the formation of organic acids occur. Although it is stated in the description of US 3431253 that it is possible to use mixed water-lower alcohol solutions, such as 90% methanol, 80% ethanol and 75% propanol solutions, in the examples only 100% aqueous solutions of glucose are contacted with alumina at 50°C.

A similar procedure is described in a journal article by H. Asaoka, Carbohydrate Research 137 (1985) 99-109, wherein the isomerization of glucose to fructose was accomplished in a 80% (v/v) methanol-water solution over a disodium pentasilicate catalyst at 45 °C. When ethanol or 1,4-dioxane was used instead of methanol, the results were worse. All attempts with propanol, acetone, acetonitrile and tetrahydrofuran gave unsatisfactory results.

Similar teachings are described in Lew et al., Ind. Eng. Chem. Res.,51 (14) (2012) 5364-5366, disclosing the conversion of glucose to fructose over Sn-containing Lewis acid zeolite beta in ethanol at 90°C. The reaction is conducted in the presence of ion exchange resin Amberlyst 131 which catalyses the reaction of fructose formed to hydroxymethyl furfurural and subsequently to ethoxymethyl furfural. In Chem. Abstr., 2011: 336348 by Canlas et al. the conversion of glucose to fructose over Ti- or Sn-containing Bronsted or Lewis acid zeolites in water or methanol at 100 to 160 °C is described. It is observed that the above-mentioned prior art documents do not relate to the formation of fructosides.

In Saravanamurugan et al., J. Amer. Chem. Soc., 135 (14) (2013) 5246-5249 the isomerization of glucose in methanol over acidic zeolites has been described. The product obtained is stated to yield fructosides, which are subsequently hydrolyzed to fructose. In the article it is shown that basic zeolites yield considerably lower amounts of fructose. Whereas acidic catalysts yield more than 20% fructose, the basic catalysts yield from 4 to 18% fructose.

Thus many documents in the prior art stipulate that the isomerization reaction medium contains water. In contrast therewith, it has now been found that it is not necessary that water is present if the reaction temperature is above 75°C. The solvent may then consist of pure alcohol. If water is absent it has been found that no undue formation of organic acids occurs. It has further been found that when the isomerization reaction is conducted in the presence of a basic catalyst and the fructoside formation is conducted in the presence of an acidic catalyst, the amount of fructoside is enhanced.

Summary of the Invention

In one aspect, the present invention provides process for producing a fructoside-rich product from a glucose-rich feedstock, which process comprises isomerizing glucose to fructose from a glucose-rich feedstock by contacting the glucose-rich feedstock in an alcoholic medium with a basic isomerization catalyst at a temperature of at least 75 °C in an isomerization zone to yield an isomerized product containing fructose, and reacting at least part of the isomerized product containing fructose with an alcohol in the presence of an acid catalyst in a

conversion zone to yield a fructoside-containing conversion product; wherein the glucose-rich feedstock is passed to the isomerization zone or the conversion zone; wherein at least a fraction of the isomerized product is passed to the conversion zone and at least a fraction of the conversion product is passed to the isomerization zone; and wherein either the isomerized product or the conversion product is split into at least two fractions, at least one fraction, i.e. the product fraction, that is split off being recovered as fructoside-rich product.

Description

Accordingly, the present invention provides a process for the manufacture of a fructoside-containing product from a glucose-rich feedstock, comprising isomerizing glucose to fructose, by contacting the glucose-containing feedstock in an alcoholic medium with a basic isomerization catalyst at a temperature of at least 75 °C to yield a fructose-containing product, and reacting at least part of the fructose-containing product obtained therefrom with an alcohol in the presence of an acid catalyst to yield a fructoside-containing product. One of the advantages of the process resides in the fact that in this way a relatively high

CLAIMS

1. Process for producing a fructoside-rich product from a glucose-rich feedstock, which process comprises isomerizing glucose to fructose from a glucose-rich feedstock by contacting the glucose-rich feedstock in an alcoholic medium with a basic isomerization catalyst at a temperature of at least 75 °C in an isomerization zone to yield an isomerized product containing fructose, and reacting at least part of the isomerized product containing fructose with an alcohol in the presence of an acid catalyst in a conversion zone to yield a fructoside-containing conversion product;
wherein the glucose-rich feedstock is passed to the isomerization zone or the conversion zone;
wherein at least a fraction of the isomerized product is passed to the conversion zone and at least a fraction of the conversion product is passed to the isomerization zone; and
wherein either the isomerized product or the conversion product is split into at least two fractions, at least one fraction, i.e. the product fraction, that is split off being recovered as fructoside-rich product.

2. Process according to claim 1, wherein the basic catalyst is selected from the group consisting of hydrotalcite, alkali-exchanged zeolites, alkali metal and alkaline earth metal oxides and hydroxides, alkali metal alcoholates, borates, boronates and borinates, and alkali metal and alkaline earth metal carbonates and mixtures thereof.

3. Process according to claim 1 or 2, wherein the isomerization is conducted at a temperature of 75 to 180 °C, preferably from 80 to 150 °C, more preferably from 80 to 130 °C.

4. Process according to any one of claims 1 to 3, wherein the isomerization is conducted at a pressure of at least the autogenous pressure at the prevailing temperature, preferably from 1 to 60 bar, more preferably 2 to 25 bar.

5. Process according to any one of claims 1 to 4, wherein the residence time of the glucose-rich feedstock in the alcoholic medium ranges from 0.1 to 10 hr.

6. Process according to any one of claims 1 to 5, wherein the alcoholic medium in the isomerization comprises an alkanol having from 1 to 8 carbon atoms, preferably having from 1 to 4 carbon atoms, more preferably being methanol.

7. Process according to any one of claims 1 to 6, wherein the alcohol with which the at least part of the fructose-containing product is reacted, is selected from C_1 to C_4 alkanols.

8. Process according to claim 7, wherein the alcohol with which the at least part of the fructose-containing product is reacted, is methanol.

9. Process according to any one of claims 1 to 8, wherein the at least part of the fructose-containing product is reacted with an alcohol in the presence of an acid catalyst, selected from acidic zeolites and acidic ion exchange resins.

10. Process according to any one of claims 1 to 9, wherein the at least part of the fructose-containing product is reacted with the alcohol at a temperature of 20 to 100 °C, preferably from 30 to 80 °C.

11. Process according to any one of claims 1 to 10, wherein the contact time of the at least part of the fructose-containing product with an alcohol ranges from 0.1 to 12 hr.

12. Process according to claim 1, wherein the glucose-rich feedstock is passed to the isomerization zone; substantially the entire isomerized product is passed to the conversion zone; and the conversion product is split into at least two fractions, at least one product fraction that is split off from the conversion product being recovered as fructoside-rich product, and at least another fraction of the conversion product being passed to the isomerization zone.

13. Process according to claim 1, wherein the glucose-rich feedstock is passed to the isomerization zone; the isomerized product is split into at least two fractions, at least one product fraction that is split off from the isomerized product being recovered as fructoside-rich product, and at least another fraction of the isomerized product being passed to the conversion zone; and substantially the entire conversion product is passed to the isomerization zone.

14. Process according to claim 1, wherein the glucose-rich feedstock is passed to the conversion zone; substantially the entire conversion product is passed to the isomerization zone; and the isomerized product is split into at least two fractions, at least one product fraction that is split off from the isomerized product being recovered as fructoside-rich product, and at least another fraction of the isomerized product being passed to the conversion zone.

15. Process according to claim 1, wherein the glucose-rich feedstock is passed to the conversion zone; the conversion product is split into at least two fractions, at least one product fraction that is split off from the conversion product being recovered as fructoside-rich product, and at least another fraction of the conversion product being passed to the isomerization zone; and substantially the entire isomerized product is passed to the conversion zone.

16. Process according to claim 1, wherein the split of the isomerization product or the conversion product, as the case may be, is into two fractions.

17. Process according to claim 1, wherein before, during or after the split of the isomerization product or the conversion product, water is removed.

18. Process according to claim 17, wherein water is removed by flashing, distillation adsorption or a combination thereof.

19. Process according to any one of the preceding claims, wherein the glucose-rich feedstock comprises the reaction product of the alcoholysis of sucrose.

20. Process according to claim 19, wherein the alcoholysis of sucrose is carried out at a temperature ranging from 25 to 150 °C, preferably, from 40 to 100°C, more preferably from 60 to 80°C.

21. Process according to claim 1, 14 or 15, wherein the glucose-rich feedstock is passed to the conversion zone, which glucose-rich feedstock further comprises fructose.

22. Process according to claim 21, wherein the glucose-rich feedstock is HFCS.

23. Process according to claim 1, further comprising the step of purifying the fructoside-rich product to yield purified fructoside.

24. Process according to claim 23, wherein the purification comprises a dewatering step, preferably an evaporation or adsorption step.