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Ghorpade , et al. **January 12, 1999**

Method and apparatus for production of levulinic acid via reactive extrusion

Abstract

The present invention relates to a continuous process for preparing levulinic acid from starch in a reactive extrusion process. In a preferred embodiment, the extrusion takes place in a twin-screw extruder having a plurality of temperature zones wherein the starch slurry is preconditioned, extruded, filter pressed, reboiled, vacuum distilled, condensed, centrifuged, whereby the waste effluent from the centrifugation is reprocessed upstream to the preconditioning stage.

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SUMMARY OF THE INVENTION

The present invention provides an improved method and apparatus for producing levulinic acid with bifunctional chemical intermediates made by hydrolyzing starch and dilute acid in an extruder.

Extrusion is generally a processing operation wherein a material is forced through a metal die. With high viscosity materials, a rotating screw of variable pitch is used to force the material through the die. Items extruded are, for example, injection molding polymers, molten glass, hot metal billet, and food items, such as spaghetti and the like.

It has unexpectedly been found that, in accordance with the present invention, extrusion processing of a starch acid slurry provides a very efficient method for the production of levulinic acid. Extrusion production, as herein described, is advantageous in that the process is continuous, requires fewer steps and reduced reaction times, and gives higher yields at a reduced cost. The present invention, in a preferred embodiment, prepares levulinic acid from starch via a twin-screw extruder having a plurality of temperature zones wherein the starch slurry is preconditioned, extruded, filter pressed, reboiled, vacuum distilled, condensed, centrifuged, and reprocessed.

Additionally, the present invention may be utilized to prepare valeric .gamma.-lactone by hydrogenation of levulinic acid. Valeric .gamma.-lactone is an excellent solvent.

Further, the present invention may be utilized to prepare 1,4-pentanediol, which on dehydration yields 1,3-pentadiene (piperylene). Piperylene is known to polymerize to an elastomer.

Despite the wide utility possessed by levulinic acid as described above, it is felt that its commercial potential is impeded by the high production costs and low yields of the currently available processes. It is therefore an object of the present invention to provide a process and apparatus for the production of levulinic acid at a cost lower than the current commercial processes.

To date, the preparation of levulinic acid from starch has been confined to batch processes. The commercial processes that operate continuously have used cellulose starting materials. It is therefore another object of the present invention to provide a continuous process and apparatus for the preparation of levulinic acid wherein starch is employed as the starting material.

It is another object of the present invention to provide a process and apparatus for the preparation of levulinic acid which requires milder reaction conditions, shorter reaction times, and fewer steps than the prior art processes.

It is a further object of the invention to provide a process and apparatus for the preparation of levulinic acid that gives increased yields over the prior art processes. The process of the present invention gives yields of about 48% which is about 70% of theoretical.

It is yet another object of the invention to provide a process and apparatus for the preparation of levulinic acid whereby the some or all of the waste stream is recycled to the beginning of the process.

Still further objects and advantages will become apparent from a consideration of the ensuing description and drawings.

For a complete disclosure of the invention please consult the full patent.