Sept. 23, 1969 H. ROTHERT 3,468,849

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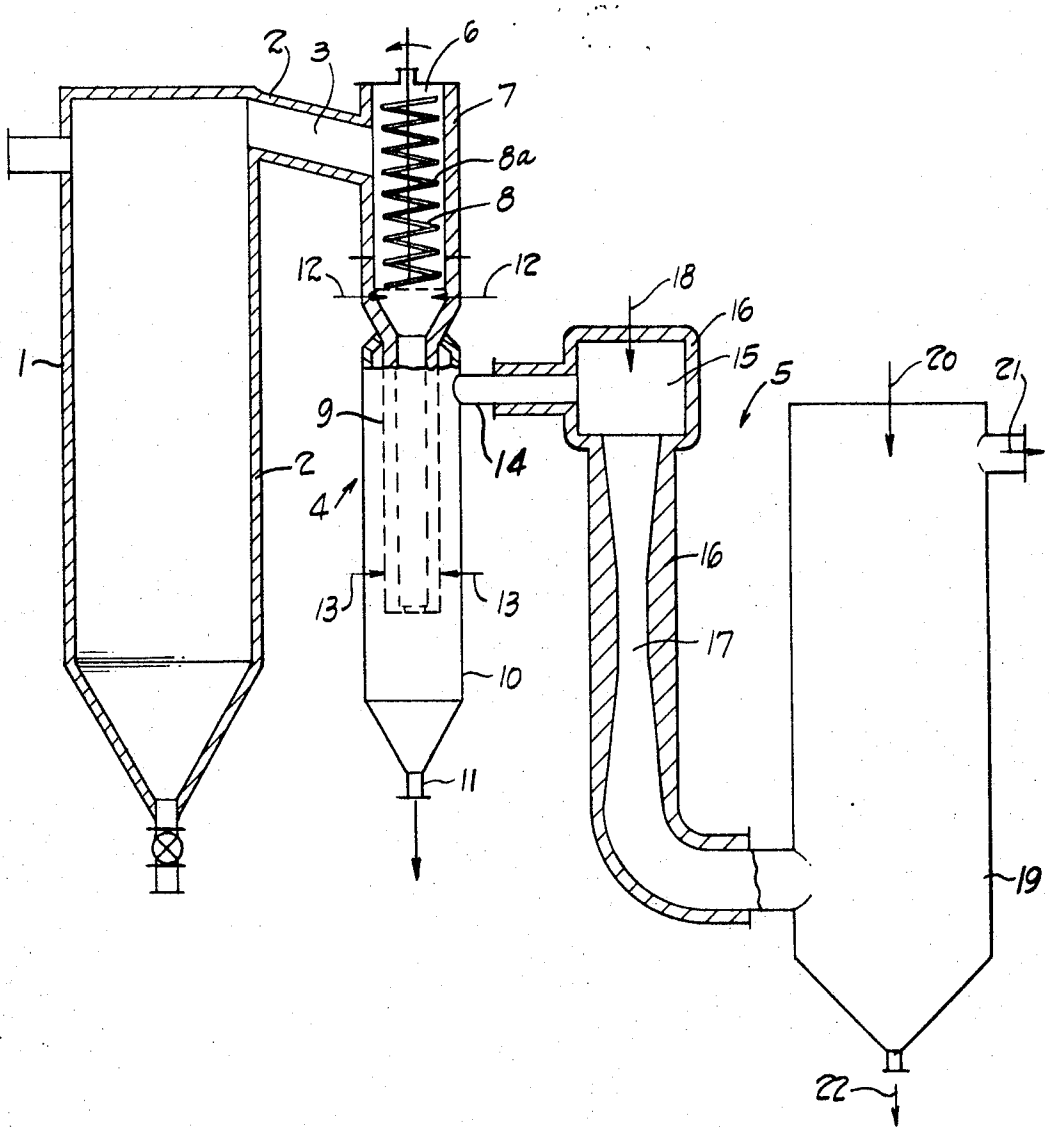
Filed Nov. 8, 1966



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United States Patent Office

3,468,849

Patented Sept. 23, 1969

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3,468,849

PROCESS FOR THE PREVENTION OF BUILD-UP OF DEPOSITS IN VAPOR CONDUITS

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Filed Nov. 8, 1966, Ser. No. 592,793
Claims priority, application Italy, Nov. 11, 1965, 24,985/65
Int. Cl. C08g *17/003, 17/04*
U.S. Cl. 260—75 3 Claims

ABSTRACT OF THE DISCLOSURE

Method for preventing build-up of poly(ethylene terepthalate) polymer deposits in vapour conduits comprising injecting a jet of vaporized glycol into a stream of vapor containing said polymer material.

The invention relates to a process for the operation of a jet pump for the conveyance of a substance which has a tendency to form annoying precipitates in a vacuum that are soluble in a non-aqueous solvent. A spray condenser is made use of when the substance to be conveyed is a vaporous medium that becomes precipitated, deposited or condensed in the spray condenser. Through this means there is formed a vacuum through which the medium is drawn, and thus conveyed onward. A jet pump is provided to supplement the condenser, and it serves the purpose of maintaining the vacuum in the spray condenser. The process, however, is also suitable for operating an installation that comprises only the jet pump, without a condenser. Such equipment for conveying the substance is of simple design, without rotating parts, and therefore it makes possible easy control of all parts of the equipment which come into contact with the substance to be conveyed. This advantage is made use of by the chemical industry in particular.

Difficulties arise, however, when the substance which is to be conveyed displays in a vacuum a tendency to form annoying precipitates that cannot be removed by water or steam. This is, for example, the case when the substance to be conveyed is a condensable vapor which contains constituents, impurities, for example, in the form of dust, or that can be condensed or can be sublimated. Examples of this are impurities which, in the region of moisture saturation, form hydrates. This applies in particular for calcium, which forms cement-like deposits in the equipment, particularly in the jet tube of the jet pump and in the piping of the equipment. The alkylene glycol propellants hereof dissolve such deposits. Another example of impurities causing disturbances are the alkali deposits of substances which in vacuum-metallurgy have to be conveyed in vapor form. In the zone of the high temperatures for melting metals, these alkali deposits evaporate, and they then deposit themselves in the cooler regions of the piping.

Disturbing deposits can also be formed from dust, and can cause erosions in the apparatus, particularly in the bends of piping.

In chemical equipment, the substance conveyed forms annoying deposits mainly during a change in its partial pressure and in its concentration in the vacuum stage of the equipment.

The fundamental problem solved by the invention is to prevent the formation of such deposits, or precipitates, and particularly cement-like deposits in the jet tube of the jet pump used in conjunction with a poly(ethylene terephthalate) reactor. The invention can be made use of in those cases where the precipitates are soluble in a non-

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aqueous, alkylene glycol, or one including a cycloaliphatic ring.

In accordance with the invention, this problem is solved in that the vapor jet of the jet pump is a vapor stream of an alkylene glycol per se forceably introduced into the system. The result of this is that this solvent prevents a formation of annoying deposits because it dissolves and washes away the condensing or sublimating precipitates directly as they are formed.

The process according to the invention can, for example, be applied to equipment wherein the substance to be conveyed is ethylene glycol, which is sucked continuously out of a reactor vessel in which monomeric bis-(2-hydroxyethyl) terephthalate is heated and thereby becomes polymerized, being sucked continuously out of the reactor by a vacuum maintained by a jet pump.

Such a process is described in application Ser. No. 519,836 filed Jan. 11, 1966. If in the way that is obvious from the prior art, the pumping jet of the jet pump is formed of steam, then, under the influence of the cooler surfaces of the jet pump there is formed a precipitate which consists of the volatile polymers or condensation products and which clings tightly to the walls of the equipment in the form of smears and gels. This deposit alters the flow cross sections, and is, therefore, a great disturbance. It also makes it more difficult, by admixture of water to the ethylene glycol, to reuse the glycol. The mixture of water and ethylene glycol cannot without further steps be separated out as waste, and, therefore, contaminates the water flowing off.

All these problems are avoided when, in accordance with the invention, the pump jet is formed of vaporized ethylene glycol with or without diluents and in which the undesirable deposits dissolve.

The glycol jet that enters the jet pump as a jet of vapor, is formed of the same substance that is conveyed by the jet pump; that is, with the preferred example of carrying out the invention, of ethylene glycol. It is, however, also possible to use aliphatic glycols other than ethylene glycol for these jets. Such materials provide a non-aqueous, preferably neutral propellant medium for the vacuum system. Specific examples of such glycols include 1,4-dimethylolcyclohexane, butylene glycol, hexamethylene glycol, or glycols having side chains, for example, 1,2-propylene glycol or the aforementioned dimethylolcyclohexane.

The invention also concerns equipment for carrying out the new process. This equipment is characterized in that, between the suction connections of the jet pump and the reactor vessel is interposed the already-mentioned spray condenser.

The vacuum vapor-jet pump can preferably be heated, because cold pump walls would promote a formation of the described deposits.

The accompanying drawing shows a preferred example of construction of the equipment for carrying out the process of the invention.

Into an upright reactor **1** is introduced a monomeric bis-(2-hydroxyethyl) terephthalate, and by means of vanes on a vertical rotating shaft, these elements not being illustrated, it is spread in a thin layer over the inner wall of the reactor **1**, out of which layer the ethylene glycol becoming liberated is vaporized under the influence of the heating of the reactor by its hot-jacket **2** and under the influence of a vacuum. These vapors are conveyed out of the reactor **1**. This objective is achieved by a spray-condenser **4** and a jet pump **5**.

The outlet connectors **3** of the reactor **1** lead to a cylindrical head part **6** of the spray condenser **4**. This head part **6** is provided with a heating jacket **7**. Into this head **6** there flow the glycol vapors conveyed through the outlet connections **3**, going then downward. In this head part **6**

is disposed a rotating shaft **8** carrying a worm **8a** that serves for cleaning the inner walls of the head part **6**. The head part **6** is open at its bottom, and merges into an inner condenser tube **9**, which runs downward and is likewise open at its bottom, and is surrounded by an outer tube **10** of greater diameter. This outer tube runs farther down than the inner tube, and at its bottom runs conically into an outlet opening, to which is connected a barometric outlet tube that is not shown in detail. The tube **9** is closely above the outer tube **10** provided with a ring of spray-nozzles **12**. Another ring of nozzles **13** is disposed in the wall of the tube **10**, closely above the lower end of the tube **9**. The nozzles **12** and **13** are supplied with a cooling fluid, whose purpose is to condense the glycol vapors. A glycol, preferably ethylene glycol, serves as the cooling fluid, although other spray media may be used if desired. Instead of a spray condenser as exemplified herein, other condensing means, e.g. surface condenser means, may be used.

The tube **10** of the spray-condenser has close below its upper end an outlet **14**, which is connected to the head **15** of the jet pump **5**. Jet pump **5** is provided with a heating jacket **16**, which extends round the head **15** and the jet venturi **17**. At the top of the head **15** is inserted a jet nozzle **18** directed downward in the axial direction, and it supplies the pumping jet of glycol vapor.

The jet pump **5** maintains the vacuum in the spray-condenser **4** and in the reactor **1**. The vapors leaving the jet pump are conveyed into a mixed-condensation tank **19**. Unprecipitated vapors or gases emerge from an outlet **21**; while the liquid ethylene glycol is drawn off from an opening **22** provided at the lower end of the tank **19**. This opening is conveniently provided with a barometric leg (not shown).

The more or less polymerized substance coming out of the bottom of the reactor **1** can be introduced into a second similar reaction vessel, which forms a second polymerization stage. The product obtained in this way can then be introduced in a third stage into a reactor vessel, corresponding to the one that is illustrated. In this case it is desirable to make the vacuum in the first stage 40 mm. Hg pressure, in the second stage 5 to 30 mm. Hg pressure, and in the third stage 0.1 mm. Hg pressure. When this is done, then there are obtained for the first stage the following operating conditions: a quantity of ethylene glycol amounting to 88 kg. hourly is liberated. In the spray-condenser **4**, 30 kg. hourly of this is separated out. The mixed condenser **19** has a diameter of about 300 mm. and a height of about 1000 mm. Its spray nozzle is fed with 660 kg. of glycol per hour. The jet pump **9** conveys to it a quantity of ethylene glycol vapor amounting to 58 kg. per hour. To outlet **21** there can be connected a conventional steam-jet vacuum pump, which sucks off the inert gases, and serves the purpose of producing a vacuum in the equipment before starting operations, and which then maintains this vacuum. The water fed to the nozzle of such a steam-jet pump has a temperature of 35° C. and the steam fed to the pump has a pressure of 5 atmospheres gage. The consumption of steam amounts to 20 kg. per hour, and the consumption of water amounts to 250 liters per hour.

In the second stage, 7.8 kg. per hour of ethylene glycol is liberated in the reactor **1**. Of this, 2.7 kg. per hour is condensed in the spray-condenser. In the mixed condenser **19** the amount of ethylene glycol sprayed in amounts to 132 kg. per hour. The dimensions of this condenser **19** are the same as in the case of the first stage. In it 5.1 kg. of residual ethylene glycol is condensed hourly. The steam-jet pump connected on consumes 35 kg. per hour of steam at 5 atmospheres gage pressure, and consumes 1.2 cubic meters per hour of water at 35° C.

In the reactor **1** of the third stage, 1 kg. per hour of ethylene glycol is liberated. The vacuum in this reactor amounts to 0.1 mm. Hg pressure. There is also sucked out of the reactor **1**, 1 kg. per hour of inert gas. In the spray-condenser **4**, 0.66 kg. per hour of ethylene glycol is condensed. The vapor-jet pump **5** is fed with 25 kg. per hour of glycol vapor hourly, at 1.05 atmospheres gage pressure. The steam-jet vacuum pump connected to the condenser **19** consumes 50 kg. per hour of steam at 5 atmospheres gage pressure, and 2 cubic meters per hour of water at 30° C.; while the spray nozzle of the mixed condenser **19** consumes 550 kg. per hour of glycol at 28° C.

For the purpose of condensing 2 kg. per hour of ethylene glycol vapor, plus about 1 kg. per hour of inert gas, from an 0.1 mm. Hg pressure to atmospheric pressure, there are needed:

When using the process claimed by the invention	When using the conventional steam-jet suction elements
Ethylene glycol vapor as the means to operate the jet-suction elements at a pressure of 1.05 atmosphere gage: 38 kg. per hour.	Steam at 5 atmospheres gage as the means for multistage jet-suction elements: 275 kg. per hour.
Ethylene glycol fluid at 28° C. for the mixed condenser: 900 kg. per hour.	Cooling water at 30° C.: 30.25 m.^{3} per hour.
Steam at 5 atmospheres gage for operating the pre-vacuum jet-suction element: 50 kg. per hour.	
Cooling water at 30° C.: 2 m.^{3} per hour.	

This comparison shows that the process according to the invention is of great economy.

A further advantage is obtained in that it is now possible to eliminate disturbing waste-water problems. Furthermore, the mixed condenser operated with ethylene glycol condenses and recovers ethylene glycol escaping from the reaction, so that it is merely necessary, in starting up the equipment, to put in the required amount of ethylene glycol.

What I claim is:

1. In a process for producing poly(ethylene terephthalate), a method for preventing the build-up of undesired deposits of the polymer in vapor conduits which comprise:

(a) moving up at subatmospheric pressure in a conduit a stream of vapor containing said polymer material which has a tendency to deposit on the walls of said conduit;

(b) forceably injecting into said stream of vapor a jet of vaporized non-aqueous, alkylene glycol sufficient to reduce the pressure in the system, and

(c) removing the mixture of said glycol and said polymer from said conduit.

2. A process in accordance with claim **1** in which the alkylene glycol is ethylene glycol.

3. A process in accordance with claim **1** in which the alkylene glycol is 1,4-dimethylolcyclohexane.

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U.S. Cl. X.R.

23—285; 55—468; 134—22; 230—102; 260—475