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Propylene Polymerization Plant and Propylene Polymerization Process

Field of the Invention

The present invention concerns a polymerization plant suitable for extra large-scale propylene polymerization. The present invention further concerns a process for polymerization of propylene using such plant.

Background

Coupling of loop and gas phase reactor is known for more than two decades under the trademark Borstar® and has found its way into practically any textbook in the field of polyolefins. The basic process layout is for example described in WO9858975A1 dealing with the preparation of propylene homopolymers and copolymers, which comprises polymerizing propylene optionally with comonomers in the presence of a catalyst at elevated temperature and pressure in at least one slurry reactor and at least one gas phase reactor, the polymerization product of at least one slurry reactor, containing unreacted monomers, being directly conducted to a first gas phase reactor essentially without recycling of the unreacted monomers to the slurry reactor.

One important aspect of loop – gas-phase reactor configurations is once-through conversion. For example, early WO9925741 describes a method of introducing polymer slurry obtained from a slurry reactor into a gas phase reactor containing a fluidized bed (C, D) with a first fluidized zone (C) on top of a second fluidized zone (D) with said fluidized zones having different flow patterns, in order to optimize the once-through conversion of the process.

Unfortunately medium size polypropylene production plants have significant higher operational costs and fixed costs per ton of produced product when compared to extra-large size polypropylene plants. However, by increasing the scale of the production, quality issues and operational issues can do rise. Increasing capacity of loop reactors can be done by adding further legs to the reactor. In industry, loop reactors have been scaled up from two legs to six legs, and even 8 legs loop reactors are not uncommon. However, the single circulation time using an 8-leg loop reactor will be roughly doubled when compared to a 4-legs loop reactor. Unfortunately, such modifications may result in drawbacks; e.g., significant concentration gradients can occur. For example, in polypropylene homo-polymerization the hydrogen to propylene ratio can vary over the length of the loop reactor, and especially in random propylene ethylene co-polymerization the ratio of ethylene to propylene can cause a significant gradient in the loop reactor. It goes without saying, when the C2/C3 ratio drops over the length of the

loop reactor, the randomness will be affected. Thus, when producing for example random propylene ethylene copolymer workarounds such as local feed of ethylene or multiple feed points to the reactor become a must. To sum up, upscaling of loop reactors is extremely cumbersome.

Another challenge for polymerization processes at very high throughput and especially for very large scale gas phase reactors is to control the temperature in a wide operating range, e.g. also at turndown ratios of 50%, and to establish a broad operation window, e.g. broad temperature ranges. Turndown ratio characterizes the ability to run a plant at reduced throughput. Turndown ratio is usually defined as $[1 - (\text{minimum capacity} / \text{design capacity})]$.

In case of turndown, the cooling water return (CWR) flow temperature can increase to high temperatures outside the normal operating window of cooling water systems. Reduced efficiency and corrosion cracking problems in the context of stainless steel equipment can be expected. Avoiding unnecessary high temperatures in the cooling water return flow is particularly important as chlorine is added to cool water circuits and/or towers for preventing biological growth.

Thus, there remains still the need for a process and a plant allowing upscaling to extra-large scale avoiding at least in part these and related problems.

Summary of the Invention

The present invention provides a plant for preparing propylene homopolymers or propylene copolymers, comprising

- (i) feed tank(s) for catalyst (1), optional co-catalyst (2), optional activator and/or optional external donor (3);
- (ii) optional a pre-contacting unit (4) for catalyst mixing being connected by feed lines (5, 5', 5'') with the feed tank(s);
- (iii) a prepolymerization reactor (6) connected with the feed tank(s) (1, 2, 3) or the pre-contacting unit (4) ;
- (iv) a propylene feed tank (7);
- (v) a first loop reactor (8) connected with the prepolymerization reactor;
- (vi) a second loop reactor (9) connected with the first loop reactor via a loop reactors connecting line (10);

- (vii) means for feeding monomer and optional comonomer (11) and hydrogen (12) to one or more of first loop reactor (8), second loop reactor (9), and/or loop reactors connecting line (10) between the loop reactors;
- (viii) a gas-phase reactor (13) equipped with a gas circulation line (14), a circulation gas compressor (15) and a circulation gas cooler (16), the gas-phase reactor being coupled to the at least second loop reactor by a direct feed line (17);
- (ix) means for feeding monomer (44) and/or comonomer (18) and/or hydrogen (19) to the gas-phase reactor (13); whereby preferably the means for feeding monomer and/or comonomer are suitable for feeding monomer and/or comonomer in condensed form;
- (x) optionally a product discharge vessel (20) connected with the gas-phase reactor;
- (xi) optionally a product outlet heater (21)
- (xii) a product receiver tank (22) connected with the optional product discharge vessel (20) or with the gas-phase reactor (13);
- (xiii) at least one purge bin (23);
- (xiv) at least one propylene nitrogen recovery unit (24);-----
- (xv) a column supply line (241) for feeding a hydrocarbon stream to a column (28)
- (xvi) a nitrogen re-feed line (243) for re-feeding a nitrogen rich stream to the purge bin (23)
- (xvii) optionally a thermal oxidizer unit (43)
- (xviii) an exhaust line (242) for discharge of an exhaust stream optionally to the optional thermal oxidizer (43)
- (xix) means for propylene homopolymer or propylene copolymer recovery (25) optionally including means for homogenization, additivation, and pelletization;
- (xx) a recovery gas treating unit (26) comprising at least one compressor (27), said column (28) and a reflux feed vessel (28a), the reflux feed vessel (28a) being connected via a recovery line (29) with the gas circulation line (14) of the gas-phase reactor (13)
- (xxi) a cooling circuit (30) for the circulation gas cooler (16);
- (xxii) a blow down unit (31) comprising a high pressure blow down bin (32), a low pressure blow down bin (33), the blow down unit (31) being optionally connected via connecting line (34) with the product receiver tank (22);

- (xxiii) a recovery feed line (35) connecting recovery gas treating unit (26) with the propylene feed tank (7), and
- wherein the circulation gas cooler (16) is a heat exchanger within a closed loop cooling water system (300) comprising a cooling water pump (301), a secondary heat exchanger (302), expansion vessel (303) and a by-pass (304) over a secondary heat exchanger.

The present invention further provides

a process for preparing propylene homopolymers and copolymers, comprising

- a) providing catalyst, optional co-catalyst, optional activator and/or optional external donor in feed tanks (1, 2, 3);
- b) feeding said catalyst, said optional co-catalyst, said optional activator and/or said optional external donor to a pre-contacting unit (4) for providing a mixed catalyst system; feeding said mixed catalyst system to the prepolymerization reactor (6)
or
feeding said catalyst, said optional co-catalyst, said optional activator and/or said optional external donor directly to a prepolymerization reactor (6);
- c) initiating prepolymerization by introducing propylene monomer and optionally introducing comonomer thereby providing a prepolymer;
- d) feeding said prepolymer to a first loop reactor (8) and polymerizing propylene optionally with comonomer yielding a first intermediate;
- e) feeding the first intermediate to a second loop reactor (9) via a loop reactors connection line (10) and further polymerizing propylene optionally with comonomer yielding a second intermediate, whereby propylene is fed from propylene feed tank (7) via propylene re-feed line (41) to the first loop reactor (8) and the second loop reactor (9);
- f) introducing optional comonomer and/or hydrogen at further feed points (11, 12) to the loop reactors;
- g) feeding the second intermediate containing unreacted monomer(s) directly to gas phase reactor (13) via direct feed line (17);
- h) further polymerizing propylene and optional comonomer in said gas phase reactor (13) by feeding propylene monomer and/or comonomer and/or hydrogen by effecting

a gas circulation via a gas circulation line (14) in upwards direction through the gas phase reactor (13), whereby the circulating gas is cooled in a circulation gas cooler (16);

- i) discharging the gas-phase reactor product into an optional product discharge vessel (20) and subsequently into a product-receiver tank (22) via an optional product outlet heater (21) for providing a raw mixture or discharging the gas-phase reactor product directly into a product receiver tank (22) for providing a raw mixture;
 - j) feeding said raw mixture to purge bin (23);
 - k) purging said raw mixture by nitrogen and catalyst deactivation agent, the catalyst deactivation agent being preferably steam,
 - l) separating nitrogen, deactivation agent and hydrocarbons from the purge bin (23) in a propylene nitrogen recovery unit (24) thereby
 - providing an essentially pure nitrogen stream being fed via nitrogen re-feed line (243) to purge bin (23) ;
 - providing a hydrocarbon lean waste stream being fed via exhaust line (242) to an optional thermal oxidizer unit (43);
 - providing a dry hydrocarbon rich stream being fed via column supply line (241) to column (28), whereby said column (28) is operated at a pressure higher than the pressure of the gas phase reactor (13) for removing oligomers, waxes and oils;
 - m) redirecting part of the non-condensed propylene back to the gas phase reactor via a first recovery line (29) and/or the condensed propylene to the propylene feed tank (7) via a second recovery line (35) and/or part of the condensed propylene is fed to a propylene propane splitter, whereby preferably, inert lights can be purged from said part of the non-condensed propylene and can be directed to a furnace or stream producing unit,
- and

wherein the circulation gas cooler (16) is a heat exchanger within a closed loop cooling water system (300) comprising a cooling water pump (301), a secondary heat exchanger (302), expansion vessel (303) and a by-pass (304) over a secondary heat exchanger.

The plant according to the present invention, in contrast to conventional Borstar plants, contains a recovery feed line (29) allowing to redirect non-condensed propylene back to the gas phase reactor (13). This setup surprisingly turned out to be beneficial with respect to the overall conversion and even further with respect to the monomer factor. For example, when producing random polypropylene copolymer huge savings are observed.

In the process according to the present invention, column (28) is operated at a pressure higher than the pressure of the gas phase reactor (13). This enables to recycle part of the hydrocarbons via recovery lines (29, 29') back to the gas phase reactor. It surprisingly turned out, that high hydrogen concentrations in the presence of ethylene are possible and the process is characterized by a high once-through conversion. As another surprising aspect, high amount of hydrogen recovery was observed. This is particularly beneficial for high melt flow rate grades: the required fresh hydrogen for the gas phase reactor can be lowered.

In a further aspect, nitrogen, steam and propylene as obtained are separated into

- a dry hydrocarbon rich stream (N₂ lean < 0.5 mol%) which is fed back to column (28),
- a pure nitrogen (>99mol%) stream which is fed back to purge bin (23) via nitrogen re-feed line (243); and
- a water waste flow and hydrocarbon lean nitrogen flow, which is fed to thermal oxidizer unit (43).

The plant according to the present invention is configured such that the circulation gas cooler (16) is a heat exchanger within a closed loop cooling water system (300) comprising a cooling water pump (301), a secondary heat exchanger (302), expansion vessel (303) and a by-pass (304) over a secondary heat exchanger. In the inventive plant and process, the gas circulation flow is cooled by a heat exchanger in order to have a very efficient temperature control in the gas phase reactor, usually and preferably a fluidized bed reactor. The polymerization heat is preferably transferred to a closed loop cooling water system (300), comprising a cooling water pump (301), a secondary heat exchanger (302), expansion vessel (303) and a by-pass over the secondary heat exchanger (304). The polymerization heat is further transferred via the closed loop cooling water system heat exchanger to a common site cooling water system, which can be for example cooling water towers. The main advantage is that the cooling water flow through the primary heat exchanger can be kept constant at a temperature above the dew point of the circulation gas and offering a wide

operating window on production rates in the gas phase reactor. Turndown ratios of 50% or more become possible. Turndown ratio characterizes the ability to run a plant at reduced throughput. Turndown ratio is defined as $[1 - (\text{minimum capacity} / \text{design capacity})]$.

In case of using a direct cooling water system cool water return temperature would rise to high values above typical designed operating temperature of the common site cooling water system, i.e. the operating window had to be limited.

The plant according to the present invention preferably comprises one or more of the following:

- (i) knock out drum (38)
- (ii) gas phase reactor area depressurization cyclone (39)
- (iii) gas phase reactor flare knock out drum (40).

Such units increase the safety of the inventive plant.

In the process according to the present invention, the circulation gas cooler (16) is a heat exchanger and the heat is transferred to a closed loop cooling water system, comprising a cooling water pump, a secondary heat exchanger, expansion vessel and a by-pass over the secondary heat exchanger. More preferably, the polymerization heat is transferred via the closed loop cooling water system to a common site cooling water system, such as a cooling water tower, for enabling a constant cooling water flow through the heat exchanger within the gas circulation at a temperature above the dew point of the circulation gas.

In a further aspect, the process according to the present invention is characterized by possible turn down ratios of more than 50%. This high turn down allows really high flexibility with respect to varying products and demands.

The split, i.e. the ratio of the amounts of material produced in loop and gas phase reactors respectively ranges from 40-60 to 60-40.

In yet a further aspect, the polymerization temperature in the first and/or the second loop reactor, preferably in both loop reactors is below 72°C, more preferably below 70°C.

Detailed Description

In the following, the invention shall be described with respect to the figures.

Reference numbering for Fig. 1

- | | |
|------------|---|
| 1 | feed tank for catalyst |
| 2 | optional feed tank for co-catalyst |
| 3 | optional feed tank(s) for activator and/or optional external donor |
| 4 | optional pre-contacting unit for catalyst mixing |
| 5, 5', 5'' | feed lines connecting feed tank connecting feed tanks and catalyst feed vessel
or prepolymerization reactor |
| 6 | prepolymerization reactor |
| 7 | propylene feed tank |
| 8 | first loop reactor |
| 9 | second loop reactor |
| 10 | loop reactors connecting line |
| 11 | means for feeding monomer and optional comonomer |
| 12 | means for feeding hydrogen |
| 13 | gas-phase reactor |
| 14 | gas circulation line |
| 15 | circulation gas compressor |
| 16 | circulation gas cooler |
| 17 | direct feed line |
| 18 | means for feeding comonomer |
| 19 | means for feeding hydrogen |
| 20 | optional product discharge vessel |
| 21 | product outlet heater |
| 22 | product receiver tank |
| 23 | purge bin |
| 24 | propylene nitrogen recovery unit |
| 25 | outlet line (for polymer powder) with means for propylene homopolymer or
propylene copolymer recovery (optionally including means for
homogenization, additivation and pelletization; |
| 26 | recovery gas treating unit |
| 27 | gas recovery compressor |
| 28 | column |
| 28a | reflux recovery feed vessel |

29, 29'	recovery feed lines
30	cooling circuit for circulation gas cooler
31	blow down unit
32	high pressure blow down bin
33	low pressure blow down bin
34	connecting line
35	recovery feed line
36	further feed line optionally containing dryer(s)
38	knock out drum
39	gas phase reactor area depressurization cyclone
40	gas phase reactor flare knock out drum
41, 41', 41''	propylene feed line
42	means for catalyst deactivation (e.g. means for introducing low pressure steam)
43	thermal oxidizer unit
44	means for feeding liquid propylene monomer
48	purge bin feed line
49	line connecting product receiver tank (22) and gas recovery compressor (27)
51	feed line from purge bin (23) to propylene nitrogen recovery unit (24)
241	column supply line
242	exhaust line
243	nitrogen re-feed line
244	external nitrogen feed line
245	feed line for catalyst deactivating agent (usually low pressure steam)
246	further nitrogen feed line
247	line for conveying nitrogen
249	outlet line for oligomers

Fig. 1 shows the inventive plant which is used for carrying out the inventive process.

Fig. 2 shows the inventive cooling setup.

Reference numbering for Fig. 2

300	gas phase reactor circulation gas cooler
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301	cooling water pump of closed loop cooling water system
302	a secondary heat exchanger
303	expansion vessel of closed loop cooling water system
304	by-pass over the secondary heat exchanger
305	site cooling water pump(s)
306	site cooling water tower(s)
310	gas circulation flow to the gas phase reactor
311	gas circulation flow after compression stage from the gas phase reactor
312	closed loop cooled cooling water flow
313	total closed loop cooling water flow in to gas phase reactor circulation cooler
314	closed loop cooling water flow out from gas phase reactor circulation cooler
315	site cooling water supply flow
316	site cooling water return flow

Fig. 3 shows a comparative cooling water setup.

Reference numbering for Fig. 3

400	gas phase reactor circulation gas cooler
401	cooling water pump of gas phase reactor cooling system
402	cooling water return control line
403	cooling water by-pass
404	cooling water supply control line
405	site cooling water pump(s)
406	site cooling water tower(s)
410	gas circulation flow to the gas phase reactor
411	gas circulation flow after compression stage from the gas phase reactor
412	fresh cool water supply flow
413	total cooling water flow to gas phase reactor circulation gas cooler

The plant according to the present invention shall be further described with respect to Fig. 1.

The plant according to the present invention for preparing propylene homopolymers and copolymers comprises feed tank(s) for catalyst (1), optional co-catalyst (2), optional activator and/or optional external donor (3). Optionally there is a pre-contacting unit (4) for catalyst mixing being connected by feed lines (5, 5', 5'') with the feed tank(s). In various embodiments and for various catalyst systems, a pre-contacting tank is not necessary.

The plant according to the present invention also includes a prepolymerization reactor (6) connected with the feed tank(s) (1, 2, 3) or the pre-contacting unit (4). Such prepolymerization is known in the art. The plant also includes a propylene feed tank (7), a first loop reactor (8) connected with the prepolymerization reactor, and a second loop reactor (9) connected with the first loop reactor via a loop reactors connecting line (10) as well as means for feeding comonomer (11) and hydrogen (12) to one or more of first loop reactor (8), second loop reactor (9), and/or loop reactors connecting line (10) between the loop reactors.

Such setup is frequently found in existing plants.

Apart from the loop reactors the plant according to the present invention also comprises a gas-phase reactor (13) equipped with a gas circulation line (14), a circulation gas compressor (15) and a circulation gas cooler (16), the gas-phase reactor being coupled to the at least second loop reactor by a direct feed line (17). means for feeding monomer (44) and/or comonomer (18) and/or hydrogen (19) to the gas-phase reactor (13). In addition to the plant optionally includes a product discharge vessel (20) connected with the gas-phase reactor. Such product discharge vessel contributes to the operational stability. Optionally a product outlet heater (21) is present. Usually a product outlet heater will be several units. The plant according to the present invention also includes a product receiver tank (22) connected with the optional product discharge vessel (20) or with the gas-phase reactor (13), at least one purge bin (23). In addition to that, the plant also includes at least one propylene nitrogen recovery unit (24) with a column supply line (241) for feeding a hydrocarbon stream to a column (28), a nitrogen re-feed line (243) for re-feeding a nitrogen rich stream to the purge bin (23), optionally a thermal oxidizer unit (43) and an exhaust line (242) for discharge of an exhaust stream optionally to the optional thermal oxidizer (43). Optionally there can be further feed lines such as a nitrogen re-feed line (243), an external nitrogen feed line (245), a feed line for catalyst deactivating agents, i.e. usually low pressure steam. Preferably, there is also an outlet for oligomers (249).

The plant according to the present invention also includes means for propylene homopolymer or propylene copolymer recovery (25) said means (25) optionally including means for homogenization, additivation, and pelletization, a recovery gas treating unit (26) comprising at least one compressor (27), said column (28) and a reflux feed vessel (28a), the reflux feed vessel (28a) being connected via a recovery line (29) with the gas circulation line (14) of the gas-phase reactor (13).

In addition to that, there is a cooling circuit (30) for the circulation gas cooler (16). Such circulation gas cooler contributes to the broad operational window.

Apart therefrom there is also a blow down unit (31) comprising a high pressure blow down bin (32), a low pressure blow down bin (33), the blow down unit (31) being optionally connected via connecting line (34) with the product receiver tank (22).

The plant according to the present invention further includes a recovery feed line (35) connecting recovery gas treating unit (26) with the propylene feed tank (7). This important recovery feed line (35) allows refeed of propylene also to the loop reactors, i.e. results in an integrated recovery system.

The cooling setup according to the present invention shall be described with reference to Fig. 2. The cooling medium, usually water, is circulated by a cooling water pump (301) in the closed loop cooling water system. The water enters a secondary heat exchanger (302) in which the heat is transferred to the site cooling water circuit. The site cooling water circuit includes site cooling water tower(s) (306), site cooling water pump(s) (305) but may also include heat consumers, i.e. re-use of the heat for residential heating or similar. The secondary heat exchanger can be of any type such as plate, shell and tube.

For properly adjusting temperature and keeping the flow through the primary heat exchanger (300) constant, there is also a by-pass (304) over the secondary heat exchanger in the closed loop cooling water system. In this closed loop cooling water system, heat is transferred from gas to water in the gas phase reactor circulation gas cooler (300). As known in the art there is also a expansion vessel of the closed loop cooling water system (303).

A direct heat exchanger on site cooling water system with recirculation pump is shown in Figure 3. Such configuration has been used for comparative purposes and is not desirable. It can be seen the site cooling water circuit is coupled directly with the gas phase reactor circulation gas cooler (400).

Experimental Part

The plant and the process according to the invention have been exemplified in the following examples. These examples are included for illustrative purposes and do not limit the invention.

Examples 1 and 2

In REF1 and REF2, no gas circulation cooler was used and there was only one loop reactor. In example IE1 a gas phase reactor and a gas circulation cooler was used. In example IE2, the recirculation was lowered.

Table 1

	REF1	REF2	IE1	IE2
Preparation of	random polypropylene ethylene copolymer			
			inventive plant/inventive process	
				product discharge vessel
Prepolymerization reactor				
production kg/h	0.9	0.9	0.9	0.9
Bar	56	56	39	39
T °C	20	20	20	20
(first) Loop reactor				
production kg/h	45	90	60	60
P Bar	55	55	38	38
T °C	85	85	70	70
Second loop reactor				
production ton/h			30	30
P Bar			37	37
T °C			70	70
GPR1				
production kg/h	30	90	90	90
P Bar	35	21	21	21
T °C	85	85	85	85
Recirculation				
kg/h	15	45	45	21
once-through conversion	83%	80%	80%	90%

It can be seen that the inventive plant and the inventive process simultaneously allowed high production in the gas phase reactor and high once-through conversion.

Example 3

In a second experimental evaluation, the catalyst mileage (IE1 versus REF2) was assessed.

Table 2

	Catalyst feed	Loop Reactor		Gas Phase Reactor		catalyst
	normalised	residence time	production normalised	residence time	production normalised	productivity
	[%]	[h]	[%]	[h]	[%]	[-]
IE1 two loops + GPR	100	0.8	100	2	100	2
REF2 one loop + GPR	100	0.5	80	2	80	1.6

Surprisingly a high catalyst mileage was obtained for the inventive process:

Example 4

In yet a further experimental evaluation using the inventive plant and the inventive process, the cooling water temperatures were monitored when producing random ethylene propylene copolymer. The inventive used a closed loop cooling water system as shown in Fig. 2, whereas the comparative examples used a configuration as shown in Fig. 3.

The results are shown in Table 3 provided below.

[illegible]

Comparative examples product		norm al rando m	40/60 rando m	50/50 rando m	60/40 rando m	turn down rando m	45/55 rando m	50/50 rando m	60/40 rando m	40/60 rando m	50/50 homo	40/60 homo	60/40 homo	50/50 homo	40/60 homo	60/40 homo
production in the GPR	kg/ h	99	108	90	72	45	99	90	72	108	90	108	72	90	108	72
Temperature GPR	°C	80	80	80	80	80	90	90	80	90	80	80	80	90	90	90
T CRG in	°C	82.77	82.77	82.77	82.77	84	92.56	92.56	92.56	92.56	82.77	82.77	82.77	92.56	92.56	92.56
T CRG out	°C	54.55	52.37	57.17	62.49	70.56	64.14	66.85	72.32	64.45	57.76	52.66	62.97	67.45	62.16	72.8
by pass flow	m ³ / h	1.6	0.0	3.0	4.5	5.3	3.6	4.2	4.9	3.0	3.3	0.0	4.6	4.3	3.0	5.0
Fresh cool water flow	m ³ / h	4.4	6.0	3.0	1.5	0.7	2.4	1.8	1.1	3.0	2.7	6.0	1.4	1.7	3.0	1.0
T CWS	°C	27	25	27	27	27	27	27	27	27	27	26.5	27	27	27	27
T CW in	°C	30.2	25.4	35.3	45.5	59.8	40.1	45.3	55.6	36.8	36.4	26.5	46.4	46.4	36.4	56.5
T CW out	°C	39.2	35.2	43.4	51.8	63.8	49.0	53.3	61.8	46.6	44.3	36.1	52.6	54.2	45.9	62.6

As can be seen from above Table 3 the temperature of the cooling water return (T CWR) stayed below 36°C for all inventive examples. Thus, a surprisingly broad operating window was found. For the comparative examples, the temperature of the cooling water out, abbreviated T CW out went up to values above 50°C and in some cases even above 60°C. Thus, many constraints are given for operation. It is needless to say temperature of above 50°C or even above 60°C cannot be accepted due to massive piping and heat exchanger restrictions.

Claims

1. Plant for preparing propylene homopolymers or propylene copolymers, comprising
 - (i) feed tank(s) for catalyst (1), optional co-catalyst (2), optional activator and/or optional external donor (3);
 - (ii) optional pre-contacting unit (4) for catalyst mixing being connected by feed lines (5, 5', 5'') with the feed tank(s);
 - (iii) a prepolymerization reactor (6) connected with the feed tank(s) (1, 2, 3) or the pre-contacting unit (4);
 - (iv) a propylene feed tank (7);
 - (v) a first loop reactor (8) connected with the prepolymerization reactor;
 - (vi) a second loop reactor (9) connected with the first loop reactor via a loop reactors connecting line (10);
 - (vii) means for feeding monomer and optional comonomer (11) and hydrogen (12) to one or more of first loop reactor (8), second loop reactor (9), and/or loop reactors connecting line (10) between the loop reactors;
 - (viii) a gas-phase reactor (13) equipped with a gas circulation line (14), a circulation gas compressor (15) and a circulation gas cooler (16), the gas-phase reactor being coupled to the at least second loop reactor by a direct feed line (17);
 - (ix) means for feeding monomer (44) and/or comonomer (18) and/or hydrogen (19) to the gas-phase reactor (13); whereby preferably the means for feeding monomer and/or comonomer are suitable for feeding monomer and/or comonomer in condensed form;
 - (x) optionally a product discharge vessel (20) connected with the gas-phase reactor;
 - (xi) optionally a product outlet heater (21)
 - (xii) a product receiver tank (22) connected with the optional product discharge vessel (20) or with the gas-phase reactor (13);
 - (xiii) at least one purge bin (23);
 - (xiv) at least one propylene nitrogen recovery unit (24);
 - (xv) a column supply line (241) for feeding a hydrocarbon stream to a column (28)

- (xvi) a nitrogen re-feed line (243) for re-feeding a nitrogen rich stream to the purge bin (23)
- (xvii) optionally a thermal oxidizer unit (43)
- (xviii) an exhaust line (242) for discharge of an exhaust stream optionally to the optional thermal oxidizer (43)
- (xix) means for propylene homopolymer or propylene copolymer recovery (25) optionally including means for homogenization, additivation, and pelletization;
- (xx) a recovery gas treating unit (26) comprising at least one compressor (27), said column (28) and a reflux feed vessel (28a), the reflux feed vessel (28a) being connected via a recovery line (29) with the gas circulation line (14) of the gas-phase reactor (13);
- (xxi) a cooling circuit (30) for the circulation gas cooler (16);
- (xxii) a blow down unit (31) comprising a high pressure blow down bin (32), a low pressure blow down bin (33), the blow down unit (31) being optionally connected via connecting line (34) with the product receiver tank (22);
- (xxiii) a recovery feed line (35) connecting recovery gas treating unit (26) with the propylene feed tank (7)., and

wherein the circulation gas cooler (16) is a heat exchanger within a closed loop cooling water system (300) comprising a cooling water pump (301), a secondary heat exchanger (302), expansion vessel (303) and a by-pass (304) over a secondary heat exchanger.

2. The plant of claim 1 further comprising one or more of the following:

- (xxiv) knock out drum (38)
- (xxv) gas phase reactor area depressurization cyclone (39)
- (xxvi) gas phase reactor flare knock out drum (40).

3. A process for preparing propylene homopolymers or propylene copolymers. comprising

- a) providing catalyst. optional co-catalyst. optional activator and/or optional external donor in feed tanks (1, 2, 3);

- b) feeding said catalyst, said optional co-catalyst, said optional activator and/or said optional external donor to a pre-contacting unit (4) for providing a mixed catalyst system; feeding said mixed catalyst system to the prepolymerization reactor
- or
- feeding said catalyst, said optional co-catalyst, said optional activator and/or said optional external donor to prepolymerization reactor (6);
- c) initiating prepolymerization by introducing propylene monomer and optionally introducing comonomer thereby providing a prepolymer;
- d) feeding said prepolymer to a first loop reactor (8) and polymerizing propylene optionally with comonomer yielding a first intermediate;
- e) feeding the first intermediate to a second loop reactor (9) via a loop reactors connection line (10) and further polymerizing propylene optionally with comonomer yielding a second intermediate, whereby propylene is fed from propylene feed tank (7) via propylene re-feed line (41) to the second loop reactor;
- f) introducing comonomer and/or hydrogen at further feed points to the loop reactors;
- g) feeding the second intermediate containing unreacted monomer(s) directly to gas phase reactor (13) via direct feed line (17);
- h) further polymerizing propylene and optional comonomer in said gas phase reactor (13) by feeding propylene and/or comonomer and/or hydrogen and by effecting a gas circulation via a gas circulation line (14) in upwards direction, whereby the circulating gas is cooled in a circulation gas cooler (16);
- i) optionally discharging the gas-phase reactor product into an optional product discharge vessel (20) and subsequently into a product-receiver tank (22) via connecting line (34) for providing a raw mixture or discharging the gas-phase reactor product directly into a product receiver tank (22) for providing a raw mixture;
- j) feeding said raw mixture to purge bin 23;
- k) purging said raw mixture by nitrogen and catalyst deactivation agent, the catalyst deactivation agent being preferably steam,
- l) separating nitrogen, deactivation agent and hydrocarbons from the purge bin (23) in a propylene nitrogen recovery unit (24) thereby

- providing an essentially pure nitrogen stream being fed via nitrogen re-feed line (243) to purge bin (23);
- providing hydrocarbon lean waste stream being fed via exhaust line (242) to a thermal oxidizer unit (43);
- providing a dry hydrocarbon rich stream being fed via column supply line (241) to column (28), whereby said column (28) is operated at a pressure higher than the pressure of the gas phase reactor (13) for removing oligomers, waxes and oils;
- m) redirecting condensed propylene and propane to one or more of the following
 - said propylene feed tank (7) via recovery feed line (35);
 - an optional propane / propylene splitter,

and feeding the non-condensed matter at least in part to the gas-phase reactor via line 29.; and

wherein the circulation gas cooler (16) is a heat exchanger and the heat is transferred via a closed loop cooling water system (300) comprising a cooling water pump (301), a secondary heat exchanger (302), expansion vessel (303) and a by-pass (304) over the secondary heat exchanger.

4. The process of claim 3, wherein the polymerization heat is transferred via the closed loop cooling water system to a common site cooling water system such as a cooling water tower for enabling a constant cooling water flow through the heat exchanger within the gas circulation at a temperature above the dew point of the circulation gas.
5. The process of claim 3 or 4, allowing a turn down ratio of 50% as to production rate in the gas-phase reactor.
6. The process according to any one of claims 3 to 5, whereby the polymerization temperature in the first and/or the second loop reactor, preferably in both loop reactors is below 72°C, more preferably below 70°C.
7. The process according to any one of claims 3 to 6, producing a homopolymer with a split between 40% and 60% produced in the gas phase reactor.

8. The process according to any one of claims 4 to 7 producing a random ethylene propylene co-polymer with a split between 40% and 60% produced in the gas phase reactor.
9. The process according to any one of claims 3 to 8 whereby the polymerization temperature in the gas phase reactor is between 80°C and 90°C and the pressure between 19 and 25 barg, more preferable 20 and 24 barg.
10. The process according to any one of claims 3 to 9 whereby the operating pressure of the column is 26 barg, more preferable 25 barg most preferably 24 barg.

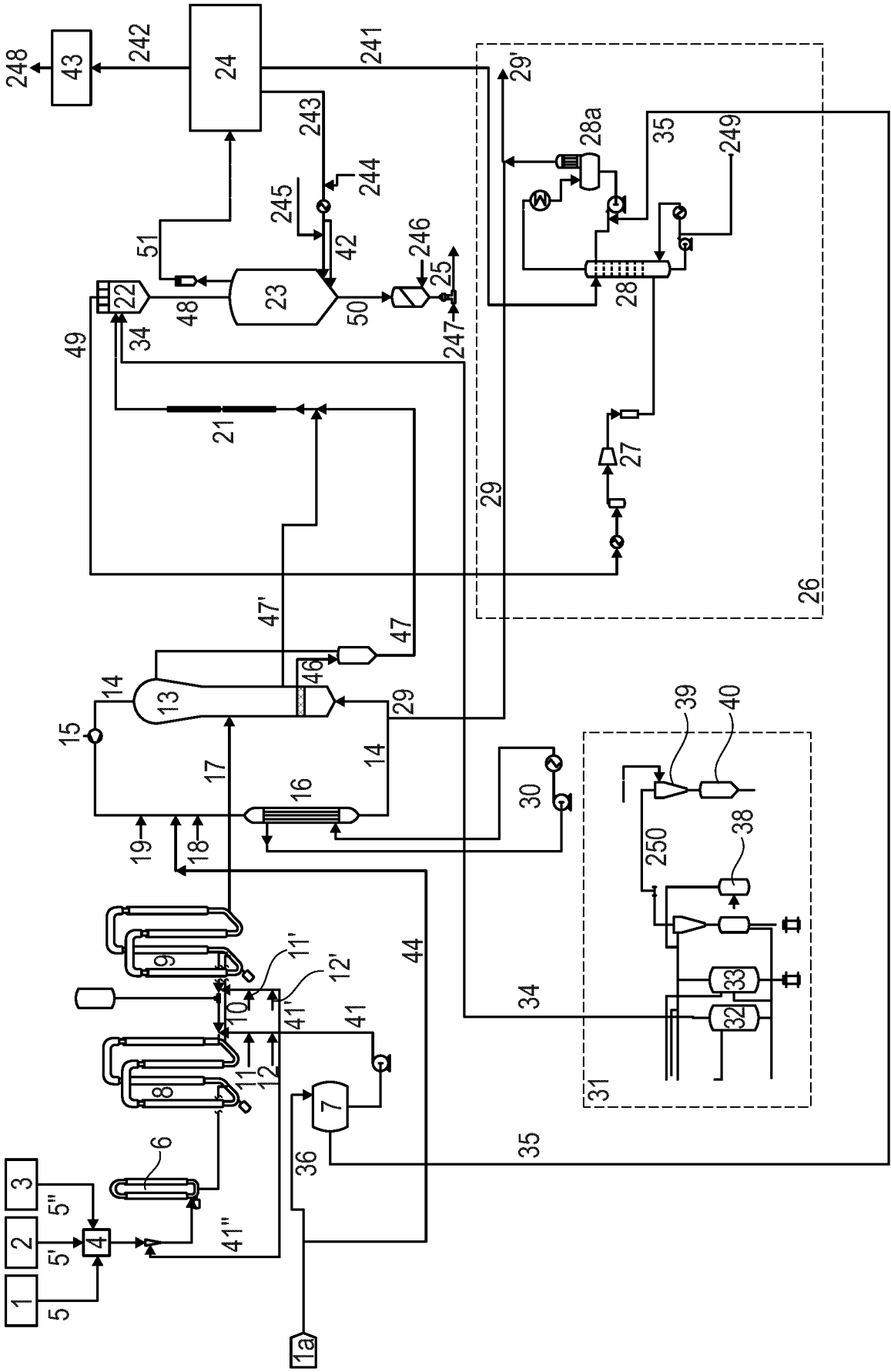


Fig. 1

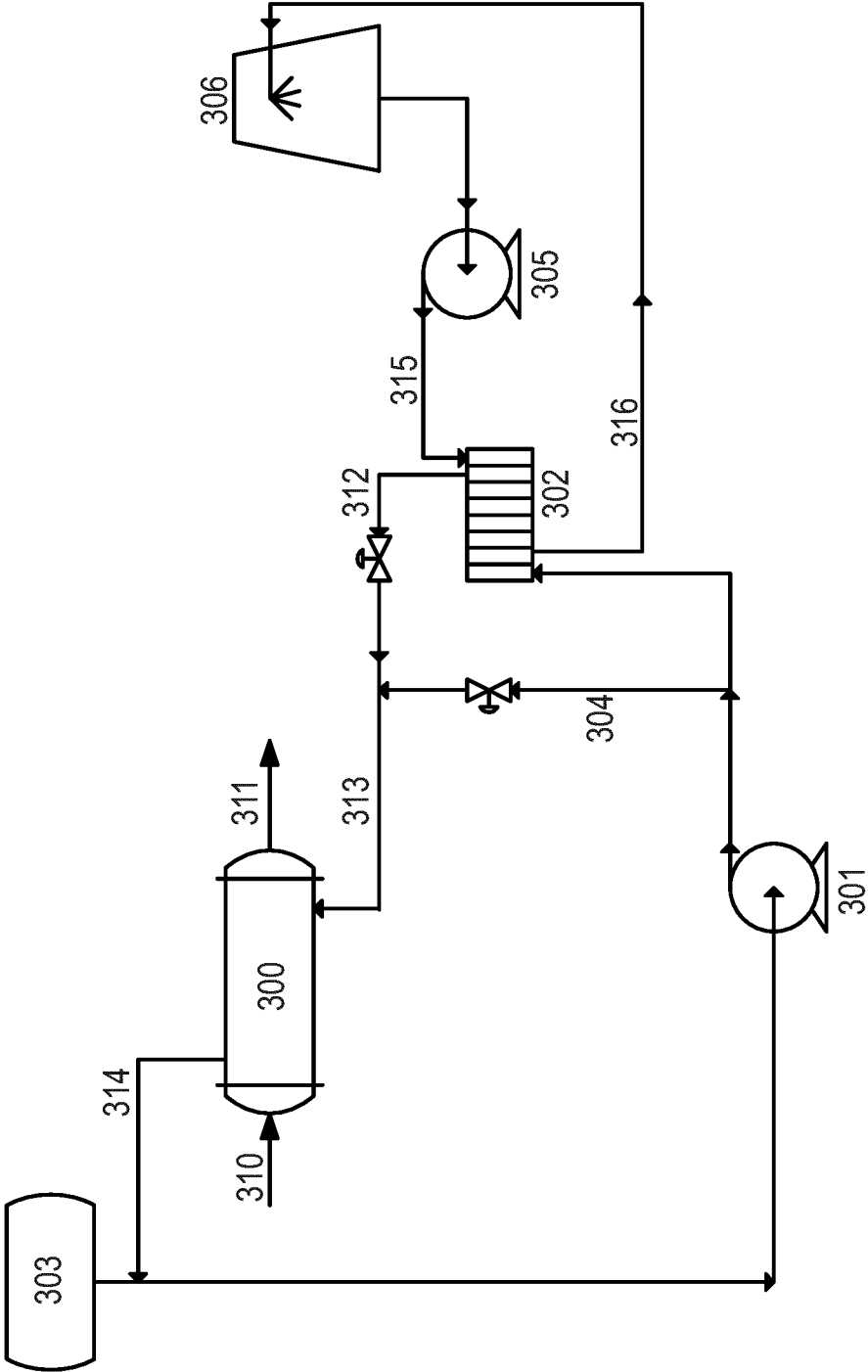


Fig. 2

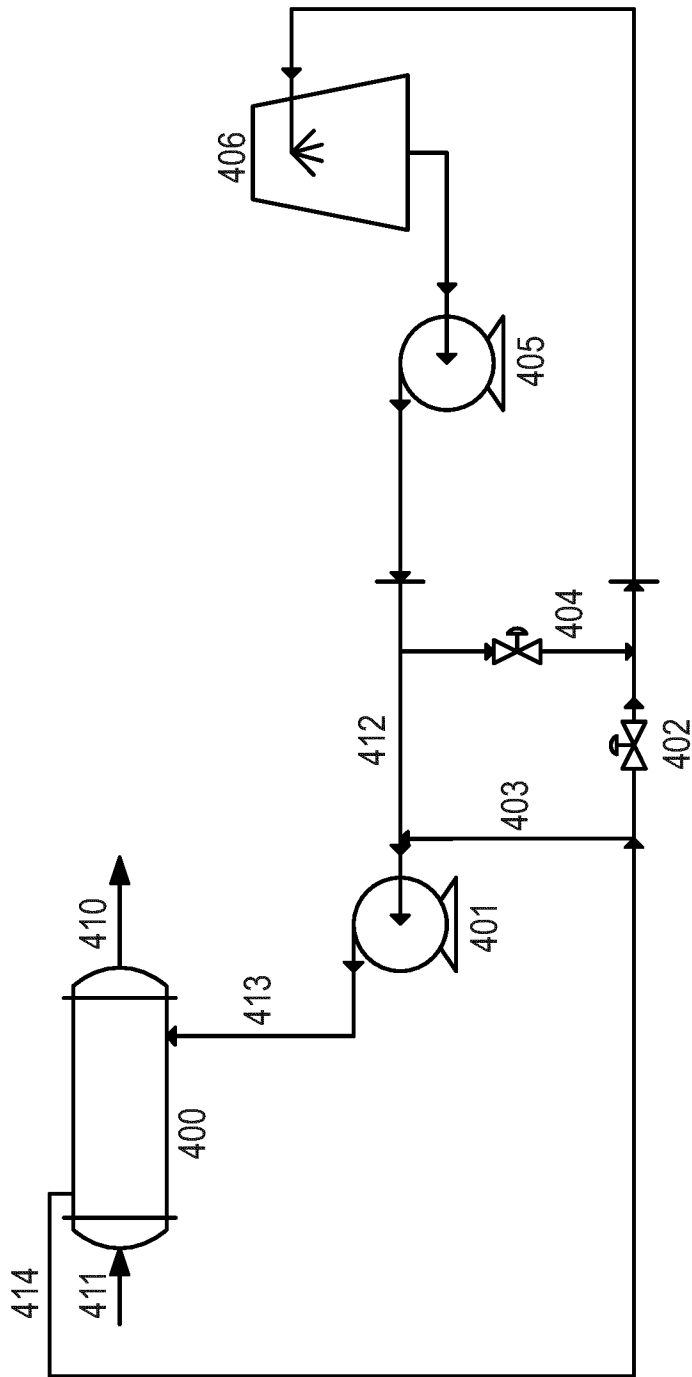


Fig. 3

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2021/082469

A. CLASSIFICATION OF SUBJECT MATTER INV. C08F2/00 C08F2/01 B01J8/24 B01J19/24 C08F10/06 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) B01J C08F		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2019/089111 A1 (EXXONMOBIL CHEMICAL PATENTS INC [US]) 9 May 2019 (2019-05-09) the whole document -----	1-10
A	CN 207 685 181 U (BEIJING HUAFU ENG CO LTD) 3 August 2018 (2018-08-03) the whole document -----	1-10
A	WO 2019/090883 A1 (BEIJING HUAFU ENG CO LTD [CN]) 16 May 2019 (2019-05-16) the whole document -----	1-10
A	EP 3 438 133 A1 (BASELL POLYOLEFINE GMBH [DE]) 6 February 2019 (2019-02-06) the whole document -----	1-10
☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance;; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance;; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 20 January 2022	Date of mailing of the international search report 01/02/2022	
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Balmer, J	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2021/082469

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2019089111 A1	09-05-2019	CN 111295399 A EP 3707174 A1 US 2020283549 A1 WO 2019089111 A1	16-06-2020 16-09-2020 10-09-2020 09-05-2019
CN 207685181 U	03-08-2018	NONE	
WO 2019090883 A1	16-05-2019	CN 109776702 A WO 2019090883 A1	21-05-2019 16-05-2019
EP 3438133 A1	06-02-2019	BR 112020002307 A2 CN 110997731 A EP 3438133 A1 EP 3638701 A1 JP 6955832 B2 JP 2020528961 A KR 20200032720 A RU 2730015 C1 US 2021102009 A1 WO 2019025560 A1	04-08-2020 10-04-2020 06-02-2019 22-04-2020 27-10-2021 01-10-2020 26-03-2020 14-08-2020 08-04-2021 07-02-2019