

PROCESS FOR SEPARATION AND PURIFICATION OF GIBBERELLIN GA3 USING MAGNETIC RESIN

BACKGROUND

Technical Field

5 The present invention relates to a process for separation and purification of gibberellin GA3, and more particularly to a process for separation and purification of gibberellin GA3 using a magnetic resin, belonging to the bioseparation field.

Related Art

10 Gibberellins (GAs) are natural plant growth regulators and are one of the six plant hormones. Gibberellins have a good effect in promoting the germination of plant seeds, can be used to control the key stages of plant growth and development such as stem elongation, flower induction and seed production to speed up the growth and development, promote premature and increase the yield, and therefore have been widely used in agricultural production. There are a large variety of gibberellins. At present, 136 gibberellins have been
15 found and identified. Gibberellins that have been put into commercial production mainly include GA3, GA4 and GA7. Among others, GA3 is the most commonly used gibberellin product at home and abroad due to its high activity, and has been widely applied to the cultivation and transplantation of hybrid rice, cotton, vegetables and garden plants. It can promote the agricultural and forestry production (especially grain production) and has
20 extremely high economic and social benefits.

 Gibberellin GA3 used in agricultural production is produced by microbial fermentation. Suitable extraction processes include a reduced-pressure compression-extraction method, an activated carbon method, a resin adsorption method, a membrane separation method and so on. However, for all such methods for extracting gibberellin GA3, there is a loss to some
25 extent in the extraction process. Taking the most frequently used reduced-pressure compression-extraction method for example, the extraction rate is only about 80%, and a large quantity of low-concentration gibberellin GA3 separation residual liquid is produced in the extraction process. The extraction of gibberellin GA3 from the separation residual

liquid requires high costs. Therefore, during commercial production, such gibberellin GA3 separation residual liquid is usually disposed of as a waste, leading to a waste of resources.

SUMMARY

5 The present invention aims to solve the defect in the prior art and provide a process for separation and purification of gibberellin GA3 using a magnetic resin.

The technical solution of the present invention is that: in the present invention, efficient enrichment of gibberellin GA3 is achieved in a low-abundance complex solution by using a magnetic resin and under the action of an external magnetic field, and then gibberellin GA3 is separated as much as possible under the joint action of physical adsorption and magnetic
10 forces by gradient elution, following by concentration and drying, to give a high-purity gibberellin GA3 product.

A specific technical solution of the present invention is as follows: a process for separation and purification of gibberellin GA3 using a magnetic resin, comprising the following specific steps:

15 (1) adjusting the PH of a solution containing gibberellin GA3 to 2.0-5.0 with an acid, adding a filtering aid and filtering to obtain a filtrate ready for use, wherein the solution containing gibberellin GA3 is a separation residual liquid containing gibberellin GA3 in commercial production of gibberellin;

(2) placing a magnetic resin into a vertically disposed resin column, and placing the
20 resin column in an adjustable magnetic field with a magnetic induction intensity of 1.3-1.8 T, and introducing the filtrate obtained in the step (1) to flow through the resin column at a rate of 2-5 BV/h;

(3) changing an external magnetic field after the adsorption, introducing an ethanol solution having a volume concentration of 15%-20% to flow through the resin column at a
25 rate of 0.5-1.2 BV/h for desorption and impurity removal, and then introducing an ethanol solution having a volume concentration of 70%-75% to flow through the resin column at a rate of 2-3 BV/h for desorption, to obtain a desorption solution; and

(4) concentrating and crystallizing the desorption solution obtained in the step (3).

Preferably, the content of gibberellin GA3 in the solution containing gibberellin GA3 in the step (1) is 5%-25% by weight. Preferably, the acid used for adjusting the PH of the solution in the step (1) is hydrochloric acid or phosphoric acid.

Preferably, the filtering aid in the step (1) is perlite and/or diatomaceous earth.
5 Preferably, the mass ratio of the filtering aid to the solution is 1:(10-20).

Preferably, the magnetic resin in the step (2) is a magnetic polypropylene quaternary ammonium ion exchange resin, and more preferably a MIEX[®] DW1101 magnetic polypropylene quaternary ammonium ion exchange resin; the diameter height ratio of the resin column is 1:(5-10); and the adjustable magnetic field in the step (2) is a longitudinal
10 external magnetic field.

The changing of the external magnetic field in the step (3) is: maintaining the direction of the external magnetic field unchanged, and adjusting the magnetic induction intensity to 0.6-1.0 T.

Preferably, the desorption time of the ethanol solution having a volume concentration
15 of 15%-20% in the step (3) is 2-3 h; and the desorption time of the ethanol solution having a volume concentration of 70%-75% is 2-5 h.

Preferably, process conditions for concentrating and crystallizing the desorption solution in the step (4) are: 0.01 MPa-0.1 MPa, 30 °C-40 °C.

Beneficial effects:

20 The present invention solves the defect in the prior art, realizes the recycling of a low-concentration separation residual liquid, greatly simplifies the technological process, achieves an obviously higher product purity than that in the prior art, and thus meets the requirements of commercial production. This process does not require additional special equipment and makes full use of resources without greatly increasing enterprise costs, and
25 also avoids the use of a large amount of organic solvents in the extraction process, thus effectively protecting the ecological environment and the health of operators.

DETAILED DESCRIPTION

The present invention is further explained below through examples, but the examples does not constitute any limit to the present invention.

Example 1

5 The pH of a separation residual liquid containing 25% of gibberellin GA3 was adjusted to 2.0 with phosphoric acid. Diatomaceous earth, of which the mass ratio to the separation residual liquid containing 25% of gibberellin GA3 is 1:10, was added, followed by uniform blending and filtering. The filtrate was collected. A MIEX[®] DW1101 magnetic polypropylene quaternary ammonium ion exchange resin was placed in a resin column
10 having a diameter height ratio of 1:7. The resin column was vertically disposed in an external longitudinal magnetic field with a magnetic induction intensity of 1.8 T. The filtrate was passed through the resin column at a rate of 2 BV/h. After the adsorption was complete, the direction of the magnetic field was maintained unchanged, the magnetic induction intensity was adjusted to 0.9 T, and a 15% ethanol solution was introduced at a
15 rate of 1.2 BV/h to desorb the resin column for 3 h. The desorption solution was discarded. Then a 75% ethanol solution was introduced at a rate of 2 BV/h to desorb the resin column for 5 h for desorption and impurity removal. The desorption solution was concentrated and crystallized at 0.01 MPa and 30 °C to give a final gibberellin GA3 product. Tests showed that the recovery rate was 92% and the product purity was 96%.

20 Example 2

 The pH of a separation residual liquid containing 5% of gibberellin GA3 was adjusted to 5.0 with hydrochloric acid. Perlite, of which the mass ratio to the separation residual liquid containing 5% of gibberellin GA3 is 1:20, was added, followed by uniform blending and filtering. The filtrate was collected. A MIEX[®] DW1101 magnetic polypropylene
25 quaternary ammonium ion exchange resin was placed in a resin column having a diameter height ratio of 1:5. The resin column was vertically disposed in an external longitudinal magnetic field with a magnetic induction intensity of 1.4 T. The filtrate was passed through the resin column at a rate of 3 BV/h. After the adsorption was complete, the direction of the

magnetic field was maintained unchanged, the magnetic induction intensity was adjusted to 0.7 T, and a 18% ethanol solution was introduced at a rate of 0.5 BV/h to desorb the resin column for 2 h. The desorption solution was discarded. Then a 70% ethanol solution was introduced at a rate of 3 BV/h to desorb the resin column for 2 h for desorption and impurity removal. The desorption solution was concentrated and crystallized at 0.05 MPa and 40 °C to give a final gibberellin GA3 product. Tests showed that the recovery rate was 91% and the product purity was 95%.

Example 3

The pH of a separation residual liquid containing 18% of gibberellin GA3 was adjusted to 3.0 with hydrochloric acid. A mixture of diatomaceous earth and perlite (the mass ratio of diatomaceous earth to perlite being 1:1), of which the mass ratio to the separation residual liquid containing 18% of gibberellin GA3 is 1:15, was added, followed by uniform blending and filtering. The filtrate was collected. A MIEX[®] DW1101 magnetic polypropylene quaternary ammonium ion exchange resin was placed in a resin column having a diameter height ratio of 1:10. The resin column was vertically disposed in an external longitudinal magnetic field with a magnetic induction intensity of 1.3 T. The filtrate was passed through the resin column at a rate of 5 BV/h. After the adsorption was complete, the direction of the magnetic field was maintained unchanged, the magnetic induction intensity was adjusted to 0.6 T, and a 20% ethanol solution was introduced at a rate of 0.9 BV/h to desorb the resin column for 2.5 h for desorption and impurity removal. Then a 73% ethanol solution was introduced at a rate of 2.5 BV/h to desorb the resin column for 4 h. The desorption solution was collected. The desorption solution was concentrated and crystallized at 0.1 MPa and 36 °C to give a final gibberellin GA3 product. Tests showed that the recovery rate was 87% and the product purity was 95%.

Example 4

The pH of a separation residual liquid containing 11% of gibberellin GA3 was adjusted to 4.0 with phosphoric acid. Diatomaceous earth, of which the mass ratio to the separation residual liquid containing 11% of gibberellin GA3 is 1:15, was added, followed by uniform blending and filtering. The filtrate was collected. A MIEX[®] DW1101 magnetic

polypropylene quaternary ammonium ion exchange resin was placed in a resin column having a diameter height ratio of 1:8. The resin column was vertically disposed in an external longitudinal magnetic field with a magnetic induction intensity of 1.5 T. The filtrate was passed through the resin column at a rate of 4 BV/h. After the adsorption was
5 complete, the direction of the magnetic field was maintained unchanged, the magnetic induction intensity was adjusted to 1.0 T, and a 15% ethanol solution was introduced at a rate of 1.0 BV/h to desorb the resin column for 2 h for desorption and impurity removal. Then a 75% ethanol solution was introduced at a rate of 2 BV/h to desorb the resin column for 3 h. The desorption solution was collected. The desorption solution was concentrated
10 and crystallized at 0.01 MPa and 30 °C to give a final gibberellin GA3 product. Tests showed that the recovery rate was 89% and the product purity was 97%.

CLAIMS

What is claimed is:

1. A process for separation and purification of gibberellin GA3 using a magnetic resin, comprising the following specific steps:

5 (1) adjusting the PH of a solution containing gibberellin GA3 to 2.0-5.0 with an acid, adding a filtering aid and filtering to obtain a filtrate ready for use, wherein the solution containing gibberellin GA3 is a separation residual liquid containing gibberellin GA3 in commercial production of gibberellin;

10 (2) placing a magnetic resin into a vertically disposed resin column, and placing the resin column in an adjustable magnetic field with a magnetic induction intensity of 1.3-1.8 T, and introducing the filtrate obtained in the step (1) to flow through the resin column at a rate of 2-5 BV/h;

15 (3) changing an external magnetic field after the adsorption, introducing an ethanol solution having a volume concentration of 15%-20% to flow through the resin column at a rate of 0.5-1.2 BV/h for desorption and impurity removal, and then introducing an ethanol solution having a volume concentration of 70%-75% to flow through the resin column at a rate of 2-3 BV/h for desorption, to obtain a desorption solution; and

(4) concentrating and crystallizing the desorption solution obtained in the step (3).

20 2. The process of claim 1, wherein the content of gibberellin GA3 in the solution containing gibberellin GA3 in the step (1) is 5%-25% by weight.

3. The process of claim 1, wherein the acid used for adjusting the PH of the solution in the step (1) is hydrochloric acid or phosphoric acid.

4. The process of claim 1, wherein the filtering aid in the step (1) is perlite and/or diatomaceous earth; and the mass ratio of the filtering aid to the solution is 1:(10-20).

25 5. The process of claim 1, wherein the magnetic resin in the step (2) is a magnetic polypropylene quaternary ammonium ion exchange resin; the diameter height ratio of the resin column is 1:(5-10); and the adjustable magnetic field in the step (2) is a longitudinal

external magnetic field.

6. The process of claim 1, wherein the changing of the external magnetic field in the step (3) is: maintaining the direction of the external magnetic field unchanged, and adjusting the magnetic induction intensity to 0.6-1.0 T.

5 7. The process of claim 1, wherein the desorption time of the ethanol solution having a volume concentration of 15%-20% in the step (3) is 2-3 h; and the desorption time of the ethanol solution having a volume concentration of 70%-75% is 2-5 h.

8. The process of claim 1, wherein process conditions for concentrating and crystallizing the desorption solution in the step (4) are: 0.01 MPa-0.1 MPa, 30 °C-40 °C.

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