



Synthesis of large-scale, monodisperse latex particles via one-step emulsion polymerization through *in situ* charge neutralization



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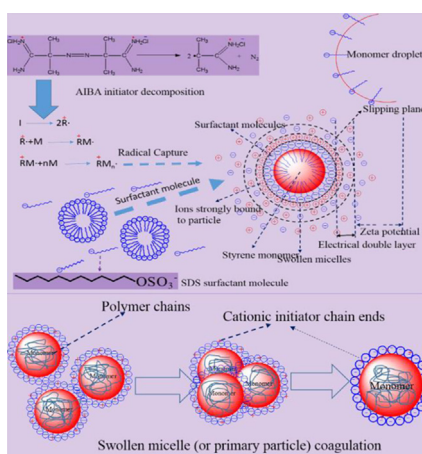
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HIGHLIGHTS

- A facile approach to prepare monodisperse polymeric nanoparticles was proposed.
- The *in situ* neutralization between surfactant SDS and initiator was adjusted to control the volume of micelle or primary particles.
- The effect of polymerization parameters on particle size distribution was investigated.

GRAPHICAL ABSTRACT



The physical model of the swollen micelle coagulation in the emulsion polymerization of styrene using AIBA and SDS as cationic initiator and anionic surfactant, respectively.

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ABSTRACT

Monodisperse sub-200 nm polystyrene nanoparticles were synthesized by a facile one-step emulsion polymerization technology using sodium dodecyl sulfonate (SDS) and 2, 2'-azobis(2-methylpropionamide) dihydrochloride (AIBA) as the surfactant and initiator, respectively. The *in situ* neutralization between negatively charged surfactant SDS and positively charged AIBA was used to control the primary particle (or swollen micelle) volume and the extent of the particle coagulation. As the oligomeric radicals with cationic AIBA chain ends were captured by anionic swollen micelles, the electrostatic stability of swollen micelles gradually reduced, further promoting the primary particle coagulation. As a result, the initial particle number decreased with the addition of AIBA. The earlier primary particle coagulation not only enlarged the particle size, but also narrowed the particle size distribution of final the latex particles.

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1. Introduction

Emulsion polymerization technology is widely applied in many fields including synthetic rubber, thermoplastics, coating, adhesive,

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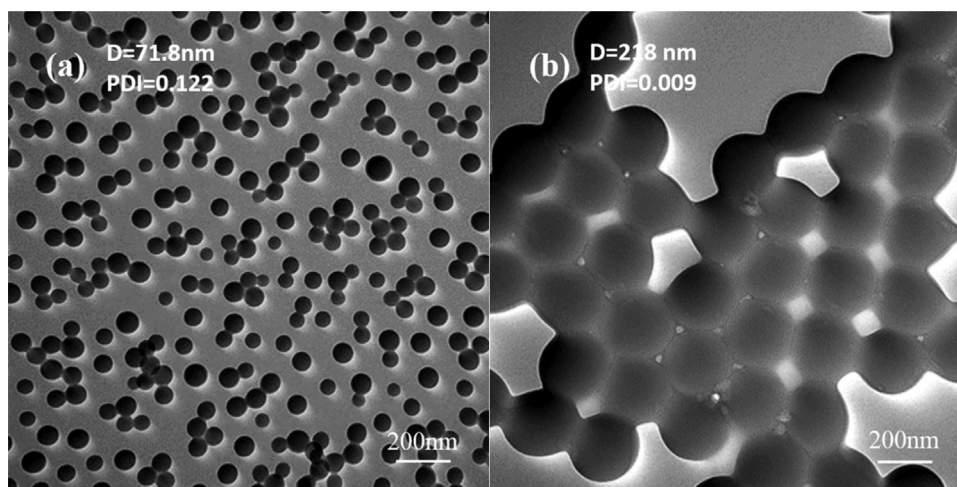


Fig. 1. The TEM images of latex particles prepared by different type initiators (a: KPS; b: AIBA).

and plastic modifier [1–4]. This approach is a very interesting as it allows the preparation of monodisperse or functional latex particles [5,6]. Until today, the challenges preparing highly monodisperse latex particles by the emulsion polymerization technology are mainly attributed to the several factors as follows: (1) the particle coagulation in the particle growth period; (2) the particle second nucleation during the polymerization process; and (3) the longer nucleation interval in the particle formation period [7,8].

With the progress of the polymerization reaction, the tremendous particle surface area gradually increased the interfacial energy of the latex particles, further promoting the particle coagulation occurred. As a result, the larger particles achieved from the aggregation of the small particles not only increase the average particle size, but also broaden the particle size distribution of the final latex particles [9,10]. In contrast to the particle coagulation during the particle growth interval, the second nucleation and longer nucleation interval decrease the average particle size [11].

To overcome these challenges, many interesting methods have been proposed to prepare monodisperse latex particles in the past several years. For instance, Konno and co workers used 2, 2'-azobis [N-(2-carboxy-ethyl)–2-methylpropionamide] hydrate as the amphoteric initiator to control the electric surface potential of particles by adjusting the pH value of the aqueous phase, further preventing the generation of new particles during the particle growth period [12,13]. Sajjadi controlled the monomer-starved condition during the particle nucleation process to decrease the polydispersity index of the latex particles [14,15]. Zang et al. used the two-stage swelling method of the seeded polymerization to prepare monodisperse latex particles by varying the swelling time of monomer and the temperature increase rate [16]. Even though these methods extremely facilitated the development of monodisperse latex particle preparation, they were also significantly difficult to be used as in industrial scale to produce massive monodisperse latex particles, because of the limit of the production charge and efficiency. Therefore, a simple method to prepare monodispersed latex particles still is required by academic and industrial community.

In recent years, cationic initiator such as 2, 2'-azobis (2-methylpropionamide) dihydrochloride (AIBA) has been gradually used in soap-free emulsion/dispersion polymerizations to adjust the charge density on the particle surface, and to further control the stability of the latex particles [17–20]. In the previous study, we prepared monodispersed latex particles by using AIBA as the initiator in 30 wt% methanol solution [21]. However, the mechanism of large-scale, monodisperse latex particles using AIBA as the

initiator was not discussed. Moreover, the effect of cationic initiator on the polymerization reaction kinetics was also ignored. To address these questions and develop a simple method to prepare monodisperse latex particles, the evolutions of the particle size, number, and zeta-potential as a function of polymerization time were investigated in this study to discuss the role of the cationic initiator in determining the polydispersity of the latex particles.

2. Experimental

2.1. Materials

The monomers styrene (St) (Jilin, China) was distilled under -0.1MPa to remove the inhibitor prior to use. All other reagents including Potassium persulphate (KPS; 99.5%), AIBA (98.5%), Sodium dodecyl sulfonate (SDS, 98%) potassium carbonate (K_2CO_3) were supplied by the Aladdin Industrial Corporation (Shanghai; China), and were used without further purification. Methanol was purchased from the Beijing Chemical Factory. Distilled deionized water (DDI) was used throughout the study.

2.2. Polymerization reaction

The polymerization reactions were carried out in a 500 mL four necked glass boiling flask equipped with a thermostat water batch cauldron, an anchor stirrer, a reflux condenser, a nitrogen (N_2) valve and a sample device. The polymerization reaction process was described in previous studies as following [22,23]: The surfactant SDS, electrolyte K_2CO_3 , Methanol and DDI were added to reaction equipment according to this sequence; monomer St was also added when the all auxiliaries dissolved in the aqueous phase. Nitrogen (N_2) purging was carried out for 30 min before the initiator was added to the reactor. When the initiator dissolved in some DDI was added into the equipment, the polymerization reaction begins. The polymerization reaction was carried out under the N_2 atmosphere, the reaction temperature and stirring rate were set as 65°C and 250 rpm, respectively. During the polymerization process, 1–2 g latex was withdrawn from the reactor using a syringe at appropriate interval to analysis the monomer conversion and particle size distribution. The detail polymerization recipes were shown as Table 1.

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