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Some Reflections On Styrene Suspension Polymerization Experiment Teaching

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Abstract. The results of suspension polymerization of styrene were studied by changing the dosage of initiator, stirring speed, size of stirring paddle, content of dispersant and reaction temperature. The results showed that when using 500 ml four flasks, styrene 40 ml and polyvinyl alcohol solution (3 g polyvinyl alcohol dissolved in 1000 ml deionized water) 200 ml, initiator 0.5 g, stirring speed 300-450 r/min, temperature range 86-95ºC, using the stepped heating method and using the impeller with a spreading blade diameter of 43 mm can ensure the success of the experiment, and the yield is relatively high.

1. Introduction

Suspension polymerization is one of the important methods to prepare synthetic resin. It is a suspension polymerization in which the initiator monomer is dispersed in a medium (usually water) which is not soluble with the monomer under the action of strong mechanical stirring force and dispersant. Polystyrene is one of the five general plastics in the world today and has a wide range of USES. Suspension polymerization is the main method of synthesis of polystyrene. Zhang Jianli et al. [1] studied the factors affecting particle size in styrene suspension polymerization. Wang qiao et al. [2] studied the effects of initiator dosage, stirring speed, temperature, dispersant dosage and ratio of water to monomer on suspension polymerization of styrene. Tang Xiaohong et al. [3] studied the effects of initiator dosage, suspension agent dosage, stirring speed and reaction temperature on the suspension polymerization of styrene. Ge Jianfang et al. [4] have studied the addition of hexagonal boron nitride to suspension polymerization system to change the size uniformity of styrene microspheres. Meanwhile, through the combination of classroom teaching and extracurricular experiments, students can cultivate individualized and innovative thinking and improve the teaching quality and effect. Zhao Dongmei et al. [5] has seven different uses. The color polystyrene microspheres were prepared with styrene as monomer, benzoyl peroxide as catalyst, polyvinyl alcohol as dispersant and divinylbenzene as crosslinking agent. Arouse students' interest. Li Qiuxia et al. [6] used polyvinyl alcohol as suspension dispersant, benzoyl peroxide as initiator, tertiary dodecyl mercaptan as chain transfer agent to prepare white granular polystyrene with controllable molecular weight by suspension polymerization. The author found that the suspension polymerization experiment was easily influenced by the reaction conditions in the process of the teaching of styrene suspension polymerization experiment.

In this paper, the success rate of styrene suspension polymerization was discussed by changing the dosage of initiator, stirring speed, the size of impeller blade, temperature range and dispersant content.

2. Experimental

2.1. Reagent and instrument

Styrene (chemical purity), guangdong shantou xilong chemical company; Sodium hydroxide (analytical purity); Pva (chemical pure), jinan hengrui chemical co., LTD. Benzoyl peroxide (BPO, analytically pure).

1 standard 4-mouth bottle (500ml);1 condensate tube (300mm); One thermocouple; One separating funnel (125ml);Beaker (100ml) two;1 electric heating package; One set of electric mixer.

2.2. Experimental processes

(1) In the beaker, 40ml styrene washed by sodium hydroxide solution and 0.5g peroxybenzoyl, the initiator, were added and stirred by hand to dissolve.

(2) Add 0.6g polyvinyl alcohol and 200ml deionized water in another beaker, heat it to boiling and dissolve it into a uniform transparent solution for later use (note: it has been dissolved in advance).

(3) Add 200ml polyvinyl alcohol solution to 500ml four-necked flask with thermometer, mixer and reflux condenser. Stir and heat up. After being up to 80 ℃, molten have joined the initiator of the styrene monomer. Careful adjustment of stirring speed is generally controlled at 300-450r/min, so that the monomer is dispersed into droplets of appropriate size.

(4) The droplet size after adjust well, up to 90 ℃, heat preservation after 3 h extracted several particles, observe whether the cooled into bead solids. If so, will raise the temperature to 95 ℃, insulation 1 h, the reaction to an end.

(5) Immediately pour the reactant into the beaker, wash with room temperature deionized water for three times, and then filter. Bead polymers in petri dishes, in 50 ℃ drum wind drying oven drying to constant weight, weighing, calculating the yield.

3. Results and discussion

3.1. Effect of initiator content on polymerization reaction

Experimental conditions: after alkali washing styrene 40ml, polyvinyl alcohol solution 200ml, stirring speed 400r/min, reaction temperature of small stirrer (blade diameter: width when blade is fully expanded, 43mm) was described in the heating step of the experimental steps. The influence of initiator BPO dosage on the polymerization reaction was examined, and the results were shown in table 1.

It can be seen from table 1 that in the case of using a small stirring propeller (blade diameter: 43mm), the yield gradually increased with the increase of initiator. When the content of initiator was 0.6g, the polystyrene balls were larger and distributed uniformly, the experiment was successful. This is because when the content of initiator increases, the concentration of free radical is higher, the reaction speed of chain growth is faster, and the resulting polystyrene balls are larger. On the other hand, as the concentration of the initiator increases, the number of active species in the reaction system increases, the probability of chain growth increases, and the yield increases. The distribution of the diameter of polystyrene is uniform. Taking into account the size of the diameter of polystyrene and the yield of polystyrene, the content of BPO is set as 0.5g in the experimental teaching.

Table 1. Influence of amount of initiator on polymerization reaction.

BPO dosage (g)	Particle size and distribution	Experiment is successful or not	Productivity (%)
0.4	Smaller, nonuniform	Yes	96.2
0.5	Smaller, uniform	Yes	96.8
0.6	Larger, uniform	Yes	97.4
0.7	Larger, nonuniform	Yes	97.6

3.2. Effect of stirring speed on polymerization reaction

Experimental conditions: after alkali washing styrene (40ml), polyvinyl alcohol (200ml) and BPO (0.5g), the reaction temperature of the small stirrer (blade diameter: 43mm) is described in the heating step of the experimental procedure. The effect of stirring speed on the polymerization reaction is examined, and the results are shown in table 2.

As can be seen from table 2, with the increase of mixing rate, the yield gradually increased, and all the experiments in the four groups were successful. However, when the mixing speed reached 400r/min, the diameter of the polystyrene ball decreased. This is because when the mixing speed is increased, the shear force is enhanced. When the droplet formed in the early stage of mixing is too large, small droplets are separated from the initial droplet, making the dispersed droplet smaller. The size and yield of the polystyrene ball diameter are taken into account comprehensively, and the mixing speed is set as 400r/min in experimental teaching.

Table 2. Influence of mixing speed on polymerization reaction.

Stirring rate(r/min)	Particle size and distribution	Experiment is successful or not	Productivity (%)
300	Larger, nonuniform	Yes	97.2
350	Larger, uniform	Yes	97.3
400	Smaller, uniform	Yes	98.5
450	Smaller, nonuniform	Yes	98.7

3.3. The influence of the size of stirrer on the polymerization reaction

Experimental conditions: 500ml four-necked flask, 40ml of styrene after alkaline washing, 200ml of polyvinyl alcohol solution, and the dosage of BPO were 0.5g, stirring speed was 400r/min. The reaction temperature was described in the heating step of the experimental steps.

It can be seen from table 3 that when the blade diameter of the agitator was 55mm and 62mm, the experimental results were all unsuccessful. That is to say, instead of forming spherical polystyrene, an agglomerate formed around the impeller, resulting in the scrapping of four-necked flask and the experiment failed. Owing to 500ml of four-necked flask of solvent volume, the use of lamina of 43mm diameter of impeller, under the regulation of speed, formed a relatively stable state of turbulence, the probability of collision between dispersed droplets is reduced, but when using a leaf of the diameter of the impeller, under the regulation of rotation speed, the form is relatively severe turbulence state, dispersed droplets collision in such a state of easy adhesive into pieces each other cause the failure of the experiment.

Table 3. Influence of the size of impeller on the polymerization reaction.

Blade diameter of stirrer(mm)	Experiment is successful or not
43	Yes
55	No
62	No

3.4. Effect of dispersant content on polymerization

Experimental conditions: after alkali washing, styrene 40ml, BPO 0.5g, small impeller (unfolding blade diameter: 43mm), stirring speed 400 r/min, reaction temperature as described in the step of heating up, the effect of dispersant content on the polymerization reaction was investigated, as shown in table 4.

From table 4, it can be seen that with the increase of dispersant content, the yield of polymerization increases at first and then decreases, but the diameter of polystyrene spheres decreases. This is because when the dispersant content is low, the whole dispersing system is unstable, and droplets collide with each other easily to form larger droplets, so the droplets appear at this time. When the content of dispersant decreases, the concentration of dispersant around the polymer droplets is higher. At this time, the existence of a certain concentration of dispersant will hinder the merging of small droplets to form large droplets. In this case, the probability of forming small droplets is higher, so the final polystyrene sphere has smaller diameter. However, the concentration of dispersant should not be too large, otherwise the growth of polymers will be affected.

Table 4. Effect of dispersant content on polymerization.

Dispersant content (ml)	Particle size and distribution	Experiment is successful or not	Productivity (%)
180	Larger, nonuniform	Yes	97.6
200	Smaller, uniform	Yes	98.2
220	Smaller, uniform	Yes	98.6
240	Smaller, uniform	Yes	97.5

3.5. Effect of reaction temperature on polymerization

Experimental conditions: after alkali washing, styrene 40 ml, BPO 0.5 g, small impeller (unfolding blade diameter: 43 mm), stirring speed 400 r/min, the effect of dispersant content on the polymerization reaction was investigated, polyvinyl alcohol solution 200 ml, the results are shown in table 5.

As can be seen from Table 5, when the reaction temperature is lower, the reaction speed is slower. At this time, there is no polymer in the system. With the increase of the system temperature, the reaction speed accelerates, and the spherical polymer begins to form. In this experiment, the spherical polystyrene microspheres with uniform spherical diameter are obtained by stepped heating method.

Table 5. Effect of reaction temperature on polymerization.

Reaction temperature (°C)	Particle size and distribution	Experiment is successful or not	Productivity (%)
68-74	No particle formation	No	-
74-80	Larger, nonuniform	Yes	97.2
80-86	Larger, nonuniform	Yes	97.9
86-95	Smaller, uniform	Yes	98.8

3. Conclusions

The effects of initiator content, stirring speed, agitator size, dispersant content and reaction temperature on suspension polymerization of styrene were studied. It was found that for the specific volume of four-necked flask, using a certain size of the impeller play a key role in the success or failure of the experiment. The content of initiator, stirring speed and dispersant had little effect on the success or failure of the experiment, but the temperature ranges and the way of heating had great influence on the suspension polymerization of styrene. Therefore, in the styrene suspension polymerization of macromolecular chemistry experiments, it is recommended to use lamina of 43mm diameter of impeller when using 500 ml of four-necked flask, the temperature range is selected at 86-95 °C, and the heating method is stepped by heating.

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