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METHOD FOR THE PRODUCTION OF SEBACIC ACID AND METHYLHEXYLCARBINOL

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It is known that castor-oil, when treated with alkalis in the presence of heat, will be split in a manner which yields sebacic acid in the form of its alkali-salt together with methylhexyl-carbinol as the chief products. The methods hitherto disclosed by the literature of obtaining sebacic acid and methylhexyl-carbinol give, however, rather poor yields of said materials, i. e., approximately 40% of the theoretical.

Now it has been found that it is possible to enhance considerably the yields of sebacic acid as well as of methylhexyl-carbinol, if instead of the castor-oil-fatty acid or of its esters and particularly instead of castor-oil we treat the ricinoleyl-alcohol, obtainable in the well known way by reduction, with alkaline agents at increased temperatures. The splitting of the ricinoleyl-alcohol may be performed, e. g., with solid alkalis such as sodium-hydroxide, potassium-hydroxide, alkali-carbonates, alkali-acetates, etc., earth-alkalis such as calcium-oxide, calcium-hydroxide, etc., or mixtures of same, or also with concentrated aqueous solutions of the various alkaline media. In this latter case we preferably work with an increased pressure of about 1 to 25 atmospheres. The temperatures applicable for this purpose are above 100° C. and preferably between 200 and 300° C.

Example

852 weight parts of ricinoleyl-alcohol (iodine number 86) obtained in reducing castor-oil-fatty acid-butyl ester with metallic sodium in a solution of butanol and freed from any solid portions by filtering after a preliminary chilling, are mixed in a suitable stirring-autoclave, resistant to alkalis, with 385 weight parts of caustic potash (96%) in the form of scales, and then heated up while stirring. As soon as the temperature has attained 220° C., an excessive pressure is caused in the reaction vessel which at 245° C. may be approximately 4 atmospheres and which is then continuously vented off to the extent, that the excessive pressure in the autoclave will never exceed about 5 atmospheres. Then the temperature is slowly increased to about 290° at which it is kept until there be no further development of pressure, i. e., until there occurs no longer a splitting of hydrogen. After a further stirring for about one hour with the valve opened in order to complete the oxidation, the reaction-mass is dissolved in several times its volume of water. During this operation an oil layer forms which in a separating-vessel is thereupon separated from the aqueous salt-solution whereupon it is washed out

several times with water. From the aqueous salt-solution and from the washing-water we obtain, by acidifying with mineral acid such as muriatic acid, sulfuric acid and the like, the thus formed sebacic acid the output of which is 535 weight parts or about 88% of the theoretical yield. The oil separated off supplies, upon distillation, the methylhexyl-carbinol with quite a satisfactory yield and it possesses a boiling-point of 174 to 176° C.

For the splitting of the ricinoleyl-alcohol we may likewise proceed by using a 50%-caustic-potash-lye instead of caustic potash itself and by heating up to temperatures of 250 to 270° C., whereby a superatmospheric pressure is produced of approximately 18 to 20 atmospheres which we maintain for 2 to 3 hours. Then the pressure is slowly reduced by blowing water-vapour through the reaction-mass. During this process an oil distills over, which, apart from small amounts of ketone, consists chiefly of methylhexyl-carbinol. The salt-residue in the autoclave upon acidification yields sebacic acid.

Ricinoleyl alcohol obtained by other processes may be used, such as that obtained by suitable high pressure catalytic hydrogenation of ricinoleic acid or its derivatives.

We claim:

1. Method for the production of sebacic acid and methylhexylcarbinol characterized by treating ricinoleyl alcohol with caustic potash at temperatures between 200 and 300° C. and under pressure of 5 atmospheres.

2. Method for the production of sebacic acid and methylhexylcarbinol characterized by treating ricinoleyl alcohol with a 50% caustic potash solution at temperatures of 250-270° C. and under pressure of 18-20 atmospheres.

3. The process for the production of sebacic acid and methylhexylcarbinol which comprises heating an alkaline agent with ricinoleyl alcohol to a temperature above 100° C.

4. The process for the production of sebacic acid and methylhexylcarbinol which comprises heating a caustic alkali with ricinoleyl alcohol to a temperature above 100° C.

5. The process for the production of sebacic acid and methylhexylcarbinol which comprises heating, under superatmospheric pressure not exceeding 25 atmospheres, a concentrated aqueous alkali with ricinoleyl alcohol to a temperature above 100° C.

6. Process for the production of sebacic acid and methylhexylcarbinol which comprises heating in a closed vessel, under autogenously de-

veloped pressure, an alkaline agent with ricinoleyl alcohol to a temperature of from above 100° C. to about 300° C.

7. In the process for the production of sebacic acid and methylhexylcarbinol by heating ricinoleyl alcohol with an alkaline agent, the step of employing, in a closed vessel, autogenously developed superatmospheric pressure upon the reactants.

8. The process for the production of sebacic acid and methylhexylcarbinol which comprises heating, in the absence of added water, a caustic alkali with ricinoleyl alcohol to a temperature above 100° C.

9. The process which comprises heating ricinoleyl alcohol with caustic potash in the absence of added water to a temperature of from about 200° C. to about 300° C. in a closed vessel under an autogenous pressure of from about for to about

five atmospheres, agitating the reaction mixture until no further pressure develops, then cooling and adding excess water, separating an upper oily layer containing methylhexylcarbinol, acidifying the aqueous residue, and separating the formed sebacic acid.

10. The process which comprises heating ricinoleyl alcohol with aqueous caustic potash solution in a closed vessel to a temperature of from about 250° C. to about 270° C., maintaining the reaction mixture under an autogenous pressure of from about eighteen to about twenty atmospheres for about two to three hours, then gradually reducing the pressure while simultaneously distilling off the formed methylhexylcarbinol, then cooling the residue, acidifying the residue and separating the formed sebacic acid.

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