylene-octene copolymer where w_{2b} is a bulk weight percent of octene comonomer. Concentrations are expressed in g/cm^3 , molecular weight is expressed in $g/mole$, and intrinsic viscosity (hence K in the Mark- Houwink equation) is expressed in dl/g unless otherwise noted. Calculation of the w_{2b} values is as discussed above.

EXAMPLES

[0155] The following examples are included to demonstrate some embodiments of the invention. It should be appreciated by those of skill in the art that the techniques disclosed in the examples which follow represent techniques discovered by the inventor to function well in the practice of the invention, and thus can be considered to constitute preferred modes for its practice. However, those of skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention.

[0156] A real-time optimization (RTO) program model ("RTO model"), including a process simulation model and optimizer having an objective function, was written in Python™ programming language (version 3.8, available from python.org open source) and implemented in the process control environment for production of polyethylene (an ethylene-hexene copolymer made using metallocene catalyst) in a gas phase fluidized bed polymerization reactor, with the optimizer operationally connected to a distributed control system (DCS). Manipulated parameters and control parameter status and limits were set by operators. Economic and process values ("objective parameters") were presented through a DCS schematic to explain unit conditions and

optimal solutions in real time. Optimum values of manipulated parameters and control parameters were obtained to achieve maximum profitability. The manipulated parameters were a make-up of an ICA component (blend ratio of iC_4/iC_5), a partial pressure of the ICA component (concentration of iC_4/iC_5 blend in reactor gas), a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor.

[0157] The optimization performed was a steady-state model with an economic objective, falling under the academic definition of Real-Time Optimization (RTO). The full process model included:

- a) reactor cooling capacity (cooling water temperature and cycle gas conditions);
- 10 b) cycle gas condensation;
- c) hydrocarbon solubility;
- d) closed-loop control relationships;
- e) resin stickiness (dMIT);
- f) catalyst productivity;
- 15 g) improved product discharge system (transfer of resin w/ dissolved hydrocarbons, reactor gas and convey gas to reactor/stripper);
- h) purging nitrogen requirements;
- i) compressor capacities;
- j) vapor liquid equilibrium (VLE) flashes;
- 20 k) vent to flare flow rates and compositions; and
- l) recycle loops and a reactor N_2 balance (including use of vent column, crossties and stripper vent operation according to U.S. Pat. No. 11,492,422).

[0158] The stand-alone process simulation model of the gas phase polymerization process assists in the identification of analyzer, instrumentation, and/or process abnormalities by comparing process parameters in the simulated operations to measured values of corresponding parameters in the operating gas phase polymerization unit.

[0159] Economic information for polyethylene profit, catalyst cost, raw material costs, and additional heuristic penalties for flaring "cost" (to further penalize flaring beyond the cost of raw materials) were also included in the model as objective parameters for the objective function in the optimizer.

[0160] Disturbances and conditions outside the scope of the RTO model (i.e., these values were considered as constants) included cooling water and ambient temperatures, product grade,

external production constraints, raw material impurities, pump/compressor downtime(s), and catalyst variability. With an accurate process simulation model, including physical and operating constraints and economic information (control parameters, as described herein), the optimizer can manipulate certain process variables (manipulated parameters, as described herein) to determine the most profitable disposition of the gas phase polymerization process. In this application, the manipulated parameters included iC₄ concentration, iC₅ concentration, reactor temperature, reactor pressure, and ethylene partial pressure in the reaction zone of the fluidized bed reactor.

[0161] Additionally, the RTO model complies with constraints (upper and/or lower limits to various control parameters, as described herein) that don't have a direct financial cost. These include a reactor temperature to cycle gas dew point delta temperature constraint, minimum cycle gas condensation levels, stickiness limits, cooling capacity, bottom-bell sweep velocity, catalyst flow rate, and polymer product withdrawal rate limits.

[0162] The optimization using the RTO model identified an optimal:

- a) balance between the two inert condensing agents (ICAs), iC₄ and iC₅, considering their relative impacts on stickiness, cooling capacity, resin solubility/purging, recovery efficiency and nitrogen venting;
- b) reactor temperature, considering its effect on cooling, stickiness, catalyst productivity and resin solubility;
- c) ethylene concentration, considering its effect on catalyst productivity and nitrogen venting; and
- d) reactor pressure, considering its effect on component partial pressures and nitrogen venting.

[0163] Some of these optimization solutions exhibit strong nonlinearities in the gas phase polymerization process that could not be optimized with traditional linear controls or with a model that does not include the recovery loop. Listed below are some surprising optimization solutions based on the combination of manipulated parameters and control parameters determined by the optimizer:

[0164] When external factors limit production rate, a higher reactor temperature decreased losses of ICA due to solubility of ICA in polymer product particles and decreased catalyst cost by increasing catalyst activity. However, in the absence of external production rate limits, production rate was often limited by cooling capacity. In this case, lowering the reactor temperature increased the cooling capacity for ICAs at the same dMIT limit, allowing increased production rates.

[0165] When solubility and production rates are high enough, the recovery loop gets overloaded and excess gas is vented to the flare from the stripper in the recovery loop. This vent is hydrocarbon-rich and very expensive (especially with the added flare penalty). Increasing reactor temperature and replacing iC_3 with iC_4 will decrease total solubility, thereby limiting
5 stripper flaring. However, this temperature move (increased reactor temperature) can sometimes counter the lowering of temperature to increase production. The balance of temperature, solubility, stickiness, venting, cooling and production is a difficult nonlinear balance that the RTO model optimized effectively.

[0166] A common operating principle is to reduce iC_3 concentration in the reactor within
10 cooling constraints to reduce dissolved iC_3 going to the stripper in the recovery loop. However, under some conditions, increasing iC_3 concentration can yield a net improvement in recovery efficiency because the change in composition makes liquids more favorable in the VLE flash. Figure 4 demonstrates this nonlinearity. For each pound of iC_3 added to a mixture of other hydrocarbons in the reactor gas (whose conditions are similar to those seen in the recovery unit),
15 the incremental increase in liquid recovery (including other hydrocarbons such as, but not limited to, unreacted monomer and comonomer) is sometimes larger than one pound, resulting in a net gain. Under other conditions, each additional pound of iC_3 yields less than one pound of additional liquid recovery, resulting in a net loss.

[0167] Although the present invention and its advantages have been described in detail, it
20 should be understood that various changes, substitutions, and alterations can be made herein without departing from the spirit and scope of the invention as defined by the appended claims. Ranges for various characteristics and attributes disclosed herein are listed as sequentially narrowing ranges. However, it should be understood that any lower endpoint of any ranges can be paired with any upper endpoint for the same characteristic or attribute, and such pairings are
25 also intended to be disclosed herein. All patents, test procedures, and other documents cited in this application are fully incorporated herein by reference for all jurisdictions in which such incorporation is permitted. Moreover, the scope of the present application is not intended to be limited to the particular embodiments of the processes, equipment, means, methods, and/or steps described in the specification. As one of the ordinary skill in the art will readily appreciate from
30 the disclosure of the present invention, processes, equipment, means, methods, and/or steps, presently existing or later to be developed that perform substantially the same function or achieve substantially the same result as the corresponding embodiments described herein, may be utilized

according to the present invention. Accordingly, the appended claims are intended to include within their scope such processes, equipment, means, methods, and/or steps.

CLAIMS

What is claimed is:

1. A method of optimizing a gas-phase polymerization process, wherein:
 - the process is implemented using a gas-phase polymerization system comprising a gas-phase fluidized bed reactor, a production loop, and a recovery loop;
 - the gas-phase fluidized bed reactor has operating conditions comprising a set of manipulated parameters;
 - the combination of the production loop and the recovery loop has operating conditions comprising a set of control parameters having constraints; and
 - each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit each related to (i) a physical configuration of equipment implementing the process and/or (ii) maintenance of thermodynamic conditions suitable for stable operation of the process, such that the lower limits and/or upper limits of the control parameters form the constraints of the set of control parameters; andthe method comprises:
 - simulating the operation of the process using a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of the set of control parameters based on selected values of the set of manipulated parameters;
 - measuring one or more inputs from the process to confirm or correct the simulated output from the process simulation model;
 - performing process optimization using an optimizer by selecting values for each manipulated parameter in the set of manipulated parameters to satisfy an objective function within the constraints of the set of control parameters; and
 - using the selected values for each manipulated parameter in the set of manipulated parameters to control the process.
2. The method of claim 1, wherein the production loop implements:
 - withdrawing a gas-phase stream from the gas-phase fluidized bed reactor, wherein the gas-phase stream comprises an induced condensing agent (ICA) component;

pressurizing the gas-phase stream in a recycle gas compressor to produce a pressurized recycle stream;
cooling the pressurized recycle stream to condense at least a portion of the ICA component to produce a condensed recycle stream; and
recycling the condensed recycle stream to the gas-phase fluidized bed reactor.

3. The method of claim 0, wherein:
a fluidized bed in the gas-phase fluidized bed reactor comprises polymer particles;
the ICA component is a mixture of two or more induced condensing agents;
the set of manipulated parameters comprises an amount of the ICA component and the relative amounts of the two or more induced condensing agents in the ICA component;
the set of control parameters comprises a stickiness of the polymer particles; and
the gas-phase polymerization process is optimized by maximizing the amount of the ICA component while maintaining the stickiness of the polymer particles below a constraint of a stickiness limit of the polymer particles, wherein the stickiness limit is determined by (i) the relative amounts of the two or more induced condensing agents in the ICA component and (ii) solubility of each of the two or more induced condensing agents in the polymer particles relative to isopentane.
4. The method of claim 0, wherein:
a fluidized bed in the gas-phase fluidized bed reactor comprises polymer particles;
the set of manipulated parameters comprises an amount of the ICA component and a reactor temperature;
the set of control parameters comprises a stickiness of the polymer particles; and
the gas-phase polymerization process is optimized by maximizing the amount of the ICA component by reducing the reactor temperature and adding additional ICA, while maintaining the stickiness of the polymer particles below a constraint of a stickiness limit of the polymer particles.
5. The method of claim 1, wherein the recovery loop implements:
withdrawing a portion of polymer particles and entrained reactor gas from a fluidized bed of the gas-phase fluidized bed reactor;

separating the polymer particles from the entrained reactor gas in a product discharge system to produce a first polymer product, wherein the first polymer product comprises a first amount of an induced condensing agent (ICA) component;

contacting the first polymer product with an inert gas in a stripper vessel to form a second polymer product and a second gas stream, wherein the second polymer product comprises a reduced amount of the ICA component, and the second gas stream comprises a gas-phase portion of the ICA component removed from the first polymer product;

cooling the second gas stream to condense at least a portion of the gas-phase fraction of the ICA component to form a third gas stream and a hydrocarbon liquid stream comprising the condensed ICA; and

recycling the hydrocarbon liquid stream to the gas-phase fluidized bed reactor.

6. The method of claim 0, wherein:

the product discharge system comprises first and second pairs of upstream and downstream lock hoppers having a crosstie between upstream lock hoppers, a crosstie between downstream lock hoppers, and valve means to enable control of reaction zone pressure based on the amount of reactor gas withdrawn in each product discharge cycle, wherein increasing withdrawal rates decreases reaction zone pressure and decreasing withdrawal rates increases reaction zone pressure;

the set of manipulated parameters comprises reaction zone pressure in the gas-phase fluidized bed reactor;

the set of control parameters comprises direct reactor venting; and

the gas-phase polymerization process is optimized by controlling reaction zone pressure through reactor gas withdrawal rates to maintain the amount of direct reactor venting below a constraint of a threshold amount of direct reactor venting.

7. The method of claim 0, wherein:

the recovery loop comprises a photoionization detector configured to ionize C₄ to C₈ hydrocarbons to measure a hydrocarbon content of the second polymer product;

the set of manipulated parameters comprises a reaction zone pressure, a reaction zone temperature, and an amount of the ICA component in the gas-phase fluidized bed reactor;

the set of control parameters comprises the hydrocarbon content of the second polymer product and the amount of effluent to flare; and

the gas-phase polymerization process is optimized by controlling the reaction zone pressure, the reaction zone temperature, and the amount of the ICA component in the gas-phase fluidized bed reactor to maintain the amount of effluent to flare below a constraint of a threshold amount of effluent to flare, and further to maintain the amount of hydrocarbon in the second polymer product below a threshold amount of hydrocarbon in the second polymer product.

8. The method of claim 1, wherein the set of manipulated parameters comprises make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor.

9. The method of claim 1, wherein the set of control parameters comprises reactor cooling capacity, cycle gas condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof.

10. The method of claim 1, wherein simulating the operation of the process comprises simulating the operation of the process concurrently with the operation of the process.

11. The method of claim 1, wherein simulating the operation of the process comprises:
measuring feedback outputs from the process, and
correcting simulation results based on the measured feedback outputs from the process.

12. The method of claim 1, wherein performing process optimization comprises performing process optimization independent of simulating the operation of the process.
13. The method of claim 1, wherein:
- the objective function relates to a quantifiable aspect of operating the gas phase polymerization process;
 - an objective parameter is a value of the quantifiable aspect corresponding to an input to or an output from the operation of the gas phase polymerization process; and
 - the objective function is satisfied by maximizing or minimizing the sum of selected objective parameters.
14. The method of claim 0, wherein the objective function is maximizing a sum of values of selected products from and the costs of selected inputs to the gas phase polymerization process.
15. The method of claim 0, wherein the objective function is minimizing an amount of energy consumed in the process to produce the polymer product, wherein energy is measured as energy per mass unit of a polymer product.
16. A process control system for controlling a gas-phase polymerization process, the process control system comprising:
- a process simulation model of steady state operation of the process to produce a simulated output comprising predicted values of a set of control parameters based on selected values of a set of manipulated parameters, the set of control parameters having constraints that comprise a lower limit and/or an upper limit of each control parameter in the set of control parameters;
 - an optimizer to select values for each parameter in the set of manipulated parameters based on one or more measured inputs from the process and the simulated output from the process simulation model to satisfy an objective function within the constraints of the set of control parameters; and
 - a multiple-input/multiple-output controller adapted to produce, during each operational cycle of the process control system, multiple control outputs configured to control the process based on the selected values of each parameter in a set of manipulated parameters

provided to the multiple-input/multiple output controller during each operational cycle of the process control system.

17. The process control system of claim 0, wherein:
 - the process comprises a gas-phase fluidized bed reactor, a production loop, and a recovery loop;
 - the gas-phase fluidized bed reactor has operating conditions comprising the set of manipulated parameters;
 - the combination of the production loop and the recovery loop has operating conditions comprising the set of control parameters; and
 - each control parameter in the set of control parameters is a function of one or more of the manipulated parameters and has a lower limit and/or an upper limit related to a physical configuration of equipment implementing the process and/or maintenance of thermodynamic conditions suitable for stable operation of the process.
18. The process control system of claim 0, wherein the production loop implements:
 - withdrawing a gas-phase stream from a gas-phase fluidized bed reactor, wherein the gas-phase stream comprises an induced condensing agent (ICA) component;
 - pressurizing the gas-phase stream in a recycle gas compressor to produce a pressurized recycle stream;
 - cooling the pressurized recycle stream to condense at least a portion of the ICA component to produce a condensed recycle stream; and
 - recycling the condensed recycle stream to the gas-phase fluidized bed reactor.
19. The process control system of claim 0, wherein the recovery loop implements:
 - withdrawing a portion of first polymer particles from a fluidized bed of a gas-phase fluidized bed reactor;
 - separating the first polymer particles from entrained reactor gas in a product discharge system to produce a first polymer product, wherein the first polymer product comprises a first amount of an induced condensing agent (ICA) component;
 - contacting the first polymer product with an inert gas in a stripper vessel to form a second polymer product and second gas stream, wherein the second polymer product

comprises a reduced amount of the ICA component, and the second gas stream comprises a gas-phase of the ICA component removed from the first polymer product; cooling the second gas stream to condense at least a portion of the gas-phase of the ICA component to form a liquid stream comprising the condensed ICA and a third gas stream; and recycling the condensed ICA to the gas-phase fluidized bed reactor.

20. The process control system of claim 0, wherein the set of manipulated parameters comprises make-up of an ICA component composition, a partial pressure of the ICA component, a partial pressure of ethylene, a temperature, a pressure, or a combination thereof, in the reaction zone of the gas-phase fluidized bed reactor.

21. The process control system of claim 0, wherein the set of control parameters comprises reactor cooling capacity, cycle gas condensation thermodynamics, hydrocarbon solubility in polymer particles, thermodynamic closed-loop control relationships, polymer particle stickiness, catalyst productivity, discharge of polymer particle containing dissolved hydrocarbons and reactor/stripper gas balance, purging nitrogen requirements, compressor capacities, VLE flashes, vent to flare flow rates and compositions, recycle loops and a reactor nitrogen balance, or a combination thereof.

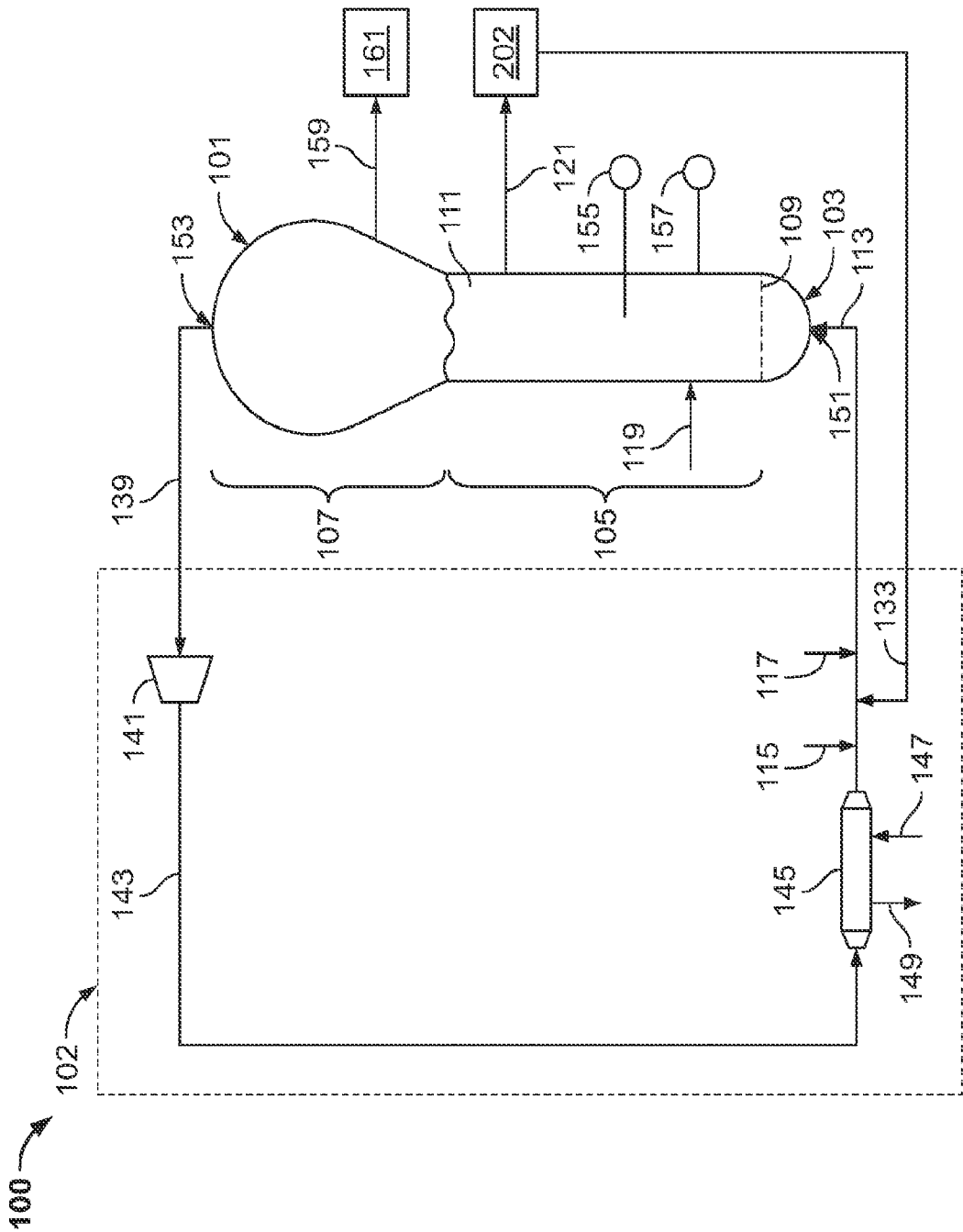


FIG. 1

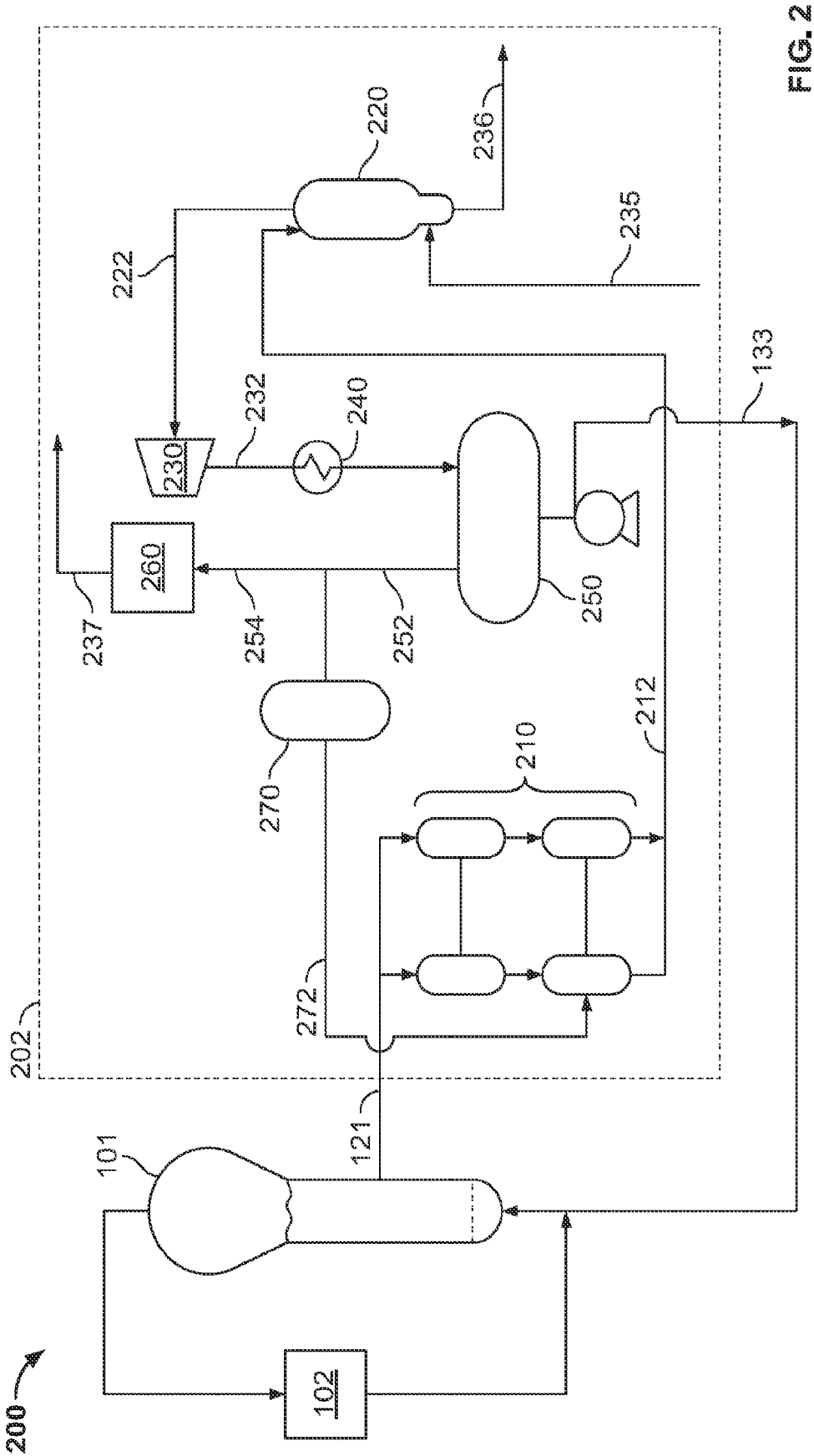


FIG. 2

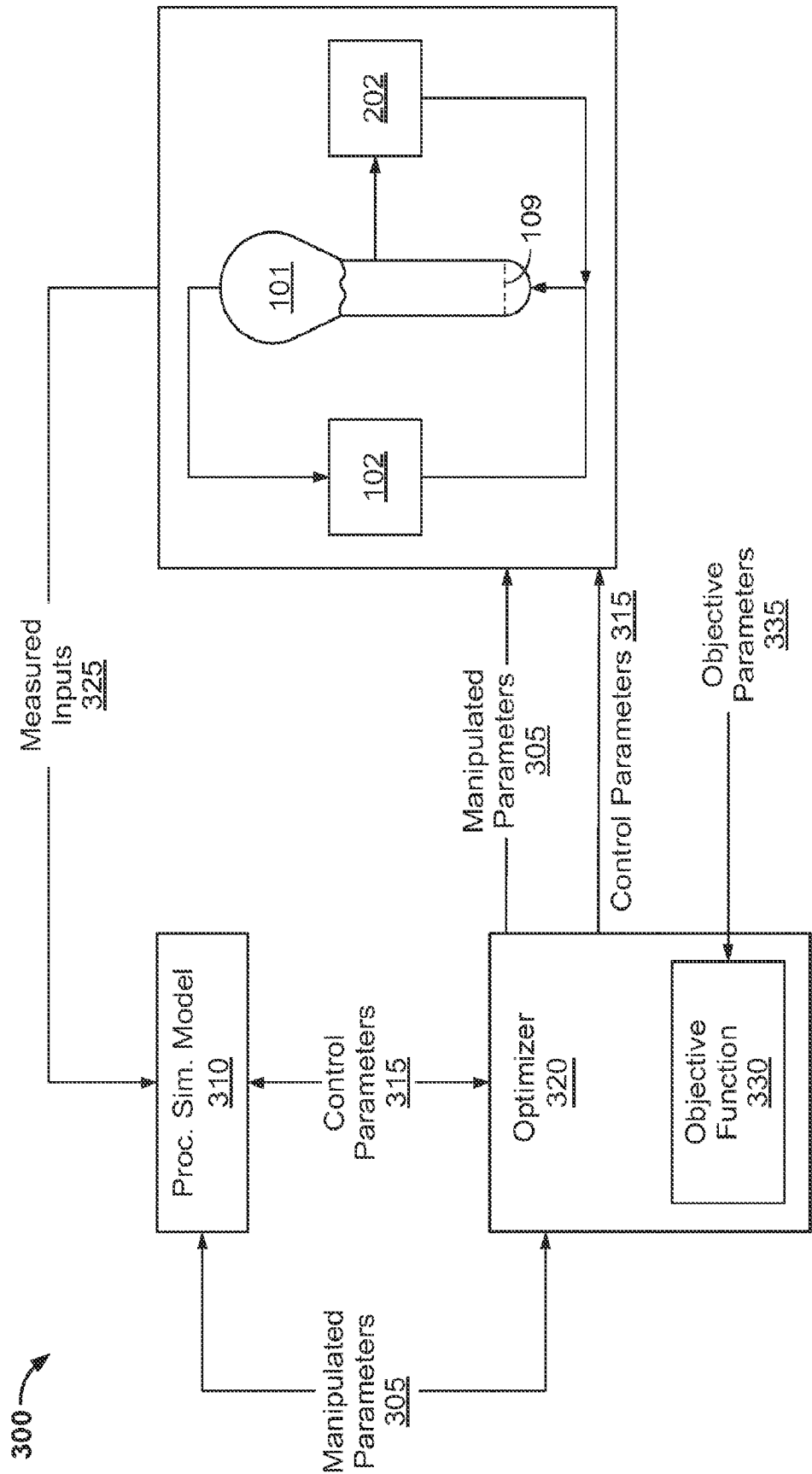


FIG. 3

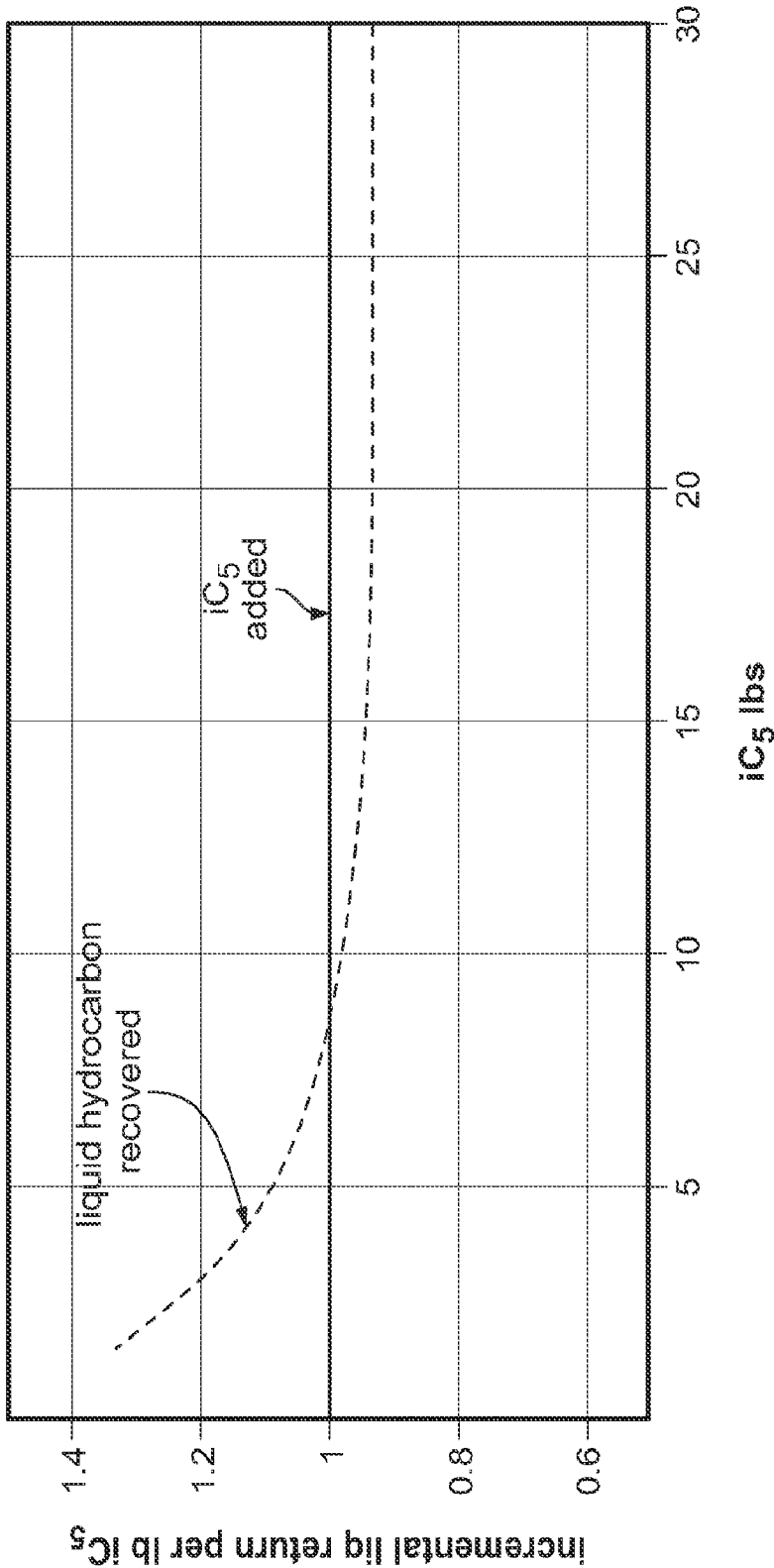


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2024/057269

A. CLASSIFICATION OF SUBJECT MATTER INV. C08F2/34 C08F2/01 G05B17/00 G05B19/00 C08F10/00 ADD.		
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) G05B 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 practicable, search terms used) EPO-Internal		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	MCAULEY K B ET AL: "NONLINEAR PRODUCT PROPERTY CONTROL IN INDUSTRIAL GAS-PHASE POLYETHYLENE REACTORS", AICHE JOURNAL, JOHN WILEY & SONS, INC, US, vol. 39, no. 5, 1 May 1993 (1993-05-01), pages 855-866, XP002056614, ISSN: 0001-1541, DOI: 10.1002/AIC.690390514 the whole document -----	1 - 21
X	WO 02/075325 A1 (EXXONMOBIL CHEM PATENTS INC [US]; DIVEKAR SUHAS V [US] ET AL.) 26 September 2002 (2002-09-26) the whole document ----- - / - -	1 - 21
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
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Date of the actual completion of the international search 26 February 2025		Date of mailing of the international search report 24/03/2025
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INTERNATIONAL SEARCH REPORT

International application No

PCT/US2024/057269

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