RESEARCH ARTICLE

Synthesis and application of high quality sorbitan monooleate (span80)

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Sorbitan monooleate (span80) is a kind of emulsifier with excellent performance, which is widely used in cosmetics, food, and medicine. In order to solve some quality problems of domestic span80 product such as dark color, uneven reaction products, lots of by-products, and much sediment, the production process of span80 synthesized by etherification followed by esterification was studied. And the selection of catalysts and the optimization of reaction conditions in the section of etherification were mainly discussed. The results showed that the optimum conditions of sorbitol etherification reaction were as follows: the concentration of catalyst Z1 was 1.1%, the reaction temperature was 150°C, the reaction time was 90 minutes, the hydroxyl value of the synthetic sorbitan was 1,375 ~ 1,399 mgKOH/g, and the degree of water loss was 0.93 ~ 0.98 under the conditions of nitrogen protection and the vacuum degree no less than 0.096 Mpa. The synthesized span80 product showed the advantages of lighter color, lower viscosity, and better fluidity, etc. And the acid value, saponification value, hydroxyl value, and other quality indicators of span80 product had reached the standard of superior grade. Moreover, when the product was used as emulsifier, its emulsifying performance was much better than that of before. The resulted products of this study demonstrated the quality improvement of span80 synthesis.

Keywords: sorbitol; etherification; hydroxyl value; Span80; oleic acid.

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Introduction

The chemical name of span80 for is sorbitan monooleate. It is synthesized by the esterification reaction of oleic acid with water-loss sorbitol. Its hydrophilic oil-wet equilibrium value (HLB value) is 4.3. It can be water-in-oil (W/O) emulsifier by mixing with oil, which belongs to non-ionic surfactant [1-4], and has excellent properties of emulsification, dispersion, foaming, and wetting. As an emulsifier with excellent performance, it has been widely used in the pharmaceutical industries [5], biological tissue engineering [6], pharmacy [7], biopolymer materials [8], and other research fields in recent years. Although there are many domestic manufacturers at present, there are some defects in different degrees such as poor color, non-uniform reaction products, large amount of sedimentation, and high freezing point [9]. In order to improve and stabilize the product quality of span80, many efforts have been made in the production process improvement [10]. In China, some researchers also proposed a synthesis reaction mathematical model for the sorbitol esterification [11]. In fact, the key technology in the preparation of Span process is the formation and protection of reasonable hydrophilic groups' structures and quantities. Previous study found that Span series products

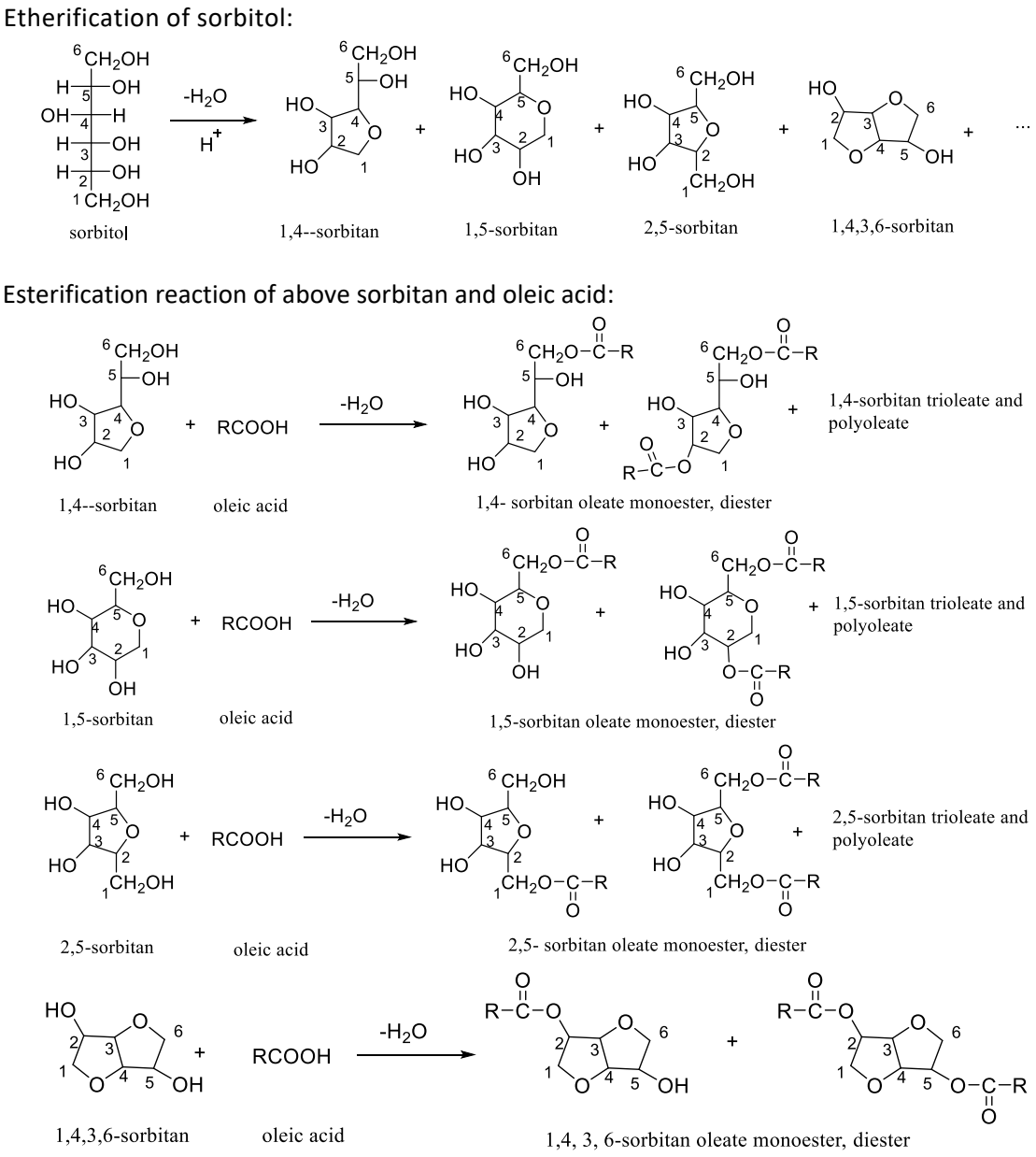

Figure 1. The specific reaction processes for span80 synthesis.

had the best practical performance when the water loss degree of sorbitol was 1.2 - 1.5 [10]. It has great significance to investigate the water loss reaction of sorbitol for the study of the synthesis technology with reasonable structures.

Span80 can be synthesized by the methods of one-step synthesis, esterification followed by etherification, and enzyme-catalyzed synthesis. Among them, the method of etherification followed by esterification is considered as the most economical and feasible method because of its mild reaction conditions, less impurities, and lighter color. The basic principle of two-steps synthesis of span80 by etherification followed by esterification is as follows. Sorbitol is first dehydrated under the action of etherification catalyst to form sorbitan, such as 1,4-sorbitan, 1,5-sorbitan, 2,5-sorbitan, and 1,4,3,6-sorbitan, etc. Then, oleic acid and esterification catalyst

are added to esterify above sorbitols and oleic acid to produce sorbitan monooleate (single ester), sorbitan dioleate (diester), sorbitan trioleate, and polyoleate (polyester), *etc*. The specific reaction processes are shown in Figure 1. The key of the synthesis is to balance the relationship between water loss and esterification. If the water loss is excessive, the hydroxyl value of the product is lower, the acid value is higher, and the esterification degree is more insufficient. On the contrary, if the esterification is complete and the dehydration is insufficient, not only too much monoester, diester, and polyester will be produced, but also the cause of easily unreasonable distribution of the esterification contents and hydroxyl value of the product will be higher. Previous study suggested that the best product was obtained when sorbitol water loss was controlled at 1.2 - 1.5 [10]. The effective control of sorbitol water loss depends on the catalyst of etherification reaction.

In order to solve the quality problems, this study focused on the synthesis process and key technical parameters of span80. The production process of span80 synthesized by etherification followed by esterification was investigated based on previous relevant studies [12-17]. The selection of catalysts and the optimization of reaction process conditions in the etherification section were mainly explored to improve the production process.

Materials and methods

Sorbitan etherification and its orthogonal experimental design

Sorbitol etherification was carried out in a 100 mL four-mouth flask with a thermometer, an oil-water separator, and a spherical condensation tube (Sichuan Shubo Group Co., LTD., Chengdu, Sichuan, China). 10 g of anhydrous sorbitol powder (Shanghai Lingfeng Chemical Reagent Co., LTD., Shanghai, China) was mixed with 0.1 g of etherification catalyst, $AlCl_3$ (catalyst Z1) (Shandong Zibo Yundi Chemical Co., LTD., Zibo, Shandong, China) with a mass ratio of 100:1. The reaction was vacuumed by using SHZ-D (ⅲ) Circulating Water Vacuum Pump (Shanghai Baoling Instrument Equipment Co., LTD., Shanghai, China) with the vacuum degree of the system set at no less than 0.096 Mpa. The system was protected by nitrogen. The system temperature was controlled by using HHS type digital display constant temperature oil bath (Jintan Jingda Instrument Manufacturing Co., LTD., Changzhou, Jiangsu, China). The reaction temperature was set at 150℃ and the reaction time was 120 min. After the reaction, samples were taken to determine the hydroxyl value and color number. The hydroxyl value of the reaction product was regulated at 1,300~1,400 mgKOH/g. The color number was about 2.0. The water loss of sorbitol was calculated by using formula (1):

$$n = \frac{1849 - V_{OH}}{482} \quad (1)$$

where n was the water loss degree of sorbitol. V_{OH} was the hydroxyl value of sorbitol after dehydration. The number 1849 was the theoretical hydroxyl value per mole of sorbitol and number 482 was the reduced hydroxyl value by dehydration of two hydroxyl groups to form a ring per mole of sorbitol.

In order to comprehensively study the influence of various factors on the sorbitol water loss and to determine the optimal reaction conditions of sorbitol water loss reaction, the influence of catalyst Z1 concentration, reaction temperature, and reaction time were studied by using three-factor and three-level orthogonal test method under the condition of certain vacuum degree and taking hydroxyl value and color number as the main indexes in this study. Orthogonal experimental factors and levels were shown in Table 1.

Synthesis of Span80 by esterification

The esterification reaction is carried out in the same reactor as the etherification reaction. At

Table 1. Factors and levels of orthogonal experiments.

Factor	Factor Code	Level Ⅰ	Level Ⅱ	Level Ⅲ
Catalyst Z_1 concentration (%wt)	A	0.9	1.1	1.3
Reaction temperature (℃)	B	120	150	180
Reaction time (min)	C	90	120	150

the end of etherification, 25 g of oleic acid (Shanghai Lingfeng Chemical Reagent Co., LTD., Shanghai, China) and 0.2 g of esterification catalyst ($NaOH:Na_2CO_3$ = 5:2) (catalyst Z2) (Nanjing Chemical Reagent CO.,LTD., Nanjing, Jiangsu, China) were added to the above reaction. The molar ratio of anhydrous sorbitol to oleic acid was 1:1.61. The amount of catalyst Z2 added to the reaction was twice more than catalyst Z1. In the same way, the system was protected by N_2 with a vacuum degree of no less than 0.096 Mpa. The reaction temperature was 200℃ and the reaction time was 150 min. After the reaction, samples were taken to determine the acid value. The acid value of the reaction product was controlled to be less than or equal to 7 mgKOH/g.

Emulsion preparation

The ratio of emulsion was set as $W_{oil}:W_{water}:W_{span80}$ = 74:24:2. The mixtures were prepared by mixing 2 g of span80 emulsifier synthesized by above method and 74 g of Dori sunflower oil (Shanghai Jiage Food Co., LTD., Shanghai, China) with 24 g of pre-heated deionized water. The mixture was stirred and heated to 65℃ by using IKA RW20 mixer (IKA®-Werke GmbH & Co. KG, Staufen, Baden-Württemberg, Germany) at 2,000 rpm for 30 min to make W/O emulsion [18-20].

Determination of quality indexes

The values of acid, hydroxyl, and saponification of the experimental products were analyzed according to national standards [21-23]. The transparency and color of the product were detected. Shimadzu LC-20AT/SPD-20A high performance liquid chromatography (HPLC) (Shimadzu Corporation, Kyoto, Japan) was used for qualitative and quantitative analysis of the products. The experimental operating conditions of HPLC were as follows:

mobile phase: $V_{CH_3OH}:V_{H_2O}$ = 90:10
flow speed: 1 mL/min
UV detection wavelength: 220 nm
detection temperature: 40℃
inertsil column: ODS-SP, 4.6 mm × 150 mm, 5 μm fixed phase filled.
injection quantity: 20 μL

The contents of various esters in the product were calculated by area normalization method.

Determination of emulsifying power

The stability of the emulsion was evaluated by centrifugal separation method. 5 mL of the emulsion prepared above was added into 10 mL graduated centrifugal tube and was centrifuged at 3,000 rpm for 15 min by using a TG16-WS high-speed centrifuge (Hunan Xiangyi Group, Changsha, Hunan, China). The volume of the precipitated water was measured thereafter. The emulsifying power was assessed by calculating the water separation rate [20] as follows:

$$R = \frac{V}{V_0} \times 100\% \quad (2)$$

where *R* was the water separation rate. *V* was the volume of precipitated water. V_0 was the total volume of water being added.

Results and discussion

Sorbitan orthogonal experiment

The orthogonal experimental results were shown in Table 2.

Table 2. The orthogonal experimental results of sorbitol water loss reaction.

Number	Catalyst Z_1 concentration (A)	Reaction temperature (B)	Reaction time (C)	Experimental results	
				Hydroxyl value	Color number
1	0.9	120	90	1,463	1.0
2	0.9	150	120	1,439	1.5
3	0.9	180	150	1,415	2.0
4	1.1	120	120	1,391	2.0
5	1.1	150	150	1,367	2.0
6	1.1	180	90	1,367	2.0
7	1.3	120	150	1,391	2.5
8	1.3	150	90	1,367	2.5
9	1.3	180	120	1,367	3.0

Table 3. Effects of different catalysts on sorbitol water loss reaction.

Catalysts dosage (mass fraction of 1%)	Hydroxyl value of sorbitan (mgKOH/g)	Water loss degree of sorbitol
concentrated sulfuric acid	162	3.5
p-toluenesulfonic acid	499	2.8
orthophosphoric acid	1,030	1.7
phosphorous acid	1,271	1.2
sodium hydroxide	1,656	0.40
Potassium hydroxide	1,666	0.38
sodium carbonate	1,632	0.45
catalyst Z1	1,386	0.96

Influence of different catalysts

Previous studies showed that sorbitol lost water faster under the action of acidic catalyst, while the esterification speed was faster, and the water loss reaction speed was slower under the action of alkaline catalyst [24]. Therefore, acid catalysts are suitable for dehydration reaction and alkaline catalysts are suitable for esterification reaction. Meanwhile, the study of Zhu, *et al*. showed that, when the sorbitol water loss reaction was completed, if the hydroxyl value of the product was controlled at 1,300 - 1,400 mgKOH/g, it was assured that the sorbitol water loss was at around 1.0 [10]. Then, during the subsequent esterification reaction, the total water loss of sorbitol could be easily controlled within 1.2 - 1.5, and finally, span80 products with reasonable structures, indexes, and good quality could be obtained. Sorbitol water loss reaction was studied by using the acid and alkaline catalysts, and the results were shown in Table 3.

The results demonstrated that the more acidic the catalyst was, the lower the hydroxyl value of sorbitan was, and the greater the water loss degree of sorbitol was. The sorbitol had a very low water loss with alkaline catalysts. According to the principles and requirements of the reaction, a suitable catalyst Z1 was selected in this study. Under the action of catalyst Z1, the water loss degree of sorbitol was effectively controlled at about 1.0.

Influence of catalyst dosage

The effects of the dosage of catalyst Z1 on the conversion rate and water loss of sorbitol were shown in Table 4. The results demonstrated that, when the other reaction conditions unchanged, conversion rate and water loss degree of sorbitol increased with the increased concentration of catalyst Z1. Moreover, the two values were equal, indicating that the catalyst Z1 had a good selectivity for sorbitol water loss reaction. Under

Table 4. Effects of catalyst Z1 dosage on the conversion and water loss of sorbitol.

Catalyst Z_1 Concentration (%wt)	Hydroxyl value of sorbitan ($mgKOH \cdot g^{-1}$)	Water Loss Degree of Sorbitol (n)	Conversion Rate of Sorbitol (%)
0.5	1,694.8	0.32	32
0.7	1,559.8	0.60	60
0.8	1,502.0	0.72	72
0.9	1,439.3	0.85	85
1.0	1,386.3	0.96	96
1.1	1,367.0	1.00	100
1.2	1,367.0	1.00	100

Table 5. Results of range analysis.

	Levels/ Factors	Catalyst Z_1 Concentration (A)	Reaction Temperature (B)	Reaction Time (C)
hydroxyl value	Ⅰ/3	1,439	1,415	1,399
	Ⅱ/3	1,375	1,391	1,399
	Ⅲ/3	1,375	1,381	1,391
	R	64	34	8
color number	Ⅰ/3	1.5	1.83	1.83
	Ⅱ/3	2.0	2.0	2.17
	Ⅲ/3	2.67	2.33	2.17
	R	1.17	0.5	0.34

the action of the catalyst Z1, sorbitol only carried out a single water loss reaction instead of producing 1, 4, 3, 6-water loss and other double water loss sorbitol. So that the water loss degree of sorbitol could be effectively controlled at about 1.0. It was also indicated that, when the catalyst concentration reached 1.1% or above, the conversion rate and water loss of sorbitol would not change.

Optimization of sorbitol water loss reaction process conditions

According to the results from orthogonal experiment (Table 2), the hydroxyl value and color number of dehydrated sorbitol were used as the indexes. The range analysis of the orthogonal experiment results was shown in Table 5. The relationship curves between different levels of each factor and the indexes were shown in Figure 2-4.

Figure 2 demonstrated that (1) catalyst Z1 concentration had the most significant effect on hydroxyl value. When the concentration of catalyst Z1 increased from 0.9% to 1.1%, the hydroxyl value decreased rapidly from 1,439 mgKOH/g to 1,375 mgKOH/g. However, continuously increasing catalyst Z1, the hydroxyl value was almost no longer changed, which indicated that the dehydration reaction would no longer lose water to a certain degree, while the corresponding water loss degree was about 1.0. The results showed that, under the effect of the catalyst, sorbitol could only undergo single water loss reactions and the catalyst Z1 had a good selectivity for sorbitol water loss reaction; (2) The effect of catalyst concentration on color was also the greatest. With the increase of catalyst concentration, the color number deepened from 1.5 to 2.6. Therefore, the precise control of catalyst concentration on sorbitol water loss reaction is very important. Combined with the

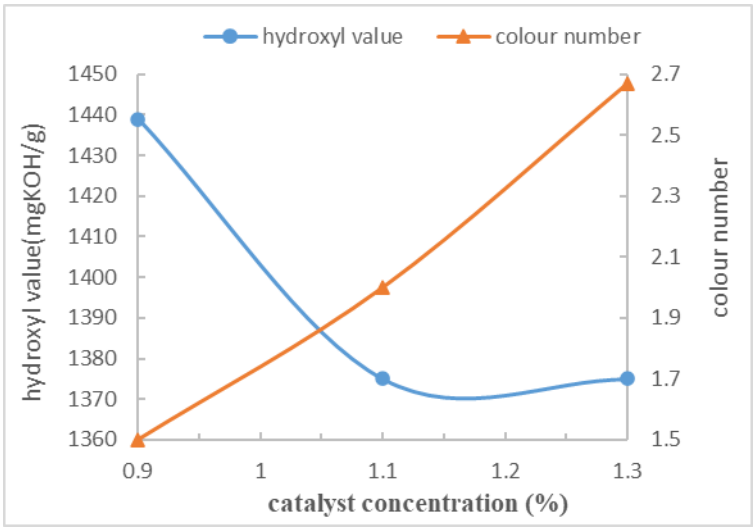

Figure 2. Effects of three levels of catalyst concentration on hydroxyl value and color of sorbitan.

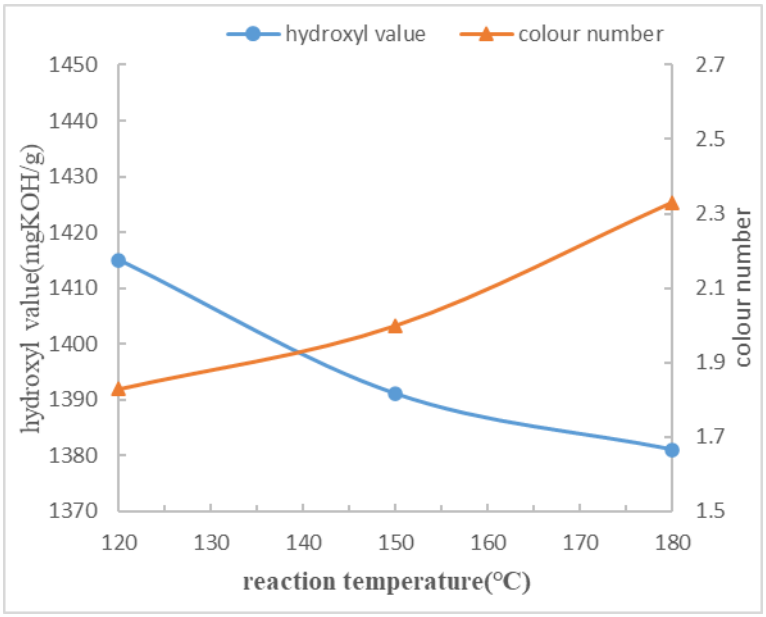

Figure 3. Effects of three levels of reaction temperature on hydroxyl value and color of sorbitan.

requirements of the evaluation index of water loss reaction, the hydroxyl value should be controlled at 1,300 mgKOH/g to 1,400 mgKOH/g, and the lower the color was, the better the product was. So, the catalyst Z1 concentration should be 1.1%.

The results demonstrated on Figure 3 showed that the reaction temperature had also a great influence on the water loss reaction of sorbitol. With the increase of temperature from 120 to 180°C, the hydroxyl value of the product decreased gradually while the color deepened gradually as well. Obviously, the effect of reaction temperature is also very important. Considering the strict requirement of hydroxyl value, the best reaction temperature should be 150°C.

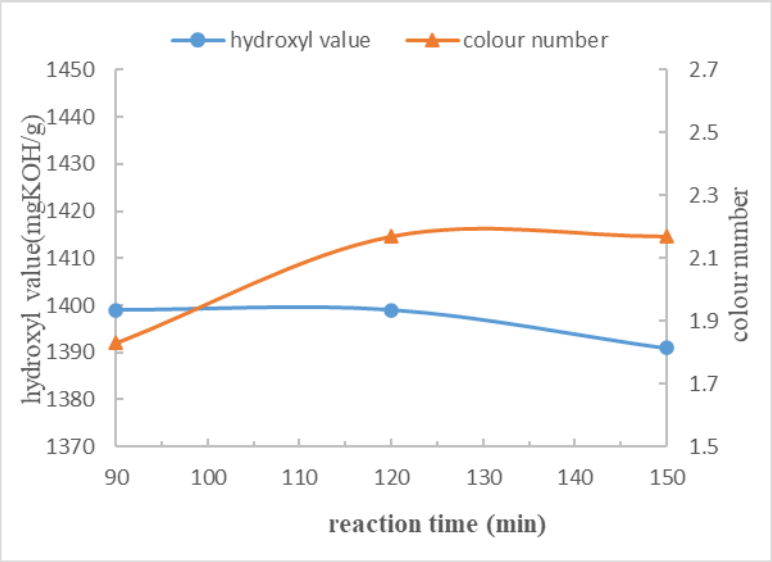

Figure 4. Effects of three levels of reaction time on hydroxyl value and color of sorbitan.

Table 6. Quality indexes of span80 products synthesized by different processes.

Sample Index	Three Quality Indexes (mgKOH/g)			Esterification Product Content (%)			Viscosity (25℃) (mPa·s)	Color Number
	Hydroxyl value	*Saponification value*	*Acid value*	*Single ester*	*Diester*	*Poly ester*		
Import sample	198	155	6.2	58	34	8	985	6
Current sample 1	202	152	5.8	62	30	8	960	6
Current sample 2	198	154	6.0	62	31	7	955	6
Original sample	215	148	8.8	45	43	12	1105	10

Figure 4 showed that the reaction time had little influence on hydroxyl value and color. Considering the control requirements of indexes and production cost saving, the reaction time should be 90 minutes.

In conclusion, the optimal control conditions of sorbitol water loss reaction were as follows: under certain vacuum degree (0.096 Mpa), the catalyst Z1 concentration was 1.1%, the reaction temperature was 150℃, the reaction time was 90 minutes, the hydroxyl value of sorbitan was 1,375 mgKOH/g to 1,399 mgKOH/g, and the water loss was 0.93 to 0.98.

Verification of reaction conditions

Span80 products were synthesized according to the optimum synthesis conditions of sorbitol water loss reaction. The following esterification reaction conditions were unchanged. The quality indexes of the products were analyzed and compared to that of the imported high-quality span80 from United States and Jiangsu Hai 'an Petrochemical Plant span80. The results were shown in Table 6. All three quality indexes of the process products met the quality standards of span80 products. The distribution of esterification was reasonable, the viscosity and color were low, and the quality indexes were close to the imported sample. Some indexes such as monoester content and viscosity were even better than that of the imported sample. In addition, the monoester content, viscosity, and

Table 7. Emulsifying properties of different span80 samples.

Sample Types	Hydroxyl Value (mgKOH/g)	Saponification Value (mgKOH/g)	Acid Value (mgKOH/g)	Single ester: Diester: Polyester	Emulsifying ability (%)
Import sample	198	155	6.2	58:34:8	20
Current sample	200	153	5.9	62:30:8	18
Original sample	215	148	8.8	45:43:12	35

color were obviously better than that of the original process sample, which further verified the rationality of the process improvement.

Application of product

The emulsifying properties of span80 products were evaluated by centrifugal separation method and compared to several other span80 samples (Table 7). According to formula 2, the higher the value of emulsifying power is, the more water is precipitated out of the emulsion under the same conditions, and the more unstable the emulsifier will be. It can thus be concluded that among the above three samples, span80 samples prepared by this process had the best emulsification performance, followed by imported samples, and the emulsifying property of the process sample was obviously improved comparing to that of the original process sample. It can also be concluded that the emulsifying power was proportional to the single ester content in the sample. Relatively, the higher the single ester content was, the more stable the emulsification performance was.

Conclusion

A suitable catalyst Z1 was selected in this study. The sorbitol water loss was effectively controlled at about 1.0 under the action of catalyst Z1. Under the action of catalyst Z1, the optimal technological conditions of sorbitol water loss reaction were as follows: the concentration of catalyst Z1 was 1.1%, the reaction temperature was 150℃, the reaction time was 90 minutes, and the hydroxyl value of sorbitan was 1,375 mgKOH/g to 1,399 mgKOH/g, the water loss was 0.93 to 0.98 under the conditions of nitrogen protection and vacuum degree not less than 0.096 Mpa. The hydroxyl value of the products synthesized by this process was 200 mgKOH/g. The saponification value was 153 mgKOH/g. The acid value was 5.9 mgKOH/g. The ratio of monoester, diester, and polyester in the product was about 62:30:8. The distribution of three esters was reasonable. The product had lower viscosity and color. All indexes met the standard of high-quality products. Moreover, when the product was used as emulsifier, the emulsifying performance of the product was significantly improved comparing to that of the original process products. This newly developed process also had the following advantages including (1) don't need to change the current production process device, only need to replace the catalyst for etherification reaction to be put into production, which is easy to industrialize; (2) due to the light color of the products produced by this process, there is no need for subsequent decolorization, which simplifies the production process and reduces the costs.

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