Ethyl Sorbate Was Prepared by Micro Reactor Low-Pressure

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1. Introduction
The micro-reactor realizes precise control of reaction time and temperature, especially in the chemical industry, the three-pass-one-counter-control master is accurate. At present, the micro-reactor equipment is expensive, and it is only used in fine chemical fields such as high temperature and high pressure, especially pharmaceutical intermediates. The application is more extensive.

Ethyl sorbate is used in livestock and poultry disinfectants to kill pathogenic bacteria and viruses. Adding ethyl sorbate to livestock water or feed can prevent disease and promote growth. Sorbic acid is mixed with pesticides such as organic phosphorus, which has good affinity and stability, and can also play a synergistic effect. Ethyl sorbate can normally participate in metabolism in the body, and finally produces carbon dioxide and water. The US Food and Drug Administration has determined that it is GRAS (basic non-toxic).

There are currently no reports on the use of microreactors to prepare ethyl sorbate. In this study, the most reasonable residence time and reaction temperature of the synthesis experiment were obtained by using the advantage experiment of Corning reactor. The material is fed in a certain proportion. When the material is made into a transparent liquid, the feed is kept warm, and a reasonable reaction time is obtained through experiments, that is, the most reasonable residence time and reaction temperature are obtained.

2. Experimental part
2.1. Experimental materials and instruments
Experimental drugs: sorbic acid (CP, Kelon Chemical Co., Ltd.), absolute ethanol (CP, Chongqing Jiangchuan Chemical Co., Ltd.), p-toluenesulfonic acid (CP, Kelon Chemical Co., Ltd.), sodium bicarbonate (CP, Chongqing Jiangchuan) Chemical Co., Ltd.).
Experimental equipment: micro-reactor (American Corning), four-necked flask, agitator, oil bath, etc.

2.2. Experiment procedure
In a 5 L four-necked flask, sorbic acid, absolute ethanol and p-toluenesulfonic acid were mixed and dissolved in a molar ratio of 1:3:0.16, and kept at 70 °C until use. The material was driven into the
microreactor at a flow rate using a KP-22 polytetrafluoro pump. When entering the microreactor, the solution density should be determined in advance. Do the pump calibration and total liquid usage. Experiments were carried out according to the microreactor operating steps. The initial product is post-treated to give a low level of ethyl sorbate.

3. Experimental results and discussion

3.1. Effect of Different Temperatures on Ethyl Sorbic Acid Content and Conversion Rate

In this group of tests, the pressure is controlled at 0.9-1.1 MPa, the material molar ratio is 1:6:0.16, and the feed is kept at 70 °C.

From Fig. 1, it can be seen that the ethyl sorbate content gradually decreases with the decrease of residence time at the same reaction temperature, which is consistent with the esterification reaction mechanism; at the same temperature, the residence time is 1-4 min, Yamanashi the content of acid ethyl ester changed slowly. When the reaction temperature was 140 °C, the content of ethyl sorbate increased more slowly during the residence time of 1-16 min. The content of ethyl sorbate was the highest at 16 min. The reaction temperature was 120 °C. The content of ethyl sorbate was higher than that of other sorbic acid at other temperatures between 1 and 16 minutes.

It can be seen from Fig. 2 that when the reaction temperature is different, the residence time of the residence time is 8min; the reaction temperature is 100, 120°C and 85°C, and the residence time is 1-16min.
3.2. Effect of Different Reaction Time on Ethyl Sorbic Acid Content and Conversion Rate

It can be seen from Fig. 3 that when the reaction temperature is controlled at 120 °C, the content of ethyl sorbate peaks at different residence time, which is optimal; when the temperature is 140 °C, sorbic acid and ethyl sorbate occur at high temperature. The reaction is inferred that the carbon-carbon double bond is broken or oxidized, resulting in a decrease in the target product content.

It can be seen from Fig. 4 that the conversion rate of the esterification reaction increases as the reaction temperature increases; at the same temperature, the relationship between the conversion rate and time is theoretically the longer the time, and the higher the conversion rate. From the 4th and 8min trends in Figure 4, the trend is quite abnormal. The reason is that there is a certain error in the conversion rate calculation formula, and the sorbic acid side reaction product is not considered.
4. Conclusion
The esterification reaction is a thermodynamically controlled reversible reaction with a long reaction time and a high reaction temperature and a high conversion rate. Under certain conditions of the catalyst, the esterification reaction is greatly affected by temperature and time. From the experimental data, it is concluded that the higher the esterification reaction temperature is, the more favorable the temperature is to the main reaction. In the experiment, the reaction temperature was optimally controlled at 120 °C; the conversion rate was the best when the residence time was 8 min.

Using Corning reactor to synthesize ethyl sorbate, the reaction temperature in the small test can only be controlled at the boiling temperature of the material at 81 °C, the reaction temperature can reach 140 °C; the reaction time is greatly shortened, and the optimal time for the small test reaction is 6 h. The reaction was completed by using the apparatus for 8 minutes. However, the solubility of sorbic acid in ethanol is greatly affected by temperature. Since there is no heat preservation measure in the feed pipe, the material is easy to crystallize and block the feed pipe during feeding, especially under the condition that the molar ratio of sorbic acid to ethanol is relatively close. If the reactor feed line cannot be insulated, it is impossible to carry out the experiment in which the molar ratio of the reaction raw materials is relatively small. With this device, the minimum conversion rate of the reaction is close to 70%. The reaction is more adequate.

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