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Contributed Paper

PE Wax Microparticle Production by External Mixing Two-Fluid Nozzle Atomization Process

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ABSTRACT

The effects of atomizing temperature, air pressure and air to liquid diameter ratio on particle size, particle size distribution and shape were studied in a polyethylene wax (PE wax) atomization process in the external mixing two-fluid nozzle atomizer. PE wax was melted and atomized at temperatures ranging from 120°C to 180°C, and atomizing air pressure was applied at pressures ranging from 1 to 7 bars. The results indicate that the particle size and bulk density of the PE wax particles decreased as the liquid cap diameter decreased and the air pressure and/or the melting temperature increased. The micrograph from a scanning electron microscope (SEM) showed that the atomization process produced smooth spherical PE wax particles.

Keywords: atomization process, microparticles, atomizer, size distribution, polyethylene

1. INTRODUCTION

Polyethylene (PE) wax has been widely applied in several processes for instance, in manufacture of rubber and plastic products, asphalt roads, paint/printing inks, production of crayons, packaging industry, manufacture of biomedical products and as a lubricant [1-3]. It was found that PE wax powder has lower heat of fusion and lower percentage of crystallization than PE wax flakes (size around 0-3 mm) [3]. Accordingly, PE wax microparticles are widely used in many applications mentioned above. This study therefore focused on PE wax powder production providing microparticles size of 50-200 μm, which are beneficial for industrial use [3].

Conventionally, to produce fine particle or powder from a solution, drum dryer can be used such as drying of natural product [4]. Unlike making powder from natural product solution, PE wax powder can be produced by either a grinding process or a ball mill process. However, these processes are energy-intensive and can cause difficulty in control of particle size and shape distribution. Additionally, the product can be contaminated in the grinding equipment [3].

In this study, an atomizing process was employed. The atomization unit consists of the heating zone, the atomizing zone (some includes the crystallizing zone) and the powder collecting zone as shown in Figure 1. The basic steps of the atomization process include liquid-air contact, atomization, and powder collection [5]. A molten polymer stream is supplied to the

atomizing nozzle and is atomized by compressed air to form polymer droplets. The droplets are rapidly cooled by cooler air to form solid polymer particles, and then fall under gravity into a cyclone or hopper located at the bottom of the unit. The atomization process can eliminate intermediate steps such as filtering, grinding and tableting which results in lowering the operating costs [5]. Moreover, this process is effective and simple. The particle size, size distribution and shapes can be controlled by manipulating a small number of input parameters such as air pressure, atomizing temperature, fluid properties, and the ratio of air to liquid mass flow rates [3, 6].

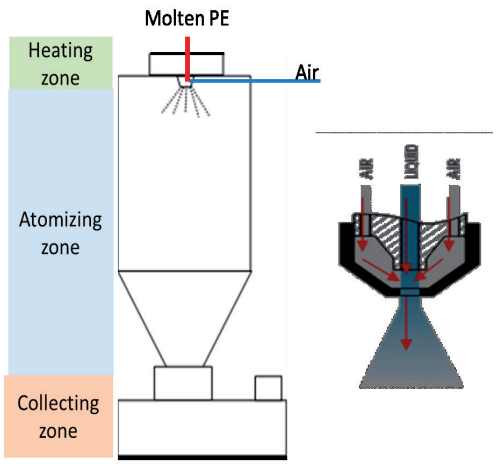


Figure 1. Schematic diagram of the atomization unit (vertical chamber) and the nozzle [5].

2. MATERIALS AND METHODS

2.1 Material and Equipment

Polyethylene wax (PE wax)

The PE wax flake (from IRPC Public Company Limited) used in this study had a density of between 0.8 and 0.9 g/cm^3 and the viscosity at 140°C about 1-30 cps. The melting and crystallizing temperatures analyzed by differential scanning calorimeter were 118°C and 78°C, respectively.

Atomizer

The atomizer unit used in this work had a 0.9-meter diameter and a 1.9-meter height. A heating pot for melting PE wax was set at the top of the atomizer as shown in Figure 1. Inside, the unit consisted of external mixing two-fluid nozzle. There was a temperature controller to control the temperatures of melting plot and nozzle at desired temperatures.

2.2 Methods

Experimental setup

Air feed rate controlled by a regulator at a design pressure was fed into the atomizer at room temperature (30°C) while the PE wax flakes were melt at the desired melting plot temperature before being fed into the atomizer by gravity flow (about 5 L/hour).

Experimental design

The experimental conditions of the three input parameters: air pressure (bar), atomizing temperature (°C) and the ratio of air to liquid mass flow rates measured in term of the air to liquid diameter (millimeter) ratio (D_A/D_L) and adjusted by changing a nozzle cap are shown in Table 1. Each experiment was performed twice.

Table 1. Conditions and units of input parameters.

Input parameters	Conditions
Air pressure (bar)	1, 2, 3, 4, 5, 6, 7
Atomizing temperature (°C)	120, 140, 160, 180
D_A/D_L	3.0:0.5, 3.0:1.0, 3.0:1.5

Determination of PE wax microparticle bulk density size and shape

The bulk density of the PE wax microparticles was determined by sampling the microparticles into a 25.1-cm^3 measuring vessel and measuring its weight. The particle sizes and size distribution

were determined by the laser diffraction method using a Malvern 2000 Droplet and Particle Sizer (Malvern, UK). (Condition: reflective index of ethanol 1.36, laser intensity 60-80% and 2500 rpm) These properties cannot be determined by sieve because heat created during sieving make the microparticles melt. The micrograph from a scanning electron microscope (SEM) was used to analyze the shape and surface of the microparticles.

Effect of input parameters on PE wax particles

The Taguchi method [7] was used to determine the effects of the input parameters on the microparticle sizes and their properties. The selected input parameters were air pressure (P), atomizing temperature (T) and air to liquid diameter ratio ($R=D_A/D_L$). Therefore, two-level-three-parameter arrays, L_4 (2^3), experiments were necessary.

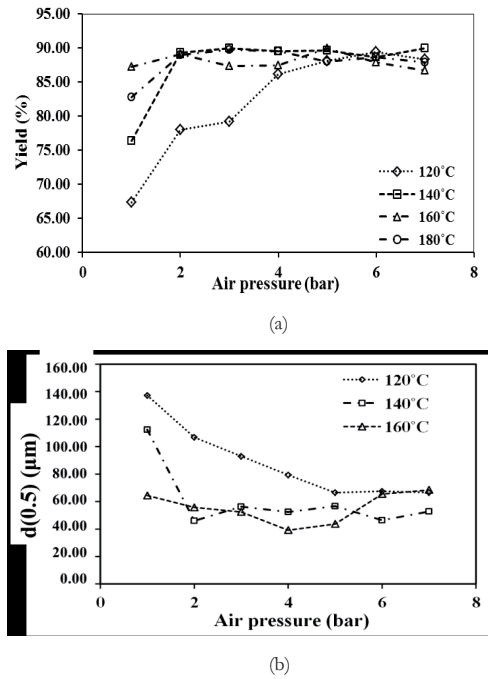
3. Results and Discussion

Since each experiment was run for a short time, therefore, the chamber temperature was about 50-60 °C.

3.1 Effect of Air Pressure and Atomizing Temperature

Considering the desired microparticle sizes smaller than 200 μm, the amount shown in term of yield (%) of PE wax microparticles (%yield = amount of micropaticles sizes smaller than 200 μm/ total amount of particle x 100) and the microparticle distribution in term of median diameter (d(0.5)) were plotted against the air pressure as shown in Figure 2(a) and 2(b), respectively. The smallest size obtained in this work was about 20 μm (d(0.1)). The amount of microparticles tended to increase with increased air pressure or temperature because increased air pressure or increased temperature produces more gas impingement energy [8] or internal energy in the stream and causes the stream of molten polymer to have a lower viscosity as well as reducing shear thinning behavior. Thus, the molten polymer breaks up and easily forms microparticles [3, 9-11]. At a low atomizing temperature (120°C), increased air pressure increased the amount of PE wax microparticles at a significantly higher level than at other temperatures, especially between 1 and 2 bars because of a pressure- dependent viscosity. This temperature also gave the largest median diameter of the microparticles.

At the temperatures above 140 °C, air pressure from 1 to 2 bar caused the PE wax median diameter to decrease and the amount of small microparticles (< 200 μm) to increase rapidly because these conditions still provide pressure-dependent viscosity behavior. Thus, it can be stated that the higher the pressure, the higher input kinetic and the lower the viscosity



(a)

(b)

Figure 2. Amount and median diameter of PE wax microparticles at various atomizing temperatures and air pressures at D_A/D_L = 3.0:1.5; (a) Yield (%) of PE wax microparticles smaller than 200 μm and (b) Median diameter.

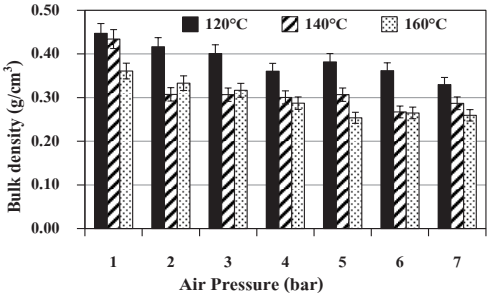


Figure 3. Effect of atomizing temperature and air pressure on the bulk density of PE wax microparticles (D_A/D_L = 3.0:1.5).

and this caused the molten PE wax stream to be simply atomized as discussed above [9- 11]. On the other hand, operating at pressures above 2 bar, air pressure does not have a significant effect on the PE wax microparticles. From Figure 3, increased air pressure and increased atomizing temperature tended to insignificantly decrease the bulk density of the PE wax microparticles because of the viscosity effect described in the previous paragraph while these increased air pressure and increased temperature make the surface of the microparticles more porous as illustrated by white spots in Figure 4 for the effect of the pressure and Figure 5 for the effect of the temperature.

This low bulk density also caused a void from adhering microparticles [12, 13]. However, both figures show spherical microparticles from this atomization.

3.2 Effect of Air to Liquid Diameter Ratio (D_A/D_L)

The effect of the ratio of the air flow rate to the liquid (molten polymer) flow rate was studied in terms of the air to liquid diameter ratio (D_A/D_L). This is one of the most important parameters affecting the microparticles in the atomization process [14- 16]. The results show that the median diameter (d(0.5)) of the PE wax microparticles were 33.30 ± 3.84, 48.60±2.37 and 112.00±1.70 operating at a D_A/D_L of 3:0.5,

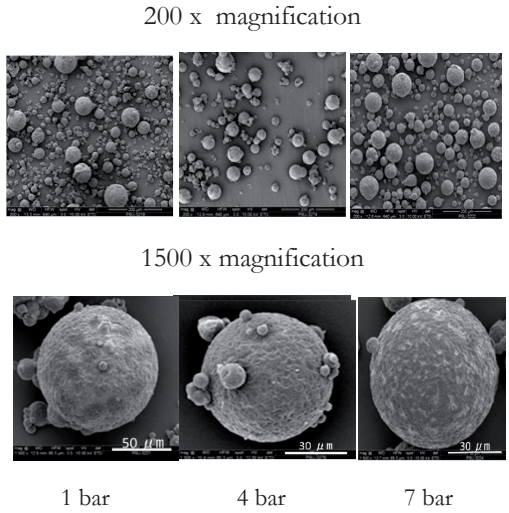


Figure 4. SEM micrographs of PE wax powder at different air pressure (atomized at 140°C and 3.0:1.5 D_A/D_L at 1500x magnification).

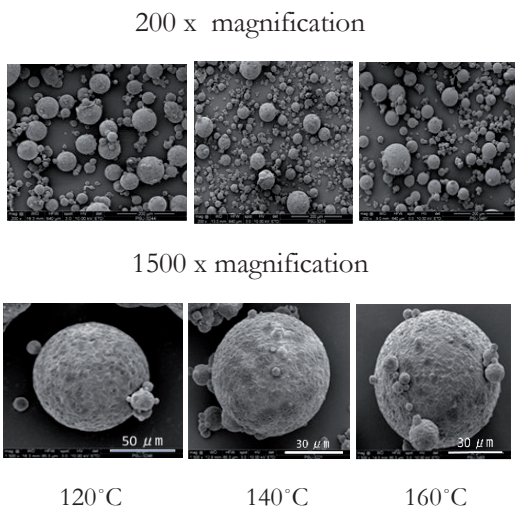


Figure 5. SEM micrographs of PE wax micropariclesat different atomizing temperature (atomized at 1 bar and 3.0:1.5 D_A/D_L).

3:1.0 and 3:1.5, respectively. Decreasing the D_A/D_L by increasing the liquid mass flow rate while keeping the air mass flow rate constant caused the PE wax droplets to disperse and form microparticles easily [17, 18].

In contrast, the bulk density of the PE wax microparticles decreased with the liquid diameter decreased as shown in Figure 6. But

too small liquid diameter was not practical to operate [19]. This may be caused by microparticle porosity or surface roughness. The higher the D_A/D_L, the higher the surface roughness as shown in Figure 7.

3.3 Effects of Input Parameters on PE Wax Microparticle Determination

The results from the Taguchi method analysis are shown in Table 2.

In contrast to the results of the atomization of molten polyethylene glycol where the flow rate has the most important effect on particle size [20] as can be seen from Table 2, the atomizing temperature has the strongest effect on the percent yield similar to that of the interaction between the air pressure and the D_A/D_L ratio, since the percentage of the PE wax microparticles smaller than 200 μm was increased to 6.28% by changing the temperature (average percent yield at T_1 - average percent yield at T_2) or by changing the interaction between the air pressure and the D_A/D_L ratio (average percent yield at P_1R_1 and P_2R_2 - average percent yield at P_1R_2 and P_2R_1). This may be caused

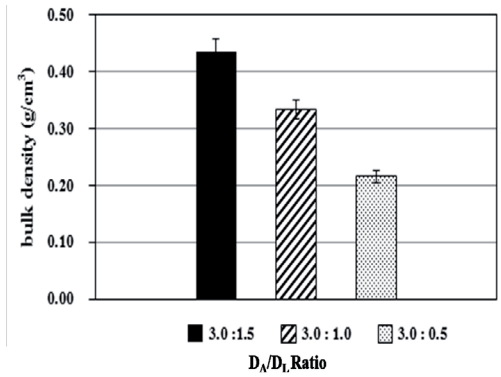
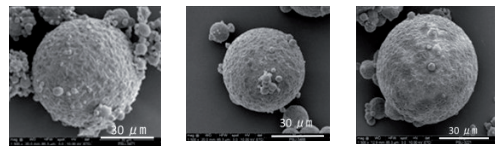


Figure 6. Effect of D_A/D_L on the bulk density of PE wax microparticles at 140 °C and air pressure of 1bar.



3.0:0.5 3.0:1.0 3.0:1.5

Figure 7. SEM micrographs of PE wax microparticles at different D_A/D_L ratios (atomized at 1 bar and 140°C atomizing temperature and 1500x magnification).

Table 2. Orthogonal array analysis of operating conditions on yield (%) of smaller than 200μm microparticles and bulk density.

Experiment	Conditions			Response (y_i)	
	Air pressure, P (bar)	Atomizing temperature, T (°C)	D_A/D_L ratio, R	Yield (%)	Bulk density (kg/m^3)
1	P_1=7	T_1= 180	R_1=3.0:1.5	y_1= 87.84	y_1= 294.4
2	P_1=7	T_2= 140	R_2=3.0:1.0	y_2= 82.82	y_2= 287.0
3	P_2=1	T_1= 180	R_2=3.0:1.0	y_3= 83.87	y_3= 318.2
4	P_2=1	T_2= 140	R_1=3.0:1.5	y_4= 76.33	y_4= 434.2

Independent variable		Effect	
		Yield (%)	Bulk density (kg/m^3)
Effect of P	$\lvert(y_1+y_2)/2-(y_3+y_4)/2\rvert$	5.23	**85.5**
Effect of T	$\lvert(y_1+y_3)/2-(y_2+y_4)/2\rvert$	**6.28**	54.3
Effect of R	$\lvert(y_1+y_4)/2-(y_2+y_3)/2\rvert$	1.26	61.7
Effect of P×T	$\lvert(y_1+y_4)/2-(y_2+y_3)/2\rvert$	1.26	61.7
Effect of P×R	$\lvert(y_1+y_3)/2-(y_2+y_4)/2\rvert$	**6.28**	54.3
Effect of T×R	$\lvert(y_1+y_2)/2-(y_3+y_4)/2\rvert$	5.23	**85.5**

by temperature-dependent viscosity. Thus, it can be said that increasing the temperature is equivalent to increasing both the air pressure and the D_A/D_L ratio.

With regard to bulk density, the value depended on the effect of the air pressure (P) or the interaction between the melting temperature and the D_A/D_L ratio. Increased air pressure decreased the bulk density as mentioned above. This allows an interaction between the atomizing temperature and the D_A/D_L ratio. However, from economic and safety considerations, it is preferable to operate the atomization process at low pressure and/or low temperature.

4. Conclusions

The atomization process using an external mixing two-fluid nozzle is considered a viable alternative to the conventional process for the production of polymer microparticles. This successful test of an atomization process for PE wax microparticles production produced spherical microparticles with a size smaller than 200 μm, and a satisfactory particle size distribution. It was found that the atomizing temperature has the strongest effect on the particle size and yield while the air pressure had the greatest effect on the bulk density. The size of the PE wax microparticles decreased with increased atomizing temperature or pressure. Operating with a higher air pressure produced a lower bulk density.

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References

[1] Wiwax Ltd. PE wax applications; Available at http://www.wiwax.eu/ APPLICATIONS. (accessed on June 1, 2011).

[2] Piringer O.G. and Baner A.L., *Plastics Packaging: Interactions with Food and Pharmaceuticals,* 2nd Edn., Wiley-VCH. 2008.

[3] Otaigbe J. U. and Mcavoy J. M., *J. Adv. Polym. Technol.*, 1997; **17**(2): 145–160. DOI: 10.1002/(SICI)1098-2329(199822)17:2<145::AID-ADV5>3.0.CO;2-T.

[4] Jittanit W., Chantara-In M., Deying T. and Ratanavong W., *Songklanakarin J. Sci. Technol.*, 2011; **33**(1): 33-41.

[5] Sh-jiyuan Ltd., Spray drying manual; Available at http://www.sh-jiyuan.com. (accessed on June 5, 2011).

[6] Thybo P., Hovgaard L., Lindelov J.S, Brask A. and Andersen S.K., *Pharm. Res.*, 2008; **25**(7): 1610-1620. DOI: 10.1007/s11095-008-9565-8.

[7] Peace G.S. *Taguchi Methods*, 2nd Edn. The United States of America: Addison-Wesley Publishing Company, 1993.

[8] Morakotjinda M., Fakpan K., Yotkaew T., Tosangthum N., Krataithong R., Daraphan A., Siriphol P. Wila P., Vetayanugul B. and Tongsri R. *Chiang Mai J. Sci.*, 2010; **37**(1): 55-63.

[9] Barnes H.A., *J. Non-Newtonian Fluid Mechanic*, 1997; **70**: 1-33. DOI: 10.1016/S0377-0257(97)00004-9.

[10] Mulhem B., Schulte G. and Fritsching U., *Chem. Eng. Sci.*, 2006; **61**(8): 2582–2589. DOI: 10.1016/j.ces.2005.11.035.

[11] Ilic I., Dreu R., Burjak M., Homar M., Kerc J. and Srcic, S., *Int. J. Pharm.,* 2009; **381**: 176-183. DOI: 10.1016/j.ijpharm.2009.05.011.

[12] Lin J., Qian L., Xiong H. and Chan T.L., *Surf. Coat. Technol.,* 2009; **203**: 1733–1740. DOI: 10.1016/j.surfcoat.2009.01.009.

[13] Bose A., *Advance in Particle Materials,* 1st Edn., United States of America: Butterworth-Heinemann, 1995.

[14] Reverchon E. and Antonacci A., *J. Supercrit. Fluids*, 2007; **39**(3): 444-452. DOI: 10.1016/j.supflu.2006.03.005.

[15] Czisch C. and Fritsching U., *Mat. Sci. Eng.*, 2007; **A. 477**(1-2): 21–25. DOI: 10.1016/j.msea.2007.06.087.

[16] Ejim C.E., Rahman M.A., Amirfazli A. and Fleck B.A., *Fuel*, 2010; **89**(8): 1872-1882. DOI: 10.1016/j.fuel.2010.03.005.

[17] Lagutkin S., Achelis L., Sheikhaliev S., Uhlenwinkel V. and Srivastava V., *Mat. Sci. Eng.*, 2004; **A 383**(1): 1–6. DOI: 10.1016/j.msea.2004.02.059.

[18] Juslin L., Antikainen O., Merkku P. and Yliruusi J., *Int. J. Pharm.*, 1995; **193**: 247–256. DOI: 10.1016/0378-5173(95)00081-S.

[19] Masua U., Prasertsit K., Innachitra P. and Keatkhunboot T., *Asean J. Chem. Eng.*, 2013; **13**(2):1-8.

[20] Borini G.B., Andrade T.C. and Freitas L.A.P, *Powder Technol.,* 2009; **189**: 520-527. DOI: 10.1016/j.powtec.2008.08.004.