



(51) International Patent Classification:
C07C 51/487 (2006.01) C07C 63/26 (2006.01)
C07C 63/15 (2006.01) B01J 8/02 (2006.01)

(21) International Application Number:
PCT/US2011/024746

(22) International Filing Date:
14 February 2011 (14.02.2011)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
61/304,567 15 February 2010 (15.02.2010) US

(71) Applicant (for all designated States except US): **IN-VISTA TECHNOLOGIES S.A.R.L.** [LU/CH]; Zweigniederlassung St. Gallen, Pestalozzistrasse 2, 9000 St. Gallen (CH).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **BICKHAM, David, Robert** [GB/GB]; 8 Berkley Drive, Hunters Hill, Guisborough TS14 7LX (GB). **O'BRIEN, Robert, John** [GB/GB]; 7 Fanacurt Road, Guisborough TS14 8BJ (GB).

PARKER, David [GB/GB]; 3 Market Hill, Whitchurch, Aylesbury HP22 4JB (GB).

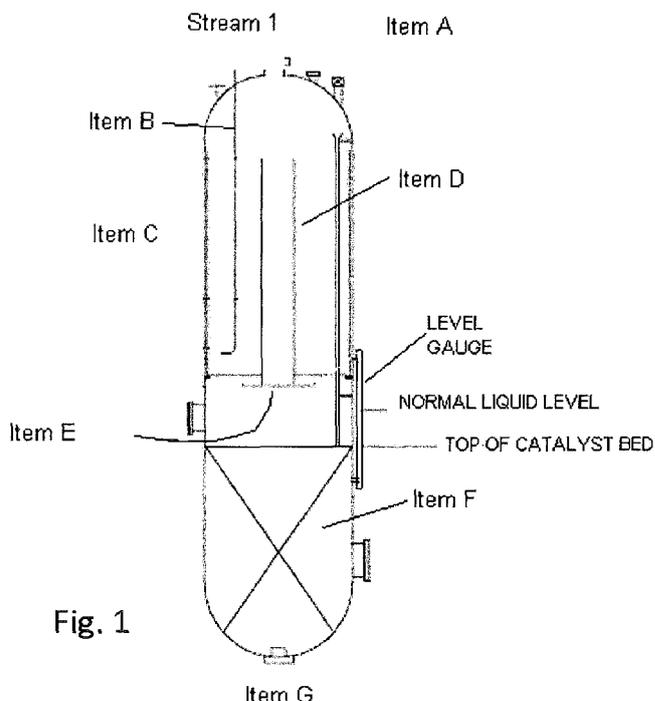
(74) Agent: **STERNER, Craig, M.**; Three Little Falls Centre/1052, 2801 Centerville Road, Wilmington, DE 19808 (US).

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

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK,

[Continued on next page]

(54) Title: PURIFICATION OF CARBOXYLIC ACIDS BY CATALYTIC HYDROGENATION



(57) Abstract: The present invention relates to a process for the production of an aromatic carboxylic acid comprising: a) introducing a crude aromatic carboxylic acid solution into a purification reactor vessel, wherein the purification reactor vessel is operating under pressure, b) introducing hydrogen gas into the purification reactor vessel, c) dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution as the solution flows down a wall of a vertical conduit onto a distributor, wherein the purification reactor vessel has a gas-liquid contact area to plant throughput (capacity) ratio of at least 0.55 m²/te/h of carboxylic acid for dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution to produce a reaction solution, and d) contacting the reaction solution with a supported catalyst bed to produce a purified aromatic carboxylic acid, wherein the supported catalyst bed is submerged in the reaction solution and a liquid level of the reaction solution is maintained above the supported catalyst bed.

WO 2011/100682 A2

SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG). **Published:**

— *without international search report and to be republished upon receipt of that report (Rule 48.2(g))*

PURIFICATION OF CARBOXYLIC ACIDS BY CATALYTIC HYDROGENATION**CROSS-REFERENCE TO RELATED APPLICATION**

5 This application claims benefit of priority from U.S. Provisional Application No. 61/304567 filed February 15, 2010.

FIELD OF THE INVENTION

10

This invention relates to a process for the purification of aromatic polycarboxylic acids, and more particularly concerns the catalytic hydrogenation reaction for the preparation of purified aromatic polycarboxylic acids.

15

BACKGROUND OF THE INVENTION

Aromatic polycarboxylic acids are commercially important chemical intermediates, especially as raw materials for polyesters, which are used for the manufacture of fibres, bottles, films and electronic applications.

20

The preparation of aromatic polycarboxylic acids, for example terephthalic acid, isophthalic acid and naphthalene dicarboxylic acid, typically include an oxidation step followed by a purification step. The oxidation step comprises a method in which a polyalkyl aromatic hydrocarbon precursor such as xylene, trialkylbenzene or dialkyl naphthalene is oxidized with molecular oxygen at a high temperature and a high pressure in the presence of a heavy metal such as cobalt or manganese and a bromine compound in an aqueous acetic acid solvent. The crude aromatic polycarboxylic acid obtained by the above oxidation reaction contains impurities such as monocarboxylic acids and aldehydes which are intermediate products of the oxidation reaction, bromine adducts and metal components which are derived from the catalyst and coloring substances having unknown structures. A purification step to produce an aromatic polycarboxylic acid having a purity sufficient for direct esterification with diols for the production of polyester polymer involves a method in

25

30

which the crude aromatic polycarboxylic acid is hydrogenated at a high temperature and pressure in the presence of a catalyst such as a Group VIII transition metal on a carbon support in water as a solvent, for example as disclosed in US patent number 3,584,039. The purified aromatic polycarboxylic acid following the hydrogenation reaction can be used
5 directly for the production of polymers or separated from the solvent typically using a method to crystallise the aromatic polycarboxylic acid, separate the crystallised product from the water solvent and drying to produce a dried, crystallised product.

10

SUMMARY OF THE INVENTION

Problems with the conventional process to purify aromatic polycarboxylic acids include i) the number of vessels required, to dissolve the aromatic polycarboxylic acid and hold a working liquid inventory of the solution and ii) the size of the reactor, to dissolve the
15 hydrogen gas, contact the solution containing crude aromatic polycarboxylic acid and hydrogen with the catalyst and to provide sufficient residence time for completion of the purification reactions. Typically, due to the limited solubility of hydrogen in the aromatic polycarboxylic acid solution a high reactor pressure has to be maintained to ensure sufficient hydrogen is dissolved in the aqueous solvent to complete the purification of the crude
20 aromatic polycarboxylic acid. A further problem that results from using a heterogeneous catalyst for the purification reactions is the shortened operational life of the catalyst, due to the breakdown of the support material.

The consequence of these combined problems is either an increase in operational
25 costs due to the higher equipment costs or the reduced catalyst life, or poorer operational control with greater purified product variation.

It is an object of this invention to reduce or avoid one or more of the above-mentioned problems. In particular, it is an object of this invention to provide an improved continuous
30 process for the purification of aromatic polycarboxylic acids as an aqueous solution at high temperature and high pressure in contact with a supported catalyst using hydrogen. A method has been found to carry out the purification reaction, comprising the conversion of reaction intermediates from an aromatic aldehyde to the corresponding aromatic

monocarboxylic acid. The present invention relates to a process for the production of an aromatic carboxylic acid comprising: a) introducing a crude aromatic carboxylic acid solution into a purification reactor vessel, wherein the purification reactor vessel is operating under pressure, b) introducing hydrogen gas into the purification reactor vessel, c) dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution as the solution flows down a wall of a vertical conduit onto a distributor, wherein the purification reactor vessel has a gas-liquid contact area to plant throughput (capacity) ratio of at least $0.55 \text{ m}^2/\text{te/h}$ of carboxylic acid processed for dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution to produce a reaction solution, and d) contacting the reaction solution with a supported catalyst bed to produce a purified aromatic carboxylic acid, wherein the supported catalyst bed is submerged in the reaction solution and a liquid level of the reaction solution is maintained above the supported catalyst bed. The catalyst bed is completely submerged and a level of dissolved crude polycarboxylic acid (crude acid) solution is maintained above the supported catalyst. The benefit of this method of operation is to extend the operational life of the supported catalyst bed, by ensuring the gaseous hydrogen does not come into direct contact with supported catalyst particles and variations in product quality are reduced by maintaining a consistent level of crude acid solution above the supported catalyst bed. It is a further object of this invention to provide improved dissolution of hydrogen in the crude acid solution as it enters the purification reactor by increasing the liquid surface area in contact with the gaseous hydrogen and reduce the rapid changes in operating pressure and fluctuations in the purification reactor liquid level that result in variable purified polycarboxylic acid (purified acid) product quality. It is a further object to reduce the number of vessels and the total residence time of the aromatic carboxylic acid solution, thereby significantly reducing the cost of the equipment necessary for the purification step of the manufacturing process.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic diagram illustrates an embodiment of the invention showing the purification reactor.

Figure 2 is a schematic diagram illustrates an embodiment of the invention showing the liquid distributor.

Figure 3 is a schematic diagram of a conventional purification reactor.

DETAILED DESCRIPTION

The present invention relates to a process for the production of an aromatic carboxylic acid comprising: a) introducing a crude aromatic carboxylic acid solution into a purification reactor vessel, wherein the purification reactor vessel is operating under pressure, b) introducing hydrogen gas into the purification reactor vessel, c) dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution as the solution flows down a wall of a vertical conduit onto a distributor, wherein the purification reactor vessel has a gas-liquid contact area to plant throughput (capacity) ratio of at least $0.55 \text{ m}^2/\text{te/h}$ of carboxylic acid for dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution to produce a reaction solution, and d) contacting the reaction solution with a supported catalyst bed to produce a purified aromatic carboxylic acid, wherein the supported catalyst bed is submerged in the reaction solution and a liquid level of the reaction solution is maintained above the supported catalyst bed. The gas-liquid contact area to plant throughput (capacity) ratio of step c) can be about $0.65 \text{ m}^2/\text{te/h}$ of carboxylic acid or more, for example about $0.75 \text{ m}^2/\text{te/h}$ of carboxylic acid or more or $1.2 \text{ m}^2/\text{te/h}$ of carboxylic acid or more. The distributor can comprise i) a perforated circular disc with a diameter about 0.3 to about 0.7 of the purification reactor vessel diameter, ii) notched weir openings arranged around the rim of the plate to retain the crude acid solution on the plate, and iii) at least one hole through which the reaction solution flows onto the liquid surface below the distributor. The distributor can also comprise i) a perforated circular open pipe or channel with a diameter about 0.3 to about 0.7 of the purification reactor vessel diameter, ii) notched weir openings arranged around the rim of the pipe or channel to retain the crude acid solution on the plate, and iii) at least one hole through which the reaction solution flows onto the liquid surface below the distributor. The distributor can be located between 0.5 to 2 metres above the liquid level of the crude acid solution, for example 0.5 to 1.0 metres and the crude acid solution flows through the holes and over the notched weir openings. Optionally, alternative liquid distributor configurations can be used to provide an increased liquid surface area in contact with the gas phase as the crude acid solution flows to the liquid volume maintained above the catalyst bed. The aromatic polycarboxylic acid can be terephthalic acid.

An embodiment of the present invention comprises feeding the crude acid solution through the inlet into the bottom of the dissolution section of the purification reactor, wherein all of the crude acid solution flows along an extended flowpath to the overflow into the vertical conduit above the distributor tray and exceeds a minimum residence time of about
5 3.0 minutes or more in the gas-liquid contact area (comprising the dissolver or dissolution section) of the purification reactor. For example the minimum residence time in the dissolution section can be about 3.5 minutes or more, or about 4.0 minutes or more. The minimum residence time in the combined dissolution section and liquid level above the catalyst can be about 3.5 minutes or more, or 4.0 minutes or more. The configuration of the
10 inlet pipe comprises a bent pipe, aligned opening or other devices known to induce flow in the dissolver or dissolution section.

In another embodiment, additional liquid distributors are located at the top of the downcomer, such that the liquid sprays down the interior of the downcomer pipe to provide
15 additional surface contact area. In this case the liquid distributors can be pipes or troughs which are oriented across the top of the downcomer, each trough or pipe with a number of holes in its base, whereby the liquid flows into the troughs or pipes and then flows through the holes which effectively generate a spray of liquid in the downcomer.

Another embodiment of the present invention relates to the control of the crude acid solution level and the gas pressure in the purification reactor. The control of both parameters comprises maintaining the gas pressure within about 0.5 bar of the set pressure by modulating the flowrate of the purified acid solution out of the purification reactor. The variation in flowrate is restricted to maintain a minimum liquid level above the catalyst bed and typically
20 this is within the range -50% to +100% of the normal flowrate. In this way the control system maintains a steady liquid level and purification reactor pressure. Level can also be controlled by modulating the flow of hydrogen into the purification reactor. The present invention can be described as further comprising the step of e) controlling the reactor pressure and reaction solution level over the supported catalyst bed in the purification reactor
25 vessel by modulating the flow of liquid out of the purification reactor vessel; or e) controlling the reactor pressure and reaction solution level over the supported catalyst bed in the purification reactor vessel by modulating the flow of liquid out of the purification reactor vessel and the flow of hydrogen gas into the purification reactor vessel, or e) controlling the
30

reactor pressure and reaction solution level over the supported catalyst bed in the purification reactor vessel by modulating the flow of hydrogen gas into the purification reactor vessel, wherein the pressure of the hydrogen gas in the purification reactor vessel is greater than 60 barA.

5

Another embodiment of the present invention can be wherein step a) further comprises heating the crude aromatic polycarboxylic acid solution to a temperature in the range of from about 275°C to about 291°C before entering the purification reactor vessel, for example about 280°C to about 289°C before entering the purification reactor vessel, or to a temperature of at least 3°C above the temperature required to dissolve the crude aromatic polycarboxylic acid in water.

10

All embodiments of the present invention can further comprise the following. The purification reactor comprises four sections: an upper dissolution section; a downcomer and distribution tray; a controlled liquid volume that is directly above and completely submerges the final section, the supported catalyst bed. The aspect ratio of the purification reactor, defined as the ratio of the length of the vessel cylindrical section to the diameter of the catalyst bed is in a range of about 2 to about 4.5, for example about 3 to about 3.5. The hydrogen gas fed to the purification reactor comprises hydrogen in a range of about 90% v/v to about 99.9%v/v, particularly about 95 % v/v to about 99.9 % v/v. The gas fed to the purification reactor can comprise condensable components or inert components that do not react with the crude acid or supported catalyst. The gas fed to the purification reactor can be at a temperature of about 40°C to about 290°C. The gas is a pressurised gas at a pressure before the purification reactor of least 70 bara and typically 1-5 bar above the gas pressure inside the purification reactor, to enable accurate control of the gas pressure inside the purification reactor. Hydrogen gas can be used as the hydrogen source in the process of the invention and, in combination with the catalyst, hydrogenates of impurities in the crude acid. The hydrogen/ crude acid solution (reaction solution) is a homogeneous liquid phase prior to contact with the supported catalyst bed.

20
25
30

The reactor of the present invention is a continuous flow reactor. "Continuous flow reactor" as used herein is defined as a reactor in which reactants are introduced and mixed and products withdrawn simultaneously in a continuous manner, as opposed to a batch-type

reactor. Whilst a suitable configuration of the purification reactor is a vertical, cylindrical vessel of constant diameter each section of the purification reactor can have a different diameter.

5 The reaction time, defined as the time corresponding to the free volume in the catalyst bed divided by volumetric flowrate of the reaction solution through the catalyst bed, can be controlled so that the crude polycarboxylic acid is converted to the purified polycarboxylic acid with high efficiency such that the purified polycarboxylic acid precipitated from the reaction medium following completion of the purification reaction contains no more than
10 about 25 ppm, for example no more than about 15 ppm aldehyde produced as an intermediate in the course of the oxidation reaction (e.g. 4-CBA in the case of terephthalic acid production). Typically, there will be at least some aldehyde present after the reaction, and usually at least 5ppm. Similarly, the aldehyde intermediate generated in the course of the oxidation reaction is converted to the corresponding aromatic monocarboxylic acid (e.g. p-
15 toluic acid in the case of terephthalic acid production) with high efficiency such that the purified acid precipitated from the reaction medium following completion of the purification reaction contains no more than about 200 ppm, for example no more than about 150 ppm aromatic monocarboxylic acid. Typically, there will be at least some aromatic monocarboxylic acid present after the reaction, and usually at least 140 ppm.

20

The present invention can be better understood by reference to Figures 1 and 2. Figure 3 is included for comparison with the present invention.

Referring to Figure 1, a feed Stream 1 to the purification reactor Item A can comprise
25 a heated solution of crude acid in water as solvent. The composition of feed Stream 1 can comprise 20% w/w – 35% w/w crude acid in water, for example 27% w/w – 33% w/w. The feed Stream 1 can come from a slurry mix tank, via a preheater to heat the slurry in order to dissolve the crude acid. The feed Stream 1 is heated to a temperature in the range from about 275 °C to about 291 °C, for example from about 280 °C to about 289 °C. The required
30 temperature at this location is determined by the need to achieve a temperature margin over that temperature required to dissolve the carboxylic acid in water. Typically a temperature margin of 3-10°C is used, for example 4-7°C. The feed Stream 1 is fed to the purification reactor Item A through an inlet Item B. The outlet of inlet Item B is located close to the

bottom of the dissolution section Item C. The configuration if the inlet Pipe B is dependent on the construction of the purification reactor vessel and Figure 1 illustrates the downcomer Item D as an axial pipe in the centre of the vessel cross section. An embodiment comprises a dissolver section constructed as a sealed can of a cylinder and circular base with a second
5 cylinder, also orientated co-axially with the purification reactor vessel, acting as a downcomer in the upper section of the purification reactor vessel and attached at its bottom opening to the distributor tray Item E. The downcomer can form a pipe or annulus.

The outlet of inlet Item B is located close to the bottom of the dissolver section Item
10 C to ensure the residence time in the dissolver section Item C for all of the crude acid exceeds a minimum duration in the range from about 3 minutes to about 10 minutes, for example about 3.5 minutes to about 5.5 minutes, and ensures no circulating flow short-circuits the flowpath from the inlet to the overflow into the downcomer. An advantage of this configuration is to ensure any solids not dissolved in the water solvent after flowing through
15 the preheater, prior to the inlet into the purification reactor, are retained in the dissolver section for sufficient duration to totally dissolve to avoid blocking the catalyst bed.

The crude acid solution flows down the downcomer Item D from the dissolver section Item C onto the distributor Item E illustrated in Figure 2. The distributor Item E collects the
20 crude acid solution distributes it evenly as it continues flowing to the next section of the purification reactor and increases the liquid surface area to advantageously improve the dissolution of hydrogen gas into the crude acid solution. Figure 2 illustrates a circular distributor Item E comprising a tray perforated with a number of holes forming the base and a vertical rim with a number of notches spaced around the periphery of the distributor.

25 The crude acid solution including dissolved hydrogen flows from the distributor Item E as a number of separate flow streams onto the surface of a volume of the solution retained above the supported catalyst bed Item F. The depth of the liquid volume is measured by a liquid level instrument, for example a nucleonic gauge, available from Tracerco, Johnson
30 Matthey plc, Pavilion 11, Belasis Hall Technology Park, Coxwold Way, Billingham, Cleveland TS23 4EA, a float gauge, a conductivity probe or other suitable instrument. The depth of the volume of crude acid solution can also be controlled to a set level by adjusting the flow of hydrogen into the vessel. The depth of the volume of crude acid solution is in the

range about 0.5 metres to about 2.0 metres, for example about 0.8 metres to about 1.5 metres above the top surface of the catalyst bed Item F. The reaction pressure is tightly controlled by modulating the liquid flowrate out of the purification reactor. The two controllers are set to maintain a smaller variation in the purification reactor pressure than in the liquid level, to
5 avoid an unstable interaction between the separate control loops.

Maintaining a liquid level above the supported catalyst bed Item F ensures an evenly distributed flow of liquid through the supported catalyst bed Item F and advantageously reduces the volume of the supported catalyst bed Item F required for the conversion of the
10 crude acid solution by reaction with hydrogen in contact with the supported catalyst to the purified acid solution. A conventional purification reactor is illustrated in Figure 3 for comparison with Figure 1.

The process beyond the outlet Item G in Figure 1 can comprise a pipe connecting the
15 purification reactor outlet Item G to a flow modulating device, such as a flow control valve and flowing downstream to product crystallisation and recovery sections. The desired product, purified acid, can be recovered by causing or allowing the crystallisation from the solution in one or more stages followed by a solids-liquid separation in one or more stages.

The modulated flow of purified acid solution can flow to a product recovery section in
20 which the carboxylic acid can be precipitated from the solution. Any suitable method of product recovery can be used. The product recovery section can comprise one or more stages of cooling or evaporative crystallisation to crystallise the purified acid product to form a slurry of crystals in aqueous mother liquor. Where the product recovery section comprises
25 one or more flashing crystallisers, the resulting flash streams from the crystallisers can be used to preheat other streams in the manufacturing process, either indirectly via conventional heat exchangers or via direct injection of the flash into the process. The slurry obtained following crystallisation can be subjected to a solids-liquid separation process using for
30 example filtration devices operating under super-atmospheric, atmospheric or sub-atmospheric conditions, with or without washing facilities, such as supplied by Andritz or Bokela, or MKK. Thus, the solids-liquid separation can be carried out using any device suitable for this purpose and arranged to operate at conditions dependent on the pressure of the final crystallisation stage. The solids-liquid separation can be carried out using an

integrated solids separation and water washing apparatus such as a belt filter unit, a rotary cylindrical filter unit, or a drum filter unit (e.g. a BHS-Fest filter drum formed with a plurality of cells to receive the slurry and in which the mother liquor is displaced from filter cake by water under hydraulic pressure supplied to the cells). After filtering the slurry, the recovered carboxylic acid product can be used directly for the production of polyester, for instance, for packaging, such as bottles, or fibres. Similarly it can be dried. If not already at atmospheric pressure, the filter cake of carboxylic acid product can be transferred to a low pressure zone (e.g. atmospheric pressure) for drying via a suitable pressure let-down device.

Following recovery of the purified acid product, at least part of the aqueous mother liquor can be recycled for re-use in the purification process, e.g. by admixture with fresh water and/or the reactants. However, the mother liquor also contains valuable reaction intermediates that can be recovered to improve the product yield for the manufacturing process. For the manufacture of terephthalic acid these comprise p-toluic acid and 4-carboxybenzaldehyde. However, color-forming species and precursors and also reaction and degradation by-products are contained in the mother liquor and a purge may be taken in order to reduce standing concentrations of these components in the process. The purge stream can be passed to effluent treatment, e.g. aerobic and/or anaerobic processing or other recovery process.

Although the invention has been described mainly with reference to para-xylene as a precursor for terephthalic acid, it will be appreciated that other precursors can be employed instead or in addition to para-xylene for the production of the corresponding carboxylic acid. Such precursors include polyalkyl aromatic hydrocarbons such as m-xylene, trialkylbenzenes or dialkyl naphthalenes for the production of aromatic carboxylic acids such as isophthalic acid and naphthalene dicarboxylic acid respectively. The invention is further illustrated below by the following non-limiting Examples.

EXAMPLES

The following examples contain data from different configurations of a purification reactor. The Comparative Example demonstrates a conventional reactor design as disclosed in

Figure 3. Examples 1 and 2 are embodiments of a purification reactor of the present invention to demonstrate the improvements in catalyst life and catalyst productivity.

5	<u>Comparative Example</u>	Conventional reactor design (Figure 3)
	Reactor diameter	3.145 m
	Capacity	52 te/h PTA capacity
	Gas-Liquid contact area	23.4 m ²
	Gas-Liquid contact area : reactor diameter	0.45 m ² /te/h PTA
10	Reactor catalyst charge	21 te
	Catalyst life	12 months
	Catalyst productivity	25,000 te PTA/te catalyst
	<u>Example 1</u>	
15	Reactor diameter	3.300 m
	Capacity	82 te/h PTA capacity
	Gas-Liquid contact area	
	In dissolver, downcomer and distributor	52 m ²
	Gas-Liquid contact area : reactor diameter	0.63 m ² /te/h PTA
20	Reactor catalyst charge	20 - 24 te
	Catalyst life	18-24 months
	Catalyst productivity	50,000 te PTA/te catalyst
	<u>Example 2</u>	
25	Reactor diameter	3.600 m
	Capacity	90 te/h PTA capacity
	Gas-Liquid contact area	
	In dissolver, downcomer and distributor	67 m ²
	Gas-Liquid contact area : reactor diameter	0.744 m ² /te/h PTA
30	Reactor surface	10.2 m ²
	Reactor catalyst charge	24 - 28 te
	Catalyst life	24 months
	Catalyst productivity	60,000 te PTA/te catalyst

While the invention has been described in conjunction with specific embodiments thereof, it is evident that the many alternatives, modifications, and variations will be apparent 5 to those skilled in the art in light of the foregoing description. Accordingly, the invention is intended to embrace all such alternatives, modifications and variations as fall within the spirit and scope of the claims.

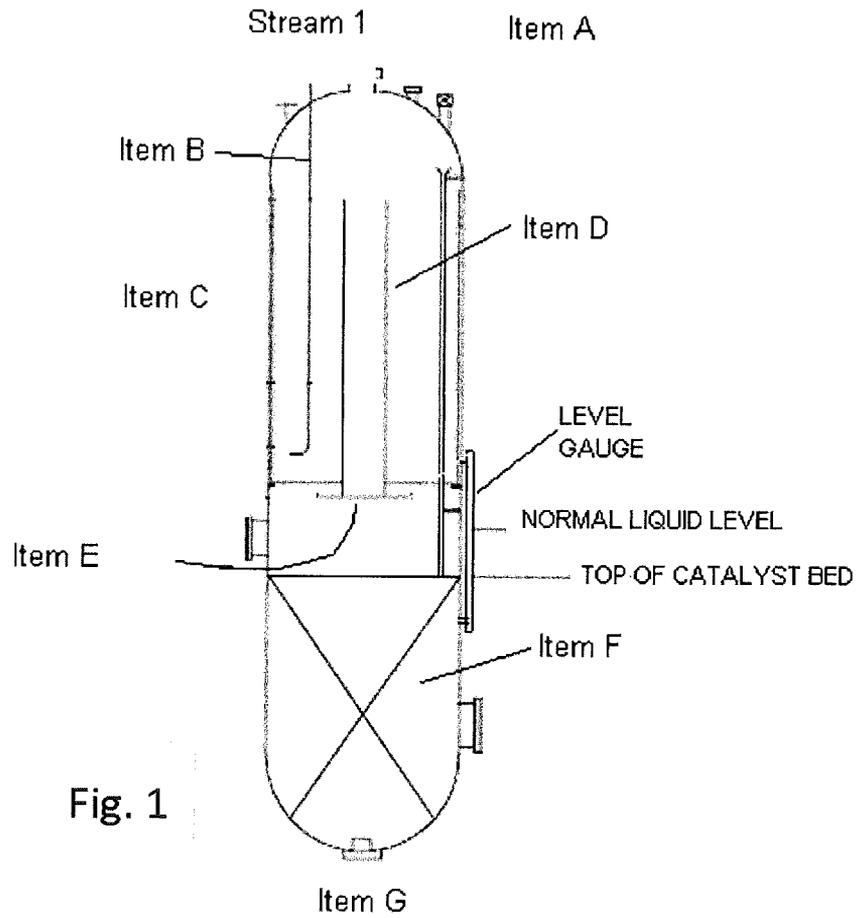
What is claimed is:

1. A process for the production of an aromatic carboxylic acid comprising:
 - a) introducing a crude aromatic carboxylic acid solution into a purification reactor vessel, wherein the purification reactor vessel is operating under pressure,
 - b) introducing hydrogen gas into the purification reactor vessel,
 - c) dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution as the solution flows down a wall of a vertical conduit onto a distributor, wherein the purification reactor vessel has a gas-liquid contact area to plant throughput ratio of at least $0.55 \text{ m}^2/\text{te/h}$ of carboxylic acid for dissolving the hydrogen gas in the crude aromatic polycarboxylic acid solution to produce a reaction solution, and
 - d) contacting the reaction solution with a supported catalyst bed to produce a purified aromatic carboxylic acid, wherein the supported catalyst bed is submerged in the reaction solution and a liquid level of the reaction solution is maintained above the supported catalyst bed.
2. The process of claim 1, wherein the gas-liquid contact area to plant throughput ratio of step c) is about $0.65 \text{ m}^2/\text{te/h}$ carboxylic acid or more.
3. The process of claim 1, wherein the gas-liquid contact area to plant throughput ratio of step c) is about $0.75 \text{ m}^2/\text{te/h}$ of carboxylic acid or more.
4. The process of claim 1, wherein the gas-liquid contact area to plant throughput ratio of step c) is about $1 \text{ m}^2/\text{te/h}$ of carboxylic acid or more.
5. The process of claim 1, wherein the distributor comprises i) a perforated circular disc with a diameter about 0.3 to about 0.7 of the purification reactor vessel diameter, ii) notched weir openings arranged around the rim of the plate to retain the crude acid solution on the plate, and iii) at least one hole through which the crude aromatic polycarboxylic acid solution flows onto the liquid surface below the distributor.

6. The process of claim 1, wherein the distributor comprises i) a perforated circular open pipe or channel with a diameter about 0.5 to about 0.9 of the purification reactor vessel diameter, ii) notched weir openings arranged around the rim of the ring to retain the crude acid solution on the plate, and iii) at least one hole through which the crude aromatic polycarboxylic acid solution flows onto the liquid surface below the distributor.
7. The process of claim 5 or 6, wherein the distributor is located about 0.5 to about 2 meters above the liquid level of the reaction solution above the supported catalyst bed.
8. The process of claim 5 or 6, wherein the distributor is located between about 0.5 to about 1 meter above the liquid level of the reaction solution above the supported catalyst bed.
9. The process of claim 1, wherein the residence time of the solution in the gas-liquid contact area is at about 3 minutes or more.
10. The process of claim 1, further comprising the step of e) controlling the reactor pressure and reaction solution level over the supported catalyst bed in the purification reactor vessel by modulating the flow of liquid out of the purification reactor vessel.
11. The process of claim 1, further comprising the step of e) controlling the reactor pressure and reaction solution level over the supported catalyst bed in the purification reactor vessel by modulating the flow of liquid out of the purification reactor vessel and the flow of hydrogen gas into the purification reactor vessel.
12. The process of claim 1, further comprising the step of e) controlling the reactor pressure and reaction solution level over the supported catalyst bed in the purification reactor vessel by modulating the flow of hydrogen gas into the purification reactor vessel, wherein the pressure of the hydrogen gas in the purification reactor vessel is greater than 60 barA.

13. The process of claim 1, wherein step a) further comprises heating the crude aromatic polycarboxylic acid solution to a temperature in the range of from about 275°C to about 291°C before entering the purification reactor vessel.
14. The process of claim 1, wherein step a) further comprises heating the crude aromatic polycarboxylic acid solution to a temperature in the range of from about 280°C to about 289°C before entering the purification reactor vessel.
15. The process of claim 1, wherein step a) further comprises heating the crude aromatic polycarboxylic acid solution to a temperature of at least 3°C above the temperature required to dissolve the crude aromatic polycarboxylic acid in water.
16. The process of claim 1, wherein the aromatic polycarboxylic acid is terephthalic acid.

1/3



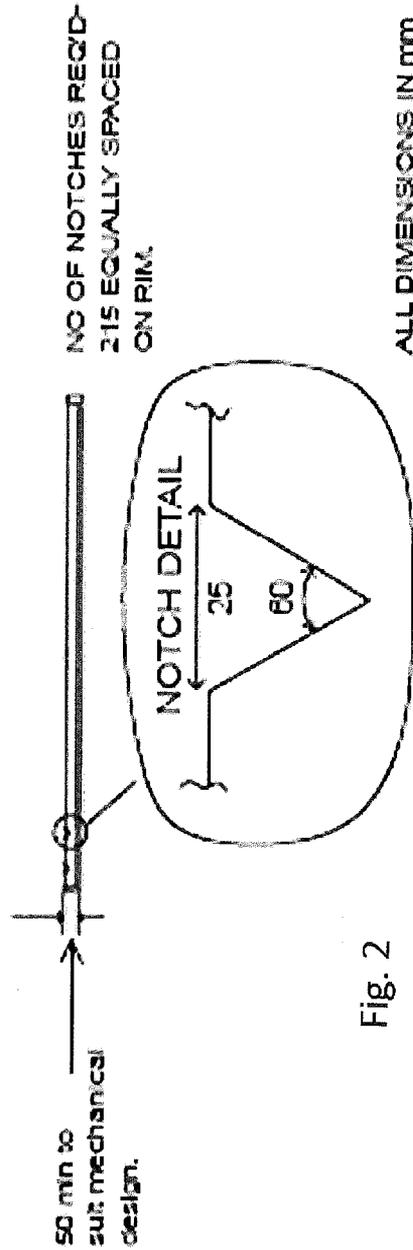
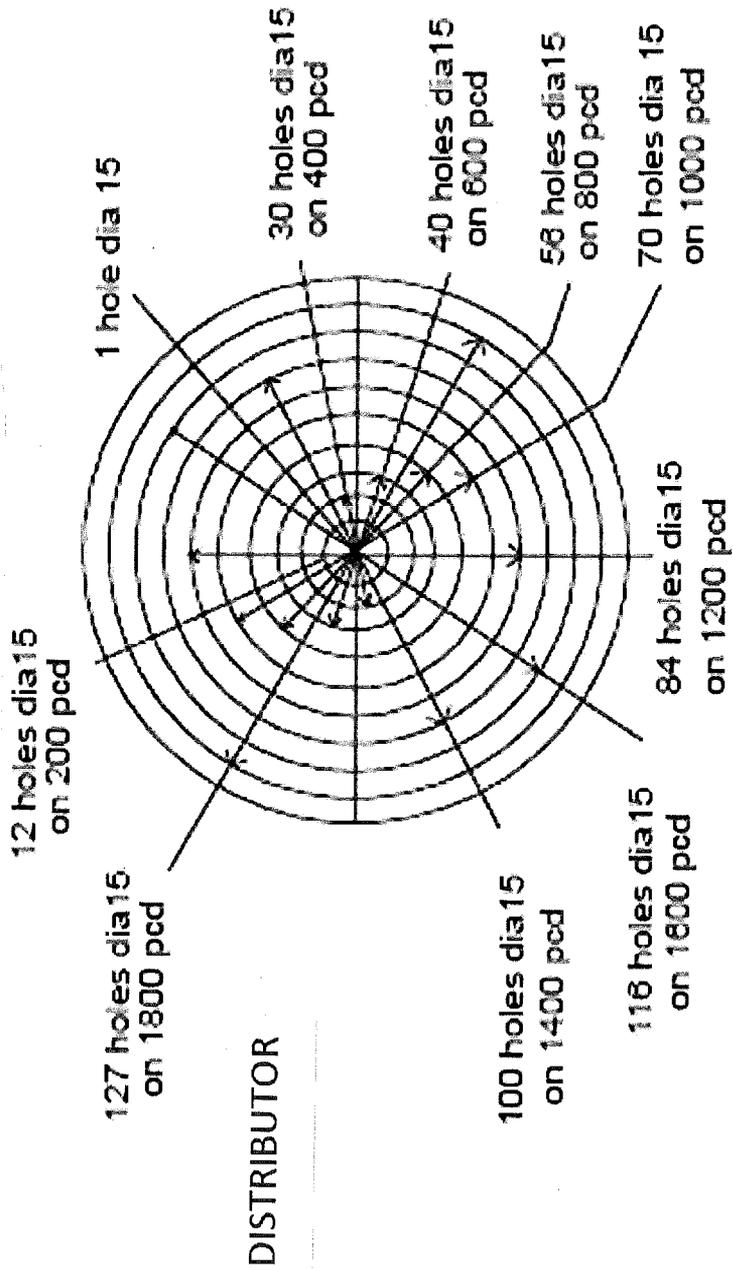


Fig. 2

ALL DIMENSIONS IN mm

MINIMUM THICKNESS OF DISTRIBUTOR PLATE TO BE 10mm

3/3

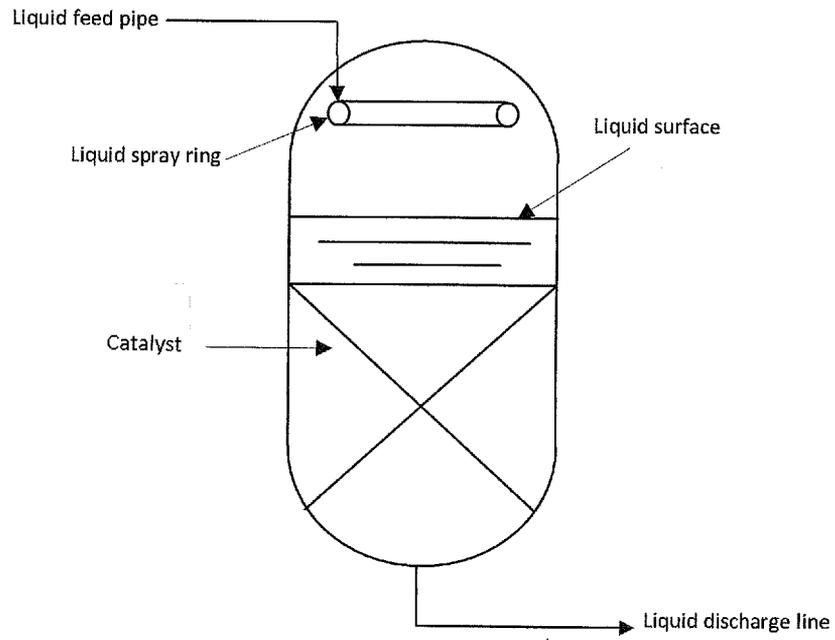


Fig. 3