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COMPLETE SPECIFICATION

Production of Terephthalic Acid by Hydrolysis of an Alkyl Terephthalate

We, HERCULES INCORPORATED, a corporation organized under the laws of the State of Delaware, one of the United States of America, of 910 Market Street, City of Wilmington, State of Delaware, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the preparation of terephthalic acid (hereinafter designated by the abbreviation "TPA") by hydrolysis of an alkyl terephthalate and, more particularly, by hydrolysis of dimethyl terephthalate (hereinafter designated by the abbreviation "DMT").

According to the present invention there is provided a method of preparing terephthalic acid from a mono- or di-alkyl terephthalate, wherein the alkyl group is a C_1 to C_4 alkyl group, which comprises contacting the alkyl terephthalate with water in the liquid phase under autogenous pressure at a temperature in the range of from 140 to 350°C. until hydrolysis to terephthalic acid has occurred and then separating the terephthalic acid from the resulting hydrolysate.

It has now been found that a mixture of a dialkyl terephthalate and water becomes a homogeneous liquid at temperatures in the range of from 140 to 350°C., that the dialkyl terephthalate undergoes hydrolysis while in solution, and that the TPA has a much lower solubility in water and crystallizes out of the hydrolysis reaction mixture.

The monoalkyl terephthalates such as the methyl or ethyl compounds as well as the dialkyl terephthalates are soluble in hot water and similarly undergo hydrolysis so that TPA is the sole acid product of the hydrolysis.

It has been found that dialkyl terephthalates are readily hydrolyzed to TPA simply by contacting with liquid water at from 140—350°C. under sufficient pressure to maintain a liquid phase and to carry the reaction to completion by using a large excess of water, or by removing one of the products, namely, the alcohol additionally formed during the hydrolysis or the TPA, from the liquid aqueous phase.

When a large excess of water is used and the amount of water is sufficient to dissolve all of the TPA produced, the TPA can be recovered by distilling off water and alcohol up to the point when TPA will crystallize from the remaining water. The distillation step aids in carrying the hydrolysis to completion, if it is not already complete, and the distillation temperature is in the hydrolysis temperature range. In its simplest aspect using a large excess of water, the alkyl terephthalate is dissolved in a volume of superheated water sufficient to dissolve all of the TPA produced at the hydrolysis temperature; the water and alcohol of hydrolysis are distilled off until the concentration of the TPA in the water is such that it crystallizes out at a temperature in the range of from 140—350°C., which temperature can be equal to or less than that at which the hydrolysis was effected. The TPA is then separated from the water while hot or after cooling. A more uniform crystalline product is obtained when the TPA is crystallized out from the water while still above 140°C. and the hot filtrate is then reusable as a hydrolysis medium for more alkyl terephthalate. The distillation process for removing water and alcohol can be carried out in the hydrolysis vessel or the aqueous solution can be transferred to another vessel, where the pressure is reduced to allow the vapors to be distilled from the aqueous TPA solution. When the TPA solution is near saturation, the heat loss can be used to reduce the temperature to a lower temperature at

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