



CONTROLLING POLYMER PARTICLE SIZE USING NANOTRAC FLEX

CONTEXT

Controlling polymer size is essential for optimizing their performance in a wide range of applications. Size directly influences key physical properties such as stability, dispersibility, mechanical strength, and viscosity - factors that significantly affect product quality in manufacturing processes like injection molding and polymer processing. In biomedical applications, particularly in polymeric drug delivery systems, polymer size is critical because it determines how effectively nanoparticles can circulate in the bloodstream and penetrate target cells. Moreover, polymer size, defined by chain length, directly determines molecular weight. Each monomer adds a fixed mass, so longer chains have higher molecular weights, while shorter chains have lower ones. This makes chain length a key factor in a polymer's physical and mechanical properties. Overall, precise control over polymer size enhances functionality, consistency, and reliability across diverse industries.

Challenges in Controlling Particle Size During Polymerization

By adjusting the feeding ratio and modifying reaction conditions like temperature and stirring rate, it is possible to control nanoparticle size in some established reactions. However, identifying the precise reaction conditions to synthesize a polymer of the desired size can be both time-consuming and, at times, impossible.



Fig. 1: Inserting Probe in Polymerization Reaction

So, to control the particle size in a polymer reaction, scientists usually monitor the growth of particle size during the ongoing reaction and quench it to stop at the desired size.

To quench the polymer reaction, scientists typically collect aliquots of the sample and measure the size outside of the reaction vessel after multiple dilutions. This often leads to inaccurate size measurements due to delays in measurement time and unstable temperatures. As a result, once the reaction is quenched after the measurement, the particle no longer remains in the same state as it was at the time of the measurement.

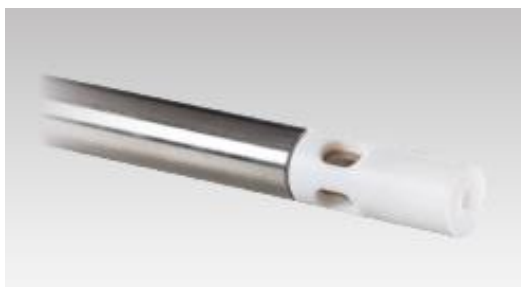


Fig. 2: NANOTRAC FLEX probe with Flow guard

Moreover, since many polymerization reactions are highly sensitive to the presence of oxygen in the air, it often becomes impossible to take aliquots of the sample for measurement without introducing oxygen into the system.

In this application note, we demonstrated the capability of **MICROTRAC's NANOTRAC FLEX** to accurately monitor and measure particle size in real time without disrupting the reaction system, even in the presence of a magnetic stirrer, by utilizing the flow guard on the probe. Furthermore, we showed that polymers of the desired size can be obtained by quenching the reaction once the target particle size is reached.

Unique Features of NANOTRAC FLEX DLS

The Nanotrac Flex features a unique probe design that allows direct immersion of the probe into the reaction vessel for particle size measurement. When paired with the flow guard, the probe can even measure particle size under stirring conditions accurately up to 250 rpm, providing real-time monitoring during the reaction. Additionally, the probe is suitable for use at temperatures up to 90°C, making it an ideal fit for measuring particle size in an ongoing

polymerization reaction, which typically occurs at around 65°C.

EXPERIMENT

In this study, we selected the synthesis of polystyrene latex via precipitation polymerization. Styrene and sodium dodecyl sulfate (SDS) were dissolved in deionized (DI) water, heated to 65°C, and maintained under nitrogen flow, with the quantities listed in **Table 1**. After 1 hour, the initiator ammonium persulfate (APS) was added. The overall reaction setup is shown in **Figure 1**. The stirring rate was maintained at 150 rpm, as increasing beyond 250 rpm appeared to cause the particles to be smaller, even with the use of a flow guard, due to the impact of external motion.

DI Water	Styrene	SDS	APS (0.1M)	Temp	Stirring Rate
90 ml	2ml	0.038g	8 ml	70 °C	150 rpm

Table 1: Quantities of Chemicals Used

Once the particle size reached the desired value, we quenched the reaction by placing the two-neck round-bottom flask in ice-cold water. One reaction was stopped at the desired particle size, while another was allowed to proceed for 5 hours until the polymerization was complete.

RESULTS

We monitored the particle size from the start, following the addition of the initiator. Initially, the particle size did not exhibit a monomodal distribution due to the presence of an excess amount of monomers and surfactants. However, after some time, we were able to track the particle size increase over time. After 35 minutes, the particle size was predominantly in the range of 63-66 nm.

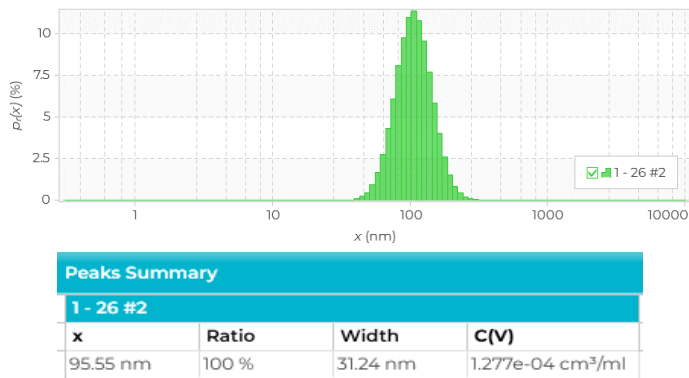


Fig. 3: Particle Size Distribution at 100 nm After 50 min.

Once the particle size reached 100 nm, as measured by the Nanotracs Flex (Figure 3), we immediately stopped the reaction by placing the reaction flask in ice-cold water. The sample was then dialyzed overnight using a membrane filter with a 12,000 Da cutoff, with repeated water changes.

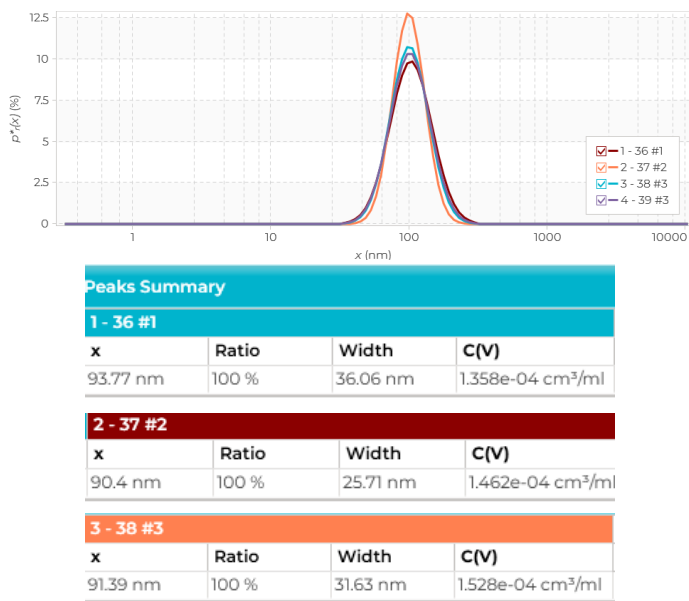


Fig. 4: Post-dialysis particle size: ~101.1 nm (triplicate)

After dialysis, the particle size was measured again, with the average particle size of 3 repeated

measurements being approximately 101.1 nm, as shown in Figure 4.

The TEM images of the sample are shown in the Figure 5.

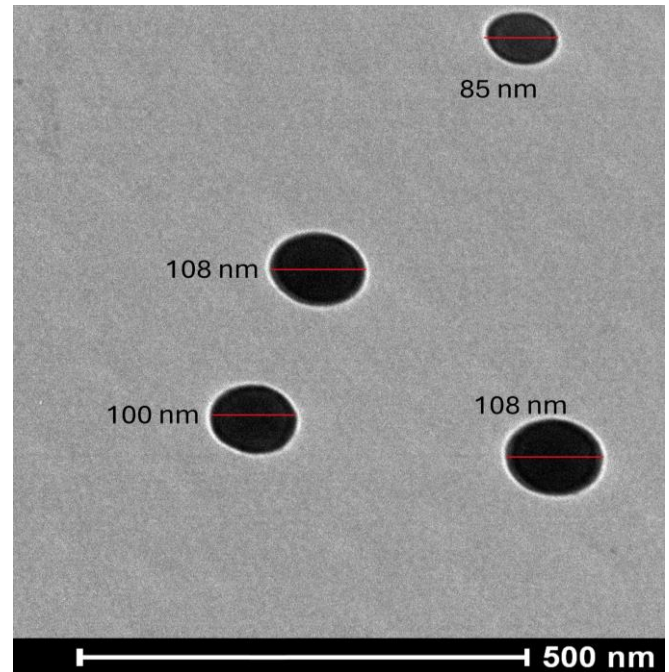


Fig. 5: TEM image of 100 nm Polystyrene latex

The other reaction was allowed to run for 5 hours until completion. The measured particle size reached 255 nm, indicating that without monitoring growth using the Nanotracs Flex with flow guard, the reaction cannot be stopped at the desired size.

CONCLUSION

Polystyrene latex particles of 100 nm were successfully synthesized by monitoring growth with Microtracs probe-based DLS equipped with a flow guard and quenching the reaction at the desired size. Without quenching at the desired particle size, the reaction proceeded further, producing particles with an average size of 255 nm.