

STRUCTURAL ANALYSIS OF SPRAY-DRIED COCONUT SHELL LIQUID SMOKE POWDER

[Analisis Struktural Bubuk Asap Cair Batok Kelapa Hasil Pengeringan Semprot]

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ABSTRACT

The research was aimed at determining spray drying conditions during the production of smoke particulate powder and its consequences on the physical properties of the product. The experiment was carried out using a complete randomized factorial design. Samples were three solution of chitosan (CS)-maltodextrin (MD) based nanoparticles i.e. CS (0.5% w/v) and MD (9.5% w/v) in acetic acid (1.0% v/v) without liquid smoke (F1), only MD (10% w/v) in liquid smoke (F2) and a mixture of CS (1.5% w/v) and MD (8.5% w/v) in liquid smoke (F5). Each sample solution was prepared at 10% solid contents with addition of 1.0% sodium tripolyphosphate. The experimental factors were inlet air temperatures (T) of 130°C (T1) and 150°C (T2) and feed flow rate (L) of 2.4 mL/minute (L1) and 5.1 mL/minute (L2). The parameters evaluated included bulk density, yield, moisture content, water activity, morphology and particle size. Results showed that the average particle sizes decreased when the inlet air temperature increased. The bulk density, moisture content and water activity of powders tended to decrease with the increase of inlet air temperature. In contrast, the powder yield increased with increasing of inlet air temperature. Furthermore, characteristics of the powder particulates were spherical with smooth surfaces for all treatments but when the inlet air temperatures was high the particles has deeper surfacial grooving and shriveled, especially for sample F1.

Keywords: chitosan, coconut shell liquid smoke powder, maltodextrin, nanoparticle, spray drying

ABSTRAK

Penelitian ini bertujuan untuk menentukan kondisi pengeringan semprot selama produksi partikel bubuk asap cair termasuk sifat-sifat fisiknya. Percobaan dilakukan dengan menggunakan rancangan acak lengkap faktorial. Sampel terdiri dari tiga larutan berbasis nanopartikel kitosan (CS)-maltodekstrin (MD) yaitu pertama CS (0,5% b/v) dan MD (9,5% b/v) dalam asam asetat (1,0% v/v) tanpa asap cair (F1), kedua hanya MD (10% b/v) dalam asap cair (F2), dan ketiga campuran CS (1,5% b/v) dan MD (8,5% b/v) dalam asap cair (F5). Setiap larutan sampel dengan total padatan 10% ditambahkan natrium tripolifosfat 1,0%. Faktor penelitian adalah suhu udara masuk (T) terdiri dari 2 tingkat (T1 = 130°C dan T2 = 150°C) dan kecepatan aliran umpan (L) terdiri dari 2 tingkat (L1 = 2,4 mL/menit dan L2 = 5,1 mL/menit). Parameter penelitian meliputi bulk density, rendemen, kadar air, aktivitas air, morfologi dan ukuran partikel. Hasil penelitian menunjukkan bahwa ukuran rata-rata partikel menurun ketika suhu udara masuk meningkat. Bulk density, kadar air dan aktivitas air dari bubuk cenderung menurun dengan meningkatnya temperatur udara masuk. Sebaliknya, hasil bubuk meningkat dengan meningkatnya temperatur udara masuk. Selain itu, karakteristik partikel bubuk berbentuk bulat dengan permukaan halus untuk semua perlakuan, tetapi ketika suhu udara masuk semakin tinggi maka permukaan bubuk lekuk dan keriput terutama untuk sampel F1.

Kata kunci: kitosan, bubuk asap cair tempurung kelapa, maltodekstrin, partikel nano, pengeringan semprot

INTRODUCTION

Production of high-tech materials using nanoparticles is of great scientific interest in order to yield products with superior characteristics. The well established method for it is dispersing nanomaterials in a suspension which successively transformed into a dry form. Spray drying technique produces micron-sized

particles from suspensions of nanoparticles and other components having dimensions in the nanometer range. Hence, the spray drying technology may establish a link between the nanomaterial synthesis and the traditional processes which can produce the actual end products using micron-sized materials. (Lindeløv and Wahlberg, 2008).

Spray drying is a process of transformation from an emulsion, suspension or dispersion system to a dry state product through atomization and dispersion in a hot gas (Oliveira *et al.*, 2010). Operationally, it is a one-step processing where a liquid feed is altered into powder which concomitantly minimising handling while reducing the bulk weight and size of

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the particles and preserving the product by reducing the water activity required for bacterial degradation (Fang *et al.*, 2005). It has also been used to produce micro-encapsulated materials by a particular carrier used in the feed slurry and selective diffusion. The common carriers include carbohydrates, gums and cellulose esters and ethers. Moreover, mixed carriers are also used as efficient and stable agents in particular feed materials (Drusch *et al.*, 2007). In this process, unstable ingredients are mixed or homogenized in a solution containing materials which envelope targeted components so that it forms a stable suspension. Such solution is called wall materials. The suspension is then fed into a spray dryer where it is converted into dried powder.

The applications of microencapsulation can be found in various food industries; the important examples are coating materials of colorants, flavors, vitamins, and other unstable food ingredients in order to prolong the shelf life (Bansode *et al.*, 2010). In addition, microencapsulation can transform liquids into free-flowing powders to ease handling. Typically, the effective wall materials for spray drying comply with the following properties: good emulsification, film forming, high solubility, low viscosity at high concentrations (Reineccius, 1988) yet biocompatible with living organism cell membranes and degradable as well as cheap.

Micro or nanoencapsulation including micro- or nanospheres or encapsulation is mostly applied in pharmaceutical industry aiming at manipulating pharmacokinetics of a drug release, promote stability and reduce toxicity (Tewa-Tagne *et al.*, 2007). The micro or nanospherical particulates have matrix architectures in which the bioactive compound can be adsorbed at the outer surface or interior pores of the particles yet the bioactive compound is enveloped or encapsulated by polymeric coat inside the particles.

Various natural polymers can be used in manufacturing nanoparticles. In the present study, chitosan (CS) and maltodextrin (MD) is chosen as the polycationic and anionic compounds. CS is a derivative of chitin, a biopolymer found in the shells of crustaceans, cell wall of fungi and other microbia (Pranoto *et al.*, 2005; Anal, 2010). It has high affinity to cross-link with the counterpart polyanions, biocompatibility, biodegradability, antibacterial properties, mucoadhesivity. Moreover, it is nontoxic with a low immunogenicity (Darmadji and Izumimoto, 1994; Anal *et al.*, 2006) and has ability to act as an adsorption improver (Vila *et al.*, 2002). On the other hand, MD is the most commonly used materials for encapsulation of bioactive materials. MD is water-soluble and capable of protecting ingredient from oxidation. Several studies have explored MD potentials in preserving vitamin C in fruit juice and stability of acerola powder (Dib *et al.*, 2003; Righetto and Netto, 2005).

Among the main factors, feeding-liquid temperature, feed rate, inlet air temperature, flow rate and outlet air temperature (Patel *et al.*, 2009) is important in process optimization. Increased feed temperature should be compensated with reduced viscosity and droplet size. However, high temperatures may cause volatilization or degradation of heat-sensitive ingredients. Controlled feed rate is also important to ensure each droplet adequately dried before it comes in contact with

the surface of the drying chamber (Loksuwan, 2007). The lower inlet air temperature, the lower evaporation rate causes the formation of microcapsules which have high density membranes, high water contents, and poor fluidity by which the particles agglomerate easily (Patel *et al.*, 2009). Meanwhile, outlet air temperature will depend on the established specification of drying characteristics. The ideal outlet air temperature for the microencapsulation of food ingredients such as flavors has been reported to be 50-80°C (Sebastien, 2004).

Coconut shell liquid smoke has been reported to contain bioactive compounds such as phenols, carbonyls and organic acids. Therefore, the coconut shell liquid smoke is potential in increasing shelf life of proteinaceous food products (Darmadji *et al.*, 2009; Zuraida *et al.*, 2011). The bioactive compounds of liquid smoke needs to be protected against deterioration during the process by mean of encapsulation. However, there is no study reporting the encapsulation of liquid smoke components. In addition, a little is known on combination CS-MD as encapsulates in different concentration into liquid smoke solution.

The objectives of this study were to produce spray-dried coconut shell liquid smoke and to determine the effects of different spray drying temperatures and suspension-feed flow rates on the structure and physical properties of the particulate powders.

MATERIALS AND METHODS

Materials

Raw coconut shell liquid smoke used in this study was obtained from Tropica Nucifera Industry, Yogyakarta, Indonesia. This material was purified using redistillation method in the laboratory. Chitosan (CS) was purchased from Biotech Surindo, Indonesia (deacetylation degree 91.5%, moisture 10.43%, ash 0.71%). Maltodextrin (MD) with Dextrose Equivalent (DE) 10.8% was from Grain Processing Corp. (Iowa, USA), Sodium tripolyphosphate (TPP) and glacial acetic acid (HOAc) were supplied by Sigma Chemicals Ltd. (Munich, Germany). The other chemicals used for analysis were of analytical grade.

Sample preparation

CS (0.5% w/v) and MD (9.5% w/v) were dispersed in an aqueous solution of glacial acetic acid (1.0% v/v) as F1 sample code. CS-MD mixed nanoparticles were prepared with a slight modification of previously described methodology (Grenha *et al.*, 2010), based on the polyelectrolyte complexation of CS with MD and additional ionic gelation of chitosan with TPP anions. Briefly, CS and MD were dissolved in coconut shell liquid smoke based on the various ratios CS : MD (0%: 10%) as F2 sample code and (1.5%: 8.5%) as F5 sample code. Sodium tripolyphosphate (1.0 mg/mL) was added in these mixtures then agitated using a magnetic stirrer at 200 rpm for 30 minute at room temperature. Nanoparticles were isolated by centrifugation (Centrifuge Damon/IEC Division, Connecticut, USA) at 3,000 rpm in a 50 mL conical tube for 30 minute at room temperature. The supernatant was discarded and nanoparticles were vacuum filtered (Gast, USA) using Whatman # 2. The solution of

nanoparticles was heated at 50°C into waterbath for 15 minute and was homogenized using a rotor–stator homogenizer (Ultraturrax T50 Basic IKA Werke, Germany) at 5.200 rpm for 2.5 minute.

Drying conditions

The sampel solution was fed into a Büchi B-290 minute spray dryer (Flawil, Switzerland) for drying. The operating conditions were as follows; aspirator rate 50%; drying inlet air temperature 130 and 150°C ($\pm 2^\circ\text{C}$), while the outlet air temperature varied between 57 and 82°C; feed flow rate was set at 2.4 and 5.1 mL/minute; atomization air rotameter 30 mm (to the bottom of the gauge ball) and the nozzle cleaner set to 4. The system was kept running without heating after the completion of the experiment until the inlet air temperature fell below 70°C. Spray-dried powders were collected, kept in amber bottles and stored under desiccated conditions at room temperature prior to powder characterization.

Bulk density

Bulk density of powder was measured by weighing 5.0 ± 0.1 g of a sample into a 10 mL graduated cylinder, rapped gently the cylinder for 3 times. The volume of the powder was recorded and the bulk density was calculated as following relationship.

$$\text{Bulk density (g/mL)} = \frac{\text{Weight of powder}}{\text{Volume of powder}}$$

Production yield

The percentage of production yield (% w/w) was calculated from the weight of dried microspheres (W1) recovered from each of 3 batches and the sum of the initial dry weight of starting materials (W2) as the following equation:

$$\text{Yield} = \frac{W1}{W2} \times 100\%$$

Determination of moisture and water activity

The moisture content of the samples was measured by hot air oven at 105°C for 16 h. The water activity of the samples was measured using Decagon Pawkit water activity meter (EminTech, Sweden).

Scanning electron microscopy

Scanning electron microscope (SEM FEI type Inspect S50, Oregon, USA) was used to study the surface properties of the spray-dried particulate powders. The particulates were attached to SEM stubs diameter of 10 mm using a two-sided adhesive tape. The samples were then sputter coated with gold and examined at 10.000 magnifications. An acceleration potential of 20 kV was used during micrograph.

Particle size measurement

The prepared microspheres suspended in distilated water were sized by using a laser particle size distribution analyzer

(Malvern Zetasizer Nanoseries Nano ZS Ver 6.20, Malvern Instruments Ltd, Malvern, UK). The size distribution was determined by the span value. Triplicate measurement was conducted.

Statistical analysis

The differences between the mean values of multiple groups were analyzed by one-way analysis of variance (ANOVA) with Tukey Methods range tests. ANOVA data with a $P < 0.05$ was classified as statistically significant. MINITABS 16.0 software, Origin 75 and Microsoft Excel 2007 program were used. Mean values from the experiments were obtained from triplicates.

RESULTS AND DISCUSSION

Visually, the coconut shell spray-dried nanoparticles were white powder especially from sample chitosan and maltodextrin in acetic acid solution 1.0% (F1), while nanoparticle with combined chitosan and malrodextrin in liquid smoke (F5) were yellowish.

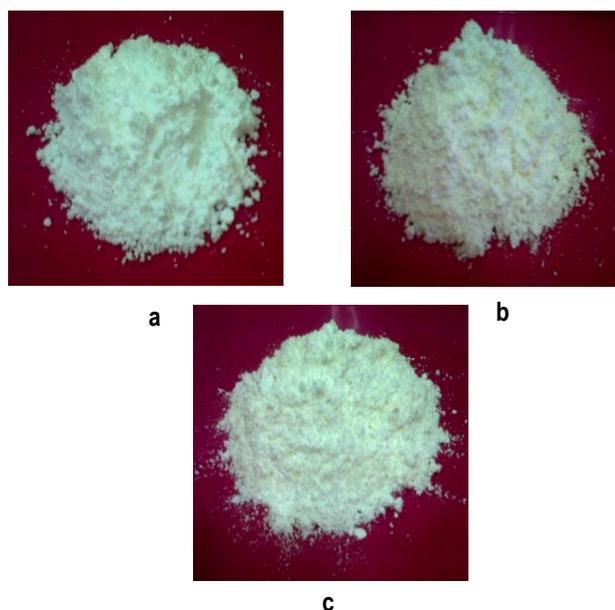


Figure 1. Spray-dried nanoparticle powders obtained from (a) chitosan (0.5% w/v) + maltodextrin (9.5% w/v) in acetic acid, (b) only maltodextrin (10% w/v) in liquid smoke, and (c) chitosan (1.5% w/v) + maltodextrin (8.5% w/v) in liquid smoke

This was due to an ionic interaction between $-\text{NH}_3^+$ group of chitosan and carbonyls groups of liquid smoke (data not shown). Carbonyls generally contribute to the color of the product once reacted with amino groups. Darmadji *et al.* (2009) mentioned that bioactive compounds in liquid smoke gave not only preservative and antioxidative effect but also color, flavor and taste to the product.

Moisture content and yield

Inlet air temperature is an important parameter for spray drying processes and can affect the final powder properties.

Table 1 shows all affecting factors in spray drying. It can be observed that the inlet air temperature is the main factor affecting powder properties. It was also found that at 150°C and high feed-flow rates, water were incompletely evaporated resulting powders with low bulk density and high water content.

The final moisture contents of powders fell into a range of 6.66 to 12.62%. The product yield increased as the inlet air temperature increased from 130 to 150°C. At 130°C, deposition of particles adhering on the cyclone wall was observed, which might be due to insufficiently dried droplet/particle within the drying chamber. Consequently, it led to a lower product yield. At inlet air temperatures of 150°C, the higher moisture removal and product yield was observed. In this systems, the moisture content of the particles was less ca 9.65% and ca 56.38% of recovery in the collection vessel and cyclon. In terms of industrial scale up, higher inlet air temperatures could be selected, due to the better water moisture removal and higher product recovery.

Bulk density

The bulk density of powder is affected by chemical composition, particle size and moisture content as well as by processing and storage conditions (Hong and Choi, 2007). Bulk density values correlated to the particle size as founded that powders derived from CS and MD in liquid smoke had smaller sizes (F5), while that from CS in acetic acid (F1) had the biggest size and hence CS can be contained most tightly and represented the highest bulk density value (Table 1). Reineccius (2004) found that spherical particles had the highest bulk density, the best packing properties and flowing ability. Moreover, according to Fuchs *et al.* (2006) spherical particulates powders can be compacted better. There is also an advantage of the high bulk density powders, i.e reduction of oxygen permeability, so the shelf life of product can be extended due to inhibition of oxidation. Therefore, the smallest bulk density

powders obtained from CS can elicit the suitable characteristic of wall material.

Water activity (a_w)

The a_w values resulted from CS and MD were in the range of 0.28-0.44. At the higher total solid contents, the drying rates were higher resulting in a lower a_w of the encapsulated powder. Newton (2007) suggests that appropriate a_w are between 0.2 and 0.3 because many chemical reactions such as oxidation, and chlorophyll degradation can occur slowly, and no microbial proliferation in such conditions. Moreover, the a_w below 0.30 ensures product stability (Drusch and Schwarz, 2006).

SEM of spray-dried powders

The sample powders were observed for granular surface properties using SEM (Figure 2).

Results clearly showed significant differences in size and shape. Nanocapsules from CS in acetic acid (F1) showed shrinkage and dimpled, while nanocapsules from liquid smoke without CS (F2) showed rounded shape, smooth surface with no obvious dents, and CS in liquid smoke powder showed spherical shape with extensive dented surface. Formation of dented surfaces of spray-dried particles was attributed to the shrinkage of the particles during the drying process (Chin *et al.*, 2010). The extensive dented surfaces of chitosan in acetic acid solution (F1) was probably attributed to maltodextrin granule disrupted resulted in more susceptible to shrinkage during the drying stages. Patel *et al.* (2009) suggests that the spherical spray dried particles had high surface/volume ratios showing appropriate characters of spray dried product. Reineccius (2004) recommends that spherical particles can retain the highest amount of flavoring agents. Based on this present result, it is considered that in a mixture of chitosan and maltodextrin were the proper wall materials for coconut shell liquid smoke powders.

Table 1. Characteristics of produced powders

Sample	Inlet Air Temperature (°C)	Feed Flow Rate (mL/minute)	Bulk Density (g/mL)	Yield*) (%)	Water Activity	Moisture (% db)
F1	130	2.4	0.42±0.01 ^a	53.83±0.16 ^d	0.31±0.01 ^{d e}	7.21±0.46 ^{gh}
F1	130	5.1	0.36±0.02 ^{abc}	62.49±0.50 ^b	0.44±0.01 ^e	8.74±0.03 ^h
F1	150	2.4	0.40±0.01 ^{cd}	72.53±1.05 ^b	0.28±0.01 ^a	6.66±0.08 ^f
F1	150	5.1	0.35±0.01 ^d	73.02±0.25 ^a	0.41±0.01 ^b	7.65±0.05 ^g
F2	130	2.4	0.42±0.01 ^a	52.63±0.38 ^d	0.35±0.01 ^c	11.24±0.03 ^b
F2	130	5.1	0.42±0.01 ^{bcd}	38.22±0.28 ^c	0.42±0.01 ^{de}	10.18±0.20 ^e
F2	150	2.4	0.40±0.01 ^a	57.25±0.27 ^h	0.31±0.01 ^{ab}	9.87±0.31 ^{de}
F2	150	5.1	0.37±0.01 ^{abc}	42.45±1.00 ^g	0.42±0.02 ^{ab}	11.22±0.07 ^{bc}
F5	130	2.4	0.42±0.03 ^{ab}	48.32±0.22 ^f	0.36±0.01 ^c	12.62±0.22 ^a
F5	130	5.1	0.42±0.01 ^{bcd}	43.78±0.44 ^e	0.43±0.01 ^d	10.55±0.34 ^b
F5	150	2.4	0.39±0.04 ^{ab}	50.42±0.31 ^g	0.32±0.01 ^{ab}	11.87±0.27 ^{cd}
F5	150	5.1	0.37±0.01 ^{abcd}	56.38±0.51 ^c	0.37±0.02 ^c	9.65±0.21 ^e

Note :

F1 = CS (0.5% w/v) and MD (9.5% w/v) in an aqueous solution of glacial acetic acid (1.0% v/v);

F2 = Only MD (10% w/v) in coconut shell liquid smoke;

F5 = CS (1.5% w/v) : MD (8.5% w/v) in coconut shell liquid smoke;

*) = data collected from cyclon and vessel fraction;

Reported means (± standard deviations) obtained from 3 replications measured triplo;

Data within column followed by different superscripts are significantly different at P<0.05 by Tukey Methods range test

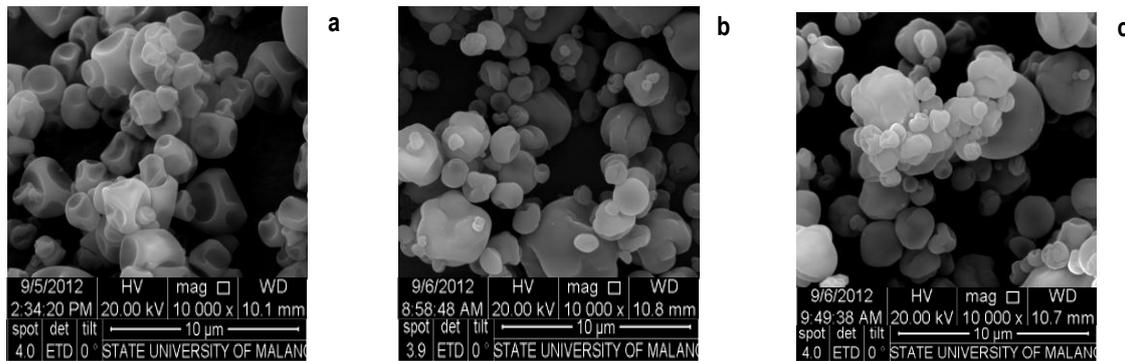


Figure 2. SME (10,000X) of spray-dried powders (a) chitosan (0.5% w/v) + maltodextrin (9.5% w/v) in acetic acid, (b) only maltodextrin (10% w/v) in liquid smoke, and (c) chitosan (1.5% w/v) + maltodextrin (8.5% w/v) in liquid smoke

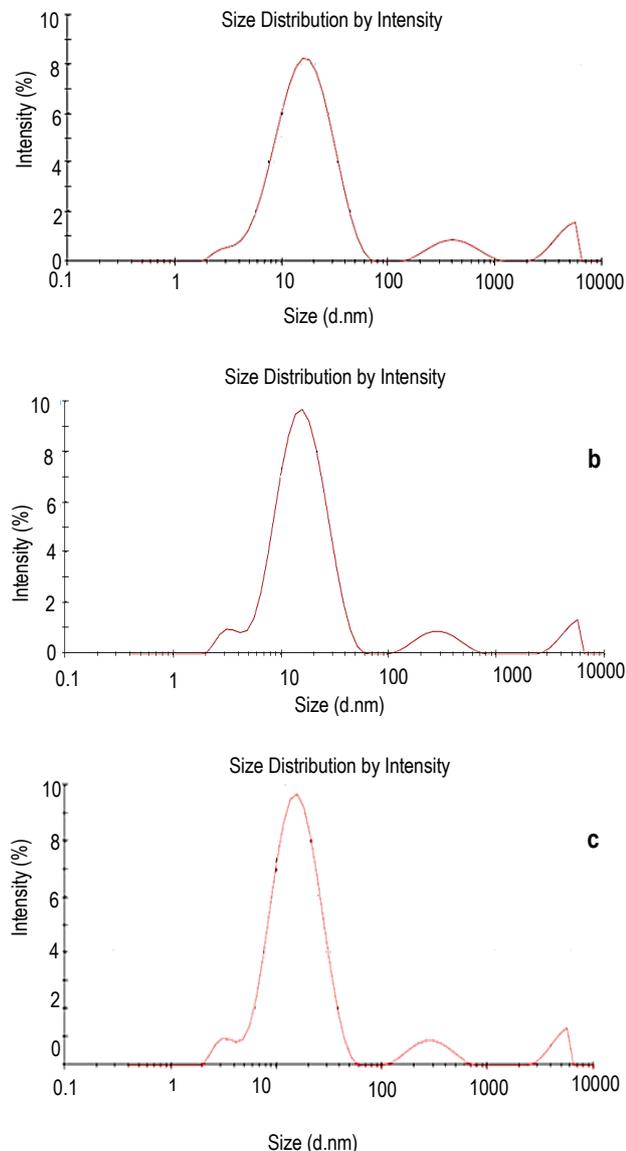


Figure 3. Particle size distribution powders generated from (a) chitosan (0.5% w/v) + maltodextrin (9.5% w/v) in acetic acid, (b) only

maltodextrin (10% w/v) in liquid smoke, and (c) chitosan (1.5% w/v) + maltodextrin (8.5% w/v) in liquid smoke

Particle size distribution

The mean particle size distribution of powders loaded as CS and MD nanoparticles are shown in Figure 3. The average size of particles formed from CS (0.5% w/v) + MD (9.5% w/v) in acetic acid was 16.21 nm, only MD (10% w/v) in coconut shell liquid smoke 14.87 nm, and CS (1.5% w/v) + MD (8.5% w/v) in coconut shell liquid smoke 13.43 nm. The differences in zeta potential with change in pH showed; at higher pH more cross-linked particles are formed compared to the lower pH (data not shown). Zhang *et al.*, (2004) publish a commercial low molecule weight CS to prepare CS nanoparticle at a concentration level of 0.1% (w/w) in a mixture of CS and TPP (weight ratio of 5:1) resulting in a bimodal particle size distribution ranging between 153 and 500 nm. The results were supported with the findings of Hu *et al.*, (2008), and they found the interactions between phenolic groups of liquid smoke and amino groups of CS (over phosphate group of TPP) may lead to decrease in the cross-linking density.

CONCLUSION

The increase in inlet air temperature up to 150°C during spray drying improved the yield. The highest particle yield of 73.02% was achieved at 150°C and also found to be dependant on the feed rate at 5.1 mL/minute. The moisture content of the powder was found to be dependent on the inlet air temperature of the drying air. Morphologically, SEM revealed the larger particles were dimpled, whereas smaller particles were more spherical. The smallest mean particle size of 13.43 nm was obtained from CS 1.5% w/v + MD 8.5% w/v in liquid smoke with the spray drying operation conditions inlet air temperature 150°C, feed flow rate 5.1 mL/minute, aspirator rate 50%, atomization air rotameter 30 mm and the nozzle cleaner set to 4. This study demonstrates that spray drying can successfully produce dried coconut shell liquid smoke powder from suspensions of nanomaterials. Therefore, it might be also be a good candidate for applying in food processing.

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