CONTINUOUS SLURRY POLYMERIZATION VOLATILE REMOVAL

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FIELD OF INVENTION

The present invention relates to an apparatus for continuously separating polymer solids from a liquid medium comprising an inert diluent and unreacted monomers in a slurry polymerization process. In particular, the present invention relates to an apparatus for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the diluent and unreacted monomers with a reduction in compression needed for diluent vapor condensation to liquid diluent for reuse in the polymerization process. In another aspect, the invention relates to a method for continuously separating polymer solids from a liquid medium. In particular, the invention relates to a method for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the inert diluent and unreacted monomers for reuse in the polymerization process.

BACKGROUND OF THE INVENTION

In many polymerization processes for the production of polymer, a polymerization effluent is formed which is a slurry of particulate polymer solids suspended in a liquid medium, ordinarily the reaction diluent and unreacted monomers. A typical example of such processes is disclosed in Hogan and Bank's U.S Pat. No. 2,285,721, the disclosure of which is incorporated herein by reference. While the polymerization processes described in the Hogan document employs a catalyst comprising chromium oxide and a support, the present invention is applicable to any process producing an effluent comprising a slurry of particulate polymer solids suspended in a liquid medium comprising a diluent and unreacted monomer. Such reaction processes include those which have come to be known in the art as particle form polymerizations.

In most commercial scale operations, it is desirable to separate the polymer and the liquid medium comprising an inert diluent and unreacted monomers in such a manner that the liquid medium is not exposed to

contamination so that the liquid medium can be recycled to the polymerization zone with minimal if any purification. A particularly favored technique that has been used heretofore is that disclosed in the Scoggin et al, U.S. Pat. No. 3,152,872, more particularly the embodiment illustrated in conjunction with Fig. 2 of that patent. In such processes the reaction diluent, dissolved monomers, and catalyst are circulated in a loop reactor wherein the pressure of the polymerization reaction is about 100 ($6.9 \times 10^5 Nm^{-2}$) to 700 psia ($48.5 \times 10^5 Nm^{-3}$). The produced solid polymer is also circulated in the reactor. A slurry of polymer and the liquid medium is collected in one or more settling legs of the slurry loop reactor from which the slurry is periodically discharged to a flash chamber wherein the mixture is flashed to a low pressure such as about 20 psia ($1.4 \times 10^5 Nm^{-2}$). While the flashing results in substantially complete removal of the liquid medium from the polymer, it is necessary to recompress the vaporized polymerization diluent (i.e., isobutane) in order to condense the recovered diluent to a liquid form suitable for recycling as liquid diluent to the polymerization zone. The cost of compression equipment and the utilities required for its operation often amounts to a significant portion of the expense involved in producing polymer.

Some polymerization processes distill the liquefied diluent prior to recycling to the reactor. The purpose of distillation is removal of monomers and light-end contaminants. The distilled liquid diluent is then passed through a treater bed to remove catalyst poisons and then on to the reactor. The equipment and utilities costs for distillation and treatment can be a significant portion of the cost of producing the polymer.

In a commercial scale operation, it is desirable to liquefy the diluent vapors at minimum cost. One such technique used heretofore is disclosed in Hanson and Sherk's U.S. Pat. No. 4,424,341 in which an intermediate pressure flash step removes a significant portion of the diluent at such a temperature and at such a pressure that this flashed portion of diluent may be liquified by heat exchange instead of by a more costly compression procedure.

BRIEF SUMMARY OF THE INVENTION

The present invention relates to an apparatus for continuously separating polymer solids from a liquid medium comprising an inert diluent and unreacted monomers In another aspect, the invention relates to an apparatus for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the diluent and unreacted monomers with a reduction in compression needed for diluent vapor condensation to liquid diluent for reuse in a polymerization process In another aspect, the invention relates to a method for continuously separating polymer solids from a liquid medium. In another aspect, the invention relates to a method for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the inert diluent and unreacted monomers for reuse in a polymerization process.

In accordance with the present invention, there is provided an apparatus for continuously recovering polymer solids from a polymerization effluent comprising a slurry of said polymer solids in a liquid medium comprising an inert diluent and unreacted monomers. The apparatus comprises a discharge valve on a slurry reactor, examples of which include slurry loop reactors and stirred tank slurry reactors, for the continuous discharge of a portion of the slurry reactor contents into a first transfer conduit: a first flash tank having a bottom defined by substantially straight sides inclined at an angle to the horizontal equal to or greater than the angle of slide of the slurry/polymer solids; wherein the pressure of the first flash tank and temperature of the polymerization effluent are such that from about 50% to about 100% of the liquid medium will be vaporized and the inert diluent component of said vapor is condensable, without compression, by heat exchange with a fluid having a temperature in the range of about 65° F (18°C) to about 135° F (57°C): a first flash tank exit seal chamber, communicating with said first flash tank, of such a length (l) and diameter (d) as to permit such a level of concentrated polymer solids/slurry to accumulate and form a pressure seal in said first flash tank exit seal chamber: a seal chamber exit reducer providing for a continuous discharge of a plug flow of concentrated polymer solids/slurry to a second transfer conduit which communicates the

concentrated polymer solids/slurry into a second flash tank wherein the pressure of said second flash tank and temperature of the concentrated polymer solids/slurry are such that essentially all of any remaining inert diluent and/or unreacted monomer will be vaporized and removed overhead for condensation by compression and heat exchange and the polymer solids are discharged from the bottom of said second flash tank for additional processing or storage.

The invention provides also a method for the continuous removal of a stream of polymerization effluent from a slurry reactor through a discharge valve; increasing the heat content of the polymerization effluent during its transit through said first transfer conduit to a temperature below the fusion point of the polymer while continuously communicating the polymerization effluent to a first flash tank having a bottom defined by substantially straight sides inclined at an angle to the horizontal equal to or greater than the angle of slide of the concentrated polymer solids/slurry; continuously vaporizing from about 50% to about 100% of the liquid medium in said first heated flash tank to yield a concentrated polymer solids/slurry and a vapor stream at such a temperature and pressure that the inert diluent content of said vapor is condensable, without compression, by heat exchange with a fluid having a temperature in the range from about 65° F (18°C) to about 135° F (57°C); continuously discharging the concentrated polymer solids/slurry from said first flash tank to a first flash tank exit seal chamber of such a length (l) and diameter (d) that a volume of concentrated polymer solids/slurry is continuously maintained so as to form a pressure seal in said first flash tank exit seal chamber; continuously discharging the concentrated polymer solids/slurry from said first flash tank seal chamber through a seal chamber exit reducer defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom; communicating a continuous plug flow of concentrated polymer solids/slurry from said first flash tank exit seal chamber through said seal chamber exit reducer to a second transfer conduit which communicates said continuous plug flow of concentrated polymer solids/slurry to a second flash tank; and

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continuously vaporizing essentially all of any remaining inert diluent and/or unreacted monomer in a second flash

tank operated at a lower pressure than said first flash tank; condensing the vaporized inert diluent and/or unreacted monomer from said second flash tank by compression and heat exchange, and continuously discharging the essentially dried polymer slurry from said second flash tank for further processing or storage.

The present invention also relates to an apparatus for capturing a higher weight percentage of polymer solids from a circulating slurry in a loop reactor than the weight percentage of solids in the circulating slurry. The apparatus includes a conduit having a first end, wherein said first end extends for a distance into the loop reactor. The conduit also has portions defining an opening wherein said opening is positioned relative to the direction of the circulating slurry. Desirably, the opening may be facing the direction of flow of the circulating slurry. Additionally, a portion of the conduit may extend outwardly from the loop reactor for discharging, continuously or otherwise the polymer solids from the loop reactor.

The present invention also provides a process for capturing a higher weight percentage of polymer solids from a circulating slurry in a loop reactor than the weight percentage of polymer solids in the circulating slurry This process includes the step of extending for a distance into a the loop reactor a conduit having portions defining an opening wherein said opening is extends into the circulating slurry. Additionally, this process may include the step of discharging, continuous or otherwise, the polymer solids from the loop reactor through a portion of the conduit extending outwardly from the loop reactor.

The present invention also provides an apparatus for purging polymer solids from a conduit connected to a loop reactor and in fluid communication with the loop reactor. This apparatus includes a sensor, a first valve in fluid communication with the conduit, a second valve positioned between a first inert diluent and the conduit, wherein the first inert diluent is in fluid communication with the conduit between the loop reactor and the first valve In response to a signal produced by the sensor, the first valve is closed and the second valve is opened allowing the first inert diluent to enter the conduit in sufficient quantities and under sufficient pressure to purge polymer solids from the conduit. This apparatus may further include a third valve positioned

between a second inert diluent and the conduit, wherein the second inert diluent is in fluid communication with the conduit between the loop reactor and the first valve In this way, when the first valve is open and the second valve is closed the third valve is opened allowing the second inert diluent to enter the conduit.

The present invention also provides a process for purging polymer solids from a conduit connected to a loop reactor and in fluid communication with the loop reactor comprising. This process includes the steps of (i) closing a first valve in response to a first signal from a first sensor, wherein the first valve is connected to and in fluid communication with the conduit, (2) opening a second valve in response to a second signal from a second sensor, wherein the second valve is fluid communication between a first inert diluent and the conduit, and wherein the first inert diluent is in fluid communication with the conduit between the loop reactor and the first valve, and (3) flowing sufficient quantities of the first inert diluent under sufficient pressure into the conduit to purge polymer solids from the conduit. In this process the first and second sensors may be a common sensor and the first and second signal may be a common signal.

The present invention also provides an apparatus for returning fines to a polymerization slurry in a loop reactor. The apparatus includes a discharge valve for discharging a portion of the polymerization slurry from the loop reactor into a first transfer conduit. The first transfer conduit communicates the polymerization slurry into a first flash tank. The first flash tank converts a portion of the polymerization slurry into a first fluid, such as a vapor. The first fluid includes a portion of the diluent and the fines from the polymerization slurry. A second transfer conduit communicates the first fluid to a first cyclone. The first cyclone converts a portion of the first fluid into a second fluid, such as a vapor. The second fluid includes a portion of the diluent and the fines. A third transfer conduit communicates the second fluid into a heat exchanger. The heat exchanger converts the second fluid into a liquid comprising the diluent and the fines. A fourth transfer conduit returns the liquid to the polymerization slurry in the loop reactor. This apparatus may

also include a first transfer conduit heater for heat exchange between the first transfer conduit heater and the polymerization slurry.

The present invention also provides a process for returning fines to a polymerization slurry in a loop reactor. The process includes (i) discharging a portion of the polymerization slurry from the loop reactor, (ii) communicating the discharge polymerization slurry into a first flash tank, (iii) converting in the flash tank a portion of the polymerization slurry into a first fluid, the first fluid comprising a diluent and the fines, (iv) communicating the first fluid from the first flash tank to a first cyclone, (v) converting in the cyclone a portion of the first fluid into a second fluid comprising the diluent and the fines, (vi) communicating the second fluid into a heat exchanger, (vii) converting in the heat exchanger the second fluid into a liquid comprising the diluent and the fines, and (viii) returning the liquid to the polymerization slurry in the loop reactor.

The present invention further provides an apparatus and process for producing polymer from a polymerization slurry in a loop reactor operating at a space time yield greater than 2.8 lbs/hr-gal (9.3 x 10^{-5}kg $s^{-1}dm^{-3}$). In this instance, the polymer is formed in the polymerization slurry which includes a liquid medium and solids. The polymerization slurry is discharged into a first transfer conduit. The polymerization slurry is referred to as a polymerization effluent upon leaving the loop reactor. The polymerization effluent is heated in the first transfer conduit to a temperature below the fusion temperature of the polymer solids. The heated polymerization effluent is communicated through said first transfer conduit to a first flash tank. In the first flash tank, from about 50% to about 100% of the liquid medium is vaporized. The vapor is condensed by heat exchange. Polymer solids are discharged from the first flash tank to a second flash tank through a seal chamber of sufficient dimension such as to maintain a volume of polymer solids in the said seal chamber sufficient to maintain a pressure seal. The polymer solids are then communicated to a second flash tank. In the second flash tank, the polymer solids are exposed to a pressure reduction from a higher pressure in the first flash tank to a lower pressure in said second flash. The polymer solids are then discharging from said second flash tank. Additionally, the weight percent of

solids in the polymerization slurry may be greater than 47. The loop reactor may be operated at a total recirculating pumping head/reactor distance of greater than 0.15 ft/ft. The loop reactor may also be operated with a recirculating pumping head greater than or equal to 200 ft (60.8m). and have more than eight vertical legs, desirably between 10 and 16 vertical legs, more desirably between 10 and 12 vertical legs, most desirably 12 vertical legs. The volume of polymerization slurry in the loop reactor may be greater than 20,000 gallon (75800dm^3).

An object of the present invention is to provide both an apparatus and method for the continuous two stage flash drying of the polymer solids following the continuous removal of the polymerization effluent comprising polymer solids and liquid medium comprising inert diluent and unreacted monomers from a slurry reactor through a point discharge valve, a continuous solids level control in the first flash tank exit seal chamber that provides a pressure seal therein which enables said first flash tank to operate under a substantially greater pressure than said second flash tank while polymer solids are continuously discharged through the seal chamber exit reducer into the second transfer conduit and further into the second flash tank which eliminates plugging in the first flash tank and the continuous liquification of from about 50% to about 100% of the inert diluent vapor by heat exchange rather than compression.

Another object of the invention is to eliminate the need for a settling leg on the slurry reactor and the intermittent high pressure pulse in the slurry reactor caused by periodic discharging of the contents of the settling leg. Another object of the present invention is to improve safety by eliminating the possibility of plugging in a settling leg.

Another object of the invention is to eliminate plugging in equipment downstream from the discharge valve. In a settling leg of a polymerization reactor polymerization continues and the heat of reaction further heats the liquid medium and a potential exists for some of the polymer solids to dissolve or to fuse together. As the contents of the settling leg exit the discharge valve, the pressure drop causes flashing of some of the liquid medium which results in cooling the remaining liquid medium causing the dissolved polymer to

precipitate which tends to plug downstream equipment. The present invention which eliminates the need for a settling leg also eliminates this potential for downstream equipment plugging by avoiding the initial dissolution or fusion of the polymer solids.

Another object of the present invention is to increase the reactor through-put by the use of continuous discharge and increased ethylene concentrations in the liquid medium, e.g., greater than or equal to 4 weight percent at reactor outlet, desirably from 4 weight percent to 8 weight percent, still more desirably from 5 weight percent to 7 weight percent. Settling legs limit ethylene concentrations due to an increased tendency to plug downstream equipment caused by accelerated reaction within the settling leg. A continuous polymerization effluent slurry flow allows ethylene concentrations to be limited only by the ethylene solubility in the liquid diluent in the reactor, thereby increasing the specific reaction rate for polymerization and increasing reactor throughput.

Another object of the present invention is to increase the weight percent (wt %) of polymer solids in the polymerization slurry circulating in the polymerization zone in the loop reactor. Desirably, the wt % of polymer solids in the polymerization slurry is greater than 45, more desirably, from 45 to 65, still more desirably from 50 to 65, and most desirably from 55 to 65.

Another object of the present invention is to increase the space time yield (STY), expressed in terms of pounds per hour-gallon (lbs/hr-gal) (1lb/hr-gal = 3.3 x 10^{-5}kgs^{-1}dm^{-3}). Desirably, the STY is greater than 2.6, more desirably from 2.6 to 4.0, and most desirably from 3.3 to 4.0.

Other aspects, objects, and advantages of the present invention will be apparent from the following disclosure and Figures 1 and 2.

The claimed apparatus and process provide several advantages over the prior art including: (1) allowing for a continuous processing of the contents of a slurry reactor from the point of discharge of the polymerization slurry effluent through a discharge valve; a first flash tank, a seal chamber; a seal chamber exit reducer; and therefrom to a second flash tank. (2) significantly increasing ethylene concentration in the loop reactor liquid medium thereby

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increasing reactor through-put, (3) significantly increasing the wt % of polymer solids in the polymerization slurry, (4) significantly increasing reactor

space time yield and (5) energy consumption is reduced by reducing the need to compress and/or distill the reactor vapor-liquid effluent. Recycling compressors and other downstream equipment can be reduced in size or eliminated.

BRIEF DESCRIPTION OF THE DRAWING

Figs. 1 and 2 are a schematic diagram illustrating an apparatus for continuously separating polymer solids from diluent and unreacted monomer in accordance with the present invention.

Fig. 3 is an enlarged, cross sectional view of the discharge conduit with opening extending a distance into the loop reactor and the circulating polymerization slurry.

Fig. 4 is a schematic view of a pressure control system.

DETAILED DESCRIPTION OF THE INVENTION

As used herein, the term "polymerization slurry" means substantially a two phase composition including polymer solids and liquid circulating within the loop reactor. The solids include catalyst and a polymerized olefin, such as polyethylene. The liquids include an inert diluent, such as isobutane, with dissolved monomer, comonomer, molecular weight control agents, such as hydrogen, antistatic agents, antifouling agents, scavengers, and other process additives.

As used herein, the term "space time yield" (STY) means the production rate of polymer per unit of loop reactor volume or polymerization slurry volume.

As used herein, the term "catalyst productivity" means weight of polymer produced per weight of catalyst introduced into the loop reactor

As used herein, the term "polymer residence time" means the average duration that a polymer particle remains within the loop reactor.

The present invention is applicable to any mixture which comprises a slurry of polymer solids and a liquid medium comprising an inert diluent and unreacted polymerizable monomers including slurries resulting from olefin polymerization. The olefin monomers generally employed in such reactions desirably include 1-olefins having from 2 up to 8 carbon atoms per molecule

Typical examples include ethylene, propylene, butene, pentene, hexene and octene Other examples include vinyl aromatic monomers, like styrene and alkyl-substituted styrene, geminally distributed monomers such as isobutylene and cyclic olefins, such as norbornene and vinyl norbornene. Typical diluents employed in such olefin polymerizations include saturated aliphatic hydrocarbons having 3 to 8, preferably 3 to 4 carbon atoms per molecule, such as propane, isobutane, propylene, n-butane, n-pentane, isopentane, n-hexane, isooctane, and the like Of these diluents those of 3 to 4 carbon atoms per molecule are preferred, and isobutane is most preferred.

The rate of discharge of the polymerization effluent is such as to allow a continuous process stream from the slurry loop reactor from the point of discharge of the liquified polymerization effluent through a single point discharge valve and also through the first flash tank and the associated vapor recovery and solids recovery systems. The rate of discharge of the polymerization effluent is such as to maintain a constant pressure in the slurry reactor and to eliminate intermittent high pressure pulses associated with a discharge of a portion of the reactor contents that occurs with settling legs on slurry reactors.

The temperature to which the polymerization effluent which is discharged from the reactor is heated during transit to the first flash tank for vaporization is below the fusion temperature of the polymer. This may be accomplished by appropriate heating of this first transfer conduit The quantity of heat to be supplied to the polymerization effluent during its transit through this first conduit to the first flash tank should preferably be at least equal to that quantity of heat which equals the heat of vaporization of that quantity of inert diluent which is to be flash vaporized in the first flash tank. This then will provide for the concentrated polymer solids formed in the first flash tank to be passed to the second flash tank to pass thereto at a higher solids temperature and thus facilitates the removal of residual diluent in the pores of such polymer solids by the operation of the second flash tank. That quantity of heat transferred to the polymerization effluent during its transit through the first transfer conduit to the first flash tank may even be greater, provided only that the quantity of heat so transferred will not cause the

polymer solids therein to become heated to such a temperature at which they will tend to fuse or agglomerate one with another

The concentrated polymer solids/slurry are discharged from the first flash tank into a first flash tank exit seal chamber of such a length (l) and diameter (d) so as to provide a volume sufficient to maintain a volume of concentrated polymer solids/slurry sufficient to maintain a pressure seal in the exit seal chamber. The concentrated polymer solids/slurry are discharged from the exit seal chamber through an exit seal chamber reducer to a second transfer conduit which communicates the concentrated polymer solids/slurry as a plug flow to a second flash tank. The exit seal chamber reducer is defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the concentrated polymer solids/slurry.

The pressure for the first flash step will vary depending on the nature of the diluent and unreacted monomers and the temperature of the polymerization effluent. Typically, pressures in the range of from about 140 psia ($9.7 \times 10^5 Nm^{-2}$) to about 315 psia ($21.8 \times 10^5 Nm^{-2}$) can be employed; more preferably from about 200 psia ($13.8 \times 10^5 Nm^{-2}$) to about 270 psia ($18.7 \times 10^5 Nm^{-2}$); and most preferably from about 225 psia ($15.6 \times 10^5 Nm^{-2}$) to about 250 psia ($17.3 \times 10^5 Nm^{-2}$).

The heat exchanging fluid used to condense the vapor from the first flash step is at a temperature in the range of from about 65° F (18°C) to about 150° F (66°C). A preferred embodiment uses a heat exchange fluid at a temperature of from about 75° F (24°C) to about 140° F (60°C). A most preferred embodiment uses a heat exchange fluid at a temperature of from about 85° F (29°C) to about 130° F (54°C).

A further understanding of the present invention will be provided by referring to Fig. 1 which illustrates a system comprising an embodiment of the invention.

In the embodiment illustrated in Fig. 1, the polymerization is carried out in a loop reactor 1. It will be understood that while the loop reactor 1 is illustrated with four vertical legs, the loop reactor 1 may be equipped with more legs, desirably eight or more legs, desirable between 8 and 20, more desirable between 8 and 16, most desirable with 12 legs. The polymerization

slurry is directionally circulated throughout the loop reactor 1 as illustrated by arrows A-D by one or more pumps, such as axial flow pumps, 2A and 2B. Desirably, the loop reactor 1 is equipped with multiple pumps wherein each

pump is dedicated to an even number of legs, such as for example, four legs, six legs, eight legs, etc Diluent comonomer and monomer are introduced into the loop reactor 1 from the diluent storage vessel 40, the comonomer storage vessel 41, and the monomer source 42 through their respective treater beds 37, 38, and 39 through conduits 5, 4 and 3, respectively, connected to conduit 6 Catalyst is added to the loop reactor 1 through one or more catalyst feed systems 7A and 7B.. Normally, catalyst is introduced in a hydrocarbon diluent.

Polymerization slurry may be removed from the loop reactor by continuous discharge through a discharge conduit 8A It will be understood that the loop reactor 1 may be equipped with one or more discharge conduits 8A. It will be also understood that the discharge conduit(s) 8A may be operated in a continuous or discontinuous mode, but desirably a continuous mode. The discharge conduit 8A extends for a distance through a portion of the wall of the loop reactor 1 and into the circulating polymerization slurry. By extending for a distance into the polymerization slurry, the discharge conduit 8A may remove polymerization effluent from the circulating polymerization slurry over an area defined from near or adjacent the inside wall of the loop reactor 1 to a distance extending into the circulating polymerization slurry. In this way, a higher weight percentage of polymer solids may be formed within the conduit 8A and ultimately removed from the loop reactor 1 than the weight percentage of polymer solids within the otherwise circulating polymerization slurry. A pressure control system (not shown in Fig. 1) operates in concert with the discharge conduit 8A. The discharge conduit 8A and the pressure control system 410 are more clearly illustrated in Fig.s 3 and 4 and will be discussed in greater detail below.

The polymerization effluent passes from the discharge conduit 8A to the discharge valve 8B to a conduit 9 which is provided with a line heater 10 and into the first flash tank 11 which separates vaporized liquid medium from polymer slurry/solids. Conduit 9 has an indirect heat exchange means such as a flash line heater 10

Vaporized liquid medium comprising diluent and unreacted monomers exit the first flash tank 11 via transfer conduit 12 through which it is passed

into a separator, such as a cyclone, illustrated by reference number 13 which separates entrained polymer solids from the vapor Polymer solids separated by the cyclone 13 are passed via conduit 14 through a dual valving assembly 14A designed to maintain a pressure seal below cyclone 13 to a lower pressure second flash tank 15

The dual valving assemble 14A includes valves 14B and 14C. The valving assemble 14A in conjunction with conduit 14 operate to periodically discharge polymer solids which have collected in the conduit 14 from the cyclone 13. The valving assembly 14A also maintains the pressure differential between the higher pressure environment in the cyclone 13 and the lower pressure environment in the second flash tank 15 In the operation of the valving assembly 14A, valves 14B and 14C are sequentially opened and closed. At the beginning of this sequence, the valve 14B is open and the valve 14C is closed allowing the polymer solids from the cyclone 13 to collect in the conduit 14. Upon the passage of time and/or the collection of sufficient polymer solids in the conduit 14, the valve 14B closes capturing a portion of the high pressure environment from the cyclone 13 in the conduit 14. After the valve 14B closes, the valve 14C opens and the polymer solids collected in the conduit 14 are forcibly discharged into the flash tank 15 by the differential pressure between the higher pressure environment in conduit 14 and the lower pressure environment in the flash tank 15. After discharging the polymer solids from conduit 14 into the flash tank 15, the valve 14C closes. Once the valve 14C closes, the valve 14B is opened at which time polymer solids will again collect in conduit 14 from the cyclone 13. The above sequence is then repeated.

Referring back to the first flash tank 11, the concentrated polymer solids/slurry in the bottom of the first flash tank 11 continuously settles by sliding along the straight line bottom surface 16 thereof into the seal chamber 17 which is illustrated in enlargement Figure 2. A polymer solids/slurry level 43 is maintained in the seal chamber 17 to eliminate plugging tendencies in first flash tank 11 and to form a pressure seal so that the first flash tank 11 can operate at a substantially higher pressure than the second flash tank 15. Polymer slurry/solids are continuously discharged from the seal chamber 17

into the lower pressure second flash tank 15 The length (l), diameter (d), and volume of the seal chamber 17 and the geometry of the seal chamber exit reducer 18 are chosen so as to provide a variable residence time and provide a continuous plug flow of concentrated polymer solids/slurry to minimize "dead" space and reduce plugging tendencies. The seal chamber 17 length must be sufficient to allow particle (polymer solids) level measurement and control.

Particle level measurement and control may be accomplished by a nuclear level indicating system 18D. The nuclear level indicating system 18D includes a nuclear radiating source (not shown) and receiver or level element 18A in signal communication with a level indicating controller 18B. In operation, the level element 18A generates a signal proportional to the particulate level in the seal chamber 17. This signal is conveyed to the level indicating controller 18B. In response to this signal and a preset value, the level indicating controller 18B sends a signal through a conduit (illustrated by broken line 18C) to a control valve 18 E which selectively controls the discharge of polymer solids into a conduit 19.

Typical residence times of the concentrated polymer solid/slurry in the seal chamber 17 are from 5 seconds to 10 minutes, preferable residence times are from 10 seconds to 2 minutes and most preferable residence times from 15 - 45 seconds. The continuous plug flow of concentrated polymer solids/slurry forms a pressure seal wherein the concentrated polymer solids/slurry have an l/d ratio inside the seal chamber 17 which is typically 1.5 to 8, preferable l/d is 2 to 6 and most preferable is 2.2 to 3. Typically the seal chamber exit reducer 18 sides are inclined, relative to the horizontal, 60 - 85 degrees, preferable 65 - 80 degrees and most preferable 68 - 75 degrees. The seal chamber exit reducer 18 geometry is defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the concentrated polymer slurry/solids and communicates the concentrated polymer solid/slurry to a second transfer conduit 19 which communicates with a feed inlet of flash tank 15. In flash tank 15 substantially all of any remaining inert diluent and unreacted monomer in the concentrated polymerization effluent is vaporized and taken overhead via conduit 20 to a second cyclone 21.

Referring now to the cyclone 13, the major portion of the liquid medium in the polymerization effluent may be been taken to cyclone 13 as vapor. The vapor after having a portion of the entrained catalyst and polymer solids removed is passed via conduit 22 through a heat exchanger system 23A wherein the vapor at a pressure from about 140 psia ($9.7 \times 10^5 Nm^{-2}$) to about 315 psia ($21.8 \times 10^5 Nm^{-2}$) is condensed by indirect heat exchange with a heat exchange fluid such as to eliminate the need for compression. The portion of the entrained catalyst and polymer solids not removed by the cyclone 13 are generally smaller in size and may be referred to as "fines", "polymer fines" and/or "catalyst fines". These fines generally include unreacted and/or under-reacted catalyst.

The heat exchanger system 23A includes a heat exchanger 23E and a tempered water circulating pump 23B connected to the heat exchanger 23E by conduit 23C. A tempered water temperature control valve 23D is connected to the heat exchanger 23E and water circulating pump 23B by conduits 23F and 23G, respectively. Cooling water from a cooling water source (not shown) is conveyed via a cooling water conduit 23H into the conduit 23G between the control valve 23D and the circulating pump 23B. A temperature indicating controller (TIC) 23J is connected between the control valve 23D and the conduit 23C. Between the controller 23J and the conduit 23C resides a temperature element 23K.

The heat exchanger system 23A operates to control the amount of vapor condensed in the heat exchanger 23E. This is accomplished by controlling the flow of cooling water introduced into the conduit 23G from the conduit 23H by exhausting heated water formed in the heat exchanger 23E. The heated water from the heat exchanger 23E is conveyed to the control valve 23D via the conduit 23F. The heated water exits the control valve 23D via the conduit 23I.

More specifically, cooling water from the conduit 23H entering the conduit 23G mixes with circulating tempered water in the conduit 23G, the mixture thereof enters the pump 23B. The water exiting the pump 23B enters the conduit 23C, a portion of which contacts the temperature element 23K, in

route to the heat exchanger 23E. The temperature element 23K generates an signal proportional to the temperature in conduit 23C. The signal is conveyed

to the temperature indicating controller 23J In response to this signal and a preset temperature value, the temperature indicating controller 23J sends a signal through a signal conduit (illustrated by the broken line 23L) to the control valve 23D which selectively controls the volume of heated water exiting the heat exchanger system 24A through the conduit 23I.

The condensed liquid medium formed at the heat exchanger 23E includes diluent, unreacted/under-reacted catalyst, polymer solids and unreacted monomers This condensed liquid medium is then passed to an accumulator 24B via a conduit 22A.

It is desirable to control the amount of vapor condensed in the heat exchanger 23E and to maintain sufficient vapor pressure in the accumulator 24B In this way, a pressure control valve 24A can maintain sufficient back pressure on the accumulator 24B. By maintaining a sufficient back pressure on the accumulator 24B, a proper operating pressure is maintained in the first flash tank 11. The pressure control valve 24A is actuated by a pressure indicating controller 24C in concert with a pressure element 24D. The pressure element 24D is in sensing communication with the accumulator 24B. The pressure element 24D generates an signal proportional to the pressure in the accumulator 24B. In response to this signal and a preset pressure value, the pressure indicating controller 24C sends a signal through a signal conduit (illustrated by the broken line 24E) to the control valve 24A which selectively controls the back pressure on the accumulator 24B

A pump 25 is provided for conveying the condensed liquid medium from the accumulator 24B back to the polymerization zone by a conduit 26. In this way, the unreacted/under-reacted catalyst and polymer solids not removed by the cyclone 13 are returned for further polymerization to the loop reactor 1.

The polymer solids in the lower pressure second flash tank 15 are passed via a conduit 27 to a conventional dryer 28. The vapor exiting the secondary cyclone 21, after filtration in a filter unit 29, is passed by a conduit 30 to a compressor 31 and the compressed vapors are passed through a conduit 32 to a condenser 33 where vapor is condensed and the condensate is passed through conduit 34 to storage vessel 35. The condensed liquid medium in the storage vessel 35 is typically vented overhead for removal of light-end

contaminants The inert diluent can be returned to the process through a treater bed 37 to remove catalyst poisons or distilled in unit 36 for more complete removal of light-ends and then returned to the process through a treater bed.

Turning now to Fig. 3, a portion of a wall 310 of the loop reactor 1 through which the discharge conduit 8A extends is illustrated The discharge conduit 8A may extend into the reactor at various angles Desirably, the discharge conduit 8A extends into the loop reactor at substantially a right angle relative to the wall 310.

The wall 310 includes an inside surface 312 and an outside surface 314. The inside surface 312 supports the circulating polymerization slurry illustrated by directional arrows 318. The discharge conduit 8A has a top 316A, and a continuous side 316B. Portions of the side 316B define an opening 320. The opening 320 has a vertical opening dimensions v1 and v2 defined by walls 320A and 320B of the side 316B. Desirably, the v1 dimension is greater than the v2 dimension. The opening 320 has horizontal opening dimensions h1 and h2 (not shown). The opening 320 may be formed in any suitable shape, such as rectangular, oval, or a combination thereof. In one embodiment, the opening 320 may be conical-shaped or scooped shaped.

The opening 320 communicates with a channel 322 defined by the inside surfaces of the top 316A and the side 316B. The channel 322 conveys captured polymerization slurry, illustrated by directional arrow 324 to the discharge valve 8B (not shown).

The opening 320 is sized and positioned relative to the direction of movement of the circulating polymerization slurry 318 Desirably, the opening 320 is in a substantially facing position to the direction of the circulating polymerization slurry 318. More desirably, the opening 320 faces the direction of the circulating slurry 318. In this way, a portion of the polymerization slurry 324 containing polymer solids is removed from the circulating polymerization slurry 318 over an area from near or adjacent the inside wall 312 of the loop reactor 1 to a distance extending into the circulating polymerization slurry 318. In this way, a higher weight percentage of polymer solids may be formed within the conduit 8A than the weight

percentage of polymer solids within the otherwise circulating polymerization slurry.

This weight percentage increase of polymer solids may depend upon the location of the discharge conduit 8A along the loop reactor 1, the insertion depth of the discharge conduit 8A within the loop reactor, the size and configuration of the opening 320, the orientation of the opening 320 relative to the direction of the circulating polymerization slurry, and the weight percentage of polymer solids in the circulating polymerization slurry 318. For example, between 1 to 5 weight percentage calculated increase is observed with a discharge conduit 8A having an $v1$ dimension of approximately 5 inches and a $h1$ dimension of approximately 1 inch (2.5cm). The discharge conduit 8A was positioned 10 ft (3.0m) downstream of a 90 degree bend in the loop reactor 1 in a portion of the loop reactor wall 314 adjacent the ground. The discharge conduit 8A extended approximately 5.5 inches (14.0cm) into the circulating polymerization slurry stream. The velocity of the circulating polymerization slurry was in the range of 28 to 34 ft/sec (8.5 to 10.3 ms^{-1}) with weight percent of polymer solids in the range of 48 to 53.

Turning now to Fig. 4, the pressure control system 410 is illustrated. The pressure control system 410 operates to maintain substantially uniform pressure within the loop reactor 1 by controlling the discharge of polymerization effluent from the loop reactor 1 via the discharge conduit 8A. The control system 410 also operates to prevent plugging of the discharge conduit 8A by polymer solids during pressure fluctuations within the loop reactor 1 and/or when the flow of polymerization effluent from the discharge conduit 8A to conduit 9 is interrupted and/or stopped.

The pressure control system 410 includes a first inert diluent source 412, such as isobutane, and an inert diluent conduit 414 in communication with a loop reactor conduit 416. The flow of inert diluent through the inert diluent conduit 414 to the loop reactor conduit 416 is controlled by the control valve 418 in concert with a flow element 420 and a flow indicator controller 422. The purpose of metering the flow of inert diluent from the first inert diluent source 412 to the loop reactor 1 is to prevent plugging of the conduit 416 by polymer solids. In this way, a loop reactor pressure element 441

(discussed below), in communication with the loop reactor conduit 416, may more accurately monitor the pressure in the loop reactor 1.

The pressure control system 410 further includes as second inert diluent source 424 and a third inert diluent source 426. Inert diluent, such as isobutane, from the second inert diluent source 424 flows into a conduit 428 towards a control valve 430 which is in fluid communication with a conduit 432. The control valve 430, in concert with a flow element 431 and a flow indicator controller 433, meters the flow of inert diluent from the second inert diluent source 424 into conduit 432. The conduit 432 is in fluid communication with a conduit 434 and the discharge conduit 8A, terminating in the discharge conduit 8A at a point between the loop reactor 1 and the discharge valve 8B. The purpose of metering the flow of inert diluent from the second inert diluent source 422 into the conduit 432 is to prevent plugging of the conduit 432 by polymer solids which might otherwise back flow into the conduit 432 from the discharge conduit 8A. Additionally, the flow of inert diluent from the second inert diluent source 422 also prevents plugging of the conduit 434 and the control valve 440 by polymer solids which might back flow into conduit 432 from the discharge conduit 8A.

Inert diluent from the third inert diluent source 426 flows into a conduit 438 towards a control valve 440 which is in fluid communication with conduit 434. As will be explained in greater detail below, in the event of a sufficient pressure fluctuation within the loop reactor 1, the control valve 440 operates to initiate a sufficient flow under sufficient pressure of inert diluent from the third inert diluent source 426 to purge and/or discharge polymer solids from the discharge conduit 8A into the loop reactor 1. In this instance, generally the flow of inert diluent from the third inert diluent source 426 into the conduit 432 will be greater than the flow of inert diluent from the second inert diluent source 424 into the conduit 432. For example, the flow of inert diluent from the second inert diluent source 424 to the discharge conduit 8A may be in a range of 0.5 to less than 2.0 gallons/min (0.03 to less than 1.3dm^3s^{-1}). The flow of inert diluent from the third inert diluent source 426 to the discharge conduit 8A may be in a range of 2.0 to 20 gallons/min (1.3 to 13dm^3s^{-1}).

The loop reactor pressure element 441 and a pressure indicating controller 442 perform several functions. As previously mentioned, the pressure element 441 monitors the loop reactor 1 pressure via the conduit 416. In response to this pressure, the loop reactor pressure element 441 generates an signal proportional to the pressure in conduit 416. This signal is conveyed to the pressure indicating controller 442. In response to this signal and a preset pressure value, the pressure indicating controller 442 sends a signal through a signal conduit (illustrated by the broken line 444) to the discharge valve 8B and the control valve 440.

During normal loop reactor operations, the discharge valve 8B is positioned to permit the flow of polymerization effluent from the discharge conduit 8A to conduit 9. At the same time, the control valve 440 is closed preventing the flow of inert diluent from the third inert diluent source 426 to the discharge conduit When sufficient pressure fluctuations occur and/or when partial depressurization in the loop reactor 1 are detected by the loop reactor pressure element 441, the signal generated by the pressure indicating controller 442 causes the discharge valve 8B to close and the control valve 440 to open By closing discharge valve 8B, thus interrupting the discharge from the loop reactor 1, pressure within the loop reactor 1 may be restored By opening the control valve 440 and flowing sufficient volumes of inert diluent from the third inert diluent source 426 into the discharge conduit 8A under sufficient pressure, polymer solids remaining in the discharge conduit 8A between the discharge valve 8B and the loop reactor 1 may be flushed out of and/or purged from the discharge conduit 8A and into the loop reactor 1. Additionally, by maintaining a sufficient flow of inert diluent, continuous or otherwise, into and/or through the discharge conduit 8A while the discharge valve 8B is closed, the polymer solids within the loop reactor 1 are prevented from entering and/or substantially collecting in the discharge conduit 8A and/or plugging the discharge conduit 8A. Upon return of normal operations, the control valve 440 closes terminating the flow of inert diluent from the third inert diluent source 426 and the discharge valve 8B opens to resume the flow of polymerization effluent through the discharge conduit 8A into the conduit 9

Having broadly described the present invention it is believed that the same will become even more apparent by reference to the following examples. It will be appreciated that the examples are presented solely for the purpose of illustration and should not be construed as limiting the invention.

EXAMPLES

Example 1

A typical ethylene polymerization process can be conducted at a temperature of about 215° F (102°C) and a pressure of 565 psia (39.1 x 10^5 Nm^{-2}). An example of such a process would result in a polymerization effluent of about 83,000 pounds per hour (10.5kg s^{-1}) comprising about 45,000 pounds per hour (5.68kg s^{-1}) of polyethylene polymer solids and about 38,000 pounds per hour (4.79kg s^{-1}) of isobutane and unreacted monomers. The continuously discharged polymerization effluent is flashed in the first flash tank at a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) and a temperature of about 180° F (82°C) to remove overhead about 35,000 pounds per hour (4.4kg s^{-1}) of diluent and unreacted monomer vapors and entrained particulates. Auxiliary heat to impart an additional quantity of heat to the polymerization effluent is supplied by appropriate heating means during the transit between the discharge valve and the first flash tank. After removal of the fines, the isobutane vapor is condensed, without compression, by heat exchange at a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) and a temperature of about 135° F (57°C). The polymer slurry/solids discharging from the bottom of the first flash tank into the seal chamber form a continuous plug flow of concentrated polymer slurry/solids, which provides a pressure seal, with an l/d ratio of the plug of polymer slurry/solids of 2.5 in an 8'4" (2.53m) long seal chamber having an l/d ratio of 5.5 and with a cone angle of about 68° on the seal chamber exit reducer. The residence time of the continuous plug flow of concentrated polymer slurry/solids is about 16 seconds. The concentrated polymer slurry/solids are continuously discharged from the bottom of the first flash tank at a temperature of about 180° F (82°C) and a pressure of about 240 psia(16.6 x $10^5 Nm^{-2}$) through a seal chamber, seal chamber exit reducer, and a second transfer conduit into a feed inlet on a second flash tank. The remaining

liquid medium in the concentrated polymer slurry/solids communicated to the second flash tank is flashed at a temperature of about 175° F (79°C) and at a pressure of about 25 psia (1.7 x $10^5 Nm^{-2}$) to remove about 4,300 pounds per hour (0.54kg s^{-1}) of isobutane and unreacted monomers which are condensed by compression and heat exchange.

Example 2

A typical ethylene polymerization process can additionally be conducted at a temperature of about 215° F (102°C) and a pressure of 565 psia (39.1 x $10^5 Nm^{-2}$). An example of such a process would result in a polymerization effluent of about 83,000 pounds per hour (10.5kg s^{-1}) comprising about 45,000 pounds per hour (5.68kg s^{-1}) of polyethylene polymer solids and about 38,000 pounds per hour (4.79kg s^{-1}) of isobutane and unreacted monomers. The continuously discharged polymerization effluent is flashed in the first flash tank at a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) and a temperature of about 175° F (79°C) to remove overhead about 23,000 pounds per hour (2.9kg s^{-1}) of diluent and unreacted monomer vapors and entrained particulates. After removal of the fines, the isobutane vapor is condensed, without compression, by heat exchange at a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) and a temperature of about 112° F (44°C). The polymer slurry/solids discharging from the bottom of the first flash tank into the seal chamber form a continuous plug flow of concentrated polymer slurry/solids, which provides a pressure seal, with an l/d ratio of the plug of polymer slurry/solids of 2.5 in an 8'4" (2.53m) long seal chamber with an l/d ratio of 5.5 and with a cone angle of about 68° on the seal chamber exit reducer. The residence time of the continuous plug flow of concentrated polymer slurry/solids in the seal chamber is about 16 seconds. About 60,000 pounds per hour (7.56kg s^{-1}) of concentrated polymer slurry/solids are continuously discharged from the bottom of the first flash tank at a temperature of about 175° F (79°C) and a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) through a seal chamber, seal chamber exit reducer and a second transfer conduit into a feed inlet on a second flash tank. The remaining liquid medium in the concentrated polymer slurry/solids communicated to the second flash tank is flashed at a temperature of about 125° F (52°C) and at a pressure of about 25 psia (1.7 x

$10^5 Nm^{-2}$) to remove about 16,000 pounds per hour (2.0kg s^{-1}) of isobutane and unreacted monomer which are condensed by compression and heat exchange

Example 3

An example of a typical ethylene polymerization process was carried out in an eight leg, 20 inch (0.50m) reactor with settling legs having an overall length of 833 ft (254m) and a volume of 11,500 gallons (43585dm^3). The reactor was equipped with a single flash tank (requiring 100% compression of all diluent discharged from the reactor), a single 460-480 kilowatt circulating pump having a pump head in the range from 85 ft to 110 ft (25.9 to 33.5m), producing a circulation rate in the range from 21,000 to 28,000 gallons per minute (gpm) (1326 to 1768$dm^3 s^{-1}$) and operated in a discontinuous discharge mode. The polymerization temperature and pressure in the reactor would be between about 215° F (101.7°C) to 218° F (103.3°C) and a pressure of 565 psia (39.1 x $10^5 Nm^{-2}$).

In the process of example 3, the reactor slurry density is in the range from 0.555 gm/cc to 0.565 gm/cc, a polymer production rate range from 28,000 pounds to 31,000 pounds per hour (3.5 to 3.9kg s^{-1}) while maintaining a reactor solids concentration weight percentage in the range from 46 to 48 with a polymer residence time in the range from 0.83 to 0.92 hours. Space time yield (STY) was in the range from 2.4 to 2.7. Example 3 data and results are further illustrated in Table 1.

Example 4

Another example of a typical ethylene polymerization process illustrating high polymer solids loading was carried out in an eight leg, 20 inch reactor having an overall length of 833 ft (254m) and a volume of 11,500 gallons (43585dm^3). The reactor in example 4 was equipped dual flash tanks, single discharge conduit, two circulating pumps in series consuming a total of between 890 and 920 kilowatts producing a total pumping head in the range from 190 ft to 240 ft (57.9 to 73.1m), producing a circulation rate in the range from 23,000 to 30,000 gpm (1453 to 1895kg s^{-1}) and operated in a continuous discharge mode. The polymerization temperature and pressure in the reactor would be between about 217° F (102.8°C) to 218° F (103.3°C) and a pressure of 565 psia (39.1 x $10^5 Nm^{-2}$).

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In the process of example 4 a polymerization effluent was produced having a reactor slurry density in the range from 0.588 to 0.592 gm/cc, a polymer production rate in the range from 38,000 to 42,000 pounds per hour (4.8 to 5.3kg s^{-1}) while maintaining a reactor solids concentration weight percentage in the range of 54 to 57 with a polymer residence time in the range of 0.68 to 0.79 hours. Space time yield (STY) was in the range of 3.3 to 3.7. Example 4 data and results are further illustrated in Table 1.

The continuously discharged polymerization effluent is flashed in the first flash tank at a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) and a temperature of about 175° F (79°) to remove overhead about 16,000 pounds per hour (2.0kg s^{-1}) of diluent and unreacted monomer vapors and entrained particulates. After removal of the fines, the isobutane vapor is condensed, without compression, by heat exchange at a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) and a temperature of about 112° F (44°C). The polymer slurry/solids discharging from the bottom of the first flash tank into the seal chamber form a continuous plug flow of concentrated polymer slurry/solids, which provides a pressure seal, with an l/d ratio of the plug of polymer slurry/solids of 2.5 in an 8'4" (2.53m) long seal chamber with an l/d ratio of 5.5 and with a cone angle of about 68° on the seal chamber exit reducer. The residence time of the continuous plug flow of concentrated polymer slurry/solids in the seal chamber is about 16 seconds. Concentrated polymer slurry/solids are continuously discharged from the bottom of the first flash tank at a temperature of about 175° F (79°C) and a pressure of about 240 psia (16.6 x $10^5 Nm^{-2}$) through a seal chamber, seal chamber exit reducer and a second transfer conduit into a feed inlet on a second flash tank. The remaining liquid medium in the concentrated polymer slurry/solids communicated to the second flash tank is flashed at a temperature of about 125° F (52°C) and at a pressure of about 25 psia (1.7 x $10^5 Nm^{-2}$) to remove about 16,000 pounds per hour (2.0kg s^{-1}) of isobutane and unreacted monomer which are condensed by compression and heat exchange.

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TABLE I

ETHYLENE POLYMERIZATION DATA

	EXAMPLE 3	EXAMPLE 4
Nominal pump(s) size, inches	20 (0.51m)	20 (0.51m)
Reactor solids concentration, wt.%	46 - 48	54 - 57
Polymer production rate, K lbs./hr.	28 – 31 (3.5 – 3.9kg s^{-1})	38 – 42 (4.8 – 5.3kg s^{-1})
Reactor circulation pump power, KW	460 - 480	890 - 920
Circulation pump head, ft.	85 – 110 (25.9 – 33.5m)	190 – 240 (57.9 – 73.1m)
Circulation rate, GPM	21,000 - 28,000 (1326 – 1768dm^3s^{-1})	23,000 - 30,000 (1453 – 1895dm^3s^{-1})
Reactor slurry density, gm/cc	0.555 - 0.565	0.588 - 0.592
Reactor temperature, degrees F	215 – 218 (101.7 – 103.3°C)	217 – 218 (102.8 – 103.3°C)
Ethylene concentration, wt. %	4.0 - 4.4	5.0 - 6.0
Hexene concentration, wt. %	0.13 - 0.19	0.13 - 0.19
Heat transfer coefficient, btu/hr-f-ft	215 - 225	230 - 245
Reactor volume, gallons	11,500 (43585dm^3)	11,500 (43585dm^3)
Reactor length, ft.	833 (254m)	833 (254m)
Circulating pump head per reactor length, ft/ft	0.100 - 0.132	0.228 - 0.288
Catalyst productivity, lb/lb	2,700 - 3,000	2,700 - 3,000
Polymer residence time, hrs.	0.83 - 0.92	0.68 - 0.79
Space time yield, lbs/hr - gal	2.4 - 2.7 (8.0 – 9.0 x 10^{-5}kg s^{-1} dm^{-3})	3.3 - 3.7 (11.0 – 12.3 x 10^{-5} kg $s^{-1}dm^{-3}$)
Isobutane compressed and recycled, %	100	45 - 60

Discussion

In view of the above description and examples, several observations relative to the apparatus and process can be made.

It has been observed that by increasing the head and flow capability of the loop reactor circulating pump(s), higher weight percent solids can be circulated in the reactor. It has also been observed that attaining the necessary head and flow from one pump is increasingly difficult as percent solids are increased above 45 weight percent and/or reactor length is increased

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Therefore, the use of two pumps in series allows a doubling of pumping head capability and a resulting percent solids increase. Increased weight percent

solids in the loop reactor increases catalyst residence time, which for chrome oxide and Ziegler-Natta catalysts, increases catalyst productivity One can choose to take advantage of higher percent solids and longer residence time by keeping production rate constant at reduced catalyst feed rate and improve the catalyst yield. Another alternative is to maintain catalyst feed rate constant and increase the reactor throughput and therefor increase STY at nearly constant catalyst productivity. Higher solids also increases the weight percent solids removed from the reactor which reduces isobutane processing cost in recycle equipment. Desirably, the higher solids are removed continuously Continuous discharge may occur through a single point discharge line.

In a loop reactor, it is not always possible to locate the continuous discharge line in an optimal location to take advantage of centrifugal force to increase the weight percent solids and therefore reduce the amount of isobutane entrained with the polymer solids. It has been observed that a specifically designed pipe as illustrated in Figure 3 inserted into the loop reactor can increase weight percent solids removed from the reactor. This pipe insert will function in any section of the loop reactor and in a straight section will increase the weight percent solids to that equal to that in a location which takes advantage of centrifugal force to concentrate solids.

With the development of high weight percent solids circulation capability in the loop reactor and two-stage flash, the need to concentrate solids in the reactor discharge is reduced compared to the conventional loop reactor operations having low solids circulation, single-stage flash, continuous discharge line, and continuous discharge or otherwise. Therefore, the conventional loop reactor settling legs, which are designed to maximize polymer solids concentration prior to discharge, can be replaced with a continuous discharge line, which simplifies the system mechanically, reduces capital cost, improves safety, reduces maintenance and improves reactor control. Settling legs require routine maintenance due to their plugging tendency and can form material which plugs downstream polymer handling equipment. Maximum loop reactor ethylene concentration is limited by settling legs due to the tendency for polymer to grow in the legs at elevated ethylene concentrations between discharges and therefore plug the leg

Continuous discharge eliminates this tendency Another advantage of continuous discharge is better response to a sudden drop in reactor pressure, which can happen if ethylene flow is quickly reduced Under this condition, settling legs will stop discharging and can plug with polymer within minutes

A development which would increases efficiency of the two-stage flash system is the continuous flash line heater. The heater would vaporize up to 100% of the diluent discharged from the reactor with the polymer which would allow greater recovery of the diluent by the intermediate pressure condenser Diluent recovery through the first flash tank would reduce utility and capital cost. Conventional low pressure single-stage diluent recovery systems include compression, distillation and treatment which have high capital and operating cost The flash line heater would increase the temperature of the polymer in the downstream dryer system and would create the potential for lower volatile levels in the final product, which would lower variable cost, improves safety and aids attainment of environmental standards

The first flash tank provides an intermediate pressure flash step which allows for simple condensation of diluent and return to the reactor. The flash line heater would be capable of supplying sufficient heat to vaporize up to 100% of the diluent in the first flash tank.

Diluent vapor and unreacted/under reacted catalyst/polymer fines go overhead from the flash tank to the cyclone The bulk of the polymer goes out the bottom of the first flash tank through the seal chamber to the second flash tank.

Connected to the bottom of the first flash tank is the seal chamber which provides for a low residence time plug flow area to control polymer level and maintain pressure in the first flash tank. The seal chamber is designed to accommodate a range of polymer forms from concentrated slurry to dry polymer.

The overhead stream from the first flash tank is received by the cyclone, which removes most of the polymer fines and returns them to the bulk of the polymer flow in the second flash tank through a two valve system which allows the fines to accumulate between the valves, then discharge through the bottom valve while maintaining pressure in the first flash system.

The overhead stream from the cyclone contains some unreacted/under reacted catalyst and polymer fines These particles are carried with the diluent vapor to the condenser, entrained with the liquid diluent after condensation, collected in the accumulator and returned to the reactor in the diluent. The condensation and accumulator systems are designed and operated to accommodate fines

The condenser provides for low variable and capital cost liquefaction of the diluent removed from the rector with the polymer via the first flash tank. Conventional single flash tank systems flash the polymerization effluent to the just above ambient pressure, which requires compression to liquefy the diluent prior to recycle to the loop reactor. An intermediate pressure flash provides for condensation with a commonly available cooling medium, such as Plant cooling water. The condenser system is flushed with diluent and designed to accommodate a level of fines without accumulation or plugging. The condenser is cooled by a tempered water system which controls the condensation temperature to achieve the proper vapor pressure in the accumulator to allow efficient pressure control by the pressure control valve on the accumulator vent. The condenser tempered water system is a pump-around loop of cooling water, the temperature of which is controlled by metering in fresh cooling water as needed

The accumulator receives the condensed diluent and catalyst/polymer fines and pumps the mixture back to the loop reactor based on level control in the accumulator. The accumulator has a bottom shape designed to accommodate fines. A vent on the accumulator purges the accumulated diluent of light-ends/non-condensables and controls pressure on the first flash system

The second flash tank, operating just above ambient pressure, receives polymer from the first flash tank seal chamber. Complete vaporization, if not already accomplished in the first flash tank, will occur in the second flash tank. Polymer leaves the bottom of the second flash tank to the dryer system. The flash-line heater would increase the temperature of the polymer which allows the dryer system to remove residual volatiles more efficiently and effectively The overhead of the second flash tank will be diluent vapor not

recovered in the first flash system and will be filtered and compressed for return to the loop reactor

While the present invention has been described and illustrated by reference to particular embodiments, it will be appreciated by those of ordinary skill in the art that the invention lends itself to many different variations not illustrated herein. For these reasons, then, reference should be made solely to the appended claims for purposes of determining the true scope of the present invention.

Although the appendant claims have single appendencies in accordance with U.S patent practice, each of the features in any of the appendant claims can be combined with each of the features of other appendant claims or the main claim.

CLAIMS

1. An apparatus for capturing a higher weight percentage of polymer solids from a circulating slurry in a loop reactor than the weight percentage of solids in the circulating slurry, the circulating slurry being circulated in a direction in the loop reactor comprising:

a conduit having a first end, wherein said first end extends for a distance into the loop reactor said conduit having portions defining an opening wherein said opening is positioned relative to the direction of the circulating slurry.

2. The apparatus of claim 1 wherein the opening is facing the direction of the circulating slurry.

3. The apparatus of claim 1 wherein a portion of the conduit extends outwardly from the loop reactor.

4. The apparatus of claim 3 wherein the polymer solids are discharged from the loop reactor through the portion of the conduit extending outwardly from the loop reactor.

5. The apparatus of claim 4 wherein the polymer solids are continuously discharged from the loop reactor through the portion of the conduit extending outwardly from the loop reactor.

6. A process for capturing a higher weight percentage of polymer solids from a circulating slurry in a loop reactor than the weight percentage of polymer solids in the circulating slurry, the circulating slurry being circulated in a direction in the loop reactor comprising the step of:

extending for a distance into the loop reactor a conduit having portions defining an opening wherein said opening extends into the circulating slurry.

7. The process of claim 6 including the step of positioning the opening in a substantially facing direction to the direction of flow of the circulating slurry.

8. The process of claim 6 including the step of extending a portion of conduit outwardly from the loop reactor.

9. The process of claim 8 including the step of discharging the polymer solids from the loop reactor through the portion of the conduit extending outwardly from the loop reactor.

10. The process of claim 9 including the step of continuously discharging the polymer solids from the loop reactor through the portion of the conduit extending outwardly from

the loop reactor.

11. An apparatus for purging polymer solids from a conduit connected to a loop reactor and in fluid communication with the loop reactor comprising:

a sensor;

a first valve in fluid communication with the conduit;

a second valve positioned between a first inert diluent and the conduit, wherein the first inert diluent is in fluid communication with the conduit between the loop reactor and the first valve;

wherein, in response to a signal produced by the sensor, the first valve is closed and the second valve is opened allowing the first inert diluent to enter the conduit in sufficient quantities and under sufficient pressure to purge polymer solids from the conduit.

12. The apparatus of claim 11 the sensor is a pressure sensor.

13. The apparatus of claim 11 wherein the conduit has a first end, wherein said first end extends for a distance into the loop reactor wherein portions adjacent to the first end define an opening.

14. The apparatus of claim 11 further comprising:

a third valve positioned between a second inert diluent and the conduit, wherein the second inert diluent is in fluid communication with the conduit between the loop reactor and the first valve;

wherein, when the first valve is open and the second valve is closed the third valve is open allowing the second inert diluent to enter the conduit.

15. A process for purging polymer solids from a conduit connected to a loop reactor and in fluid communication with the loop reactor comprising the steps of:

closing a first valve in response to a first signal from a first sensor, wherein the first valve is connected to and in fluid communication with the conduit;

opening a second valve in response to a second signal from a second sensor, wherein the second valve is fluid communication between a first inert diluent and the conduit, and wherein the first inert diluent is in fluid communication with the conduit between the loop reactor and the first valve;

flowing sufficient quantities of the first inert diluent under sufficient pressure into the conduit to purge polymer solids from the conduit.

16. The process of claim 15 wherein the first and second sensors are the same.

17. The process of claim 15 wherein the first and second signals are the same.

18. An apparatus for returning fines to a polymerization slurry in a loop reactor comprising,

a discharge valve for discharging a portion of the polymerization slurry from the loop reactor into a first transfer conduit;

the first transfer conduit communicating said polymerization slurry therein into a first flash tank, the first flash tank converting a portion of the polymerization slurry into a first fluid, the first fluid comprising a diluent and the fines;

a second transfer conduit communicating the first fluid therein to a first cyclone, the first cyclone converting a portion of the first fluid into a second fluid comprising the diluent and the fines;

a third transfer conduit communicating the second fluid into a heat exchanger, the heat exchanger converting the second fluid into a liquid comprising the diluent and the fines; and

a fourth transfer conduit communicating the liquid to the polymerization slurry in the loop reactor.

19. The apparatus of claim 18 further comprising a first transfer conduit heater for heat exchange between the first transfer conduit heater and the polymerization slurry.

20. A process for returning fines to a polymerization slurry in a loop reactor comprising the steps of:

discharging a portion of the polymerization slurry from the loop reactor;

communicating the discharge polymerization slurry into a first flash tank;

converting in the flash tank a portion of the polymerization slurry into a first fluid, the first fluid comprising a diluent and the fines;

communicating the first fluid from the first flash tank to a first cyclone;

converting in the cyclone a portion of the first fluid into a second fluid comprising the diluent and the fines;

communicating the second fluid into a heat exchanger;

converting in the heat exchanger the second fluid into a liquid comprising the diluent and the fines; and

returning the liquid to the polymerization slurry in the loop reactor.

21. An apparatus for continuously capturing a higher weight percentage of polymer solids from a circulating slurry in a loop reactor than the weight percentage of solids in the circulating slurry, the circulating slurry being circulated in a direction in the loop reactor comprising:

a conduit having a first end, wherein said first end extends for a distance into the loop reactor said conduit having portions defining an opening wherein said opening is facing the direction of the circulating slurry.

22. A process for continuously capturing a higher weight percentage of polymer solids from a circulating slurry in a loop reactor than the weight percentage of polymer solids in the circulating slurry, the circulating slurry being circulated in a direction in the loop reactor comprising the step of:

extending for a distance into the loop reactor a conduit having portions defining an opening wherein said opening extends into the circulating slurry.

23. An apparatus for returning fines to a polymerization slurry in a loop reactor comprising,

a discharge valve for continuously discharging a portion of the polymerization slurry from the loop reactor into a first transfer conduit;

the first transfer conduit communicating said polymerization slurry therein into a first flash tank, the first flash tank converting a portion of the polymerization slurry into a first fluid, the first fluid comprising a diluent and the fines;

a second transfer conduit communicating the first fluid therein to a first cyclone, the first cyclone converting a portion of the first fluid into a second fluid comprising the diluent and the fines;

a third transfer conduit communicating the second fluid into a heat exchanger, the heat exchanger converting the second fluid into a liquid comprising the diluent and the fines; and

a fourth transfer conduit communicating the liquid to the polymerization slurry in the loop reactor.

24. A process for returning fines to a polymerization slurry in a loop reactor comprising the steps of:

continuously discharging a portion of the polymerization slurry from the loop reactor;

communicating the discharge polymerization slurry into a first flash tank;

converting in the flash tank a portion of the polymerization slurry into a first fluid, the first fluid comprising a diluent and the fines;

communicating the first fluid from the first flash tank to a first cyclone;

converting in the cyclone a portion of the first fluid into a second fluid comprising the diluent and the fines;

communicating the second fluid into a heat exchanger;

converting in the heat exchanger the second fluid into a liquid comprising the diluent and the fines; and

returning the liquid to the polymerization slurry in the loop reactor.

25. An apparatus for purging polymer solids from a conduit connected to a loop

reactor and in fluid communication with the loop reactor comprising:

a sensor;

a first valve in fluid communication with the conduit;

a second valve positioned between a first inert diluent and the conduit, wherein the first inert diluent is in fluid communication with the conduit between the loop reactor and the first valve, wherein, in response to a signal produced by the sensor, the first valve is closed and the second valve is opened allowing the first inert diluent to enter the conduit in sufficient quantities and under sufficient pressure to purge polymer solids from the conduit; and

a third valve positioned between a second inert diluent and the conduit, wherein the second inert diluent is in fluid communication with the conduit between the loop reactor and the first valve, wherein, when the first valve is open and the second valve is closed the third valve is open allowing the second inert diluent to enter the conduit.