

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
29 January 2009 (29.01.2009)

PCT

(10) International Publication Number
WO 2009/014682 A2

(51) International Patent Classification:
B01J 8/24 (2006.01) **B01J 19/00** (2006.01)

TX 77345 (US). **HAGERTY, Robert, O.** [US/US]; 2601 South Broadway #48, La Porte, TX 77571 (US).

(21) International Application Number:
PCT/US2008/008871

(74) Agents: **ARECHEDERRA, III, Leandro** et al.; Univation Technologies, LLC, 5555 San Felipe, Suite 1950, Houston, TX 77056-2746 (US).

(22) International Filing Date: 22 July 2008 (22.07.2008)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/961,770 24 July 2007 (24.07.2007) US

(71) Applicant (for all designated States except US): **UNIVATION TECHNOLOGIES, LLC** [US/US]; 5555 San Felipe, Suite 1950, Houston, TX 77056-2746 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **MUHLE, Michael, E.** [US/US]; 5902 Leafy Aspen Court, Kingwood, TX 77345 (US). **PANNELL, Richard, B.** [US/US]; 3902 Hill Springs Drive, Kingwood, TX 77345 (US). **MARKEL, Eric, J.** [US/US]; 4534 Natural Bridge Drive, Kingwood,

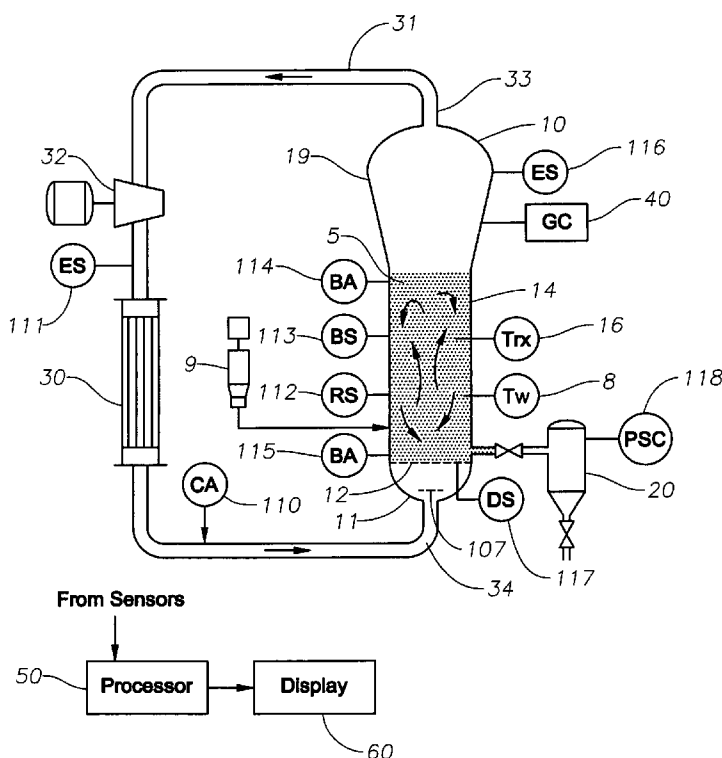
(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM,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

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),

[Continued on next page]

(54) Title: METHOD FOR MONITORING A POLYMERIZATION REACTION

Fig. 1
Fluidized Bed Reaction System



(57) Abstract: In some embodiments, a method of monitoring a resin-producing polymerization reaction in a fluid bed reactor system to generate reaction parameter data in on-line fashion, wavelet transforming the reaction parameter data to generate frequency-domain data or determining kurtosis of each of at least two subsets of the reaction parameter data, and optionally also determining from the frequency-domain data or kurtosis values an indication of at least one of degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking. Optionally also, the reaction is controlled in response to the kurtosis values or frequency-domain data, for example, in an effort to prevent the occurrence of sheeting or another discontinuity event or to maintain the reactor in a stable, non-sticking condition.



European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

- *without international search report and to be republished upon receipt of that report*

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))*

METHOD FOR MONITORING A POLYMERIZATION REACTION

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] The application claims the benefit of Serial No. 60/961,770, filed on July 24, 2007, the disclosure of which is incorporated by reference in its entirety.

FIELD OF THE INVENTION

[0002] The invention pertains to methods for monitoring at least one parameter of a resin-producing polymerization reaction (e.g., an olefin polymerization reaction) in a fluidized-bed reactor and processing the resulting data in on-line fashion to generate processed data indicative of approach to or degree or imminence of resin stickiness, or resin sticking temperature, or likelihood of an unsafe reactor operating condition that can lead to sheeting or chunking. Optionally also, the reaction is controlled in response to the processed data (e.g., to prevent occurrence of a reactor discontinuity event or resin stickiness).

BACKGROUND

[0003] The expression “on-line generation” of data during a reaction is used herein to denote generation of the data sufficiently rapidly that the data is available essentially instantaneously for use during the reaction. The expression “generation of data in on-line fashion” during a reaction is used synonymously with the expression on-line generation of data during a reaction. Generation of data from a laboratory test (on at least one substance employed or generated in the reaction) is not considered “on-line generation” of data during the reaction if the laboratory test consumes so much time that parameters of the reaction may change significantly during the test. It is contemplated that on-line generation of data can include the use of a previously generated database that may have been generated in any of a variety of ways including time-consuming laboratory tests.

[0004] With reference to a product being produced by a continuous reaction, the expression “instantaneous” value of a property of the product herein denotes the

value of the property of the most recently produced quantity of the product. The most recently produced quantity typically undergoes mixing with previously produced quantities of the product before a mixture of the recently and previously produced product 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 herein denotes the value of the property of the product that exits the reactor at time T. This bed average property is determined from the residence time weighted average of the instantaneous property.

[0005] Throughout this disclosure, the expression “diluent” (or “condensable diluent” or “condensable diluent gas”) denotes condensable gas (or a mixture of condensable gases) present in a polymerization reactor with polymer resin being produced. The diluent is condensable at the temperatures encountered in the process heat exchanger. Examples of diluents include induced condensing agents (ICAs), comonomers, isomers of comonomers, and combinations thereof. Exemplary ICAs are typically aliphatic C₄-C₆ hydrocarbons.

[0006] The expression “dry polymer resin” (or “dry version” of polymer resin) is used herein to denote polymer resin that does not contain substantial amounts of dissolved gas. An example of dry polymer resin is polymer that had been previously produced in a polymerization reactor and then purged to eliminate all (or substantially all) unreacted comonomers and ICAs that had been dissolved in the polymer at the time of production. As will be discussed herein, a dry version of polymer resin has significantly different melting behavior than would the same polymer resin if it were in the presence of a significant amount of condensable diluent gas and comonomer.

[0007] The expression polyethylene denotes a polymer of ethylene and optionally one or more C₃-C₁₀ α -olefins while the expression polyolefin denotes a polymer of one or more C₂-C₁₀ α -olefins.

[0008] Throughout this disclosure, the abbreviation “MI” denotes melt index.

[0009] One commonly used method for producing polymers is gas phase polymerization. A conventional gas phase fluidized bed reactor, during operation to produce polyolefins by polymerization, contains a fluidized dense-phase bed including a mixture of reaction gas, polymer (resin) particles, catalyst, and (optionally) catalyst modifiers or continuity aides. Typically, any of several process control variables can be controlled to cause the reaction product to have desired characteristics.

[0010] Generally in a gas-phase fluidized bed process for producing polymers, a gaseous stream containing one or more monomers is continuously passed through a fluidized bed under reactive conditions in the presence of a catalyst. This gaseous stream is withdrawn from the fluidized bed and recycled back into the reactor. Simultaneously, polymer product is withdrawn from the reactor and new monomer/comonomer is added to replace the polymerized monomer/comonomer. The recycled gas stream is heated in the reactor by the heat of polymerization. This heat is removed in another part of the cycle by a cooling system external to the reactor.

[0011] It is important to remove heat generated by the reaction in order to maintain the temperature of the reactor, generally, at a temperature below the polymer melting point and/or catalyst deactivation temperature. Further, heat removal is important to prevent excessive stickiness of polymer particles that if left unchecked, may result in loss of fluidization or agglomeration of the sticky particles which may lead to formation of chunks or sheets of polymer that cannot be removed as product. This phenomenon is commonly referred to as sheeting or chunking. Further, such chunks or sheets may fall onto the distributor plate causing impaired fluidization, and in many cases forcing a reactor shutdown. Prevention of such stickiness and/or sheeting has been accomplished by controlling the temperature of the fluid bed to a temperature below the fusion or sintering temperature of the polymer particles. Above this fusion or sintering temperature, empirical evidence suggests that such fusion or sintering leads to agglomeration or stickiness, which in turn can, if left unchecked, may lead to the above conditions including sheeting. It is also important to provide good mixing

of individual catalyst particles since clusters of these particles tend to overheat and reach temperatures in excess of the indicated bed temperature, an ensemble average of the individual resin particle temperatures.

[0012] It is understood that the amount of polymer produced in a fluidized bed polymerization process is directly related to the amount of heat that can be withdrawn from the fluidized bed reaction zone since the exothermic heat generated by the reaction is directly proportional to the rate of polymer production. In steady state operation of the reaction process, the rate of heat removal from the fluidized bed must equal the rate of rate of heat generation, such that the bed temperature remains constant. Conventionally, heat has been removed from the fluidized bed by cooling the gas recycle stream in a heat exchanger external to the reactor.

[0013] A requirement of a fluidized bed process is that the velocity of the gaseous recycle stream be sufficient to maintain the reaction zone in a fluidized state. In a conventional fluidized bed polymerization process, the amount of fluid circulated to remove the heat of polymerization is greater than the amount of fluid required for support of the fluidized bed and for adequate mixing of the solids in the fluidized bed. The excess velocity provides additional gas flow to (and through) the fluid bed for additional cooling capacity and more intensive mixing of the reactor bed. However, to prevent excessive entrainment of solids in a gaseous stream withdrawn from the fluidized bed, the velocity of the gaseous stream must be regulated.

[0014] For a time, it was thought that the temperature of the gaseous stream external to the reactor, otherwise known as the recycle stream temperature, could not be decreased below the dew point of the recycle stream without causing problems of polymer agglomeration or plugging of the reactor system. The dew point of the recycle stream is that temperature at which liquid condensate first begins to form in the gaseous recycle stream. The dew point can be calculated knowing the gas composition and is thermodynamically defined using an equation of state.

[0015] A recycle stream can be cooled to a temperature below the dew point in a fluidized bed polymerization process resulting in condensing a portion of the recycle gas stream. The resulting stream containing entrained liquid is then returned to the reactor without causing agglomeration and/or plugging phenomena. The process of purposefully condensing a portion of the recycle stream is known in the industry as "condensed mode" operation in a gas phase polymerization process.

[0016] When a recycle stream temperature is lowered to a point below its dew point in "condensed mode" operation, an increase in polymer production is possible, as compared to production in a non-condensing mode because of increased cooling capacity. Consequently, a substantial increase in space-time yield, the amount of polymer production in a given reactor volume, can be achieved by condensed mode operation with little or no change in product properties.

[0017] Cooling of the recycle stream to a temperature below the gas dew point temperature produces a three-phase solid/gas/liquid mixture. The liquid phase of this three-phase gas/liquid mixture in "condensed mode" operation remains entrained or suspended in the gas phase of the mixture. Vaporization of the liquid occurs only when heat is added or pressure is reduced. In some conventional processes, vaporization occurs when the three-phase mixture enters the fluidized bed, with the reacting (warmer) resin providing the required heat of vaporization. The vaporization thus provides an additional means of extracting heat of reaction from the fluidized bed. The heat removal capacity is further enhanced in condensed mode operation by the lower gas temperatures of the gas stream entering the fluidized bed. This heat removal capability is commonly referred to as "sensible heat". Both of these factors increase the overall heat removal capability of the system and thereby enable higher space-time yields (higher reactor production rates per unit volume of the fluidized bed).

[0018] The cooling capacity of recycle gas can be increased further while at a given reaction temperature and a given temperature of the cooling heat transfer

medium. One option described is to add non-polymerizing, non-reactive materials to the reactor, which are condensable at the temperatures encountered in the process heat exchanger. Such non-reactive, condensable materials are collectively known as induced condensing agents (ICAs). Increasing concentrations of ICA in the reactor causes corresponding increases in the dew point temperature of the reactor gas, which promotes higher levels of condensing for higher (heat transfer limited) production rates from the reactor. Suitable ICA materials are selected based on their specific heat and boiling point properties. In particular, an ICA compound is selected such that a relatively high portion of the material is condensed at the cooling water temperatures available in polymer production plants, which are typically 20-40°C. ICA materials include hexane, isohexane, pentane, isopentane, butane, isobutane and other hydrocarbon compounds that are similarly non-reactive in the polymerization process.

[0019] U.S. Patent No. 5,352,749, teaches that there are limits to the concentrations of condensable gases, whether ICA materials, comonomers or combinations thereof, that can be tolerated in the reaction system. Above certain limiting concentrations, the condensable gases can cause a sudden loss of fluidization in the reactor, and a consequent loss in ability to control the temperature in the fluid bed. U.S. Patent Nos. 5,352,749, 5,405,922, and 5,436,304, disclose upper limits of ICA in the reactor, depending on the type of polymer being produced. For example, U.S. Patent No. 5,352,749 discloses that a limiting concentration of ICA (isopentane) exists, beyond which the reactor contents suddenly lose fluidization. The authors characterized this limit by tracking the ratio of fluidized bulk density to settled bulk density. As the concentration of isopentane was increased, they found that the bulk density ratio steadily decreased. When the concentration of isopentane was sufficiently high, corresponding to a bulk density ratio of 0.59, they found that fluidization in the reactor was lost. They therefore determined that this ratio (0.59) was a point of no return, below which the reactor will cease functioning due to loss of fluidization.

[0020] As described in WO 2005/113615(A2), attempts to operate polymerization reactors with excessive ICA concentrations cause polymer particles suspended in

the fluid bed to become cohesive or “sticky,” and in some cases cause the fluid bed to solidify in the form of a large chunk. This stickiness problem is characterized by undesirable changes in fluidization and mixing in the fluid bed, which if left unchecked, may develop into a reactor discontinuity event, such as sheeting in the straight sided reaction section, sheeting in the dome of such a reactor, or chunking, any of which can lead to reactor shut-downs, which in large scale reactors are expensive. These solid masses (sheets or chunks) of polymer eventually become dislodged from the walls and fall into the reaction section and settle on the distributor plate, where they interfere with fluidization, block the product discharge port, and usually force a reactor shut-down for cleaning. The term “discontinuity event” is used to describe a disruption in the continuous operation of a polymerization reactor caused by sheeting, chunking or distributor plate fouling. The terms “sheeting and/or chunking” while used synonymously herein, may describe different manifestations of problems caused by excessive polymer stickiness in the fluid bed. In either manifestation (sheeting or chunking) the excessive polymer stickiness can lead directly to a reactor discontinuity event with the associated loss production.

[0021] It would be desirable to provide a method of determining a stable operating condition for fluidized bed polymerization, especially if operating in condensed mode, to facilitate optimum design of the plant and determination of desirable process conditions for optimum or maximum production rates for a given plant design.

[0022] It would also be desirable to have a mechanism in commercial gas-phase reactors to detect the onset of stickiness that is a better (e.g., more reliable or earlier) indicator of the onset of stickiness than are conventional techniques (e.g., monitoring the fluidized bulk density as described in above-mentioned U.S. Patent No. 5,352,749 or monitoring the standard deviation of fluidized bulk density as described in below-discussed U.S. Patent No. 6,384,157). Such a mechanism would allow the operators to determine when conditions of limiting stickiness are being approached, and enable them to take corrective action before discontinuity events (e.g., sheeting and chunking) occur, while keeping the reactors at or near

conditions of maximum ICA concentration to permit higher production rates with substantially less risk.

[0023] In some cases, data generated by monitoring a reaction in a fluidized bed reactor are wavelet transformed to generate frequency-domain data (comprising frequency coefficients associated with time values), and a degree of resin stickiness, or an approach to or imminence of resin stickiness, or an unsafe or undesired reactor operating condition leading to sheeting or chucking, or a resin sticking temperature is identified from the frequency coefficients. Since desired, smooth operation of a fluidized bed reactor system is typically characterized by efficient, random mixing of particles in the fluidized bed, an undesired operating condition is often characterized by non-uniformities, including hot spots, cold bands (caused by insulating layers of fines), and polymer agglomerates (e.g., chunks or sheets) in the reactor.

[0024] Kurtosis of reaction parameter data (generated by monitoring a reaction) is determined as a function of time, and a degree of resin stickiness, or an approach to or imminence of resin stickiness, or an unsafe or undesired reactor operating condition, or a resin sticking temperature is identified using the kurtosis data.

[0025] Kurtosis is a measure of the degree of “peakedness” of a distribution. In the literature, the kurtosis of a non-random distribution is often defined as follows:

$$\gamma_2 = \mu_4 / \sigma^4 - 3, \text{ where}$$

μ_4 / σ^4 is the fourth standardized moment of the distribution, μ_4 is the fourth moment about the mean, and σ is the standard deviation (so that σ^2 is the “variance” or second moment about the mean). In the formula, “3” is subtracted from μ_4 / σ^4 to normalize the defined value so that the defined kurtosis of a normal (Gaussian) distribution is zero. With this definition, kurtosis can range from -2 to +infinity.

[0026] U.S. Patent No. 6,384,157, teaches monitoring fluidized bulk density in a polymerization reactor and determining when the standard deviation of the

monitored data drops (e.g., below a threshold). Such a drop is interpreted as an indication of defluidization of the fluidized bed. However, the inventors have recognized that the standard deviation (in contrast with the kurtosis) of monitored polymerization reaction parameter data is not as sensitive an indicator of the degree (or imminence) of resin stickiness or of unsafe reactor operating conditions leading to sheeting or chunking.

[0027] The kurtosis of various types of data has been determined in contexts other than that of the present invention. For example, U.S. Patent No. 5,673,026, teaches measuring kurtosis of sensor data (indicative of some flow parameter of flowing fluid) to determine when the fluid flow ceases. However, until the present invention, the kurtosis of monitored polymerization reaction parameter data had not been determined and used as an indicator of the degree (or imminence) of resin stickiness and/or of unsafe reactor operating conditions and/or other conditions.

[0028] Polymerization reaction parameter data to be processed in some cases is indicative of a sequence of static charge values. Such data can be generated by monitoring a polymerization reaction using one or more static probes in any of a variety of conventional ways (e.g., as described in U.S. Patent Application Publication No. 2005/0148742). U.S. Patent Application Publication No. 2005/0148742 describes use of static probes positioned in the entrainment zone of a fluidized bed polymerization reaction system to monitor “carryover static” during a polymer resin-producing polymerization reaction in the reactor system, and describes control of the reaction in response to the results of such monitoring to prevent discontinuity events such as chunking and sheeting (e.g., to reduce carryover static and thereby prevent such discontinuity events). The expression “entrainment zone” of a fluidized bed reactor system is used in U.S. Patent Application Publication No. 2005/0148742 and the present disclosure to denote any location in the reactor system outside the dense phase zone of the system (i.e., outside the fluidized bed). However, U.S. Patent Application Publication No. 2005/0148742 does not suggest wavelet transforming static charge data (or other

data) or determining the kurtosis of a set of static charge data values (or other data values).

[0029] The expression “carryover static” is used in U.S. Application Publication No. 2005/0148742 to denote static charging that results from frictional contact by particles (e.g., catalyst particles and resin particles) against the metal walls of a gas recycle line, or against other metal components in a reactor entrainment zone. Carryover static can be measured by suitable static probes positioned in various sections of the entrainment zone of a reaction system, including the expanded (disengagement) section, the recycle line, and the distributor plate.

[0030] The expression “entrainment static” denotes carryover static that results from frictional contact between entrained particles and a static probe located in a gas recycle line of a fluidized bed reactor system. Thus, the term “entrainment static” represents a specific means of measuring the carryover static generated by frictional contact of entrained particles that occur throughout the gas recycle system.

[0031] It is conventional to perform a Fourier transform on time-domain reaction parameter data (e.g., temperature data) generated by monitoring a polymerization reaction. A wavelet transform (performed in a class of embodiments of the present invention) on reaction parameter data generated by monitoring a polymerization reaction is a very different operation than a Fourier transform. The inventors have recognized that frequency coefficients generated by Fourier-transforming polymerization reaction parameter data cannot to the extent desired be used as indicators of the degree (or imminence) of resin stickiness or unsafe or undesired reactor operating conditions. The inventors have recognized that, for a number of reasons (some of which are discussed below), frequency coefficients (having associated time values) generated by wavelet-transforming polymerization reaction parameter data can be used reliably as indicators of the degree (or imminence) of resin stickiness or unsafe or undesired reactor operating conditions, and are reliably indicative of rapid changes in monitored reaction parameters.

[0032] The expression “wavelet transform” is used in a conventional sense to denote a well-known type of transform on time-domain data. The expression “inverse wavelet transform” is used in a conventional sense to denote a transform which is the inverse of a wavelet transform. In essence, a wavelet transform fits a set of wavelets to the data, for each of many different wavelet frequencies and each of a number of different times for each wavelength frequency. Each wavelet is a short duration function having a characteristic center time and frequency (e.g., a function similar to a truncated sine function). By contrast a Fourier transform has no time characteristic since it is time independent and is purely a frequency metric.

[0033] Various types of wavelet transforms are well known. One widely used wavelet transform is the discrete one-dimensional wavelet transform which produces a set of wavelet coefficients, $d_{j,k}$, in response to a function $X(t)$ (which can be indicative of a time series of data values):

$$d_{j,k} = \int X(t) \Psi_{j,k}(t) dt, \text{ where}$$

j indicates a frequency (one of a predetermined range of frequencies), k is a translation index corresponding to a time value associated with a time-shifted version of a wavelet function $\Psi(t)$ (i.e., the center time of the time-shifted wavelet function), t denotes time, the integral is over the range $-\infty$ to ∞ , $\Psi_{j,k}(t) = 2^{-j/2} \Psi(2^{-j}t - k)$, and $\Psi(t)$ is the mother wavelet function. Since a power of 2 is used for the time shifting this is often referred to as a dyadic wavelet. It is a very practical filtering algorithm since it yields a highly efficient computational algorithm for the wavelet transformation.

[0034] Another widely used wavelet transform is the continuous wavelet transform which convolves a time-domain function $X(t)$ with each of a sequence of wavelet functions (e.g., a first wavelet function, a sequence of time-shifted versions of the first wavelet function, each shifted by a different time, and scaled versions of the first wavelet function and time-shifted versions of each scaled wavelet function) to produce a set of wavelet coefficients, $d(\text{scale}, \text{position})$,

where “scale” denotes the time interval over which a corresponding wavelet function extends (the scale of a wavelet function determines a frequency associated with the wavelet function) and “position” denotes a time value (e.g., a center time) associated with the wavelet function. A continuous wavelet transform of function $X(t)$ can produce a set of wavelet coefficients:

$$d(scale, position) = \int X(t) \Psi(scale, position, t) dt, \text{ where}$$

t denotes time, the integral is over the range $-\infty$ to ∞ , and $\Psi(scale, position, t)$ is a scaled wavelet function associated with a particular scale and position. To perform a continuous wavelet transformation on a time domain signal, the signal can be convolved with a sequence of wavelet functions as follows: the signal is convolved with a first wavelet function and also with a sequence of time-shifted versions of the first wavelet function (each shifted by a different time); a scaled wavelet function (having a different frequency than that associated with the first wavelet function) is determined by scaling the first wavelet function; the signal is also convolved with the scaled wavelet function and with a sequence of a time-shifted versions of the scaled wavelet function (each shifted by a different time); and these steps are repeated for each of a sequence of differently scaled wavelet functions (each having a different characteristic frequency) and time-shifted versions thereof. A continuous wavelet transform differs from a discrete wavelet transform in two ways: a continuous wavelet transform is continuous in terms of shifting of the wavelet function (during computation, the analyzing wavelet function is shifted smoothly over the full domain of the analyzed time-domain function); and a continuous wavelet transform can operate at every scale (from the scale of a first wavelet function up to a predetermined maximum scale).

[0035] It is known how to program processors to perform both discrete wavelet transforms and continuous wavelet transforms efficiently. Commercially available software (e.g., the MATLAB software available from MathWorks, Inc.) can be used to program processors to perform discrete and continuous wavelet transforms.

[0036] The result of a wavelet transform on time domain data is a set of frequency coefficients. However, unlike the frequency coefficients resulting from a Fourier transform (or sine transform or cosine transform) on the data, each of the frequency coefficients resulting from a wavelet transform has a time value associated with it (i.e., the center time of a time-shifted version of a wavelet function), and different ones of the frequency coefficients are associated with different time values. Thus, the frequency coefficients resulting from a wavelet transform can be graphed as a three dimensional graph with time on one axis, frequency on another axis, and signal amplitude on a third axis.

[0037] It has been proposed to perform a wavelet transform on time-domain, reaction temperature data (sequences of temperature values) generated using skin temperature sensors (or other temperature sensors) which monitor a reaction in a fluidized bed reactor, as one step in a process of generating a high-pass filtered version of the reaction temperature data. However, the resulting high-pass filtered temperature data are also time-domain data. The high-pass filtering step removes noise from the time-domain data and thus allows fluidization quality to be determined from the high-pass filtered, time-domain data in an improved manner than from the original (non-high-pass filtered) time-domain data. Wavelet transforms have similarly been performed on time-domain data in other contexts to generate a noise-reduced, filtered version of the time-domain data (e.g., a high-pass filtered version of the time-domain data from which low frequency noise has been removed, or a low-pass filtered version of the time-domain data from which high frequency noise has been removed). In these methods, noise is removed from the frequency coefficients generated by wavelet transforming the time-domain data, and an inverse wavelet transform is then performed on the filtered frequency coefficients to generate filtered time-domain data.

[0038] U.S. Patent No. 6,826,513, issued November 30, 2004, discloses an example of the methods described in the previous paragraph. U.S. Patent No. 6,826,513 discloses wavelet transformation of a processed version of sensor data (e.g., pressure data or flow data generated during monitoring of polymerization and fluidized bed reactions or other reactions) to remove noise from the data,

filtering the resulting wavelet coefficients, and performing an inverse wavelet transformation on the filtered wavelet coefficients to generate filtered (denoised) sensor data. The resulting denoised sensor data are used to identify unsafe reactor operating conditions. Wavelet transforms are used in accordance with the present invention in a very different manner than that disclosed in U.S. Patent No. 6,826,513. For example, a wavelet transform is performed on time-domain data generated by monitoring a reaction, and the resulting wavelet coefficients are analyzed, optionally after they undergo additional processing but without performing an inverse wavelet transformation thereon, to identify undesirable reaction conditions (e.g., to identify anomalies indicative of degree of resin stickiness or to identify likelihood of imminent resin stickiness). U.S. Patent No. 6,826,513 neither teaches use of denoised data (generated in accordance with methods disclosed therein) to identify conditions indicative of degree of resin stickiness or likelihood of imminent resin stickiness, or use of wavelet coefficients (resulting from wavelet transformation of time-domain data) to identify conditions indicative of degree of resin stickiness or imminent resin stickiness or to identify unsafe reactor operating conditions leading to sheeting or chunking. Denoised data generated in accordance with U.S. Patent No. 6,826,513's teaching may be useful to characterize overall stability of (or progressive change in) a process, but would not be useful to identify singular events of short duration (e.g., spiking or short-duration events indicative of decreased randomness) in a noisy signal (e.g., a signal characteristic of random interactions occurring in a well-mixed fluidized bed), or to identify conditions indicative of degree of resin stickiness or imminent resin stickiness in a reactor, or to identify unsafe reactor operating conditions leading to sheeting or chunking.

SUMMARY

[0039] In a first class of embodiments, the inventive method includes the steps of: (a) monitoring at least one reaction parameter of a polymerization reaction (e.g., a polyethylene polymerization reaction) in a fluidized bed reactor system to generate (in on-line fashion) time-domain reaction parameter data; (b) wavelet

transforming the reaction parameter data to generate frequency-domain data comprising frequency coefficients associated with time values; and (c) determining from the frequency-domain data at least one indication of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition leading to sheeting or chucking. Step (c) can include the step of generating a signal (or data or a display) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event). The reaction parameter data transformed in step (b) can be acoustic, absolute pressure or differential pressure data indicative of vibration of an element of the reactor system, or carryover static data or bed static data, or other time-domain reaction parameter data. When an element of the reactor system is exposed to a pressure fluctuation, the element may “ring” and a spike in wavelet-transformed frequency-domain acoustic (or differential pressure) data may be indicative of this ringing.

[0040] The inventors have recognized that wavelet-transformed reaction parameter data are more reliably indicative of the likelihood of imminent resin stickiness or an unsafe reactor operating condition than Fourier-transformed (or sine- or cosine-transformed) reaction parameter data, because a transform of the latter type essentially fits sinusoidal functions to time-domain data and thus Fourier-transformed (or sine- or cosine-transformed) reaction parameter data are indicative only of synchronous events (i.e., a Fourier transform cancels out information indicative of two events that are out-of-phase with each other). In contrast, a wavelet transform essentially fits wavelet functions (e.g., truncated sine functions or other functions of a type having a characteristic frequency and center time) to the data being transformed, and thus wavelet-transformed reaction parameter data are indicative of asynchronous events as well as synchronous events. The inventors have further recognized that in predicting imminent resin stickiness or resin sheeting (or other unsafe reactor operating conditions),

asynchronous events in the reactor system are typically the type of events of interest.

[0041] Polymerization reaction parameter data generated during smooth, steady state operation of a fluidized bed reactor (with efficient, random mixing of particles in the fluidized bed) using a static, acoustic, temperature, absolute pressure, or differential pressure (DP) sensor with a sufficiently high sample rate (e.g., 100 Hz) are typically indicative of a noisy signal characteristic of the random interaction of the well-mixed bed with the sensor. Undesired non-uniformities in the bed cause subtle changes in the randomness of the data values. Undesired non-uniformities in the bed are typically indicated by spikes in the data which occur randomly with characteristic time scales of less than one second, or by other manifestations of decreased randomness in the data. Such spiking (or decreased randomness) can be caused by the undesired presence of resin sheets or conditions leading to sheeting. In some cases, undesired non-uniformities in the bed are indicated by spiking in entrainment static data, which can occur when a continuity additive clears up excess entrainment to reveal discharge events. Because of the random, asynchronous nature of the spiking or other manifestations of decreased randomness, and because the spikes (or other manifestations) are typically infrequent and phase-shifted, frequency coefficients generated by conventionally Fourier-transforming (or sine- or cosine-transforming) measured reaction parameter data and/or denoised wavelet transforms of the derivatives of reaction parameter data are not reliably indicative of the spikes (or other manifestations).

[0042] In a second class of embodiments, the inventive method includes the steps of: (a) monitoring at least one reaction parameter of a polymerization reaction (e.g., a polyethylene polymerization reaction) in a fluidized bed reactor system to generate (in on-line fashion) time-domain reaction parameter data (i.e., a time sequence of reaction parameter values), which may be, for example, carryover static or bed static data values or other reaction parameter data values; and (b) determining kurtosis of each of at least two subsets of the reaction parameter data, each of said subsets of the reaction parameter data including data values in a

different one of a sequence of different time intervals. Optionally, the method also includes a step of determining from kurtosis values generated in step (b) at least one indication of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition leading to sheeting or chunking, and typically also a step of generating a signal (or data or a display) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event).

[0043] Because kurtosis values are not prone to baseline fluctuations and can be self-normalizing, they are especially useful to determine degree of resin stickiness, or approach to or imminence of resin stickiness (or an unsafe or undesired reactor operating condition leading to sheeting or chunking). For example, if one simply uses time-averaged values of sensor data for this purpose (i.e., an average of the sensor data values in each of a sequence of subsets of data values, each subset generated in a different one of a sequence of time windows) rather than a sequence of kurtosis values of the sensor data (one kurtosis value for each of a sequence of subsets of the data values, each subset generated in a different one of a sequence of time windows), the baseline average of a sequence of the time-averaged values will typically change over time so that fast fluctuations (among individual ones of the time-averaged values) cannot reliably be identified unless the time-averaged values are corrected for baseline average changes.

[0044] Some preferred embodiments in the second class determine a sequence of kurtosis values of a sequence of subsets of monitored static data values, and use the resulting kurtosis values to predict an approach to or imminence of resin sheeting (e.g., kurtosis values determined from product chamber static charge data are used to predict wall sheeting, or kurtosis values determined from static data from one or more static sensors in an expanded section of the reactor above the fluidized bed are used to predict dome sheeting).

[0045] Other embodiments in the second class determine a sequence of kurtosis values of a sequence of subsets of monitored reaction parameter data values other than static data values (e.g., temperature or acoustic data values) and use the resulting kurtosis values to predict an approach to or imminence of resin sheeting, or a discontinuity event, or an unsafe or undesired reactor operating condition.

[0046] Kurtosis is a measure of the degree of “peakedness” of a distribution. In describing examples of the invention, the expressions “kurtosis” and “excess kurtosis” of a distribution are used herein as synonyms to denote the following quantity:

$$\gamma_2 = \mu_4 / \sigma^4 - 3, \text{ where}$$

μ_4 / σ^4 is the fourth standardized moment of the distribution, μ_4 is the fourth moment about the mean, and σ is the standard deviation (second moment about the mean). In the formula, “3” is subtracted from μ_4 / σ^4 to normalize the defined value of kurtosis so that the defined kurtosis of a normal (Gaussian) distribution is zero. With this definition, kurtosis can range from -2 to +infinity. In the claims, the expression “kurtosis” of a distribution (e.g., a set of data values) is used in a broader sense to denote “ $A\gamma_2 + B$,” where γ_2 is defined as in the above formula, and A and B are arbitrary constants.

[0047] It is known how to determine kurtosis of a set of data values and how to program processors to determine kurtosis of a set of data values (e.g., a time series of data values) efficiently, although kurtosis has not been determined on subsets of a set of reaction parameter data values generated in accordance with the invention (each subset including reaction parameter data values in a different interval of a sequence of time intervals). Commercially available software (e.g., the MATLAB software available from MathWorks, Inc. or Labview software available from National Instruments) can be used to program processors to determine kurtosis of a set of data values.

[0048] In some implementations of the above-mentioned first class and second class of embodiments, “high speed” reaction parameter data are generated and

processed in accordance with the invention. The expression “high speed” data is used herein to denote a sequence (time series) of data values collected with a sample rate greater (and typically much greater) than 1 Hz (the sample rate is on the order of 100 Hz in typical embodiments of the inventive method). For example, in some embodiments at least one high speed skin thermocouple is used to generate time-domain reaction parameter data that are processed in accordance with the invention. A “high speed” (or “fast”) skin thermocouple is configured to sense reactor temperature excursions of shorter duration than can a conventional skin thermocouple, has sufficiently fast response to be sensitive to temperature spikes of duration on the order of a second (e.g., spikes having duration of one second or a few seconds), and is typically positioned not more than one half inch from the reactor wall.

[0049] In some implementations of the above-mentioned first class and second class of embodiments, the time-domain reaction parameter data processed in accordance with the invention are (or include) one or more of: bed static data indicative of static charge in the fluidized bed of a fluidized bed reactor (such static charge is sometimes referred to herein as reactor or “bed” static charge); carryover static data indicative of carryover static (e.g., entrainment static); acoustic or pressure (e.g., differential pressure) data; and temperature data. The carryover static data can be generated using at least one static probe positioned to monitor static charge outside the fluidized bed (i.e., in the entrainment zone). Typically, the reactor has a gas recycle line and the carryover static data are entrainment static data, generated using at least one entrainment static probe positioned to monitor static charge in the gas recycle line. Static probes suitable for generating carryover static (e.g., entrainment static) data in many embodiments of the invention are described in above-referenced US Patent Application Publication No. 2005/0148742. Static probes suitable for generating carryover static data in some embodiments of the invention are static current probes; others are static voltage probes. Alternatively, the carryover static data are generated by other instruments (e.g., using a Faraday cup to measure static

charge of samples of entrained material collected from the recycle line with an isokinetic or other sampler).

[0050] In some embodiments in which bed static data are generated and processed in accordance with the invention, the bed static data are generated using at least one static probe (e.g., a static probe of the type described in U.S. Patent Nos. 4,532,311, 5,648,581, and 6,008,662 or another conventional reactor static probe) positioned to monitor static charge in the reactor at or near a portion of the reactor wall that bounds the fluidized bed.

[0051] In some embodiments, the reaction parameter data processed in accordance with the invention are or include at least one of: acoustic data (generated using one or more acoustic emission sensors) that are indicative of a time series of readings of acoustic emissions of contents of the reactor, absolute pressure data from absolute pressure sensors, differential pressure data from differential pressure sensors, static charge data from static sensors, fluidized bulk density data from appropriate sensors, and temperature data from one or more skin temperature sensors, wall temperature sensors, and/or other temperature sensors.

[0052] Reaction monitoring in accordance with the invention can be used to identify when reaction conditions deviate from desirable operating conditions which are typically characterized by efficient, random mixing of particles in the reaction zone. Undesirable operating conditions are typically characterized by non-uniformities, including hot spots, cold bands caused by insulating layers of fines, and fused polymer sheets.

[0053] In some implementations of the above-mentioned first class and second class of embodiments, the inventive method also includes the step of controlling the reaction in response to kurtosis values or frequency-domain data generated in step (b) or in response to at least one indication determined in step (c), typically in an effort to prevent (and preferably to prevent) the occurrence of sheeting or another discontinuity event and/or to maintain the reactor in a stable, non-sticking condition. For example, the control may be implemented by adjusting reaction

temperature, or changing the superficial velocity of fluidizing gas, or controlling the feed rate of a continuity additive (e.g., aluminum distearate (A) or ethoxylated amine (B) additive, either neat or in oil slurry) or an ICA into the reactor.

[0054] Other aspects of the invention are systems including (and methods of using) sensors and optionally also other instruments for monitoring reaction parameters (e.g., parameters indicative of static charge, acoustic, temperature, pressure, and/or DP values, where “DP” values are pressure differences two different locations along the length of the fluid bed), and processors programmed to process the resulting reaction parameter data in accordance with any embodiment of the inventive method (typically to generate data, or signals, or a display indicative of imminent occurrence of resin sheeting or another discontinuity event). In preferred embodiments, relevant measured data from all reaction monitoring instruments, and relevant calculated values, are combined into an integrated computer display for presentation to users (e.g., plant operators). Such a computer display can be supplemented by process alarms or advisory notices to warn the users of conditions in the process that may be approaching those that will lead to sheeting (e.g., wall or dome sheeting) or other discontinuity events. The alarms or advisory notices can also be combined with recommended control actions to avoid the discontinuity event.

BRIEF DESCRIPTION OF THE DRAWINGS

[0055] Figure 1 is a simplified cross-sectional view of a reaction system, including fluidized bed reactor 10, whose operation can be monitored and optionally also controlled in accordance with the invention.

[0056] Figure 2 is a set of two graphs: a one-dimensional plot (at the top of Fig. 2) of a time series of measured reaction parameter data values versus time; and a two-dimensional plot (at the bottom of Fig. 2) of wavelet coefficients generated by performing a wavelet transform on these data values. In both plots, the horizontal axis indicates the same time range. In the two-dimensional plot, the magnitude of each wavelet coefficient is indicated by a degree of darkness, and

the vertical axis indicates a frequency associated with each subset of the wavelet coefficients.

[0057] Fig. 3 is a graph of a times series of static charge data values (curve "A") generated during a polymerization reaction using a static probe in the expanded section of a fluidized bed reactor (e.g., expanded section 19 of Fig. 1) plotted as a function of time, and the degree of resin sheeting (curve "B") in the same reactor during the reaction (in units of pans of sheeted resin material collected during the reaction as marked along the vertical axis at the right) also plotted as a function of time.

[0058] Fig. 4 is a graph of a times series of kurtosis values (curve "C") generated from the static charge data values plotted in Fig. 3, superimposed on the resin sheeting curve (curve "B") also plotted in Fig. 3.

[0059] Fig. 5 is a set of four graphs: a graph (labeled "Mean") of the mean value (computed in a sequence of time windows) of a time series of entrainment static charge values measured during a polymerization reaction using an entrainment static probe in the recycle line of a fluidized bed reactor (e.g., recycle line 31 of Fig. 1) as a function of time (the center time of each window); a graph (labeled "Kurtosis") of kurtosis (one kurtosis value computed in each window in the same sequence of time windows) as a function of time of the same entrainment static charge values; a graph (labeled "RMS") of the root mean square or "RMS" (one RMS value computed in each window in the same sequence of time windows) as a function of time of the same entrainment static charge values; and a graph (labeled "Std Dev") of the standard deviation (one standard deviation computed in each window in the same sequence of time windows) as a function of time of the same entrainment static charge values.

[0060] Fig. 5a is a graph of continuity additive feed rate versus time during the reaction.

[0061] Fig. 6 is a graph of kurtosis (computed in a sequence of time windows) of a time series of entrainment static charge values measured during a

polymerization reaction using an entrainment static probe in the recycle line of a fluidized bed reactor (e.g., recycle line 31 of Fig. 1) as a function of time (the center time of each window), and a graph of continuity additive feed rate versus time during the reaction.

DETAILED DESCRIPTION

[0062] Before the present compounds, components, compositions, and/or methods are disclosed and described, it is to be understood that unless otherwise indicated this invention is not limited to specific compounds, components, compositions, reactants, reaction conditions, ligands, metallocene structures, process steps, methodologies, or the like, as such may vary, unless otherwise specified. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments only and is not intended to be limiting.

[0063] It must also be noted that, as used in the specification and the appended claims, the singular forms "a," "an" and "the" include plural referents unless otherwise specified.

[0064] For the sake of brevity, terminology, expressions, definitions, etc. will not be repeated in this section but are incorporated herein where applicable and relevant.

[0065] A reactor system whose operation can be monitored and optionally also controlled in accordance with the invention will be described with reference to Figure 1. The Figure 1 system includes fluidized bed reactor 10. Reactor 10 has a bottom end 11, a top expanded section 19, a cylindrical (straight) section 14 between bottom end 11, and a distributor plate assembly 12 (sometimes referred to as a distributor plate) within section 14. A fluidized bed 5 of granular polymer and catalyst particles is contained within the straight section 14. The bed is fluidized by the steady flow of recycle gas through distributor plate 12. The flow rate of fluidizing gas is regulated to provide the fluidized bed with relatively good mixing, as illustrated in the figure.

[0066] The reactor system also has a catalyst feeder 9 for controlled addition of polymerization catalyst to the fluidized bed (the reaction zone). Within the reaction zone, the catalyst particles react with the monomer (e.g., ethylene) and comonomer and optionally other reaction gas to produce granular polymer particles. As new polymer particles are produced, other polymer particles are continually withdrawn from the fluidized bed through product discharge system 20. After passing through the product discharge system, the polymer granules are degassed (or “purged”) with a flow of inert nitrogen to remove substantially all of the dissolved hydrocarbon materials.

[0067] The reactor system of Figure 1 also has a cooling control loop which includes a recycle gas line 31, a circulating gas cooler 30 and compressor 32, coupled with reactor 10 as shown. During operation, the cooled circulating gas from cooler 30 flows through inlet 34 into reactor 10, then propagates upward through the bed and out from reactor 10 via outlet 33.

[0068] The expanded section 19 is also known as the “velocity reduction zone,” and is designed to minimize the quantities of particle entrainment from the fluidized bed. Each diameter of each horizontal cross-section of the expanded section 19 is greater than the diameter of straight section 14. The increased diameter causes a reduction in the speed of the fluidizing gas, which allows most of the entrained particles (catalyst and resin particles) to settle back into the fluidized bed, thereby minimizing the quantities of solid particles that are “carried over” from the fluidized bed (at a given value of fluidizing gas velocity) through the recycle gas line 31.

[0069] Continuity additive feed 110 introduces a continuity additive (e.g., a slurry of additive A or B material in mineral oil in one embodiment) into the fluid stream within line 31.

[0070] Fluidized bed reactor 10 has an annular disk flow deflector 107 at its bottom end (at reactor inlet 34, which is the outlet of recycle line 31).

[0071] One or more temperature sensors 16 are located in the fluidized bed, and are used with a control system (which can include processor 50 of Fig. 1) and an external cooling loop to control the fluidized bed temperature T_{rx} near the process set-point. Relatively warm reactor gas (whose temperature has increased during its flow through reactor 10) is withdrawn from outlet 33 and is pumped by compressor 32 to cooler 30, wherein the temperature of the gas (the cooling fluid) is reduced. Relatively cool fluid (which may contain condensed liquid) flows out from cooler 30 to the reactor inlet 34, to cool the fluidized bed. Temperature sensors (not shown) near the inlet and outlet of cooler 30 provide feedback to the control system regulate the amount by which cooler 30 reduces the temperature of the fluid entering reactor 10. During operation, cooled circulating gas from cooler 30 flows through annular disk 107 into reactor 10, then propagates upward through the bed and out from reactor 10 into inlet 33 of recycle line 31.

[0072] Reactor 10 can be implemented as a mLLDPE (metallocene-catalyzed, linear low-density polyethylene) reactor with straight section 14 having height 47 feet, six inches.

[0073] In addition to bed temperature sensor(s) 16 noted above, the Fig. 1 system also includes other sensors for generating reaction parameter data during a polymerization reaction. The data are processed in accordance with the invention in processor 50, typically to generate a display (60) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of or an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event). The sensors include entrainment static probe 111, distributor plate static sensor 117, upper and lower bed acoustic emission sensors 114 and 115, reactor static probes 112 and 113, one or more skin temperature sensors 8, expanded section static probe 116, product discharge chamber static sensor 118, and optionally also differential pressure sensors (not shown) configured to sense differential pressure in bed 10.

[0074] Each skin temperature sensor 8 is configured and positioned to sense the temperature T_w of the resin (or other reactor contents) near the wall of reactor 10

during operation. Each skin temperature sensor 8 is typically implemented as a thermocouple sensor having fast response design and mounted along the reactor wall so as to protrude into fluidized bed 5 (and/or the volume above bed 5) from the reactor wall by a small amount (e.g., one eighth to one quarter of an inch).

[0075] Each bed temperature sensor 16 is positioned in the fluidized bed, and is used with a control system (which can include processor 50 of Fig. 1) and the external cooling loop to control the fluidized bed temperature T_{rx} to be near to the process set-point. Each bed temperature sensor 16 can be a resistance temperature sensor positioned and configured to sense bed temperature during reactor operation at a location within reactor 10 away from the reactor wall. The resistance temperature sensor can be mounted so as to protrude into the bed (e.g., eight to eighteen inches away from the reactor wall) more deeply than does skin temperature sensor 8. In some preferred embodiments, each skin temperature sensor 8 is a high speed skin thermocouple. A high speed thermocouple can sense reactor temperature excursions of shorter duration than can a conventional thermocouple. Detection of such short duration temperature excursions can be necessary to generate reaction parameter data that are useful to perform typical embodiments of the inventive method.

[0076] In operation, entrainment static probe 111 positioned between compressor 32 and cooler 30 generates reaction parameter data by monitoring the static charge of entrained particles within line 31.

[0077] Reactor static probes 112 and 113 (and optionally also at least one other reactor static probe) are used to monitor the static charge at or near the reactor wall within fluidized bed 5. Reactor static probe 113 is located in the upper portion of fluidized bed 5 and is thus sometimes referred to herein as an “upper bed” reactor static probe (or “upper bed” static probe).

[0078] Additional static probes can also be used to monitor the static charge at other locations of the reactor system. For example, distributor plate static probe 117 can be positioned near to (e.g., on or at) distributor plate 12 to monitor the static charge at or near to distributor plate 12, and an annular disk static probe can

be positioned near to (e.g., on or at) annular disk 107 to monitor the static charge at or near to disk 107.

[0079] Electrostatic activity in a reactor system can be monitored by the specific static probes described herein or other static probes to generate reaction parameter data to be processed in accordance with the invention. A static probe typically includes a metallic probe tip, one or more signal wires, an electrical feed-through, and a measuring instrument. The probe tip may comprise a cylindrical rod, but could be any cross sectional form such as square, rectangular, triangular, or oblong. In various embodiments, the probe tip is made of any of a variety of conductive materials. With respect to the signal wires, any conventional insulated wire may be used. With respect to the electrical feed-through, any suitable feed-through may be used as long as it provides the necessary electrical isolation from ground (and the reactor walls), and provides the required pressure seal to prevent leakage of high pressure reactor gases from the reactor. Electrical feed-through suitable for use in typical embodiments are available commercially, e.g., a transducer gland assembly from Conax Buffalo Technologies.

[0080] In the Fig. 1 system, reactor gas composition may be measured using gas chromatograph system 40. Monitored reactor gas composition parameters can be or include concentrations (and partial pressures) of all reactant gases and induced condensing agents (ICAs), as well as all inert gases (such as nitrogen, hydrocarbon inerts, etc.) that are present in relevant quantities.

[0081] The Fig. 1 system optionally also employs other sensors and also other apparatus to measure other reaction parameters during a polymerization reaction. Such other reaction parameters can include instantaneous and bed-averaged resin product properties (e.g., melt index and density of the polymer resin product being produced by the Figure 1 system during a polymerization reaction). Resin product properties are conventionally measured by periodically sampling the resin as it exits the reactor (e.g. once per hour), and performing the appropriate tests in a quality control laboratory.

[0082] It is known how to control various process control variables (e.g., to control gas phase composition within reactor 10, the concentration of induced condensing agents (ICAs) and comonomer introduced into reactor 10, partial pressure of at least one reactant (e.g., ethylene) introduced into reactor, and the type and properties of each catalyst introduced into reactor 10, and to use elements 30 and 32 in the manner described above to control temperature) to control various reactions performed by the Figure 1 system. For example, it is known how to control a polymerization reaction during a transition by controlling process control variables such that the product (granular polymer 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.

[0083] In typical embodiments of the invention, a reaction (e.g., a steady-state reaction and/or a reaction transition) performed by a polymerization reactor is controlled by adjusting (or regulating) controlling process variables in response to at least one control variable determined from reaction parameter data processed in accordance with the invention. Each such control variable is determined based on a processed version of data output from sensors (and optionally also other apparatus) that measure reaction parameters. Processor 50 of Figure 1 is an example of a processor programmed to process reaction parameter data in on-line fashion in accordance with the invention and to generate one or more of such control variables in on-line fashion in accordance with any of various embodiments of the invention in response to the processed reaction parameter data (e.g., a processed version of the output of one or more of temperature sensor(s) 16, skin temperature sensor(s) 8, sensors 111, 112, 113, 114, 115, 116, 117, and 118, and gas chromatograph 40, and optionally also a processed version of resin density and/or MI or other resin properties measured during the reaction). Optionally also, processor 50 is programmed to control (or cause other elements of the Fig. 1 system to control) the reaction in response to each control variable. Processor 50 may be a separate, stand alone processor, or it may be integral with

other process control computers that are conventionally used to monitor and control the reactor system.

[0084] Preferably, processor 50 is configured and programmed to process reaction parameter data from all or some of the system's reaction monitoring instruments, and optionally also to combine the resulting processed data with relevant calculated parameters into an integrated computer display for presentation to users (e.g., plant operators). An example of such a computer display is display 60 of Fig. 1. The computer display can be supplemented by process alarms or advisory notices (e.g., an "excessive static indication" notice) to users to warn of conditions in the process that may be approaching those that will lead to sheeting (e.g., wall or dome sheeting) or another discontinuity event. Such alarms or advisory notices can also be combined with recommended control actions to avoid the discontinuity event (e.g., recommended control actions displayed as part of the display 60). For example, in response to kurtosis values generated (or data generated from wavelet transforms) in accordance with the invention, an advisory could be generated with a displayed (or otherwise promulgated) recommendation to reduce the reactor temperature and/or isopentane concentration to avoid dome sheeting.

[0085] With reference to Fig. 2, we next describe a class of embodiments of the inventive method in which a wavelet transform is performed on reaction parameter data. Each such embodiment includes the steps of: (a) monitoring at least one reaction parameter of a polymerization reaction in a fluidized bed reactor system to generate (in on-line fashion) time-domain reaction parameter data; (b) wavelet transforming the reaction parameter data to generate frequency-domain data comprising frequency coefficients associated with time values; and (c) determining from the frequency-domain data (e.g., from spikes or other anomalies in a plot of the frequency-domain data) at least one indication of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition leading to sheeting or chunking. Processor 50 of Fig. 1 can be programmed to perform steps (b) and (c). The reaction parameter data generated in step (a) can be

the output of one or more sensors of any of a variety of different types, including but not limited to bed static data (e.g., bed static data generated using reactor static probe 112 or 113), entrainment static data (e.g., entrainment static data generated using probe 111) and other carryover static data (e.g., static data generated using distributor plate static sensor 117 or expanded section static probe 116), other static data (e.g., static data generated using product discharge chamber static sensor 118), acoustic emission data (e.g., acoustic data generated using acoustic emission sensors 114 and 115), temperature data (e.g., temperature data generated using bed temperature sensor 16 or skin temperature sensor 8), and differential pressure data generated using differential pressure sensors. The reaction parameter data generated in step (a) can be either the raw output of one or more sensors or a processed version of such raw sensor output that has undergone high-pass (or other) filtering or other preliminary processing.

[0086] Step (c) can include the step of generating a signal (or data or a display) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event). For example, processor 50 of Fig. 1 can be programmed in accordance with the invention to perform step (c) including by generating display 60 (and also data) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event).

[0087] The inventors have recognized that frequency-domain data generated by wavelet-transforming reaction parameter data are more reliably indicative of the likelihood of imminent resin stickiness or sheeting (and of many unsafe fluidized bed reactor operating conditions) than are frequency coefficients generated by Fourier-transforming (or sine- or cosine-transforming) the data. This is because a transform of the latter type essentially fits sinusoidal functions to time-domain data and thus the Fourier-transformed (or sine- or cosine-transformed) reaction parameter data are indicative only of synchronous events (i.e., a Fourier transform

cancels out information indicative of two events that are out-of-phase with each other). In contrast, a wavelet transform essentially fits wavelet functions (e.g., Haar, Daubechies, Coiflet or other functions of a type having a characteristic frequency and center time) to the data being transformed, and thus wavelet-transformed reaction parameter data are indicative of asynchronous events as well as synchronous events. The inventors have further recognized that in predicting imminent resin stickiness or resin sheeting (or other unsafe reactor operating conditions), asynchronous events in the reactor system are typically the type of events of interest.

[0088] For example, consider the time series of approximately 30,000 reaction parameter data values plotted versus time (in minutes) in the graph at the top of Fig. 2. Wavelet coefficients generated in accordance with the invention by performing a continuous wavelet transform on these data values are plotted in the two-dimensional plot at the bottom of Fig. 2 as a function of time (over the same time range as in the upper graph of Fig. 2), with the magnitude of each wavelet coefficient indicated by a degree of darkness (lighter coefficients have higher magnitudes and darker coefficients have lower magnitudes). There is a set of wavelet coefficients (each associated with a different frequency) for each time value.

[0089] If one considers the time series of data values plotted in the upper graph of Fig. 2 as a time domain function $X(t)$, the continuous wavelet transform produces a set of wavelet coefficients (plotted in the lower graph of Fig. 2), each having a scale index and a translation index (described below), as a result of convolutions of the function $X(t)$ with wavelet transform functions. To generate the wavelet coefficients of Fig. 2, commercially available software (the MATLAB software available from MathWorks, Inc.) was used to program a processor to perform the wavelet transform.

[0090] It is known how to program a processor (e.g., processor 50 of Fig. 1) to perform a continuous or discrete wavelet transform (or other wavelet transform) efficiently. One of ordinary skill in the art would be able readily to program a

processor to perform such a wavelet transform if commercially available software were not used for this purpose.

[0091] To implement the wavelet transform, scaled versions of the mother wavelet function $\Psi(t)$ are used. These are scaled versions in the sense that the time scale of the mother wavelet function (the time range over which it has nonzero values) is stretched by various amounts to define a set of differently scaled versions of the mother wavelet function. Each wavelet coefficient plotted in the lower graph of Fig. 2 has a scale index which corresponds to a frequency along the vertical axis. The frequency values marked along the vertical axis correspond to the “time scale” of the scaled mother wavelet function used to generate all wavelet coefficients plotted in the same row of the graph.

[0092] To implement the wavelet transform, a set of differently time-shifted versions of the scaled mother wavelet function is used. Each scaled version of the mother wavelet function is time-shifted in the sense that its time range (the time range over which it has nonzero values) is translated by one of a predetermined set of time values. Each of the wavelet coefficients plotted in one column of the lower graph of Fig. 2 (having time coordinate T) is generated from a subset of the reaction parameter data values (of the upper graph of Fig. 2) plotted within a time interval centered at this time T and from one time-shifted, scaled mother wavelet function whose center time is the time T . Each wavelet coefficient plotted in the lower graph of Fig. 2 thus has a translation index which corresponds to a time value marked along the graph’s horizontal axis.

[0093] The fluidized bed reactor system operators observed that three resin sheeting events occurred in the reactor during generation of the data plotted in the upper graph of Fig. 2, at the approximate times 2.75, 2.84, and 2.88 along the graph’s horizontal axis. The wavelet coefficients plotted in the lower graph of Fig. 2 are clearly indicative of these three sheeting events. However, the reaction parameter data values plotted in the upper graph of Fig. 2 are less not clearly indicative of the three sheeting events.

[0094] Typically, polymerization reaction parameter data generated during smooth, steady state operation of a fluidized bed reactor (with efficient, random mixing of particles in the fluidized bed) using a sensor (e.g., a static charge, acoustic, temperature, pressure or differential pressure sensor) with a sufficiently high sample rate (e.g., 100 Hz) are indicative of a noisy signal (e.g., as are portions of the signal plotted in the upper graph of Fig. 2) characteristic of the random interaction of the well-mixed bed with the sensor. Undesired non-uniformities in the bed cause subtle changes in the randomness of the data values. Undesired non-uniformities in the bed are typically indicated by spikes in the data which occur randomly with characteristic time scales of less than one second, or by other manifestations of decreased randomness in the data. Such spiking (or decreased randomness) can be caused, for example, by the undesired presence of resin sheets or conditions leading to sheeting. In some cases, undesired non-uniformities in the bed are indicated by spiking in entrainment static data, which can occur when a continuity additive clears up excess entrainment to reveal discharge events. Because of the random, asynchronous nature of the spiking or other typical manifestations of decreased randomness, and because the spikes (or other manifestations) are typically infrequent and phase-shifted, frequency coefficients generated by conventionally Fourier-transforming (or sine- or cosine-transforming) measured reaction parameter data are not reliably indicative of the spikes or other manifestations since the corresponding time of the event is missing. The inventors have recognized that, in contrast, wavelet coefficients generated by wavelet transforming such reaction parameter data in accordance with the invention are reliably indicative of such spikes or other manifestations.

[0095] With reference to Figs. 3-6, we next describe a class of embodiments of the invention in which kurtosis of measured reaction parameter data is determined. In each such embodiment, the inventive method includes the steps of: (a) monitoring at least one reaction parameter of a polymerization reaction in a fluidized bed reactor system to generate (in on-line fashion) time-domain reaction parameter data (i.e., a time sequence of reaction parameter values); and (b) determining kurtosis of each of at least two subsets of the reaction parameter data,

each of said subsets of the reaction parameter data including data values in a different one of a sequence of different time intervals. Optionally, the method also includes a step of: (c) determining from kurtosis values generated in step (b) at least one indication of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition, and typically also a step of generating a signal (or data or a display) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event).

[0096] The reaction parameter data generated in step (a) can be the output of one or more sensors of any of a variety of different types, including but not limited to bed static data (e.g., bed static data generated using reactor static probe 112 or 113), entrainment static data (e.g., entrainment static data generated using probe 111) and other carryover static data (e.g., static data generated using distributor plate static sensor 117 or expanded section static probe 116), other static data (e.g., static data generated using product discharge chamber static sensor 118), acoustic emission data (e.g., acoustic data generated using acoustic emission sensors 114 and 115), temperature data (e.g., temperature data generated using bed temperature sensor 16 or skin temperature sensor 8), absolute pressure data generated using pressure sensors, and differential pressure data generated using differential pressure sensors. The reaction parameter data generated in step (a) can be either the raw output of one or more sensors or a processed version of such raw sensor output that has undergone high-pass (or other) filtering or other preliminary processing.

[0097] Step (c) can include the step of generating a signal (or data or a display) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event). For example, processor 50 of Fig. 1 can be programmed in accordance with the invention to perform step (c) including by generating display

60 (and also data) indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition (e.g., likelihood of an imminent discontinuity event).

[0098] The inventors have recognized that because kurtosis values are not prone to baseline fluctuations and can be self-normalizing, they are especially useful to determine degree of resin stickiness, or approach to or imminence of resin stickiness (or an unsafe or undesired reactor operating condition or an imminent sheeting event unrelated to resin stickiness). For example, if one simply uses time-averaged values of sensor data for this purpose (i.e., an average of the data values in each of a sequence of subsets of sensor data values, each subset generated in a different one of a sequence of time windows) rather than a sequence of kurtosis values of the sensor data (one kurtosis value for each of a sequence of subsets of the data values, each subset generated in a different one of a sequence of time windows), the baseline average of a sequence of the time-averaged values will typically change over time so that fast fluctuations (among individual ones of the time-averaged values) cannot reliably be identified unless the time-averaged values are corrected for baseline average changes.

[0099] For example, consider the time series of reaction parameter data values (curve "A") plotted in Fig. 3, and the time series of kurtosis values (curve "C") generated from these data values plotted in Fig. 4. The reaction parameter data values plotted in Fig. 3 are static charge data values generated while monitoring a polymerization reaction using a static probe in the expanded section of a fluidized bed reactor (a static probe of the same type as static probe 116, positioned in expanded section 19 of reactor 10 of Fig. 1). The degree of resin sheeting in the same reactor (in units of pans of sheeted resin material collected during the reaction as marked along the vertical axis at the right, at each of a sequence of sample times with a sampling frequency of two per day) is plotted as curve "B" in Fig. 3. Curve B indicates that the onset of a sheeting event occurred between the times labeled as hours 0:00 and 12:00 on June 6, 2004.

[00100] Fig. 4 is a graph of kurtosis values (curve “C”) generated from the static charge data values (curve “A”) plotted in Fig. 3, superimposed on the same resin sheeting curve (curve “B”) that is also plotted in Fig. 3. Each kurtosis value of curve C was generated from a subsequence of the static charge values of Fig. 3 within a time window including (and corresponding to) the time value at which the kurtosis value is plotted in Fig. 4. Specifically, each kurtosis value is:

$$\gamma_2 = \mu_4 / \sigma^4 - 3, \text{ where}$$

μ_4 / σ^4 is the fourth standardized moment, μ_4 is the fourth moment about the mean, and σ is the standard deviation, of the relevant subsequence of static charge values. To generate the kurtosis values of Fig. 4, commercially available software (the MATLAB software available from MathWorks, Inc.) was used to program a processor to perform the calculation of kurtosis. It is known how to program a processor (e.g., processor 50 of Fig. 1) to determine efficiently the kurtosis of a set of data values. One of ordinary skill in the art would be able readily to program a processor to determine each kurtosis value plotted in Fig. 4 if commercially available software were not used for this purpose.

[00101] The time series of static charge data values (curve “A” of Fig. 3) exhibits baseline fluctuations and has a relatively low signal to noise ratio. The time series of kurtosis values (curve “C” of Fig. 4) does not exhibit baseline fluctuations and has a relatively high signal to noise ratio. The inventors have recognized (consistent with Figs. 3 and 4 and with other experimentation and analysis) that a time series of kurtosis values (e.g., the Fig. 4 values) is an excellent predictor of resin sheeting events (e.g., the sheeting event which occurred between the times labeled in Figs. 3 and 4 as hours 0:00 and 12:00 on June 6, 2004) and is typically a much better predictor of the sheeting event than a corresponding time series of reaction parameter data values (e.g., the static charge data values plotted as curve “A” of Fig. 3).

[00102] The inventors have also recognized based on experimentation and analysis that in general, in analyzing reaction parameter data values generated by

monitoring a polymerization reaction in a fluidized bed reactor, a times series of kurtosis values generated from a time series of the reaction parameter data values is more clearly indicative of degree of resin stickiness, and of an approach to or imminence of resin stickiness in the reactor (or an unsafe or undesired reactor operating condition) than are the reaction parameter data values themselves, or root mean square or standard deviation values generated from the reaction parameter data values. More specifically, given a times series of such reaction parameter data values, and a set of subsequences of the reaction parameter data values (each within a different time window of a set of time windows having different center times but equal duration), and a kurtosis value, a root mean square, a mean value, and a standard deviation value for each such subsequence, the inventors have recognized that in general the kurtosis values are better predictors (and are more clearly indicative) of degree of resin stickiness and of approach to or imminence of resin stickiness in the reactor (or of unsafe or undesired reactor operating conditions) than are the reaction parameter data values themselves, the root mean square values, the mean values, or the standard deviation values. Sequences of such kurtosis values do not exhibit baseline fluctuations and typically have better signal to noise ratio and are better indicative of maximum and minimum type events than are sequences of the corresponding reaction parameter data values themselves or of corresponding root mean square, mean, or standard deviation values.

[00103] As an example, consider the values plotted in Fig. 5 which were generated from a set of approximately 13,000,000 entrainment static charge values measured during one day and a half of a polymerization reaction using an entrainment static probe in the recycle line of a fluidized bed reactor (i.e., a static probe of the same type as probe 111 positioned along recycle line 31 of the Fig. 1 reactor system). This sequence of entrainment static charge values was windowed, in the sense that subsequences of the entrainment static charge values were determined, with each subsequence including values within a different one of a set of time windows having different center times but equal duration. A kurtosis value, a root mean square value, a standard deviation value, and a mean value was determined for

each subsequence of the entrainment static charge values. In Fig. 5, the graph labeled "Mean" plots the mean values versus time (over the time range having 1.5 day duration), the graph labeled "Kurtosis" plots the kurtosis values versus time over the same range, the graph labeled "RMS" plots the root mean square or "RMS" values versus time over the same range, and the graph labeled "Std Dev" plots the standard deviation values versus time over the same range. During the 1.5 day reaction monitoring period, a resin sheeting incident occurred at approximately the time labeled 125 along the horizontal axis of each graph of Fig. 5. The inventors have recognized (consistent with inspection of the data plotted in Fig. 5) that the kurtosis values of Fig. 5 were an excellent predictor of the resin sheeting event and were a better predictor of the sheeting event than the mean, RMS, or standard deviation values of Fig. 5.

[00104] As another example, consider the kurtosis values plotted versus time as the upper set of data in Fig. 6 (in which one kurtosis value is plotted for each interval of 5 minutes over the 6.4-day period indicated along the horizontal axis). These kurtosis values were generated from a time series of entrainment static charge values measured during a polymerization reaction using an entrainment static probe in a fluidized bed reactor system's gas recycle line (i.e., a static probe of the same type as probe 111 positioned along recycle line 31 of the Fig. 1 system). Each plotted kurtosis value was computed from a different subsequence of the static charge values in a different one of a sequence of time windows. During the reaction monitoring period, a continuity additive was fed into the reactor and the flow rate of this additive into the reactor is plotted (in units of pounds per hour) as the lower set of data in Fig. 6 (labeled "Feed Rate of Continuity Additive"). As shown in Fig. 6 the flow of the continuity additive into the reactor results in sharp increases in entrainment static kurtosis. In addition, when continuity aid was lost for a period of about 18 hours commencing on the day labeled 3/29, the kurtosis of entrainment static kurtosis responded promptly. In this way, the kurtosis of entrainment static is a responsive measure of the impact on continuity aid on solids entrainment and may be used in a feedback loop for control of continuity aid flow rate to the reactor.

[00105] During this period of loss of continuity additive flow, a resin sheeting incident occurred just after the time at which a peak in kurtosis values appears in Fig. 6 (the largest peak in kurtosis values in the interval of continuity additive flow loss, which occurs just before the start of the day labeled 3/30). The inventors have recognized (consistent with inspection of the data plotted in Fig. 6) that the kurtosis values of Fig. 6 were an excellent predictor of the resin sheeting event. Fig. 6 shows that immediately after the loss of the continuity additive the kurtosis begins to increase several hours before the actual sheeting event. These data show that the kurtosis of the entrainment static is an excellent predictor of an imminent sheeting event well in advance of the actual event.

[00106] Some preferred embodiments of the inventive method include the steps of determining a sequence of kurtosis values from measured reaction parameter data, and predicting from the kurtosis values an approach to or imminence of resin sheeting (e.g., kurtosis values determined from product chamber static charge data are used to predict wall sheeting, or kurtosis values determined from static data from one or more static sensors in an expanded section of the reactor above the fluidized bed are used to predict dome sheeting, or kurtosis values determined from one or more differential pressure sensors in the fluid bed reactor are used to predict resin chunking).

[00107] Other embodiments of the inventive method include the steps of determining a sequence of kurtosis values from measured reaction parameter data values other than static data values (e.g., temperature or acoustic data values) and predicting from the kurtosis values an approach to or imminence of resin sheeting or a discontinuity event, or an unsafe or undesired reactor operating condition.

[00108] We next describe examples of commercial-scale reactions (e.g., commercial-scale, gas-phase fluidized-bed polymerization reactions) that can be monitored and optionally also controlled in accordance with the invention. Some such reactions can occur in a reactor having the geometry of reactor 10 of Figure 1. In different embodiments of the invention, performance of any of a variety of

different reactors is monitored and optionally also controlled in accordance with the invention.

[00109] In some embodiments, a continuous gas phase fluidized bed reactor is monitored and optionally also controlled in accordance with the invention while it operates to perform polymerization as follows: The fluidized bed is made up of polymer granules. Gaseous feed streams of the primary monomer and hydrogen together with liquid or gaseous comonomer are mixed together in a mixing tee arrangement and introduced below the reactor bed into the recycle gas line. For example, the primary monomer is ethylene and the comonomer is 1-hexene. The individual flow rates of ethylene, hydrogen and comonomer are controlled to maintain fixed gas composition targets. The ethylene concentration is controlled to maintain a constant ethylene partial pressure. The hydrogen is controlled to maintain a constant hydrogen to ethylene mole ratio. The hexene is controlled to maintain a constant hexene to ethylene mole ratio (or alternatively, the flow rates of comonomer and ethylene are held at a fixed ratio). The concentration of all gases is measured by an on-line gas chromatograph to ensure relatively constant composition in the recycle gas stream. A solid or liquid catalyst is injected directly into the fluidized bed using purified nitrogen as a carrier. The feed rate of catalyst is adjusted to maintain a constant production rate. The reacting bed of growing polymer particles is maintained in a fluidized state by the continuous flow of make up feed and recycle gas through the reaction zone (i.e. the fluidized bed). In some implementations, a superficial gas velocity of 1 to 3 ft/sec is used to achieve this, and the reactor is operated at a total pressure of 300 psig. To maintain a constant reactor temperature, the temperature of the recycle gas is continuously adjusted up or down to accommodate any changes in the rate of heat generation due to the polymerization. The fluidized bed is maintained at a constant height by withdrawing a portion of the bed at a rate equal to the rate of formation of particulate product. The product is removed semi-continuously via a series of valves into a fixed volume chamber, which is simultaneously vented back to the reactor. This allows for highly efficient removal of the product, while at the same time recycling a large portion of the unreacted gases back to the

reactor. This product is purged to remove entrained hydrocarbons and treated with a small stream of humidified nitrogen to deactivate any trace quantities of residual catalyst.

[00110] In other embodiments, a reactor is monitored and optionally also controlled in accordance with the invention while it operates to perform polymerization using any of a variety of different processes (e.g., slurry, or gas phase processes). For example, the reactor can be a fluidized bed reactor operating to produce polyolefin polymers by a gas phase polymerization process. This type of reactor and means for operating such a reactor are well known. In operation of such reactors to perform gas phase polymerization processes, the polymerization medium can be mechanically agitated or fluidized by the continuous flow of the gaseous monomer and diluent.

[00111] In some embodiments, a polymerization reaction that is a continuous gas phase process (e.g., a fluid bed process) is monitored and optionally also controlled in accordance with the invention. A fluidized bed reactor for performing such a process typically comprises a reaction zone and a so-called velocity reduction zone. The reaction zone comprises a bed of growing polymer particles, formed polymer particles and a minor amount of catalyst particles fluidized by the continuous flow of the gaseous monomer and diluent to remove heat of polymerization through the reaction zone. Optionally, some of the re-circulated gases may be cooled and compressed to form liquids that increase the heat removal capacity of the circulating gas stream when readmitted to the reaction zone. This method of operation is referred to as "condensed mode". A suitable rate of gas flow may be readily determined by simple experiment. Make up of gaseous monomer to the circulating gas stream is at a rate equal to the rate at which particulate polymer product and monomer associated therewith is withdrawn from the reactor and the composition of the gas passing through the reactor is adjusted to maintain an essentially steady state gaseous composition within the reaction zone. The gas leaving the reaction zone is passed to the velocity reduction zone where entrained particles are removed. Finer entrained particles and dust may be removed in a cyclone and/or fine filter. The gas is

compressed in a compressor and passed through a heat exchanger wherein the heat of polymerization is removed, and then returned to the reaction zone.

[00112] The reactor temperature (T_{rx}) of the fluid bed process is normally operated at the highest temperature that is feasible, given the stickiness or sintering characteristics of the polymer in the fluid bed. Although there is no generally recognized method for establishing the upper limit of reactor temperature, the upper limit is believed to be related to the sintering temperature of the polymer product. The present method provides a quantitative for setting the temperature limits based on the MIT_R (the temperature at which the onset of melting is expected to occur in the reactor) or MRT_R (the reduced melt reference temperature, which is typically a temperature at which the onset of melting is expected to occur in the reactor). The upper limit of reactor temperature is preferably set by the limiting value of ΔMIT (or ΔMRT), defined above. The limiting value of ΔMIT , as defined herein, is the maximum amount by which the reactor temperature can exceed the MIT_R without inducing excessive stickiness in the product. The limiting value of ΔMRT , in preferred embodiments, is the maximum amount by which the reactor temperature can exceed the MRT_R without inducing excessive stickiness in the product.

[00113] In other embodiments, a reactor whose operation is monitored and optionally also controlled in accordance with the invention effects polymerization by a slurry polymerization process. A slurry polymerization process generally uses pressures in the range of from 1 to 50 atmospheres, and temperatures in the range of 0°C to 120°C, and more particularly from 30°C to 100°C. In a slurry polymerization, a suspension of solid, particulate polymer is formed in a liquid polymerization diluent medium to which monomer and comonomers and often hydrogen along with catalyst are added. The suspension including diluent is intermittently or continuously removed from the reactor where the volatile components are separated from the polymer and recycled, optionally after a distillation, to the reactor. The liquid diluent employed in the polymerization medium is typically an alkane having from 3 to 7 carbon atoms, a branched alkane in one embodiment. The medium employed should be liquid under the conditions

of polymerization and relatively inert. When a propane medium is used the process must be operated above the reaction diluent critical temperature and pressure. In one embodiment, a hexane, isopentane or isobutane medium is employed.

[00114] In other embodiments, a reaction monitored and optionally also controlled in accordance with the invention is or includes particle form polymerization, or a slurry process in which the temperature is kept below the temperature at which the polymer begins to melt and/or goes into solution. In other embodiments, a reaction monitored and optionally also controlled in accordance with the invention is a loop reactor or one of a plurality of stirred reactors in series, parallel, or combinations thereof. Non-limiting examples of slurry processes include continuous loop or stirred tank processes.

[00115] A reaction monitored and optionally also controlled in accordance with some embodiments of the invention can produce homopolymers of olefins (e.g., homopolymers of ethylene), and/or copolymers, terpolymers, and the like, of olefins, particularly ethylene, and at least one other olefin. The olefins, for example, may contain from 2 to 16 carbon atoms in one embodiment; and in another embodiment, ethylene and a comonomer comprising from 3 to 12 carbon atoms in another embodiment; and ethylene and a comonomer comprising from 4 to 10 carbon atoms in yet another embodiment; and ethylene and a comonomer comprising from 4 to 8 carbon atoms in yet another embodiment. A reaction monitored and optionally also controlled in accordance with the invention can produce polyethylenes. Such polyethylenes can be homopolymers of ethylene and interpolymers of ethylene and at least one α -olefin wherein the ethylene content is at least about 50% by weight of the total monomers involved. Exemplary olefins that may be utilized in embodiments of the invention are 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. Also utilizable herein are polyenes such as 1,3-hexadiene, 1,4-hexadiene, cyclopentadiene, dicyclopentadiene, 4-vinylcyclohex-1-ene, 1,5-cyclooctadiene, 5-vinylidene-2-norbornene and 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.

[00116] In the production of polyethylene or polypropylene, comonomers may be present in the polymerization reactor. When present, the comonomer may be present at any level with the ethylene or propylene monomer that will achieve the desired weight percent incorporation of the comonomer into the finished resin. In one embodiment of polyethylene production, the comonomer is present with ethylene in a mole ratio range in the gas phase of from 0.0001 (comonomer:ethylene) to 50, and from 0.0001 to 5 in another embodiment, and from 0.0005 to 1.0 in yet another embodiment, and from 0.001 to 0.5 in yet another embodiment. Expressed in absolute terms, in making polyethylene, the amount of ethylene present in the polymerization reactor may range to up to 1000 atmospheres pressure in one embodiment, and up to 500 atmospheres pressure in another embodiment, and up to 100 atmospheres pressure in yet another embodiment, and up to 50 atmospheres in yet another embodiment, and up to 10 atmospheres in yet another embodiment.

[00117] Hydrogen gas is often used in olefin polymerization to control the final properties of the polyolefin. For some types of catalyst systems, it is known that increasing concentrations (or partial pressures) of hydrogen increase the molecular weight or melt index (MI) of the polyolefin generated. The MI can thus be influenced by the hydrogen concentration. The amount of hydrogen in the polymerization can be expressed as a mole ratio relative to the total polymerizable monomer, for example, ethylene, or a blend of ethylene and hexane or propylene. The amount of hydrogen used in some polymerization processes is an amount necessary to achieve the desired MI (or molecular weight) of the final polyolefin resin. In one embodiment, the mole ratio in the gas phase of hydrogen to total monomer (H_2 :monomer) is greater than 0.00001. The mole ratio is greater than 0.0005 in another embodiment, greater than 0.001 in yet another embodiment, less than 10 in yet another embodiment, less than 5 in yet another embodiment, less than 3 in yet another embodiment, and less than 0.10 in yet another embodiment, wherein a desirable range may comprise any combination of any upper mole ratio

limit with any lower mole ratio limit described herein. Expressed another way, the amount of hydrogen in the reactor at any time may range to up to 10 ppm in one embodiment, or up to 100 or 3000 or 4000 or 5000 ppm in other embodiments, or between 10 ppm and 5000 ppm in yet another embodiment, or between 500 ppm and 2000 ppm in another embodiment.

[00118] A reactor monitored and optionally also controlled in accordance with some embodiments of the invention can be an element of a staged reactor employing two or more reactors in series, wherein one reactor may produce, for example, a high molecular weight component and another reactor may produce a low molecular weight component.

[00119] A reactor monitored and optionally also controlled in accordance with the invention can implement a slurry or gas phase process in the presence of a bulky ligand metallocene-type catalyst system and in the absence of, or essentially free of, any scavengers, such as triethylaluminum, trimethylaluminum, triisobutylaluminum and tri-n-hexylaluminum and diethyl aluminum chloride, diethyl zinc and the like. By "essentially free", it is meant that these compounds are not deliberately added to the reactor or any reactor components, and if present, are present to less than 1 ppm in the reactor.

[00120] A reactor monitored and optionally also controlled in accordance with the invention can employ one or more catalysts combined with up to 10 wt% of a metal-fatty acid compound, such as, for example, an aluminum stearate, based upon the weight of the catalyst system (or its components). Other metals that may be suitable include other Group 2 and Group 5-13 metals. In other embodiments, a solution of the metal-fatty acid compound is fed into the reactor. In other embodiments, the metal-fatty acid compound is mixed with the catalyst and fed into the reactor separately. These agents may be mixed with the catalyst or may be fed into the reactor in a solution, a slurry, or as a solid (preferably as a powder) with or without the catalyst system or its components.

[00121] In a reactor monitored and optionally also controlled in accordance with some embodiments of the invention, supported catalyst(s) can be combined with activators and can be combined by tumbling and/or other suitable means, with up to 2.5 wt% (by weight of the catalyst composition) of an antistatic agent, such as an ethoxylated or methoxylated amine, an example of which is Kemamine AS-990 (ICI Specialties, Bloomington Delaware). Other antistatic compositions include the Octastat family of compounds, more specifically Octastat 2000, 2500, 3000, and 5000.

[00122] Metal fatty acids and antistatic agents can be added as either solid slurries, solutions, or as solids (preferably as powders) as separate feeds into the reactor. One advantage of this method of addition is that it permits on-line adjustment of the level of the additive.

[00123] Examples of polymers that can be produced in accordance with the invention 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; and polychloroprene; norbornene homopolymers and copolymers with one or more C₂ - C₁₈ alpha olefin; terpolymers of one or more C₂ - C₁₈ alpha olefins with a diene.

[00124] Monomers that can be present in a reactor monitored and optionally also controlled in accordance with the invention include one or more of: C₂ - C₁₈ alpha olefins such as ethylene, propylene, and optionally at least one diene, for example, hexadiene, dicyclopentadiene, octadiene including methyloctadiene (e.g., 1-methyl-1,6-octadiene and 7-methyl-1,6-octadiene), norbornadiene, and ethylidene norbornene; and readily condensable monomers, for example, isoprene, styrene, butadiene, isobutylene, chloroprene, acrylonitrile, cyclic olefins such as norbornenes.

[00125] Fluidized bed polymerization can be monitored and optionally also controlled in accordance with some embodiments of the invention. The reaction can be any type of fluidized polymerization reaction and can be carried out in a single reactor or multiple reactors such as two or more reactors in series.

[00126] In various embodiments, any of many different types of polymerization catalysts can be used in a polymerization process monitored and optionally also controlled in accordance with the present invention. A single catalyst may be used, or a mixture of catalysts may be employed, if desired. The catalyst can be soluble or insoluble, supported or unsupported. It may be a prepolymer, spray dried with or without a filler, a liquid, or a solution, slurry/suspension or dispersion. These catalysts are used with cocatalysts and promoters known in the art. Typically these are alkylaluminums, alkylaluminum halides, alkylaluminum hydrides, as well as aluminoxanes. For illustrative purposes only, examples of suitable catalysts 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.

[00127] In various embodiments, a polymerization reaction monitored and optionally also controlled in accordance with the invention can employ other additives, such as (for example) inert particulate particles.

[00128] The phrases, unless otherwise specified, "consists essentially of" and "consisting essentially of" do not exclude the presence of other steps, elements, or materials, whether or not, specifically mentioned in this specification, as long as such steps, elements, or materials, do not affect the basic and novel characteristics of the invention, additionally, they do not exclude impurities normally associated with the elements and materials used.

[00129] For the sake of brevity, only certain ranges are explicitly disclosed herein. However, ranges from any lower limit may be combined with any upper limit to recite a range not explicitly recited, as well as, ranges from any lower limit may be combined with any other lower limit to recite a range not explicitly recited, in the same way, ranges from any upper limit may be combined with any other upper limit to recite a range not explicitly recited. Additionally, within a range includes every point or individual value between its end points even though not explicitly recited. Thus, every point or individual value may serve as its own lower or upper limit combined with any other point or individual value or any other lower or upper limit, to recite a range not explicitly recited.

[00130] All priority documents are herein fully incorporated by reference for all jurisdictions in which such incorporation is permitted and to the extent such disclosure is consistent with the description of the present invention. Further, all documents and references cited herein, including testing procedures, publications, patents, journal articles, etc. are herein fully incorporated by reference for all jurisdictions in which such incorporation is permitted and to the extent such disclosure is consistent with the description of the present invention.

[00131] While the invention has been described with respect to a number of embodiments and examples, those skilled in the art, having benefit of this disclosure, will appreciate that other embodiments can be devised which do not depart from the scope and spirit of the invention as disclosed herein.

CLAIMS

What is claimed is:

1. A method for monitoring a resin-producing polymerization reaction in a fluidized bed reactor system, including the steps of:
 - (a) monitoring at least one reaction parameter of the reaction to generate in on-line fashion time-domain reaction parameter data;
 - (b) wavelet transforming the reaction parameter data to generate frequency-domain data comprising frequency coefficients associated with time values; and
 - (c) determining from the frequency-domain data at least one indication of at least one of degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.
2. The method of claim 1, wherein the reaction parameter data transformed in step (b) are static data.
3. The method of claim 1, wherein the reaction parameter data transformed in step (b) are carryover static data.
4. The method of claim 1, wherein the reaction parameter data transformed in step (b) are entrainment static data.
5. The method of claim 1, wherein the reaction parameter data transformed in step (b) are acoustic emission data.
6. The method of claim 1, wherein the reaction parameter data transformed in step (b) are differential pressure data.
7. The method of claim 1, wherein the reaction parameter data transformed in step (b) are high speed reaction parameter data.

8. The method of claim 7, wherein the high speed reaction parameter data are bed temperature data.

9. The method of claim 7, wherein the high speed reaction parameter data are skin temperature data.

10. The method of any one of the preceding claims, wherein step (c) includes the step of generating a signal indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

11. The method of any one of claims 1-9, wherein step (c) includes the step of generating data indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

12. The method of any one of claims 1-9, wherein step (c) includes the step of generating a display indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

13. The method of any one of the preceding claims, also including the step of controlling the reaction in response to the at least one indication determined in step (c) in an effort to prevent occurrence of a discontinuity event.

14. The method of any one of the preceding claims, also including the step of:
(d) controlling the reaction in response to the at least one indication determined in step (c).

15. The method of claim 14, wherein step (d) includes the step of adjusting reaction temperature.

16. The method of claim 14, wherein step (d) includes the step of controlling a feed rate of a continuity additive.

17. The method of claim 14, wherein step (d) includes the step of controlling a feed rate of an induced condensing agent.

18. The method of any one of claims 1-13, also including the step of:
(d) controlling the reaction in response to the frequency-domain data determined in step (c).

19. The method of any one of the preceding claims, wherein the reaction polymerizes ethylene and at least one comonomer in the presence of a catalyst selected from a group consisting of Ziegler-Natta, chromium, chromium oxide, AlCl_3 , cobalt, iron, palladium, and metallocene catalyst.

20. The method of any one of the preceding claims, wherein the reaction produces polyethylene.

21. The method of any one of the preceding claims, wherein the reaction produces a polyolefin.

22. A method for monitoring a resin-producing polymerization reaction in a fluidized bed reactor system, including the steps of:
(a) monitoring at least one reaction parameter of the reaction to generate in on-line fashion time-domain reaction parameter data; and
(b) determining kurtosis of each of at least two subsets of the reaction parameter data, each of said subsets of the reaction parameter data including data values in a different one of a sequence of different time intervals.

23. The method of claim 22, wherein the reaction parameter data generated in step (a) are static data.

24. The method of claim 22, wherein the reaction parameter data generated in step (a) are carryover static data.

25. The method of claim 22, wherein the reaction parameter data generated in step (a) are entrainment static data.

26. The method of claim 22, wherein the reaction parameter data generated in step (a) are acoustic emission data.

27. The method of claim 22, wherein the reaction parameter data generated in step (a) are differential pressure data.

28. The method of claim 22, wherein the reaction parameter data generated in step (a) are high speed reaction parameter data.

29. The method of claim 28, wherein the high speed reaction parameter data are bed temperature data.

30. The method of claim 28, wherein the high speed reaction parameter data are skin temperature data.

31. The method of any one of claims 22-30, also including the step of:
(c) determining from kurtosis values generated in step (b) at least one indication of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

32. The method of any one of claims 22-31, wherein step (c) includes the step of generating a signal indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

33. The method of any one of claims 22-31, wherein step (c) includes the step of generating data indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

34. The method of any one of claims 22-31, wherein step (c) includes the step of generating a display indicative of at least one of a degree of resin stickiness, an approach to or imminence of resin stickiness, and an approach to or imminence of an unsafe or undesired reactor operating condition that can result in sheeting or chunking.

35. The method of any one of claims 22-31, also including the step of controlling the reaction in response to the at least one indication determined in step (c) in an effort to prevent occurrence of a discontinuity event.

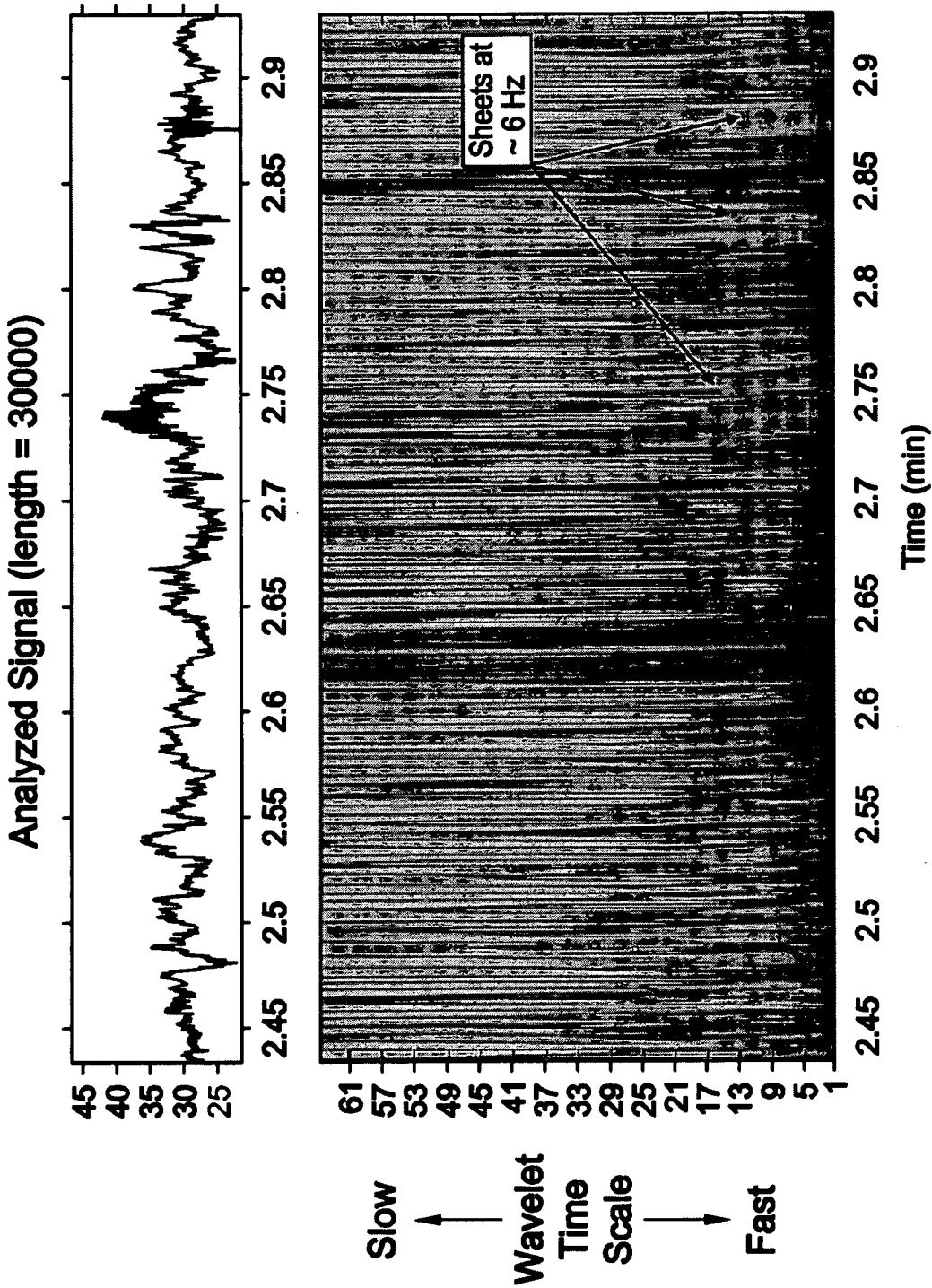
36. The method of any one of claims 31-35, also including the step of:
(d) controlling the reaction in response to the at least one indication determined in step (c).

37. The method of claim 36, wherein step (d) includes the step of adjusting reaction temperature.

38. The method of claim 36, wherein step (d) includes the step of controlling a feed rate of a continuity additive.

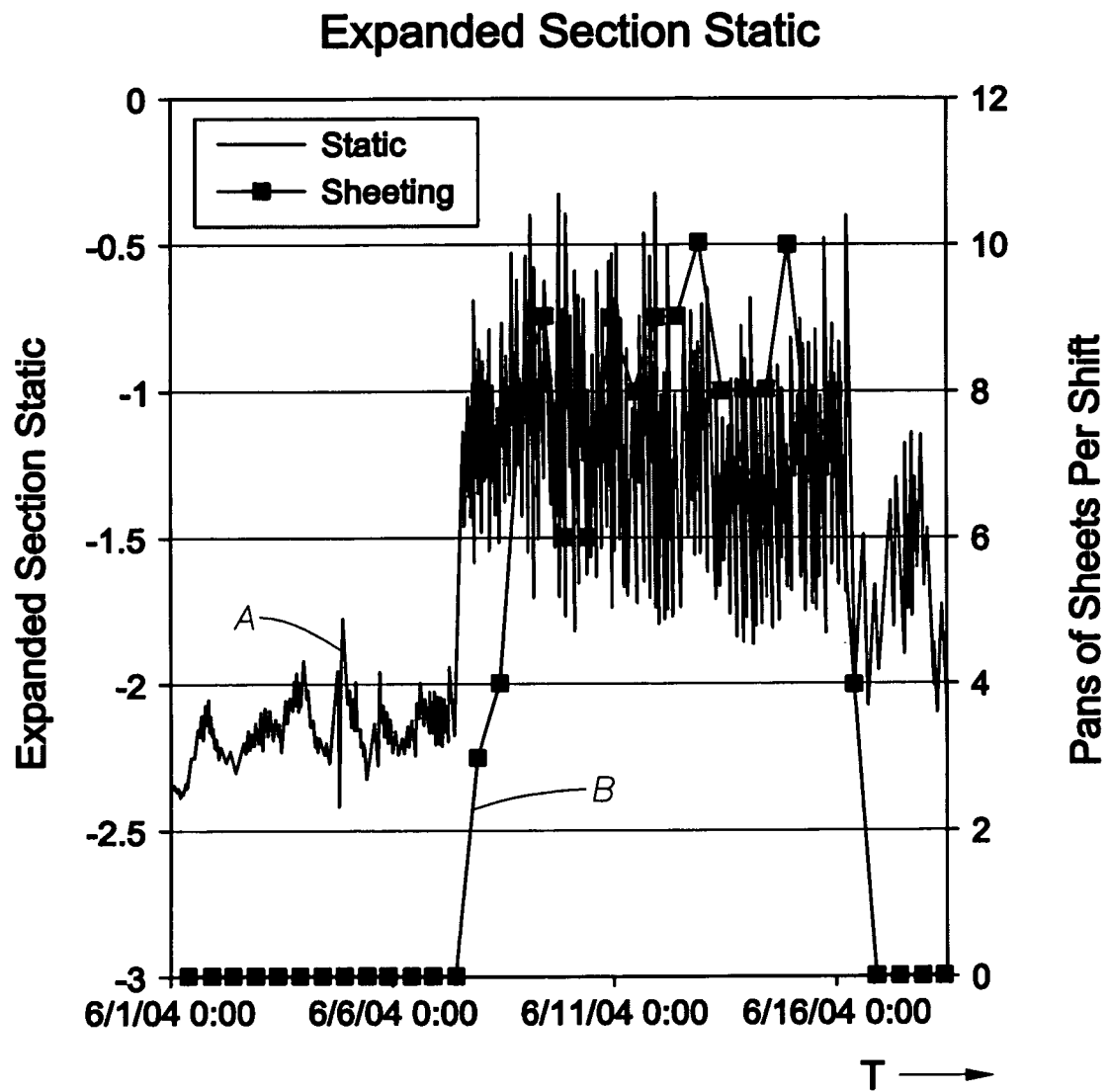
39. The method of any one of claims 22-38, also including the step of:
(c) controlling the reaction in response to kurtosis values generated during step (b).
40. The method of any one of claims 22-39, wherein the reaction polymerizes ethylene and at least one comonomer in the presence of a catalyst selected from a group consisting of Ziegler-Natta, chromium, chromium oxide, AlCl_3 , cobalt, iron, palladium, and metallocene catalyst.
41. The method of any one of claims 22-40, wherein the reaction produces polyethylene.
42. The method of any one of claims 22-41, wherein the reaction produces a polyolefin.

Fig. 2 Sheet Detection: Signal Processing to Detect Asynchronous, High Frequency Events



3/7

Fig. 3



4/7

Fig. 4

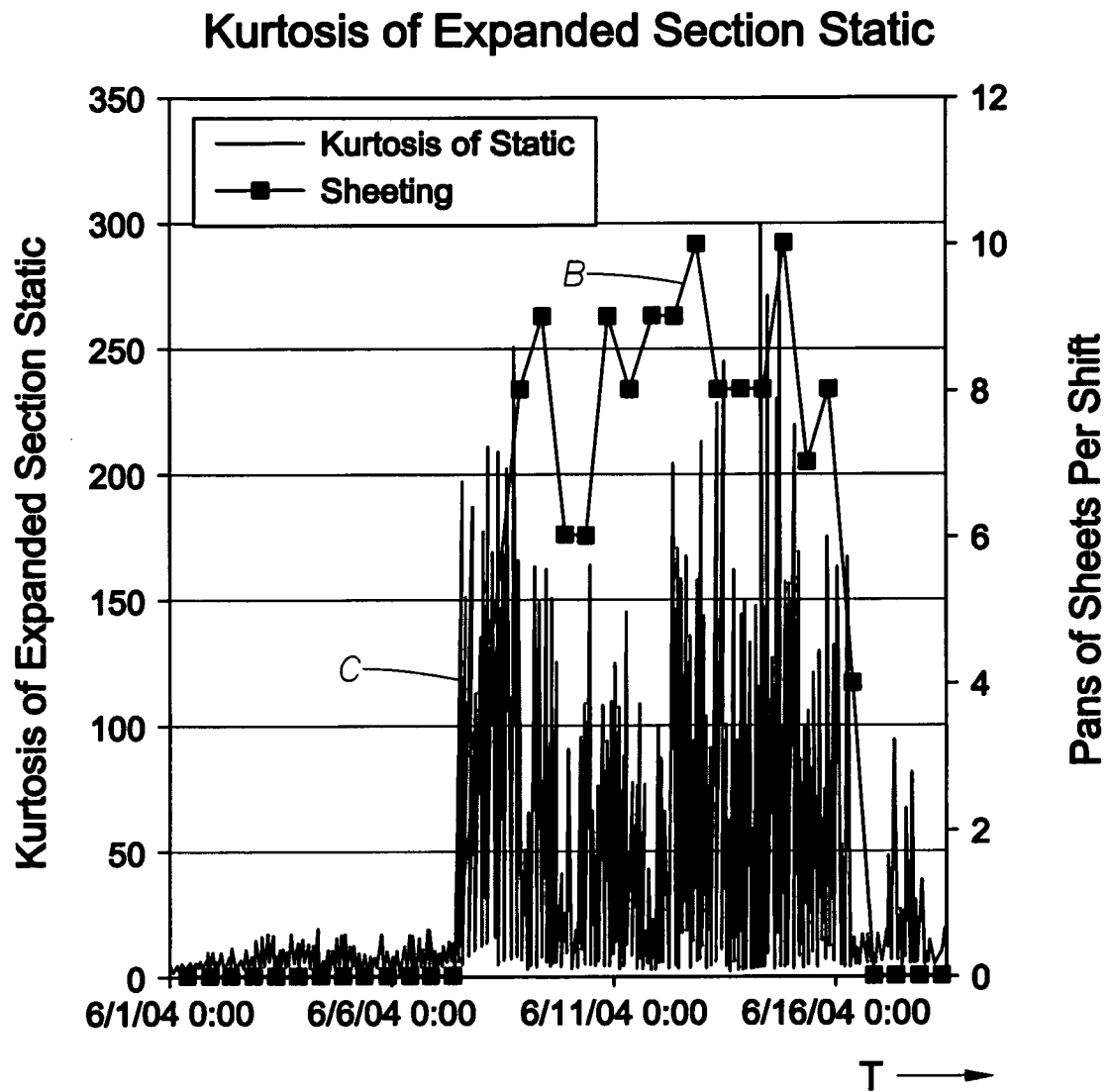
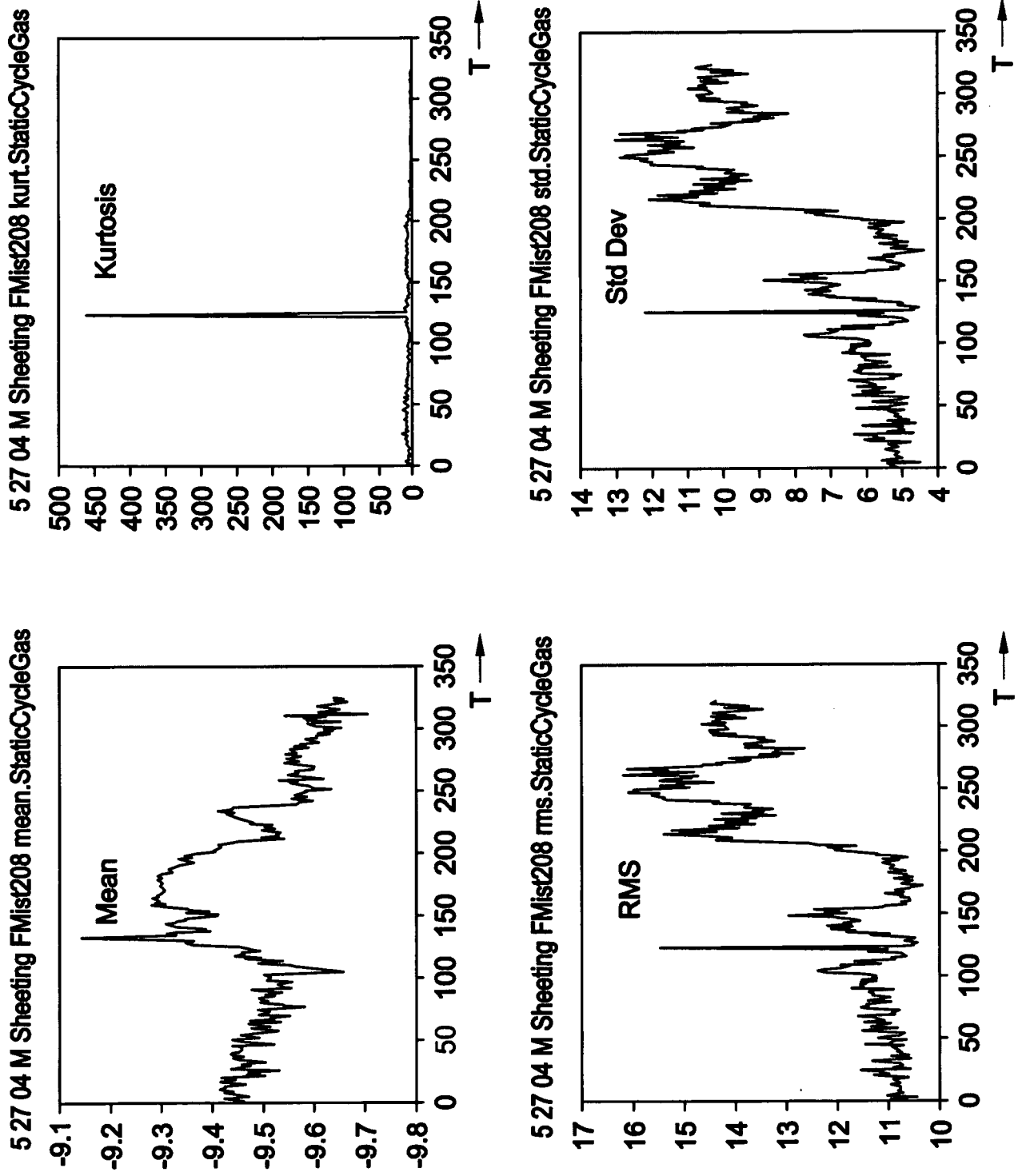


Fig. 5



CofeedTestB kurt.StaticCycleGas

Fig. 5a

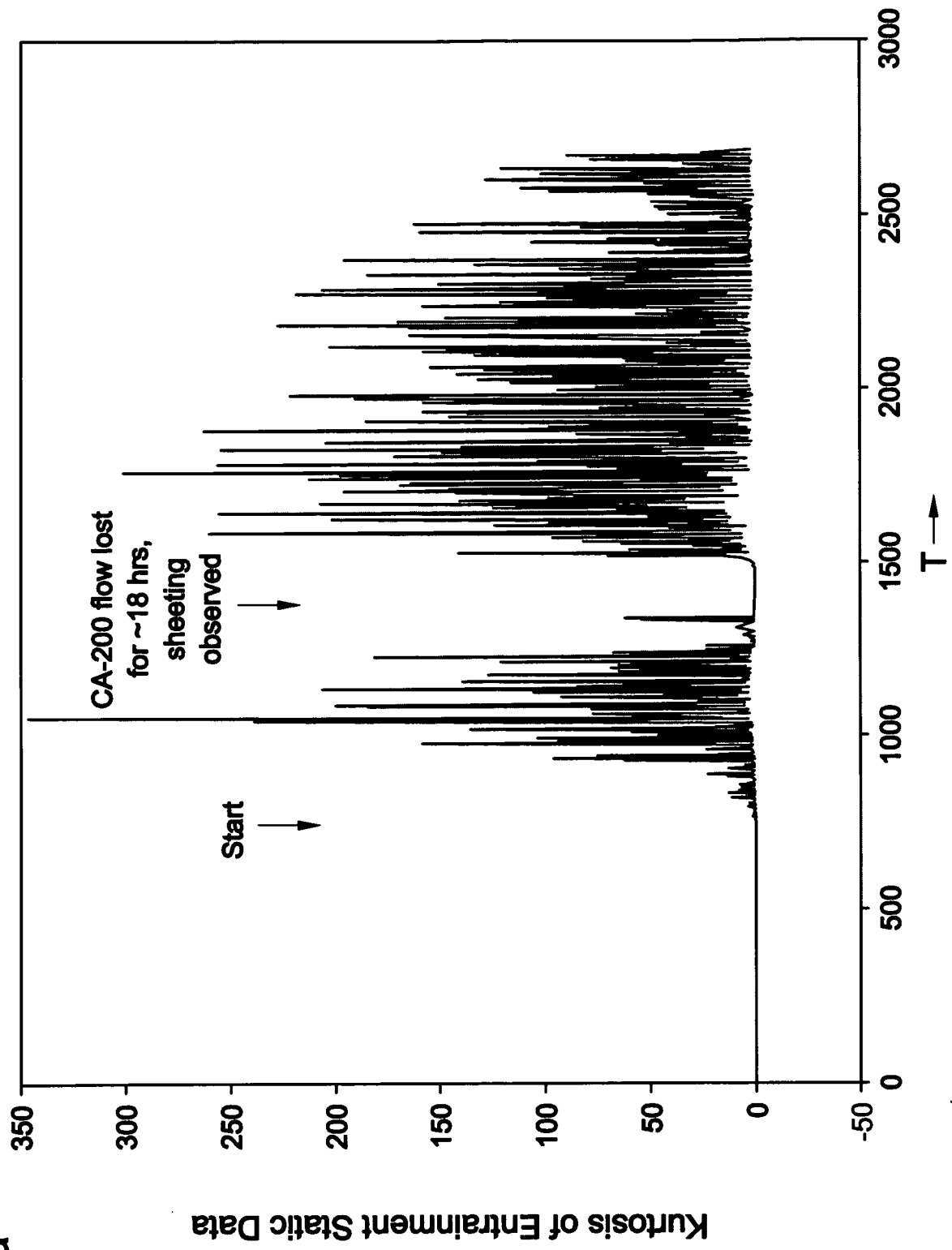


Fig. 6

Continuity Aid Test, Unipol PE Reactor

