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Preparation of monodisperse polystyrene spheres by physical method

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Abstract. The preparation of monodisperse polystyrene spheres was carried out by physical method, i.e. milling and gamma irradiation processes, which used the solid commercial polystyrene. The solid polystyrene was dissolved by ethyl acetate at a concentration of 10%. The monodisperse particle arrangement was obtained by milling process which in this research the time duration varied by 8 and 60 minutes. Whereas the spherical particle structure was achieved by gamma irradiation at a dose varied by 5 KGy or 15 KGy. The yielded solution was characterized using Particle Size Analyser (PSA) and Scanning Electron Microscopy (SEM). The results showed that the optimum monodisperse polystyrene spheres solutions were produced by dissolving the solid polystyrene using ethyl acetate and milling the solution for 8 minutes and radiating the solution using gamma radiation at a dose of 15 KGy. The resulting solution has a particle diameter range of 23-57.8 nm and a span of 0.49-0.59.

Keywords: monodisperse, polystyrene, sonication, milling, gamma radiation

1. Introduction

Monodisperse polymer particles can be a good candidate for fascinating applications such as combinatorial synthesis, biondiagnostics, biotechnology, electronic devices and information technology [1-4]. Monodisperse nanopolystyrene solutions are needed to calibrate Particle Size Analyser (PSA) that is closely related to nanomaterials. As nanoparticles research grows rapidly due to its unique properties that are more beneficial [5-7], the presence of this solution becomes meaningful. Likewise, the monodisperse particle arrangement, the polystyrene has to meet the structure requirements of spherical particles.

In this work, synthesis of polystyrene solution which was intended to be used as a PSA calibrator performed by changing the properties of the existing industrial polystyrene solution particles. Generally, polystyrene synthesis is carried out by chemical methods, namely polymerization process which consists of polymerization of suspensions, precipitation and emulsions [8, 9]. One type of synthetic polymers that is widely developed is an emulsion polymer that produces polystyrene with a size of 10–100 nm.



However, it is difficult to obtain a high purity polymer by the chemical synthesis process. In addition to the chemical methods, the synthesis process can also be carried out by the physical methods which overcome this problem due to the mechanically synthesis process.

It is important to consider the change in particle size, particle structure, and particle arrangement. Commercial polystyrene materials were created to be a monodisperse nano-sized particle with the spherical particle structure. Polystyrene solution be prepared by dissolving solid polystyrene using a solution of ethyl acetate at concentration of 10%. The nano-sized scale, monodisperse arrangement and spherical structure of particle were attempted by milling process and gamma irradiation process [10], respectively. The spherical structure of polystyrene particles can be obtained by performing gamma irradiation which has been widely used to synthesis several nano-materials through electron collisions and the activation of ions. The gamma irradiation process that was carried out at a dose of 0.2-1.5 Mrad can lead to degradation of polystyrene solutions [8]. Further, the resulting polystyrene samples were characterized using Particle Size Analyser (PSA) to determine the size and arrangement of particles and Scanning Electron Microscopy (SEM) to determine the particle structure. Finally, the characterization results of the obtained sample were compared with standard polystyrene solutions.

2. Method

The polystyrene solution was prepared by dissolving solid polystyrene (99%, sigma aldrich) in ethyl acetate to attain a concentration of 10%. This process was performed by magnetic stirrer (SLMSH 300) for 60 minutes with an angular speed of 300 rpm. The solution was then milled by Cosmos CB 180 miller with a frequency of 50 Hz. The milling process began by inserting distilled water into the miller, and then the polystyrene solution was dripped into it during the machine was working to achieve a concentration of 20% for 8 or 60 minutes. The operating format of the miller was 1-minute rest time for each 2 minutes working time. The next process was the gamma irradiation.

The radiation dose given in this study was 0.5 Mrad (5 KGy) and 1.5 Mrad (15 KGy) by using the gamma irradiation source of Co 60. Gamma ray that irradiates the polystyrene particles will cause a Compton effect. The resultant energy due to collision will change to thermal energy and will break down polystyrene particles to attain a spherical structure. Further, PSA (Delsa Nano Beckman Counter) and SEM characterization was then carried out and compared with the standard polystyrene solutions. For the purposes of SEM characterization, it is necessary to crystallize the polystyrene solution first. This process was carried out by the method of deep coating and heating procedure. In this case the glass substrate was dipped in the polystyrene solution and then heated to a temperature of 100°C for 60 minutes.

3. Results and Discussion

The polystyrene solution has met the homogeneous standard so that it can be continued to the stage of preparing the nano polystyrene monodisperse by milling process. The polystyrene solution of 8 minutes milling time has a more turbid colour than the solution with 60 minutes milling time. This fact may be due to more homogeneous and smaller particle sizes of the polystyrene solution with 60 minutes milling time. The milling process was followed by the aging procedure in order to realize its stability. Further, the PSA characterization was performed to confirm the particle structure. The monodisperse state of the sample was adjusted by the width of the sample distribution which is attain by using span analysis following equation (1) [11] and by considering the particle diameter range.

$$Span = \frac{D_{90}-D_{10}}{D_{50}} \quad (1)$$

Span is the range of particle size, D_{90} and D_{10} are the particle diameter of 90% and 10% of particles, respectively; whilst D_{50} is the diameter of the sample between D_{90} and D_{10} . Further, D_{90} certainly represent the particle diameter range in the sample [12].

The PSA characterization results of polystyrene solution of 8 minutes milling time are presented in following Fig. 1 and table 1.

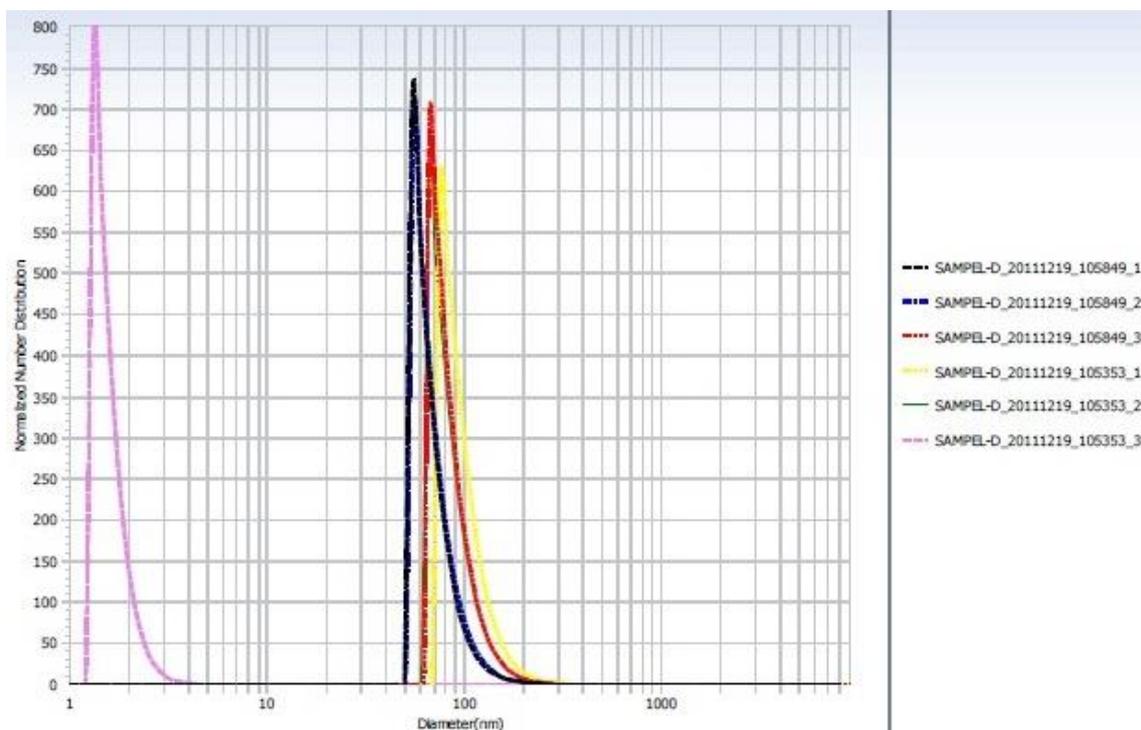


Figure 1. Graph of PSA characterization results of polystyrene solution of 8 minutes milling time.

Table 1. The results of the PSA characterization of polystyrene solution of 8 minutes milling time.

No	Sample	D_{10} (nm)	D_{50} (nm)	D_{90} (nm)	Span
1	Sample-D_20111219_105353_1	1.2	1.4	1.9	0.50
2	Sample-D_20111219_105849_1	51.2	59.6	86.1	0.59
3	Sample-D_20111219_105849_1	52.1	61.0	88.3	0.59
4	Sample-D_20111219_105353_1	62.4	74.0	108.2	0.62
5	Sample-D_20111219_105849_1	64.5	75.9	109.6	0.59
6	Sample-D_20111219_105353_1	70.7	84.4	123.6	0.63

The results of the PSA characterization of polystyrene solution of 8 minutes milling time denoted that the polystyrene solution has a particle diameter range between 1.9-123.6 nm and span between 0.50-0.63. Despite the diameter range is rather wide but the polystyrene solution of 8 minutes milling time has a narrow span.

Henceforth, the results of the PSA characterization was described by its value solely. The PSA characterization results of polystyrene solution of 60 minutes milling time exhibited that the polystyrene solution has a diameter range between 2.9-69.6 nm and span between 0.45-0.60. Increasing the milling time duration renders the reduction of particle size. Based on the span achieved from the milling process it can be concluded that the monodisperse structure has been successfully accomplished, therefore proceed with the gamma irradiation process to realize the spherical structure. After it, the sample was characterized by PSA equipment for confirmation the particle arrangement and the resume of the PSA characterization results depicted on table 2, which was coupled with the PSA characterization results of the standard polystyrene solution. The standard polystyrene solution is a solution that has been used as a test standard for PSA calibration. The gamma irradiation process turned out to affect the particle

arrangement of the polystyrene solution. The sample of 8 minutes milling time with 5 KGy dose gamma irradiation and the sample of 60 minutes milling time with 15 KGy dose gamma irradiation turned into polydisperse sample. On the other hand, the higher level of the irradiation dose led the particle size become smaller.

Table 2. The results of the PSA characterization of polystyrene solution after gamma irradiation process.

	Sample	Diameter (nm)	Span
8 minutes milling time	5 KGy gamma irradiation	40.6-2224.8	0.25–0.58
	15 KGy gamma irradiation	23.3-57.8	0.49–0.59
60 minutes milling time	5 KGy gamma irradiation	199.0-418.9	0.53–0.58
	15 KGy gamma irradiation	2.4-59.7	0.37–0.60
Standard polystyrene solution		85.8-109.8	0.46–0.60

Based on the table 2, it can be concluded that the solution sample of 8 minutes milling time with 15 KGy dose gamma irradiation is the best sample which fulfilled the standard of the monodisperse particle arrangement. Therefore, it was necessary to confirm the spherical structure of this sample by means of SEM images (Fig. 2). Fig. 2 confirmed that the spherical structure of the prepared polystyrene solution has been achieved. These facts indicated that this solution can be used as a calibrator for the PSA equipment. The particle size that seemed large and different from each other which is seen in SEM image (Fig. 2. (a)), probably due to the crystallization process in the sample. Further, the Fig. 2. (b), displayed the spherical monodisperse polystyrene particles of 300 nm in diameter.

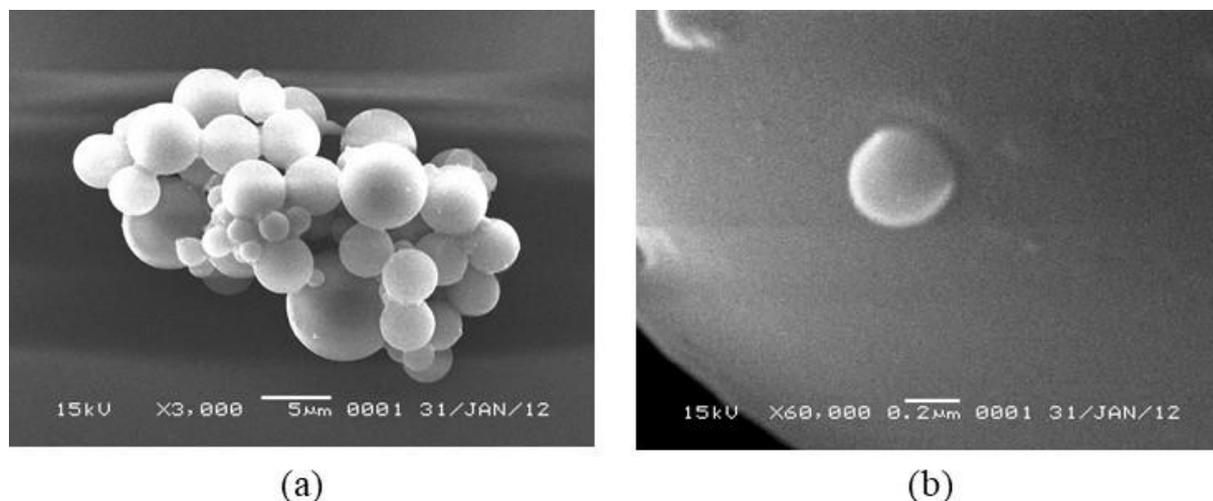


Figure 2. SEM images of the polystyrene solution sample of 8 minutes milling time with 15 KGy dose gamma irradiation: (a) 3000 x magnification, (b) 60000 x magnification.

4. Conclusions

The polystyrene solution with monodisperse particle composition and spherical structure was successfully synthesized through a simple physical method, that is by milling process followed by gamma irradiation. The most optimum polystyrene monodisperse and spherical solution which was successfully synthesized was a solution with a particle diameter range of 23.3-57.8 nm and a span of 0.49-0.59 which was achieved by performing the milling method for 8 minutes and continued with gamma irradiation at a dose of 15 KGy.

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References

- [1] Chen C, Zhu Z-G, Shih W-H, Ge Q-Q, Liu M-J and Zhu X-R 2015 Facile preparation and self-assembly of monodisperse polystyrene nanospheres for photonic crystals *J. Nanosci. Nanotechnol.* **15** 4 3239-43
- [2] Lee J, Ha J U, Choe S, Lee C-S and Shim S E 2006 Synthesis of highly monodisperse polystyrene microspheres via dispersion polymerization using an amphoteric initiator *J. Colloid Interface Sci.* **298** 2 663-71
- [3] Zang Z, Wen M, Chen W, Zeng Y, Zu Z, Zeng X and Tang X 2015 Strong yellow emission of ZnO hollow nanospheres fabricated using polystyrene spheres as templates *Mater. Des.* **84** 418-21
- [4] Zhang J, Chen Z, Wang Z, Zhang W and Ming N 2003 Preparation of monodisperse polystyrene spheres in aqueous alcohol system *Mater. Lett.* **57** 28 4466-70
- [5] Cao G 2004 *Nanostructures & nanomaterials: synthesis, properties & applications*: Imperial college press)
- [6] Widanarto W, Jandra M, Ghoshal S, Effendi M and Cahyanto W 2015 BaCO₃ mediated modifications in structural and magnetic properties of natural nanoferrites *J. Phys. Chem. Solids* **79** 78-81
- [7] Effendi M, Yokoi H and Kuroda N 2010 Optical Study on Electronic Transport Properties of Single-Walled Carbon Nanotubes at High Temperature *J. Nanosci. Nanotechnol.* **10** 6 4074-7
- [8] WT Tan, MM Radhi, Rahman M A and Kassim A 2010 Synthesis and Characterization of Grafted Polystyrene with Acrylonitrile using Gamma-Irradiation *J. Appl. Sci.* **10** 2 139-44
- [9] Troughton M J 2008 *Handbook of plastics joining: a practical guide*: William Andrew)
- [10] Wang S, Wang X and Zhang Z 2007 Preparation of polystyrene particles with narrow particle size distribution by γ -ray initiated miniemulsion polymerization stabilized by polymeric surfactant *Eur. Polym. J.* **43** 1 178-84
- [11] Webb P A 2002 Interpretation of particle size reported by different analytical techniques. Micromeritics)
- [12] Hackley V A and Ferraris C F 2001 *The use of nomenclature in dispersion science and technology* vol 960: US Department of Commerce, Technology Administration, National Institute of Standards and Technology)