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[54] **PROCESS FOR CLEANING SLURRY COOLERS**

[75] Inventors: **Anders E. Andersen,** Berwyn; **Alan R. Hirsig,** Wallingford; **Walter A. Mameniskis,** Drexel Hill; **Frank W. Melpolder,** Wallingford, all of Pa.

[73] Assignee: **Atlantic Richfield Company,** Los Angeles, Calif.

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[51] **Int. Cl.²**..... **C07C 51/33**

[58] **Field of Search**..... 62/58; 260/525, 524 R; 134/5, 22 C

[56] **References Cited**

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Primary Examiner—James A. Patten
Assistant Examiner—Richard D. Kelly
Attorney, Agent, or Firm—John R. Ewbank

[57] **ABSTRACT**

Plating deposits are removed from the surface of tubes of heat exchangers used to control exothermic reactions and cool hot slurries in which the quantity of reaction product exceeds the solubility limit at surface temperature by periodically substituting a hot fluid for the cooling liquid.

4 Claims, No Drawings

PROCESS FOR CLEANING SLURRY COOLERS

BACKGROUND OF THE INVENTION

1. Field of Invention

This invention relates to a process for maintaining heat exchangers free of plating deposits.

2. Description of the Prior Art

Control of the reaction temperature of certain exothermic reactions is ordinarily accomplished by cooling the reactants and the reaction product at a rate necessary to maintain the desired temperatures. When the reaction product is the form of a slurry such as in the manufacture of isophthalic acid by the air oxidation of metaxylene in a catalyst in solvent system such as cobalt in acetic acid, and one cooling means is a conventional shell and tube heat exchanger, the cooling results in a supersaturated solution of reaction product in solvent which is passing through the tubes of the heat exchanger. While it is possible with such a system to minimize the temperature driving force or degree of supersaturation, some amount of "plating" or deposition of the product on the inside of the heat exchanger tubing is unavoidable. This plating reduces the heat transfer capacity of the cooler and leads, eventually, to physical plugging of the slurry flow spaces within the exchanger. Various solutions to this problem of cleaning a plated exchanger have been suggested by prior artisans. Chemical cleaning involves draining and flushing the tubes of the exchanger and thereafter introducing foreign substances such as sodium bicarbonate or sodium carbonate in the slurry side of the heat exchanger to dissolve the plating and thereafter further draining and flushing after cleaning. See Frank, U.S. Pat. No. 2,676,180, Apr. 20, 1954. Mechanical means have also been proposed but these also result in substantial shutdown time for draining, flushing, and cleaning.

SUMMARY OF THE INVENTION

It is therefore an object of this invention to provide a process for cleaning the process side of heat exchangers which are subject to plating by supersaturated solutions which are being cooled. It is a further object to provide a more efficient process for maintaining the reaction temperature of exothermic reactions by improved operation of heat exchangers.

These and other objects as will become apparent from the following description are accomplished by the process of this invention which comprises periodically substituting a hot fluid for the cooling liquid in a heat exchanger to remove plating deposits from the process side of the heat exchanger tubes.

DETAILED DESCRIPTION OF THE INVENTION AND PREFERRED EMBODIMENTS

While this invention is applicable to any exothermic reaction system wherein reaction product crystallizes from a process solution and wherein continuous cooling is necessary to control the exothermic reaction, it will be described in detail only with respect to one reaction system, namely the air oxidation of metaxylene to produce isophthalic acid using acetic acid as solvent and a solution of cobalt as catalyst.

An exothermic reaction system such as described above must be controlled as to temperature by continuous cooling. The process of this invention is applicable in systems using any conventional type heat exchanger, for example, shell and tube or double pipe coolers. The cooling fluid may be air, water or any other fluid normally used for cooling. In a preferred embodiment water is the cooling fluid and is flowed through the shell at a rate depending upon the water temperature and the amount of cooling necessary to maintain the reactants at the desired temperature.

The reaction system is a slurry of isophthalic acid solids suspended in a solution of isophthalic acid and m-xylene in acetic acid solvent.

The reaction system is introduced in the tube inlet and is withdrawn from the tube outlet at a lower temperature. It is preferred to introduce the reactants in the tube section rather than the shell section to avoid shell dead space where solids may collect. In either embodiment plating occurs over the passage of time on the side of the tube in contact with the reaction system. The reason for the plating is thought to be due to the change in solubility of the reaction product in the solvent at different temperatures. That is to say as the temperature of the hot reaction system is lowered in the heat exchanger the solution becomes supersaturated Furthermore, the heat of crystallization being removed at the surface causes a tight adherence of the crystal to the metal surface. The resultant plating of precipitated product on the cold tube walls reduces the heat transfer capacity of the heat exchanger and leads to physical plugging of the flow spaces.

In carrying out the process of this invention the flow of cooling fluid is stopped and a warm or hot fluid is substituted. The hot fluid is preferably steam but may alternatively be any hot fluid such as hot water or hot air or hot oil. We have found that this reversal of temperature gradient results in rapid reduction and removal of the plating deposits on the tube walls by dissolving and flaking off. Removal and draining of the reactants is not necessary.

The flow of process fluid through the exchanger may either be temporarily halted or continued during the periodic deplating process.

It is our theory of this invention that the periodic reversal of the temperature gradient causes localized increase in the solubility of the plated material and flaking from the surface due to a combination of factors such as mechanical stresses due to different coefficients of expansions between the metal surface and plated material, solubility increase at higher temperature at the interface, and mechanical shearing due to normal slurry flow. This invention does not operate by melting of the plated material; for example, even though the melting point of isophthalic acid is about 600°F. low pressure steam can be used effectively to remove deposits of isophthalic acid in accordance with the process of this invention.

The following examples are presented to illustrate but a few embodiments of the invention and are not to be construed as limiting:

EXAMPLE I

A heat exchanger used to control the temperature of a reaction system wherein metaxylene is oxidized in acetic acid solvent to isophthalic acid using cobalt catalyst in solution and a heat transfer coefficient of about 260 BTU/ft.^{2}-hour-°F. at the beginning of a production run. Cooling water was run through the shell of the heat exchanger at a rate necessary to control the exothermic reaction at a temperature of about 225° F. The process fluid run through the tubes of the exchanger was a

slurry having an average of about 19 weight percent solids. After a run of about 10 days the heat transfer coefficient was down to 50 BTU/ft.²-hour-°F. due to isophthalic acid plating on the inside of the tubes. The flow of process fluid in the tubes was continued and the cooling water was shut off. The reactant flow to the reactor was shut off at this point to prevent a temperature runaway since no cooling was available. Steam was introduced in the shell at a rate of 700 pounds per hour which resulted in an initial overall ΔT of about 15°F. After 7 hours the slurry reached a temperature of 200°F. after which the steam was stopped and cooling water begun through the shell of the exchanger. The heat transfer coefficient was measured to be about 260 BTU/ft.²-hour-°F., its original clean condition value.

This example demonstrated successful removal of plating deposits on heat exchanger surfaces in contact with a slurry of oxidation product.

The concepts of this invention are applicable to any system having a heat exchanger subject to plating type clogging and is thought to include any reaction system wherein product continuously crystallizes from a solution causing a slurry. The invention is especially applicable to systems wherein an exothermic reaction must be controlled by continuous cooling, and a high melting point reaction product is caused to precipitate. Examples of classes of reactions are oxidation or organic compounds to acids. Exemplary organic acids are isophthalic acid, terephthalic acid, orthophthalic acid, salicylic acid, and other acids having a high melting point and produced by processes involving slurry in reactors.

While the invention has been described in detail with regard to various preferred embodiments, it is to be understood that it is not limited thereto but is to be construed broadly so as to include various modifications, alternatives, and improvements which should become readily apparent.

We claim:

1. The process for operating a heat exchanger wherein a reaction system comprising a slurry is cooled by passing said slurry through one side of said heat exchanger and passing a cooler liquid through the other side of said heat exchanger, the improvement which consists of:

regulating the composition of said slurry by conducting the oxidation of metaxylene by air to isophthalic acid in an aqueous acetic acid solvent containing a solution of cobalt as catalyst, said slurry tending to deposit plating deposits of slurried material onto the surface of the process side of the exchanger, said plating deposits comprising organic compounds and acids;

regulating the temperature of the reaction system in the heat exchanger to maintain a reaction temperature of from about 200°F. to 275°F. by circulating cooling water in said heat exchanger; and

operating the heat exchanger in an improved manner by periodically substituting low pressure steam for said cooling water to remove plating deposits of slurried material from the surface of the process side of the exchanger, the temperature of the low pressure steam being less than the melting point of isophthalic acid but sufficiently hot to alter the adhesion of such deposits to permit such deposits to become dispersed and withdrawn as a component of withdrawn slurry, said low pressure steam heating the heat exchanger and circulating slurry to a temperature of about 200°F.

2. The process of claim **1** wherein said heat exchanger is a shell and tube type.

3. The process of claim **1** wherein said heat exchanger is a double pipe type wherein the process liquid is passed through the inside and the cooling fluid is passed through the annular space.

4. An improved process of controlling the temperature of a circulating slurry of reaction system in the exothermic oxidation reaction of metaxylene to isophthalic acid in the presence of acetic acid as solvent and a soluble catalyst comprising:

cooling the reaction product in a heat exchanger so as to maintain a reaction temperature of from about 200°F. to 275°F. by circulating cooling water in said heat exchanger; and

periodically substituting low pressure steam for said cooling water in said heat exchanger to heat the heat exchanger and circulating slurry to a temperature of about 200°F., thereby dispersing into said circulating slurry of reaction system the organic deposits previously formed in said heat exchanger, said deposits comprising organic compounds and acids.

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