

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property

Organization

International Bureau

(43) International Publication Date

29 July 2021 (29.07.2021)



(10) International Publication Number

WO 2021/151071 A1

(51) International Patent Classification:

C08J 11/24 (2006.01)

C08L 67/00 (2006.01)

C08J 11/16 (2006.01)

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

(21) International Application Number:

PCT/US2021/014896

(22) International Filing Date:

25 January 2021 (25.01.2021)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

62/964,948

23 January 2020 (23.01.2020) US

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

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

Declarations under Rule 4.17:

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

Published:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(54) Title: PROCESS AND SYSTEM FOR DEPOLYMERIZING WASTE PLASTIC

(57) Abstract: A continuous flow process and system for depolymerizing plastic. A heterogeneous mixture of plastic particles, a solvent, and a catalyst are pumped continuously through a heating zone at a flow rate just high enough to maintain a particle speed great enough to keep the plastic particles in suspension. The temperature of the heterogeneous mixture is raised in the heating zone and maintained in a hold zone to complete depolymerization of the mixture into a homogeneous solution containing a liquefied reaction product. The homogeneous solution is cooled to solidify and precipitate a solid reaction product. The solid reaction product is separated from the solvent to be recycled. The solvent is recirculated to be reused as a constituent of the heterogeneous mixture.



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PROCESS AND SYSTEM FOR DEPOLYMERIZING WASTE PLASTIC

CROSS-REFERENCE TO RELATED APPLICATION

This application claims priority to U.S. Provisional Application No. 62/964,948, filed on January 23, 2020, which is hereby incorporated by reference.

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BACKGROUND

The invention relates generally to the depolymerization of resin, plastic, or polymer. More particularly, it relates to the depolymerization of waste plastic in a continuous process.

Plastic is conventionally depolymerized in large reaction vessels usually equipped with a heating jacket and an agitator. The depolymerization reaction is sequestered in the vessel until depolymerization is complete. After depolymerization the vessel is emptied and then refilled. Each batch is heated to speed up depolymerization and then cooled to produce viable raw material for new polymers. The batch process typically takes between 20 min and 800 min. Continuous operation is simulated by sequentially emptying and refilling a group of reaction vessels in round-robin fashion. The constant need to fill, heat, cool, empty, and repeat wastes energy and requires additional equipment to maintain the illusion of actual continuous flow in a parallel batch process.

SUMMARY

A process embodying features of the invention for depolymerizing plastic comprises: (a) continuously flowing a mixture containing solid plastic particles in a solvent through a line in a heating chamber at a particle speed great enough to maintain the plastic particles suspended in the solvent and prevent the plastic particles from agglomerating and clogging the line; and (b) transferring heat through the line in the heating chamber to heat the mixture to a reaction temperature to start the depolymerization of the plastic particles in the solvent into a homogeneous solution including a liquefied reaction product.

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A system embodying features of the invention for the continuous depolymerization of plastic comprises a pump operating at a pump flow rate and a line through which the pump continuously feeds a heterogeneous mixture including particles of plastic in a solvent at a particle speed. A heating zone raises the temperature of the heterogeneous mixture flowing through the line to a reaction temperature of at least 150°C. The conversion of the heterogeneous mixture containing the plastic particles into a homogeneous solution

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containing a liquefied reaction product including monomer, dimer, oligomers and/or reaction side-products is started in the heating zone.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a block diagram of a system embodying features of the invention for depolymerizing plastic.

FIG. 2 is a flowchart showing the progression of a volume of plastic undergoing a depolymerization process in the system of FIG. 1.

DETAILED DESCRIPTION

A system and a process for depolymerizing plastic are shown in FIGS. 1 and 2. The system and process may be used with various plastics such as, but not limited to, PET, modified PET, PET blends, PEN, PBT, PET-G, PLA, PGA, PLGA, PEF, copolyesters, polycarbonates, polyamides (Nylon), polyurethanes and combinations and blends. Depolymerization of the plastic into the following, but not limited to: (bis (2-hydroxyethyl) terephthalate (BHET), dimethyl terephthalate (DMT), terephthalic acid (TA), (bis (2-hydroxyethyl) naphthalate (BHEN), (bis (2-hydroxyethyl) Furanoate (BHEF) their respective oligomers, acids, half-esters, or mixed esters. Additionally, chemically useful compounds such as dioctyl terephthalate (DOTP), diisobutyl terephthalate (DITP), dibutyl terephthalate (DBTP), bisphenol A (BPA), lactates, bis (2-hydroxyethyl) terephthalamide (BHETA), and other terephthalamides.

Solid plastic particles of waste polyester material, in the form of flakes, fines, grain, granules, granola, lumps, chunks, and/or powder, are mixed with a solvent and a catalyst in a mixer 10 to produce a heterogeneous mixture 12. The mixer 10 can use an agitator, such as a propeller 13, stirrer, or other agitator or a recirculating solvent to do the mixing. Or the mixture can be premixed. Examples of solvents are, but not limited to, ethylene glycol (EG), diethylene glycol (DEG), glycol ethers, methanol, ethanol, propanol, butanol, 2-ethyl hexanol, tetramethyl cyclobutanediol (CBDM), cyclohexanedimethanol (CHDM), alcohols, ethanol amine, ionic liquids, polar protic solvents, polar aprotic solvents, and water. Examples of suitable catalysts include but not limited to: zinc salts, zinc acetate; zinc chloride; titanium salts; manganese salts; magnesium salts; sodium hydroxide; potassium hydroxide; 1, 5, 7-Triazabicyclo [4.4.0] dec-5-ene (TBD); 1, 8-Diazabicyclo [5.4.0] undec-7-ene (DBU); magnesium acetate, 4-dimethylaminopyridine (DMAP); amine; trialkyl amine; and combinations of those catalysts. The heterogeneous mixture 12 is pumped through a

series of connected lines, such as tubes or pipes, by a pump 14. No agitator, auger, or extruder is needed to advance the mixture through the system. The pump 14 operates at a flow rate great enough to move the mixture 12 through the system with a particle speed great enough to maintain the particles suspended in the solvent and to prevent the particles from agglomerating and clogging the lines. By operating continuously without stopping, the pump 14 flows the heterogeneous mixture through the system at a steady rate that makes the conversion of plastic into liquified product a function of position within the system rather than a function of time—as in batch systems.

An optional preheating heat exchanger (preheater) 16 is used to preheat the heterogeneous mixture 12. The preheater 16 can heat the heterogeneous mixture 12 by a heat source, such as a flame, steam, hot-oil or a circulated heat transfer fluid. Or the hot homogeneous solution containing the liquified product after the depolymerization reaction can be used in the preheater 16 to transfer heat to the heterogeneous mixture and, in the process, cool itself down.

The preheated heterogeneous mixture 12' flows continuously into and through a downstream heating chamber 18 in which depolymerization starts. The heating chamber 18 may be realized as a reactor heat exchanger that raises the temperature of the heterogeneous mixture to a reaction temperature of at least 150°C. The heterogeneous mixture is heated in the reaction heat exchanger 18 by a heat source 20. The heat source 20 may directly heat the heterogeneous mixture with microwave radiation, direct flame, electrically heated pipe, inductively heated pipe, geothermal, magnon-drag thermoelectricity, or ohmically, as a few examples. Or the heat source 20 may indirectly heat the heterogeneous mixture by directly heating a heat transfer fluid external to the heating chamber 18. Examples of suitable transfer fluids are hot oil, a thermal fluid, a molten salt, and steam. The heated heat transfer fluid is then pumped past the line containing the heterogeneous mixture in the heating chamber 18. Heat is transferred from the heat transfer fluid to the heterogeneous mixture to start depolymerization. The heterogeneous mixture flowing through the heating chamber 18 is not contacted directly by the heat transfer fluid.

A hold tube 22 after the heating chamber 18 maintains the reaction temperature for at least one minute to complete the conversion of the heterogeneous mixture containing plastic to a homogeneous solution 24 containing the liquified product. The hold tube 22 may be realized by an insulated spool or coil of pipe or tube or as a jacketed pipe or vessel. Or the hold tube can be part of the heating chamber rather than a stand-alone component. The reaction is completed in the hold tube. The exiting homogeneous solution contains the

solvent, the spent catalyst, and depolymerized plastic in the form of a liquefied reaction product that typically includes monomers, oligomers, and/or minor side-products from the reaction (e.g. half-esters, half-amides, mixed esters, mixed amides).

The homogeneous solution 24 is pumped continuously through the optional preheating heat exchanger 16 to cool itself and preheat the incoming heterogeneous mixture 12. A backpressure regulator 26 maintains a system pressure, e.g., 100 psi to 400 psi, above the vapor pressure of the solvent at the reaction temperature.

After flowing through the backpressure regulator 26, the homogeneous solution 24 flows through an optional chilling heat exchanger (chiller) 28 that uses cold water or other cooling heat transfer fluid from a chilled reservoir 30 to remove any excess heat that the preheater 16 did not reclaim.

After the solution is cooled, it is poured into precipitation or crystallization tanks and cooled until the liquefied product precipitates as a solid reaction product 34. The solvent is then decanted, filtered, centrifuged or distilled away from the solid reaction product. The solid reaction product may be subsequently filter-pressed to further separate it from any remaining solvent. The decanting, filtration, centrifugation or distillation of solvent, followed by the pressing to separate the solid reaction product 34 in the solution 24 from the solvent 36 is represented in FIG. 1 by a separator 32.

The separated solvent 36 is recirculated back to the mixer 10 for reuse. An optional solvent cleaning, purification or regeneration step may be required to remove reaction contaminants from the solvent feeding the subsequent heterogeneous mixture 12. Reaction contaminants may include particulate, ionic salts, anions, cations, spent catalyst, dyes, adhesives, components from blends, fillers and/or decomposed solvent. Contamination removal 42 may occur by passing the separated solvent 36 through filters and/or over sorbents such as activated charcoal, ion exchange resin, diatomaceous earth, sand, zeolites, clay, silica, alumina, oxides, size exclusion and/or tangential flow filtration. Contamination removal 42 of solvent 36 may be an in-line or off-line process. Contamination removal 42 may occur at the separated solvent step 36 or at the homogeneous solution step 24.

Thus, the system moves the heterogeneous mixture 12 through four zones: Z1 – a cold entry zone in which the mixture is fed into the system by the pump 14; Z2 – a preheating zone in which the mixture is heated in the preheater 16; Z3 – a heating zone in which the mixture is heated to raise its temperature to the reaction temperature; and Z4 - a hold zone in which the mixture is maintained at the reaction temperature to complete the conversion of the heterogeneous mixture into the homogeneous solution 24. The homogeneous solution 24 is

moved through a cooling zone Z5 in which the homogeneous solution is cooled in the chiller 28 or by the transfer of heat to the incoming heterogeneous mixture 12 in the preheater 16.

The pump 14 maintains a continuous flow rate through the system that ensures a particle speed of the heterogeneous mixture great enough to keep the particles in suspension. In that way the plastic particles do not settle in the lines and clog the system.

The size of plastic particles pumped through the system can vary, but they are typically between 0.1 μm and 20,000 μm in at least one dimension. To maintain the particles in suspension, the flow rate of the pump 14 is set to ensure a particle speed of at least 20 cm/s through the system. Particle speeds above 20 cm/s or 30 cm/s provide a safety margin. The pump flow rate is set equal to the product of the desired particle speed and the cross-sectional area of the lines (pipes or tubes) through which the mixture is pumped. If mixers are installed in the lines between the pump 14 and the regulator 26, lower particle speeds are possible.

In the heating zone Z3, the heating chamber 18 raises the temperature to the reaction temperature or higher to start the depolymerization reaction, which is completed in the hold zone Z4. The length L of the hold tube 22 in the hold zone Z4 depends on its cross-sectional area A, the pump's flow rate Q, and the hold time T required at the reaction temperature to complete the reaction: $L = QT/A$. The hold time can range from 5 min to 10 min or even from 1 min to 60 min. The diameter of the lines running through the zones is 1 cm to 10 cm, but can be as great as 100 cm. If jacketed piping is used, the diameter of the jacket may range from 1.1 to 5.0 times the diameter of the inner pipe through which the mixture is pumped.

WHAT IS CLAIMED IS:

1. A continuous flow process for depolymerizing plastic, the process comprising:
 - (a) continuously flowing a mixture containing solid plastic particles in a solvent through a line in a heating chamber at a particle speed sufficient to maintain suspension
5 of the plastic particles in the solvent and to prevent the plastic particles from agglomerating and clogging the line, wherein the solid plastic particles consist of modified PET, PET blends, PEN, PBT, PET-G, PLA, PGA, PLGA, PEF, copolyesters, polycarbonates, polyamides, polyurethanes, or any combination thereof;
 - (b) transferring heat through the line in the heating chamber to heat the mixture to a
10 reaction temperature to start the depolymerization of the plastic particles in the solvent into a homogeneous solution including a liquefied reaction product.
2. The continuous flow process of claim 1, wherein the solvent consists of ethylene glycol, diethylene glycol, glycol ethers, methanol, ethanol, propanol, butanol, 2-ethyl hexanol, tetramethyl cyclobutanediol, cyclohexanedimethanol, alcohols, ethanol amine, ionic
15 liquids, polar protic solvents, polar aprotic solvents, water, or any combination thereof.
3. The continuous flow process of claim 1, wherein the liquefied reaction product includes monomers, dimers, or oligomers.
4. The continuous flow process of claim 1, wherein the liquefied reaction product includes (bis (2-hydroxyethyl) terephthalate, dimethyl terephthalate, terephthalic acid, (bis (2-
20 hydroxyethyl) naphthalate, (bis (2-hydroxyethyl) Furanoate, their respective oligomers, acids, half-esters, mixed esters, dioctyl terephthalate, diisobutyl terephthalate, dibutyl terephthalate, bisphenol A, lactates, bis (2-hydroxyethyl) terephthalamide, other terephthalamides, or any combination thereof.
5. The continuous flow process of claim 1, wherein the reaction temperature is at least 150°C.
- 25 6. The continuous flow process of claim 1, wherein step (b) further includes holding the mixture at the reaction temperature for at least one minute.
7. The continuous flow process of claim 1, wherein step (a) further includes preheating the mixture in a preheating heat exchanger before flowing it into the heating chamber.
8. The continuous flow process of claim 7, further comprising:

(c) flowing the homogeneous solution through a passage in the preheating heat exchanger after the homogeneous solution exits the heating chamber, wherein the homogeneous solution transfers heat to the mixture in the preheating heat exchanger.

9. The continuous flow process of claim 1, further comprising maintaining a system
5 pressure above the vapor pressure of the solvent at the reaction temperature to prevent the solvent from evaporating.
10. The continuous flow process of claim 1, wherein step (a) further includes mixing the solid plastic particles and the solvent with a catalyst to form the mixture.
11. The continuous flow process of claim 10, wherein the catalyst consists of zinc salts, zinc
10 acetate, zinc chloride, titanium salts, titanium (IV) isopropoxide, titanium (IV) n-butoxide, manganese salts, magnesium salts, sodium hydroxide, potassium hydroxide, 1, 5, 7-Triazabicyclo [4.4.0] dec-5-ene, 1, 8-Diazabicyclo [5.4.0] undec-7-ene, magnesium acetate, 4-dimethylaminopyridine, amine, trialkyl amine, or any combination thereof.
12. The continuous flow process of claim 1, further comprising:
15 (c) cooling the homogeneous solution in a chilling heat exchanger to a temperature below 50°C.
13. The continuous flow process of claim 12, further comprising:
(d) settling the cooled homogeneous solution at room temperature for a time between
about 0.5 hour and 100 hours to allow the liquefied reaction product to solidify into a solid
20 reaction product.
14. The continuous flow process of claim 13, further comprising:
(e) separating the solid reaction product from the solvent by one or more of decanting, filtering, centrifuging, pressing, and distillation.
15. The continuous flow process of claim 14, further comprising:
25 (f) reusing the solvent separated from the solid reaction product in the process.
16. The continuous flow process of claim 1, further comprising:
(c) separating the solvent from the reaction product;
(d) removing contaminants from the solvent by filtration, sorbents, or a combination thereof; and

(e) reusing the solvent in the process by mixing solid plastic particles in the reused solvent to form the mixture.

17. The continuous flow process of claim 16, wherein the sorbent consists of activated charcoal, ion exchange resin, diatomaceous earth, sand, zeolites, clay, silica, alumina, oxides, or any combination thereof.

18. The continuous flow process of claim 1, wherein the plastic particles have a size between 0.1 μm and 20,000 μm in at least one dimension.

19. The continuous flow process of claim 1, wherein the solid plastic particles are in the form of flakes, fines, grain, granules, granola, lumps, chunks, powder, or any combination thereof.

20. The continuous flow process of claim 1, wherein the particle speed is at least 30 cm/s through the line.

21. The continuous flow process of claim 1, wherein in step (b) the mixture is indirectly heated in the line of the heating chamber by pumping a hot heat transfer fluid past the line.

22. A system for continuous depolymerization of plastic, comprising:

a pump operating at a flow rate;

a line through which the pump continuously feeds a heterogeneous mixture including solid plastic particles in a solvent at a particle speed, wherein the solid plastic particles consist of modified PET, PET blends, PEN, PBT, PET-G, PLA, PGA, PLGA, PEF, copolyesters, polycarbonates, polyamides, polyurethanes, or any combination thereof;

a heating zone raising the temperature of the heterogeneous mixture flowing through the line to a reaction temperature of at least 150°C;

wherein the conversion of the heterogeneous mixture containing the solid plastic particles into a homogeneous solution containing a liquefied reaction product is started in the heating zone.

23. The system of claim 22, a hold tube receiving the heated heterogeneous mixture from the heating zone to maintain the reaction temperature for a hold time of at least one minute at

the flow rate to completely convert the heterogeneous mixture containing the solid plastic particles into the homogeneous solution containing the liquefied reaction product.

24. The system of claim 23, wherein the hold tube is an insulated pipe or tubing.

25. The system of claim 24, wherein the length of the hold tube is long enough to ensure that the conversion of the heterogeneous mixture into the homogeneous solution containing liquefied reaction product is complete.

26. The system of claim 25, wherein the hold time in the hold tube is between 1 minute and 60 minutes.

27. The system of claim 25, wherein the hold time in the hold tube is between 5 minutes and 10 minutes.

28. The system of claim 22, further comprising a mixer upstream of the heating zone using an agitator or recirculating solvent to stir the heterogeneous mixture.

29. The system of claim 22, wherein the heterogeneous mixture includes the solid plastic particles, the solvent, and a catalyst.

30. The system of claim 22, wherein the solid plastic particles have a size between 0.1 μm and 20,000 μm in at least one dimension.

31. The system of claim 22, wherein the pump maintains the flow rate so that the particle speed of the solid plastic particles exceeds 30 cm/s.

32. The system of claim 31, wherein the flow rate is set equal to the product of the desired particle speed and the cross-sectional area of the line.

33. The system of claim 22, further comprising a preheating heat exchanger that preheats the heterogeneous mixture indirectly with the homogeneous solution containing the liquefied reaction product to decrease a hold time of the heterogeneous mixture in the heating zone and to cool the homogeneous solution.

34. The system of claim 22, further comprising a reactor heat exchanger in the heating zone that raises the temperature of the heterogeneous mixture to the reaction temperature.

35. The system of claim 34, wherein a heat source is configured to heat a heat transfer fluid that flows past the heterogeneous mixture in the reactor heat exchanger to transfer heat to the heterogeneous mixture.
36. The system of claim 22, further comprising a back-pressure regulator downstream of the heating zone that maintains a system pressure above the vapor pressure of the solvent at the reaction temperature.
37. The system of claim 22, further comprising a chiller downstream of the heating zone and including a chilling heat exchanger with the homogeneous solution on one side cooled indirectly by a cold liquid on the other side of the heat exchanger, wherein the chiller lowers the temperature of the homogeneous solution to below 50°C
38. The system of claim 22, further comprising a separator that includes a precipitation or crystallization tank in which the liquefied reaction product in the homogeneous solution solidifies into a solid reaction product and precipitates.
39. The system of claim 38, wherein the separator separates the solvent from the solid reaction product by one or more of decanting, filtering, centrifuging, pressing, and distillation.
40. The system of claim 38, further comprising a contaminant removal portion downstream of the separator, wherein the contaminant removal portion removes reaction contaminants from the solvent, and wherein the contaminant removal portion includes a filter or a sorbent.
41. The system of claim 40, wherein the filter includes a size exclusion filter or a tangential flow filter.
42. The system of claim 40, wherein the sorbent consists of activated charcoal, ion exchange resin, diatomaceous earth, sand, zeolites, clay, silica, alumina, oxides, or any combination thereof.
43. The system of claim 22, further comprising a preheater upstream of the heating zone and a hold tube after the heating zone; wherein the preheater, the heating zone, and the hold tube each include a heat exchanger.

44. The system of claim 43, wherein each heat exchanger is a tube-in-shell, tubes-in-shell, coil-in-shell, tube-in-tube, jacketed piping, platular, plate-and-shell, or plate-and-frame heat exchanger.
- 5 45. The system of claim 22, further comprising a preheater upstream of the heating zone and a hold tube after the heating zone; wherein the preheater, the heating zone, and the hold tube include multiple lengths of jacketed piping having a jacket around an inner pipe, wherein the jacketed piping is connected.
- 10 46. The system of claim 45, wherein the inner diameter of the inner pipe in the jacketed piping is between 1 cm and 100 cm and the diameter of the jacket is between 1.1 and 5.0 times the diameter of the inner pipe.

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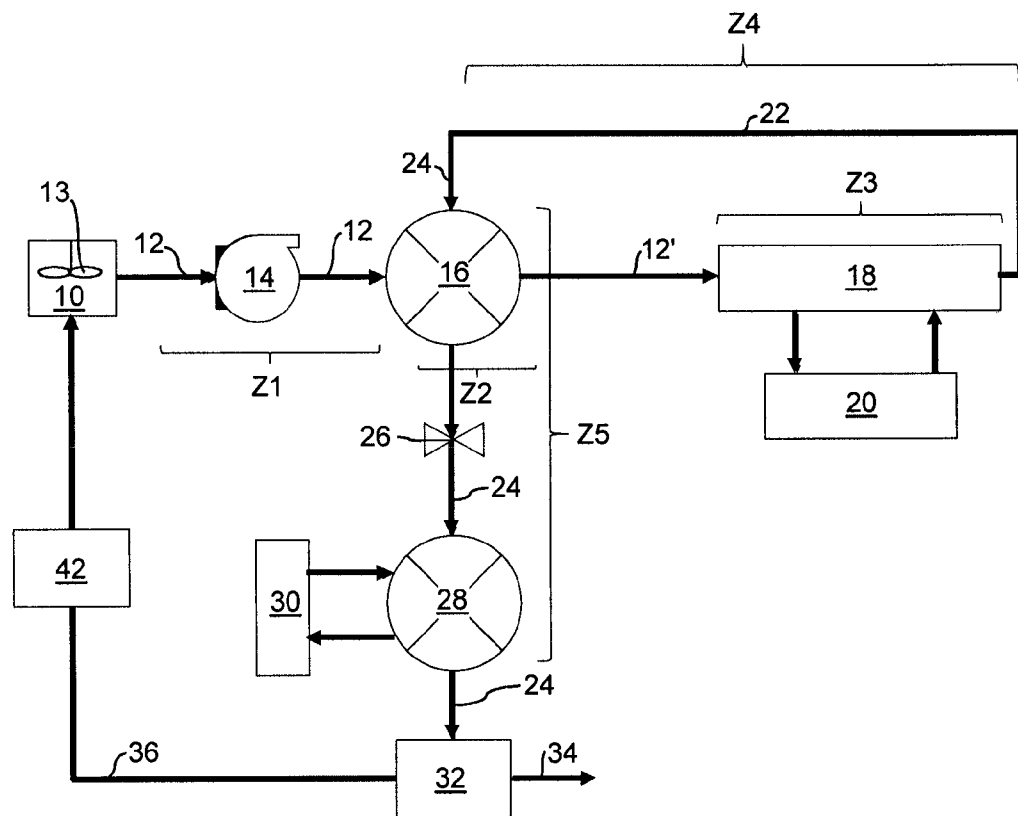


FIG. 1

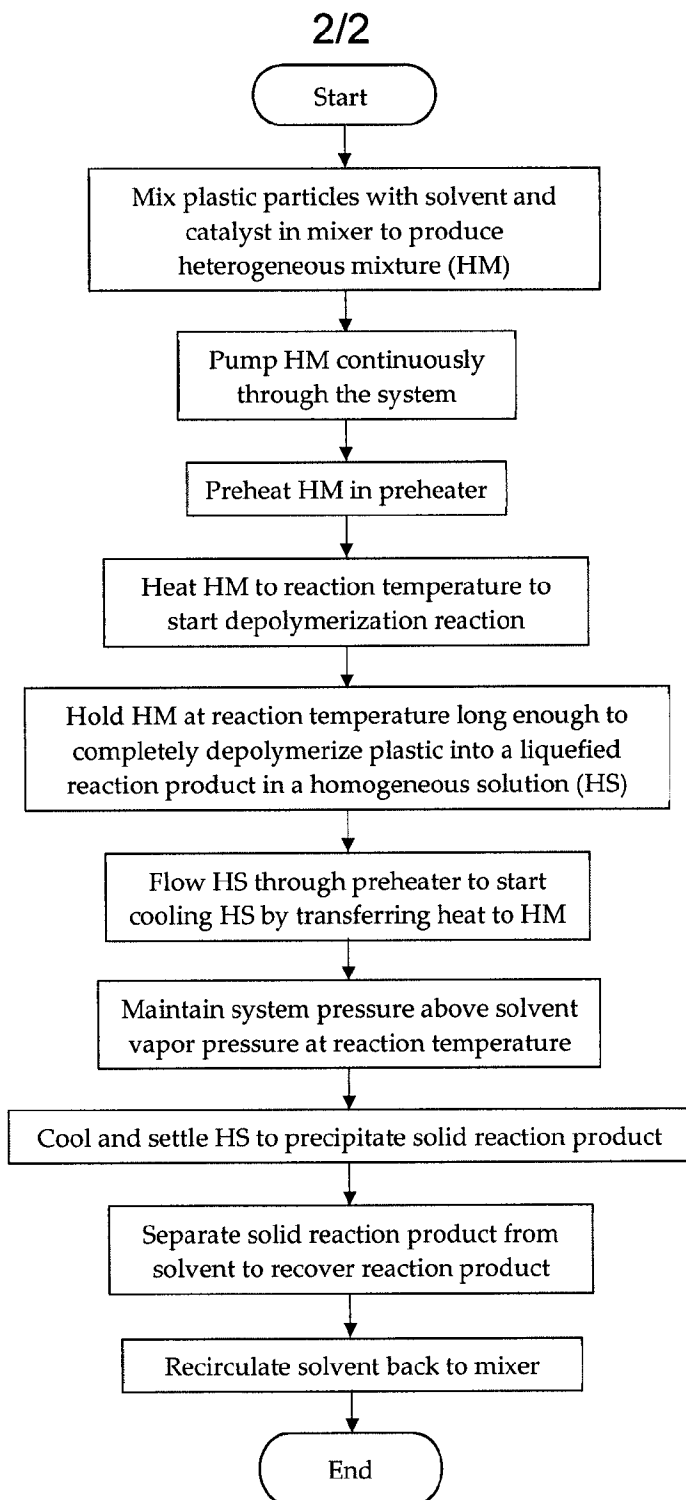


FIG. 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2021/014896

A. CLASSIFICATION OF SUBJECT MATTER

INV. C08J11/24 C08J11/16
ADD. C08L67/00

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)
C08J

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 2017/087752 A1 (UNIV NORTH CAROLINA CHAPEL HILL [US]) 26 May 2017 (2017-05-26) claims 1,4,11,12	1-46
A	EP 1 134 211 A1 (BROCCATELLI MASSIMO [IT]) 19 September 2001 (2001-09-19) claims 1,12,13,18	1-46



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search

28 May 2021

Date of mailing of the international search report

09/06/2021

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2021/014896

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