

Description

The invention relates to a new method of preparing lactulose by basic isomerisation of lactose.

A conventional method of preparing lactulose from lactose comprises the basic isomerisation of lactose at elevated temperature. During the said isomerisation reaction are formed - in addition to lactulose - decomposition products thereof, and a brown-coloured reaction mixture is obtained.

It is known that the brown colouring of the mixture during the isomerisation can be mitigated by carrying out the isomerisation in the presence of sodium sulphite, sodium bisulphite or sodium phosphite. It is assumed that the sulphite ion, bisulphite ion or phosphite ion reacts with the decomposition products of lactulose while forming sugar sulphonc acids and sugar phosphonic acids which can then be removed by means of ion exchangers.

Such a method is known from Netherlands Patent Specification no. 165467 in which the use of sodium sulphite, from United States Patent Specification no. 4,536,221 in which the use of sodium bisulphite, and from Netherlands Patent Application No. 7907259 in which the use of sodium phosphite as an oxidant is described. However, the formed sugar sulphonc acids and sugar phosphonic acids are hard to decompose on purifying the waste water in a water treatment plant and hence they are ecologically unsound.

In EP-A-0132509 the purification of lactulose syrup is described. This purification is accomplished by oxidising the syrup under controlled conditions, e.g. chemically by bromine or electrochemically in the presence of calciumcarbonate, followed by a passage over ion exchange resins. To reduce the presence of byproducts, distinguishable as extraneous peaks, in the purified product, it is recommended to pass the solution successively through a column of anionic resin in bisulphite form at 5°C, followed by passages through a strong cationic resin column and through a weak anionic resin column, indeed a laborious purification process.

It has now been found that the above-mentioned problems do not present themselves when hydrogen peroxide or sodium chlorite is used in the method mentioned in the preamble, instead of sulphite, bisulphite or phosphite.

Hydrogen peroxide both in an acid medium and in a basic medium may serve as an oxidant and consequently can oxidize the decomposition products of lactulose which are formed during the isomerisation to (carboxylic) acids which can be removed by means of ion exchangers.

Excellent results are also obtained with sodium chlorite as a coreagent besides the sodium hydroxide required for the isomerisation, but as a result of the formation of corrosive compounds, for example, chlorine and chlorodioxide, during the reaction, higher requirements are imposed on the apparatus than when hydrogen peroxide is used.

According to a preferred embodiment of the method according to the invention hydrogen peroxide is used as an oxidant and is added after the basic isomerisation reaction of lactose to lactulose. Preferably 0,5-5 g of H_2O_2 are used per kg of lactose. $1H_2O$.

It is recommendable in particular to add hydrogen peroxide, for example, in the form of 17.5% solution, to the isomerisation mixture at a temperature of approximately 50°C, viz. at 40-80°C, after the pH of the mixture has decreased to below 7, viz. to 5,0-7,0. However, it is also possible to add hydrogen peroxide at a higher temperature of approximately 95°C, viz. at 90-100°C, under basic conditions (pH 7.5-8.0).

In case sodium chlorite is used instead of hydrogen peroxide, preferably 1-5 g of $NaClO_2$ per kg of lactose. $1H_2O$ are used at a temperature of 90-100°C and under basic conditions.

The reaction mixture, after isomerisation and treatment with H_2O_2 or $NaClO_2$, is processed in the conventional manner. The non-converted lactose is removed by crystallisation and is used again. The mixture comprising lactulose and decomposition products is purified by means of ion exchangers.

By using H_2O_2 or $NaClO_2$ instead of Na_2SO_3 , $NaHSO_3$ and Na_3PO_3 used so far, the decomposition products of lactulose which are formed during the isomerisation are converted into readily decomposable sugar carboxylic acids which cause no problems ecologically.

EXAMPLE I

The data and results of a number of experiments on a laboratory scale are recorded in table A hereinafter. In all the experiments the isomerisation of lactose to lactulose was carried out with NaOH, optionally in the presence of Na_2SO_3 . The addition of H_2O_2 according to the invention after the isomerisation reaction was carried out in two different manners:

method I: in basic conditions (pH 7.5-8) at approx. 95°C

method II: in acid conditions (pH <7) at approx. 50°C.



Table A

Exp. No.	NaOH g/kg lactose.1H ₂ O	Na ₂ SO ₃ (ditto)	H ₂ O ₂ (ditto)	Method	Colour after isomerisation *
1	2.5	-	-	-	13-16
2	2.0	2.0	-	-	3-10
3	2.5	-	2.4	I	2-3
4	2.5	-	2.4	II	6-7

* The colour is compared with the Gardner scale, a colour scale from 1 (colourless) to 20 (reddish brown).

On the basis of the colourindex the use of H₂O₂ according to method I were to be preferred. On the basis of results of stability experiments (ageing tests), however, it seems that method II leads to a slightly better result.

EXAMPLE II

The data and results of a number of experiments on a laboratory scale are recorded in table B. As in example I in all the experiments the isomerisation was carried out with NaOH, optionally in the presence of Na₂SO₃. The addition of NaClO₂ was carried out simultaneously with the addition of NaOH at about 95 °C.

Table B

Exp. No.	NaOH g/kg lactose.1H ₂ O	Na ₂ SO ₃ (ditto)	NaClO ₂ (ditto)	Colour after isomerisation *
5	2.5	-	-	13-16
6	2.0	2.0	-	3-10
7	1.9	-	2.7	3-8
8	3.5	-	1.2	13-16
9	3.5	-	3.9	3-8

* Gardner scale

Claims

1. A method of preparing lactulose by basic isomerisation of lactose, characterised in that the reaction mixture is treated with hydrogen peroxide or sodium chlorite.
2. A method as claimed in Claim 1, characterised in that 0.5-5 g of H₂O₂ are used per kg of lactose.1H₂O.
3. A method as claimed in Claims 1-2, characterised in that H₂O₂ is added at a pH 5.0-7.0, and at a temperature of 40-80 °C.
4. A method as claimed in Claims 1-2, characterised in that H₂O₂ is added at a pH of 7.5-8.0 and at a temperature of 90-100 °C.
5. A method as claimed in claim 1, characterized in that 1-5 g of NaClO₂ are used per kg of lactose.1H₂O at a temperature of 90-100 °C and under basic conditions.

Patentansprüche

1. Verfahren zur Herstellung von Lactulose durch basische Isomerisierung von Lactose, dadurch gekennzeichnet, daß die Reaktionsmischung mit Wasserstoffperoxid oder Natriumchlorit behandelt wird.
2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß 0,5 bis 5 g H₂O₂ pro kg Lactose.1H₂O verwendet werden.



3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß H_2O_2 bei einem pH von 5,0 bis 7,0 und bei einer Temperatur von 40 bis 80 °C zugesetzt wird.
- 5 4. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß H_2O_2 bei einem pH von 7,5 bis 8,0 und bei einer Temperatur von 90 bis 100 °C zugesetzt wird.
5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß 1 bis 5 g NaClO_2 pro kg Lactose. $\cdot\text{H}_2\text{O}$ bei einer Temperatur von 90 bis 100 °C und unter basischen Bedingungen verwendet werden.

10 Revendications

1. Procédé de préparation du lactulose par isomérisation basique du lactose, caractérisé en ce que le mélange réactionnel est traité avec du peroxyde d'hydrogène ou du chlorite de sodium.
- 15 2. Procédé selon la revendication 1, caractérisé en ce que 0,5 à 5 g d' H_2O_2 sont utilisés par kg de lactose. $\cdot\text{H}_2\text{O}$.
3. Procédé selon l'une des revendications 1 ou 2, caractérisé en ce que le H_2O_2 est ajouté à un pH de 5,0 - 7,0, et à une température de 40-80 °C.
- 20 4. Procédé selon l'une des revendications 1 ou 2, caractérisé en ce que H_2O_2 est ajouté à un pH de 7,5 - 8,0, et à une température de 90-100 °C.
5. Procédé selon la revendication 1, caractérisé en ce que 1 à 5 g de NaClO_2 sont utilisés par kg de lactose. $\cdot\text{H}_2\text{O}$ à une température de 90-100 °C et dans des conditions basiques.
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