

# Fabrication and Characterization of Monodisperse Polystyrene Latex (PSL) with Various Diameters

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**Abstract.** The focus of this study was the synthesis of polystyrene latex (PSL) particles and their diameter control for the application of aerosol filtration test. Simple emulsion polymerization was used as the method to produce uniformly spherical PSL particles. The diameter of PSL particles ranging from (160 to 941) nm was obtained by controlling the reaction parameters during the polymerization process. The reaction parameters were the concentration of styrene monomer, the concentration of potassium persulphate initiator, and the solution temperature. The diameter of PSL particles was larger as the concentration of styrene monomer, and initiator concentration increased, as analyzed from their SEM images. The higher the solution temperature, the resulting particles diameter would be smaller.

**Keywords.** Aerosol filtration test, polymerization process, polystyrene latex (PSL), and SEM.

## 1. Introduction

In recent years, research about aerosol filter has become the center of attention of many researchers, especially for those working on the air filter. One efficient method for the aerosol filtration is to use a nanofiber membrane as a filter produced by electrospinning method [1, 2], and a constant current electrospinning system was employed to obtain highly uniform nanofibers [3, 4]. The efficiency of the nanofiber filter can be determined by the performance to filter particles [5, 6]. Hence, it is necessary to have appropriate test particles for the filtration investigation. On the application of air and water filtration, the commonly used test particles are polystyrene latex (PSL). PSL particles that are spherical and uniform [7–11] make them possible for applications in many areas, such as the fabrication of hollow template [8, 9] and standard particles for calibrating aerosol instrument [12, 13].

PSL particles with nanometer size can be used for air filtration while those with nanometer-micrometer size can be applied in water filtration. The need of PSL particles with various sizes was limited by the expensive production cost. Therefore, there must be an innovative way to produce PSL particles more efficiently at less expensive cost. In previous studies, many synthesis methods have



been developed [14–17]. One commonly used method is a simple emulsion polymerization method without any additive substance. It is a synthesis method in the liquid phase using styrene monomers being diluted in distilled water [9]. The advantage of this method is that the size of particles can be controlled by varying the parameters such as the process temperature, the concentration of styrene monomer, and the initiator concentration [8–11]. However, the polymerization process takes 24 h, which is relatively time-consuming.

This paper reports the synthesis of PSL particles that was conducted using additive-free emulsion polymerization method for 10 h. Potassium persulphate was used as the initiator of the polymerization process in order to reach the larger size of particles instead of using 2,2-azobis dihydrochloride (AIBA) [8]. The size of PSL particles was expected to be from nanometer to micrometer, which could be achieved by adjusting the concentration of styrene monomer. Based on the data, an equation could be obtained to predict the size of particles. The predictive equation for the AIBA initiator has been obtained [9]. However, for the potassium persulphate initiator, there is no report. Therefore, we will discuss in more detail about the equation.

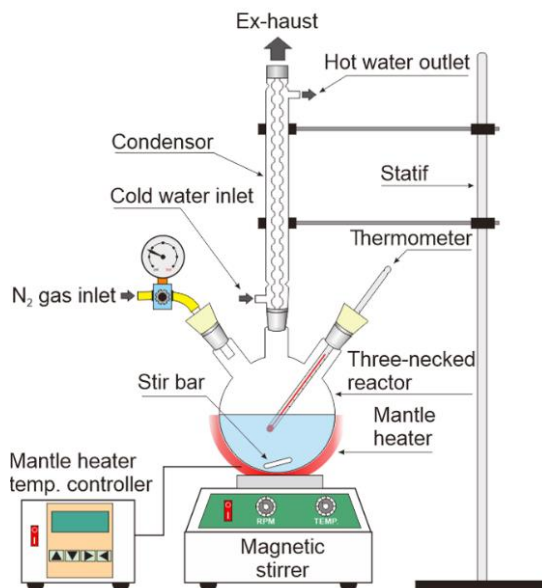
## 2. Materials and methods

### 2.1. Materials

The material used in this study was styrene monomer while the chemicals were sodium hydroxide (NaOH) and potassium persulphate. Styrene monomer and sodium hydroxide were purchased from PT. Brataco (Bandung, Indonesia) while potassium persulphate was obtained from Merck. The solvent was distilled water, which was obtained from Chemistry Laboratory of Institut Teknologi Bandung (ITB).

### 2.2. Synthesis of PSL particles

PSL particles were synthesized using the liquid phase method. This method is also called as the conventional emulsion polymerization in which the styrene monomer was dissolved in distilled water, and potassium persulphate was used as the initiator [8, 9]. The polymerization process was conducted in a batch reactor system and under the condition of nitrogen atmosphere. The batch reactor system is shown in Figure 1 consisting of a four-necked batch-glass reactor with a capacity of 300 mL, a magnetic stirrer, a mantle heater and a temperature controller, a thermometer, and nitrogen gas. The first step was to purify the styrene monomer from the inhibitor by washing it using a solution of NaOH (3 M). The NaOH solution was obtained by dissolving NaOH powder into the distilled water until homogeneous. It was then inserted into the Erlenmeyer flask and bubbled with nitrogen gas for 15 min (flow rate of 1 lpm). The styrene monomer was then mixed with the base solution in the Erlenmeyer flask and then shaken for 30 min. There were two different phases seen after shaking, the clear solution at the bottom and yellow solution on the top. The yellow solution was the pure solution of styrene monomer that will be used in the subsequent polymerization process. The distilled water was inserted into the reactor system while stirred and heated to a specified temperature under the atmospheric condition of nitrogen gas for approximately 15 min. After reaching the intended temperature, the previously purified solution of styrene monomer was then added to the reactor system and then waited for 10 min for styrene monomer to be uniformly dispersed in the solution. To start the polymerization process, potassium persulphate was added to the solution of styrene in the distilled water. The solution was maintained at a constant temperature for 10 h under nitrogen gas atmosphere. Finally, the solution was then cooled until it reached the ambient temperature.



**Figure 1.** Scheme of the batch reactor system.

### 2.3. Characterization of PSL particles

The morphology and size of the obtained PSL particles were observed using a scanning electron microscope (SEM) (Hitachi, SU3500). The measurement and analysis of the average diameter and the size distribution of PSL particles were performed using SEM image analysis with the help of ImageMIF software version 3.0 developed in the Integrated Laboratory of Materials and Instrumentation, ITB. The diameter was measured at two hundred different particles in the SEM image. The size homogeneity of PSL particles could be determined by calculating the coefficient of variation (CV). Note that the coefficient of variation is the ratio of the standard deviation ( $\sigma$ ) of diameter to the average diameter ( $\mu$ ) of the PSL particles. If the CV is greater than 0.3, then the particles are classified as inhomogeneous; otherwise, it will be homogeneous particles [1].

## 3. Results and discussion

Table 1 shows three reaction parameters, which are the concentration of styrene monomer, the concentration of potassium persulphate initiator, and the solution temperature in the reactor-batch, that were used to obtain PSL particles with several diameters. The adjustment of the parameters is very influential in controlling the size of PSL particles produced by the conventional emulsion polymerization methods [10, 11, 17]. However, this study focused on the synthesis of PSL particles by varying the styrene monomer concentration while the initiator concentration and the solution temperature were kept constant. Figure 2 shows the SEM images and the distribution of PSL particles for several different styrene monomer concentrations at the solution temperature of 80 °C. It can be seen that the morphological structure of the PSL particles was spherical and uniform. The increase in the concentration of styrene monomer had no effect on the shape of PSL particles. Similar results were also reported by previous papers [8, 9] that the shape of PSL particles was spherical and uniform, even when other parameters such as the solution temperature and the initiator concentrations were varied [8–11]. Table 1 gives the average diameters of the PSL particles along with the CVs for different styrene monomer concentrations and different temperatures. The calculated CVs implied that the obtained PSL particles were homogeneous or monodisperse.

**Table 1.** The sizes of PSL particles and their synthesis parameters.

Sample	Concentration of Monomer (wt.%)	Concentration of Initiator (wt.%)	Temperature (°C)	Average Diameter of Particles (nm)	Coefficient of Variation (CV)
PSL 1	4	0.04	55	281	0.05
PSL 2	9	0.04	55	295	0.03
PSL 3	11	0.04	55	451	0.08
PSL 4	12	0.04	55	941	0.14
PSL 5	0.5	0.04	80	160	0.05
PSL 6	1.2	0.04	80	216	0.13
PSL 7	2.0	0.04	80	295	0.06

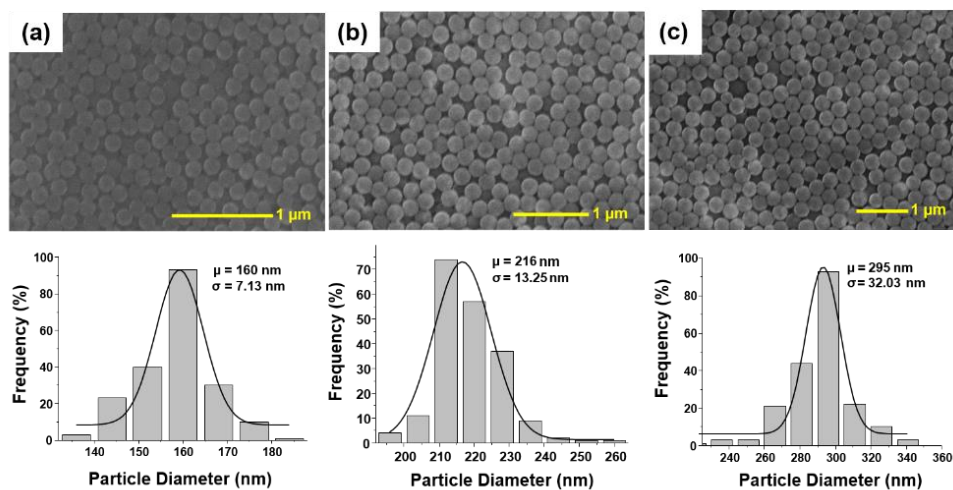
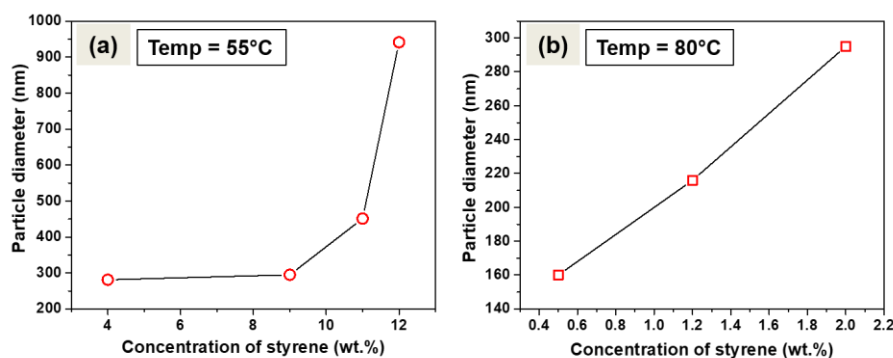
**Figure 2.** SEM images and distribution of PSL particles at temperature of 80 °C: (a) PSL 5, (b) PSL 6, (c) PSL 7.

Figure 3 depicts the relationship between the styrene monomer concentration and the diameter of PSL particles at different temperatures. The large PSL particles within (281 to 941) nm were obtained under the low polymerization temperature (temperature of 55 °C). Otherwise, the size of PSL particles was within (160 to 295) nm when the polymerization temperature as high as 80 °C, which are similar to the previous reports [7, 8]. However, the time and the number of styrene monomers used could be reduced by synthesizing at different temperatures.

**Figure 3.** The particle size of PSL with varying concentration of styrene monomer at temperatures of (a) 55 °C and (b) 80 °C.

In Figure 3 (a), the high polymerization temperature (80 °C) resulted in an increased interaction and change in the reactant components, which were styrene monomer and initiator [16]. Therefore, the core or the nucleus was formed appropriately. This process requires a lot of monomers and oligomers in the early stage of polymerization since the number of free monomers and oligomers will decrease significantly. This results in the retardation in the formation of particles and production of small particles [10, 11]. For the process using the low polymerization temperature (55 °C) as depicted in Figure 3 (b), the decomposition of the initiator took a longer time that caused the formation of the oligomer to be slower and then resulted in a low number of the nuclei [18]. At this condition, the core would capture a larger amount of oligomers/monomers hence larger particles were produced [9]. It can be seen from the graph that when the temperature used was 55 °C, the size of PSL particles increased but the data obtained were an anomaly. The results agreed to a certain degree with the previous paper [9]. It was found that the use of polymerization temperature below 60 °C caused the polymerization to be imperfect and the produced trend data were less satisfying. The low polymerization temperature also led to lower interactions between components reaction (initiator, styrene monomer, distilled water) so that the process of particles formation became worst.

The size of PSL particles could be predicted by considering Equation 1 [10].

$$D_p = 71.80(S) + 15.85\ln(I) - 170.30\ln(T) + 847.87 \quad (1)$$

where  $D_p$  is the particle diameter (nm),  $S$  is the concentration of styrene monomer (wt%),  $I$  is the initiator concentration (wt%), and  $T$  is the temperature of solution (°C). However, the diameters of the synthesized PSL particles were different from the predictive equation in Equation 1. The PSL particles synthesized at the polymerization temperature of 80 °C had an average diameter larger than that predicted by Equation 1. This is because the present experiment used potassium persulphate as the initiator while Equation 1 was obtained using the 2,2-azobis dihydrochloride (AIBA) initiator. This diameter difference has also been reported previously [8] where the average diameter of the PSL particles synthesized using the potassium persulphate initiator was greater than that done using the AIBA one.

#### 4. Conclusions

Monodisperse particles of polystyrene latex (PSL) have been synthesized using a simple emulsion polymerization method (without the additive substance) with distilled water as the solvent and potassium persulphate as the initiator. Using this method, the average diameter of PSL particles could be controlled by varying the solution temperature and the styrene monomer concentration. It has been found that the diameter of the synthesized PSL particles was in the range of (160 to 941) nm. The higher the temperature of the solution, the smaller the diameter of the PSL particles was. The greater the styrene monomer concentration, the greater the diameter of the PSL particles was. Spherical and homogeneous PSL particles can, therefore, be used as pollutants for the applications of air and water filtration systems.

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