The Study of Nozzle Type Application on Polystyrene Microsphere Processing Using Anti – Solvent Technology

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Abstract

Micronization has become the interest of scientists to develop advanced performance materials. The efforts have been conducted in many applications, such as in adsorbents, catalyst supports, pharmaceuticals and food industries. This work aimed to investigate the effect of contact technique by varying nozzle dimensions (diameter and type) and operating condition in polystyrene production using supercritical anti – solvent technology. The samples were characterized in the term of morphology and particle distribution. The analysis results showed that the usage of co – axial nozzle could produce smaller discrete particles comparing to that of the axial nozzle usage. Approximately 5 – 10 µm particle size was produced using 4.6 mm ID nozzle, and less than 5 µm of particles were precipitated when 3.6 mm ID nozzle was used. The application of higher precipitation pressure at 40 bars suggested more effective micronization than that at atmospheric pressure though there have been some shrinkage occurrences.
Keywords: microsphere, polystyrene, nozzle type, anti solvent technology

Introduction

Polymers have been used as alternative materials for different purposes, due to their wide properties availability provided by the vast molecular weight range. Polymers have found practical application in the form of microsphere for adsorbent, catalyst support and drug delivery systems.

Microspheres can be produced using several methods. The conventional ones such as spray drying, grinding and crushing have become the most common application to process microspheres. However these technologies possess several drawbacks which retard their application for wider scope such as high energy consumption and highly organic solvent residue after processing (Ginty et al 2007). The anti solvent technology (SAS) becomes the interest of scientists to overcome those disadvantages. The anti solvent technology provides a more effective way to produce microparticles through its single step, clean, and relatively low temperature – pressure processing features (Ginty et al 2005). The antisolvent technology also offers the possibility to produce materials with narrower particle size distribution with smaller average of diameters (York 1999).

In this work, polymer of styrene in toluene was processed using supercritical CO_2 at 75 - 80 bars and precipitated at 30^{o}C. Pressure application of precipitation chambers were at atmospheric and 40 bars.

Methodology

Material

Polymer model chosen was polystyrene (PS) purchased from Dow Chemical Co. Toluena was purchased from Brataco Chemica Indonesia. Gas of CO_2 99% purity was supplied by Tri Gases Inc. Indonesia.

Experiments

The microsphere polymers processing was conducted using Aerosol Solvent Extraction System (ASES) technique. Various concentration of polystyrene from 3% to 15% wt were sprayed through nozzles 0.5 mm ID 20 cm long. The nozzles used were axial and coaxial types (3.6 mm and 4.6 mm ID annulus). Polymer solution was pushed with CO_2 flow and heated up to 45^{o}C. The antisolvent gas of 100^{o}C was contacted with solution through the coaxial nozzle. Precipitation chamber were pressurized at atmospheric and 40 bars pressure (gauge) to find out chamber pressure effect on the products. Samples were collected in the filter for morphology characterization using scanning electron microscopy JEOL JSM – T330A.

Result and Discussion

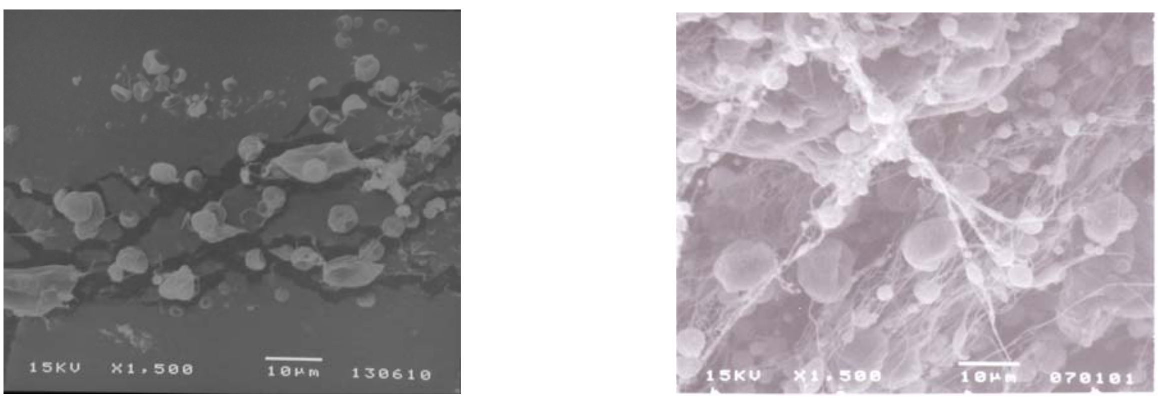
Nozzle Type

Two type of nozzles, axial and coaxial nozzles were used to investigate their effects on particle processed. Both nozzles were in 0,5 mm ID dimension, with 3.6 mm ID of annulus. The SEM analysis results were summarized in table 1.

wt %	**PS Morphology, $ID_{inner\ pipe}$ 0,5 mm Coaxial**	**PS M orphology, Φ_{nozzle} 0,5 mm Axial**
3	*Microsphere*, discrete	*Microsphere*,*fibril*,agglomerate
5	*Microsphere*, discrete	*Microsphere*,*fibril*,agglomerate
7	*Microsphere*, discrete	*Microsphere*,*fibril*,agglomerate
9	*Microsphere*, discrete	*Microsphere*,*fibril*,agglomerate
11	*Microsphere*, discrete	*Microsphere*,*fibril*,agglomerate
13	*Microsphere*, *fibril*, discrete	*Microsphere*,*fibril*,agglomerate
15	*Microsphere*, *fibril*, discrete	*Microsphere*,*fibril*,agglomerate

Table 1 Summary of SEM Analysis Result

The overall results showed that coaxial nozzle could produce more dizcrete particle than the axial one. Hydrodynamic effect from coaxial spraying might have assisted atomization and particle reduction. The result is in agreement with Sze Tu et al report which stated the coaxial nozzle usage could reduce particle size due to mechanical and hydrodynamic effect from the spraying (Sze Tu et al 1998).



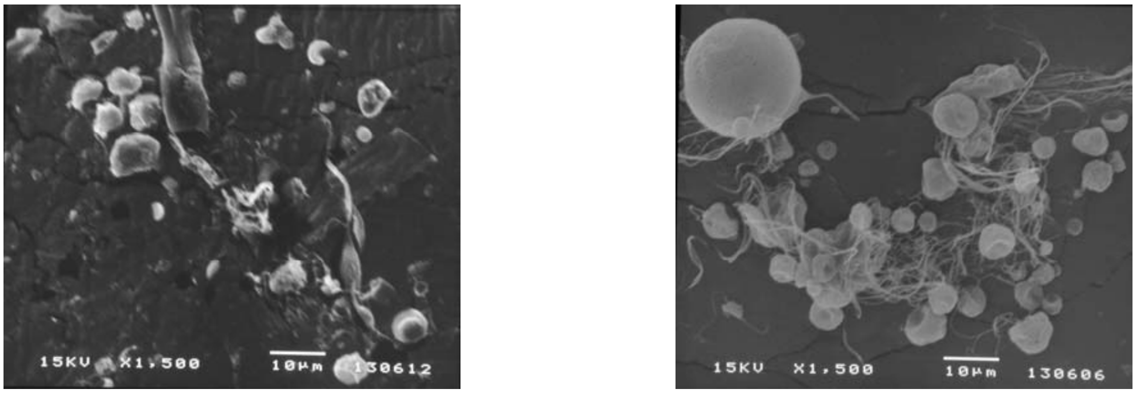
(a) (b)

Figure 1 SEM Images of Processed Polystyrene from 7% wt Solution
(a) using Coaxial Nozzle ID = 0.5 mm
(b) using Axial Nozzle ID = 0.5 mm

Electron microscopy images of figure showed the samples characterization of polystyrene processed with different type of nozzles. Minimum of fibril formation were produced from coaxial nozzle usage.

Coaxial Nozzle Application

Coaxial nozzle with ID annulus 3.6 mm and 4.6 mm were used to study the processing of polystyrene microspheres.



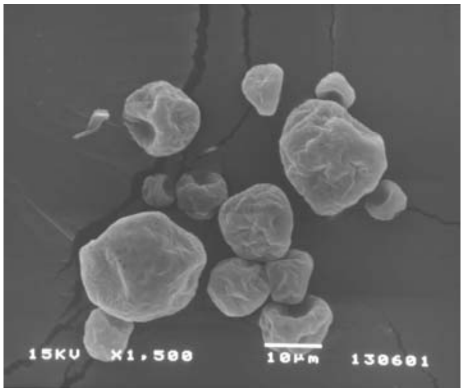
(a) (b)

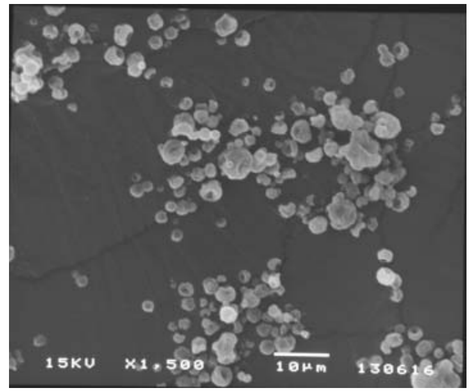
Figure 2 SEM Images of Processed Polystyrene from 11 % wt Solution
(a) using Coaxial Nozzle ID annulus 3.6 mm
(b) using Coaxial Nozzle ID annulus 4.6 mm

Figure 2 showed the examples of processing by different coaxial nozzles. The 3.6 ID mm type could produce discrete particles with fibril appearance after 13 % wt of original solution. The 4.6 mm ID type image showed the presence of fibril starting at 11% wt solution. The result suggested that the smaller the ID annulus the more discrete the microspheres produced. The smaller the ID annulus, the faster CO_2 flowrate in the nozzle. The increase of CO_2/solution ratio might have increased the amount of solvent uptake by gas. As a result, the possibility of droplets formation could have increased.

Precipitation Chamber Pressure

Precipitation were carried out in two different conditions i.e. at atmospheric and 40 bars gauge. The SEM analysis showed that at higher precipitation pressure, more discrete particles were produced.





(a) (b)

Figure 3 SEM Images of Processed Polystyrene from 3% wt Solution
(a) atmospheric precipitation
(b) 40 bars precipitation

The application of high precipitation pressure could produce narrower particle size distribution.

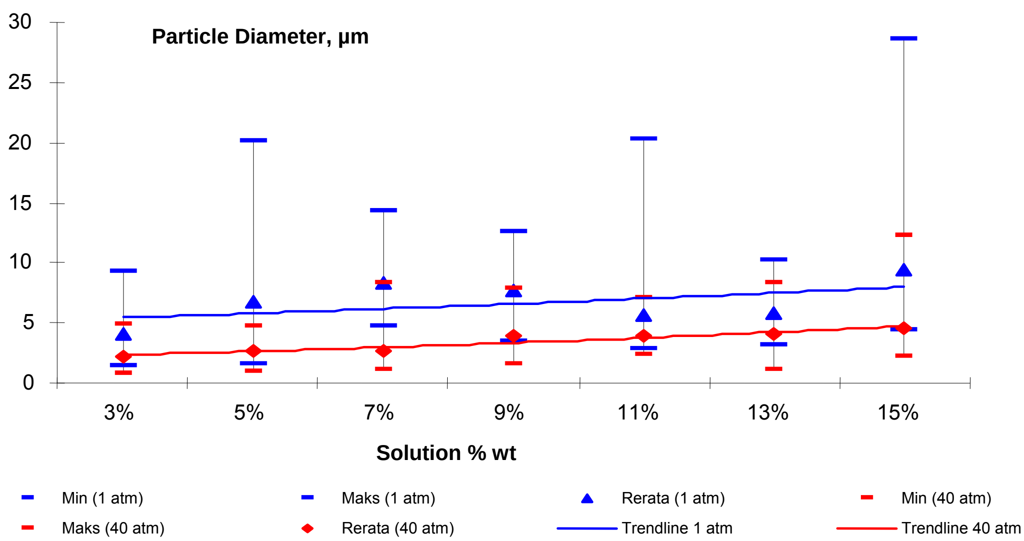


Figure 4 Diameter Particle Distribution of Polystyrene Processed
(Coaxial Nozzle 3.6 ID type)

Figure 4 showed that mean diameter particle of atmospheric precipitation was larger than that of the 40 bars operating condition. It was recorded that average particle size was c.a 5 – 10 µm for atmospheric precipitation while less than 5 µm particle size was produced for 40 bars precipitation. The result suggested that the increasing pressure in precipitation chamber could have affected the precipitates formation. High pressure precipitation chamber was loaded heavily by antisolvent CO2 which may yield a more effective precipitates formation during the contact, comparing to the lower density of antisolvent at atmospheric pressure operation.

Some shrinkage occured both at atmospheric and elevated pressure precipitation. The shrinkage might have been caused by the mechanical forces generated during the spraying. The slow release of solvent by the gas might have caused 'collapse' occurence in the particles produced.

Conclusion

The study has succeded in producing microparticle of polystyrene. The usage of coaxial nozzle could produce more discrete particles comparing than that of the axial one. The particles produced could achieve more uniform and smaller size when annulus nozzle diameter of the nozzle was smaller in elevated precipitation pressure.

Acknowledgment

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