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Characteristics of liquid smoke nanoencapsulation with a combination of maltodextrin and gum arabic as co-encapsulant

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ABSTRACT

The development of powder-type liquid smoke synthesis by the nanoencapsulation method was proposed to overcome the difficulties in handling liquid smoke. In the liquid form, liquid smoke was unpractical and difficult to distribute and transportation. Otherwise, phenolic compounds were easy to deteriorate (oxidize) during storage. The aims of this research to determine the character of nanocapsules by the spray drying process, involving water content, water activity (Aw), color, solubility, total phenolic compounds, functional group (FTIR), phenolic compounds staining, nanocapsules morphology profiles, and also efficiency phenolic compounds in the spray drying process. Resulted nanocapsule has a color intensity 85.17 ± 1.13; a -0.45 ± 0.11 and 9.2 ± 0.13 b; water content 5.92 ± 0.12%, water activity 0.26 ± 0.01 and 85.02 ± 0.33% solubility. Scanning Electron Microscope (SEM) results showed nanocapsule form was round and wrinkled. This optimization process obtained particles with nanometer dimensions and in recovery efficiency of phenolic compounds in the spray drying process of 47.36%. These results have the potential to be applied to the development of the food industry.

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INTRODUCTION

Liquid smoke is a mixture of solutions and colloidal dispersions of wood smoke vapor in water obtained from the pyrolysis of wood (Dewi et al., 2019). According to Chairudin et al. (2022), liquid smoke originating from coconut shells has a chemical composition consisting of 5.13% phenol, 13.28% carbonyl, and 11.39% acid. These compounds play a significant role in forming the desired food properties and have different functional properties. One of the compounds in liquid smoke that acts as an antioxidant and antimicrobial is a phenolic compound. However, handling liquid smoke in liquid form is considered less practical, especially distribution and transportation, because it requires a special container/place. In addition, the content of phenolic compounds in liquid smoke is easily damaged (oxidized) during storage.

Nanoencapsulation consists of an active ingredient enveloped or surrounded by encapsulation on a size scale of 1 to 1000 nanometers (Zielińska et al., 2020). Another definition of nanoencapsulation is the development of technological innovations by encapsulating or absorbing the active compound in an encapsulation at a tiny size on the nanometer scale or 10-9 meters (Dawit Moges et al., 2020). Compared with microencapsulation techniques, nanoencapsulation can provide several advantages, including improving taste, color, texture, flavor, product consistency, absorption, and stability of bioactive components (Ashraf et al., 2021). According to Gharibzahedi and Smith (2021), nanoencapsulation can maintain the stability of bioactive components during processing and storage, protect active ingredients against interactions with the environment such as oxidation and hydrolysis, improve and enhance sensory properties such as taste, color, and texture, increase product stability and facilitate handling. Nanoencapsulation can also protect phenolic compounds with antioxidant activity, prevent material damage from interactions with other materials, and increase stability to light and oxygen (Albuquerque et al., 2021).

Produce nanometer-sized products (10-9m); several things must be considered: selecting the proper coating method and material. The coating material must be food grade and GRAS (Generally Recognized as Safe). One of the methods used to produce nanocapsules is spray drying. According to Piñón-Balderrama et al. (2020), the encapsulation process by spray drying must use a coating material with high solubility properties, forming a dry layer and low viscosity. One of the materials with high solubility and low viscosity, commonly used as an encapsulant, is maltodextrin.

Research by (Saloko et al., 2012) showed that the morphological profile of spray-dried smoked flour coated with maltodextrin was wrinkled on the outside of the wall. Bucurescu et al. (2018) encapsulated curcumin oleoresin with gum arabic coating, maltodextrin, and modified starch. The results of this study showed that curcumin oleoresin microcapsules coated with a combination of maltodextrin encapsulants with a higher ratio of gum arabic and modified starch had morphological forms that were not as salty, dented and there were cracks or breaks in the capsule walls that could trigger the release of the active ingredient.

To increase the encapsulation efficiency of phenolic compounds, this study, a combination of encapsulants using maltodextrin and gum arabic was carried out in this study. According to Łupina et al. (2021), gum arabic is an excellent film-forming agent in trapping or absorbing encapsulated components. In addition, gum arabic contains arabinogalactan protein (AGP) and glycoprotein (GP) groups, which act as emulsifiers and thickeners that effectively protect colloids and prevent colloids brittleness, wall cracks, and leakage of active ingredients to protect the active components in liquid smoke.

Ensure that the nanoparticles produced are nanometer in size; a homogenization process is carried out that considers the speed and length of time the homogenization occurs. Homogenization is a process that aims to uniform and reduce the size of the dispersed globules or particles. Wang (2015) researched the manufacture of liquid smoke nanoparticles from coconut shells using chitosan and maltodextrin encapsulants with homogenization using an ultra-turrax homogenizer at a speed of 4000 rpm for 2.5 minutes, capable of producing nanometer-sized particles. The average particle size formed from chitosan (0.5% w/v) and maltodextrin (9.5% w/v) in acetic acid was 16.21 nm. This study aims to determine the characteristics of liquid smoke nanoencapsulation with maltodextrin and gum arabic as co-encapsulant before being applied to the food matrix.

MATERIALS AND METHODS

Materials

Liquid smoke from Coconut shell grade 1 was obtained from PT Tropica Nucifera Industry, Depok, Sleman, Yogyakarta. The coating material used is maltodextrin combined with gum arabic as a co-encapsulant. Maltodextrin has a dextrose equivalent (DE) of 10.8% in small and fine white granules and a moisture content of 6.67% obtained from Grain Processing Corp., Jowa, USA. Gum arabic is the form of yellowish-white granules and has a water content of 13.45%. Chemicals used for analysis: Aquadest, Toluene, NaOH (Merck, Germany), Phenol (Merck, Germany), FeCl3 (JT Baker, USA), Na2CO3 (Merck, Germany), Folinciocalteau (Merck, Germany).

Method

The analysis carried out in this study was divided into three stages, namely liquid smoke analysis, nanoparticle analysis, and liquid smoke nanocapsule analysis. The liquid smoke analysis includes water content (AOAC international, 2019), total phenolic compounds (Delgado et al., 2019), and total dissolved (AOAC international, 2019). Nanoparticle analysis included particle size distribution, total dissolved solids (AOAC international, 2019), total phenolic compounds (Delgado et al., 2019), observation, and staining of phenolic compounds with FeCl3 (A Badria and S Aboelmaaty, 2019). Analysis of nanocapsules included water content, water activity (Aw), color, solubility (Sun et al., 2020), total phenolic compounds, observation and staining of phenolic compounds with $FeCl_3$ using a microscope with optilab software (Umar et al., 2021) functional (FTIR).

Water content

The toluene distillation method determined the water content of liquid smoke (AOAC international, 2019). A total of 3 grams of liquid smoke was weighed, and then added 50 ml of toluene was. Furthermore, it is heated using a distillation apparatus that has been connected to a reservoir of evaporated water (Stark-Dean). Heating is carried out until all the water in the solution evaporates, which is indicated by not adding water to the container (± 1 hour). The water content is calculated based on the percentage of the volume of water that evaporates by the weight of the liquid smoke solution. The moisture content of liquid smoke nanocapsules was measured using a moisture balance device (Ohaus MB 35 halogen, Switzerland) by drying a sample of at least 0.5 gram at a temperature of 105°C until it reached a constant weight. The percentage of water content will be printed on the device.

Total phenolic compounds

Analysis of total phenolic compounds was carried out by weighing 0.5 grams of nanoparticles or nanocapsules diluted with distilled water to 50 ml. 1 ml of the dilution is taken, diluted again to 10 ml so that the total dilution is 1000 times. The dilution results were taken at 1 ml and added with a solution of 5 ml of 2% alkaline Na_2CO_3 left for 10 minutes at room temperature. Then 0.5 ml of Folin Ciocalteau solution was added, vortexed, and left for 30 minutes. Next, the absorbance was measured at a wavelength of 750 nm. The phenol concentration of the sample solution was calculated based on the standard curve obtained from the pure phenol solution.

Staining of Phenolic Compounds

Staining of Phenolic Compounds (Badria and Aboelmaaty, (2019), with modification) was carried out by preparing 5% (w/v) FeCl3 solution and dripping it on 3 ml of nanoparticle solution and many liquid smoke nanocapsules then homogenized and placed on a glass slide. Furthermore, it was observed using an Olympus CX 21 light microscope, Olympus Corp Japan, with the help of optilab software at a magnification of 100x.

Color

Color testing is carried out based on the Hunter Color Notation System. This color measurement was carried out objectively using a chromameter. Three-parameter values will be generated from the color test, namely parameters L, a, and b. Hunter tristimulus data are L (brightness value), a (red or greenish), and b (yellowish or bluish).

Morphological profiles

Morphological profiles of liquid smoke nanocapsules were observed using a Scanning Electron Microscope (SEM FEI type Inspect S50, Oregon, USA). The nanocapsules were attached to SEM stubs or sample holders with a diameter of 10 mm using double-sided adhesive tape. Then the sample was coated with gold and viewed at a magnification of 1000 to 10,000 times at a voltage of 20 kV.

Functional group analysis

Functional group analysis (FTIR) uses the principle of absorbing functional groups using infrared absorption. Wavelength measurements were carried out using a Shimadzu Infra-Red Spectrometer.

RESULTS AND DISCUSSION

Determination of treatment

The primary material must be encapsulated and protected from environmental influences in the encapsulation process and required coating material. The main ingredient used in this research is liquid smoke grade 1, containing total phenol, carbonyl, and acid components. Each has an essential role in food product preservation, antioxidant, antibacterial, flavor enhancement, flavor, and aroma of smoked products. The ratio of the concentration of gum arabic and maltodextrin and the amount of liquid smoke used are presented in Table 1.

The manufacture of nanocapsules and the characterization of nanocapsules were only carried out on nanoparticles that had been optimized (concentration ratio of gum arabic and Maltodextrin 12:88, homogenization speed of 4000 Rpm for 3 minutes) (Utami, 2015) and compared with control nanocapsules (maltodextrin encapsulation only).

Table 1 Rasio of encapsulation concentration and amount of liquid smoke

Number test	Concentration ratio gum arabic: Maltodextrin	Liquid smoke (m)
1	8: 92	225
2	12 : 88	225
3	16 : 84	225
4	20 : 80	225
5	24 : 76	225
6	28 : 72	225
7	32 : 68	225
8	36 : 64	225
9	40 : 60	225

Note: the concentration rasio of gum arabic and maltodextrin is based on the total amount of encapsulant used

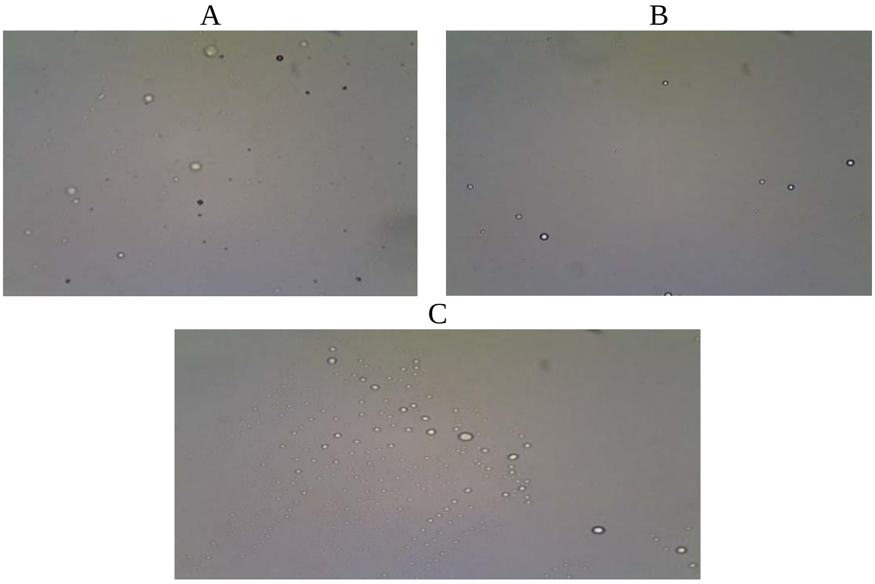

Figure 1 Observation of phenolic compounds in liquid smoke without coloring, magnification 400x (A) Nanoparticles of liquid smoke encapsulated by gum arabic only; (B) Maltodextrin encapsulated liquid smoke nanoparticles only; (C) Optimized nanoparticles

Phenolic Compound Staining

Liquid smoke appears as a clear yellowish liquid with a slightly pungent smoke-like aroma. Liquid smoke also has a phenolic aroma because it contains phenolic compounds with an aromatic ring due to lignin degradation. One of the qualitative methods to prove the presence of phenolic compounds and see whether the encapsulant encapsulates liquid smoke phenolic compounds is to react with ferric chloride ($FeCl_3$).

Observations using a light microscope with optilab software showed that the nanoparticles and nanocapsules of liquid smoke with the addition of $FeCl_3$ would give a brownish-red color. In contrast to the nanoparticles not added with $FeCl_3$, they would only appear as colorless transparent spheres. The brown color indicates a reaction between ferric chloride and phenolic compounds (guaiacol) contained in the liquid smoke. This result indicates that the liquid smoke phenolic compounds are encapsulated by gum arabic and maltodextrin. The coloring of liquid smoke using $FeCl_3$ follows the research of Badria dan Aboelmaaty (2019), namely phenolic compounds and $FeCl_3$ react to produce a brownish-red color.

Based on Figure 1 above, the liquid smoke nanoparticles with gum arabic encapsulation only, maltodextrin encapsulation only, and the optimized nanoparticles are in the form of colorless transparent spheres surrounded by purple-black circles. The purple-black circle is the encapsulation that surrounds the liquid smoke phenolic compounds. Based on observations, it also appears that the particles have a non-uniform spherical shape. This non-uniformity strengthens the results of the particle distribution index, which shows that the resulting nanoparticles have different sizes, which are also shown in the three peaks of the z-average analysis. (Utami, 2015).

Based on Figure 2, it can be seen that the nanoparticles and nanocapsules are red-brown. The red-brown color indicates the encapsulated or adsorbed phenolic compounds, while the coating material's purple-black circle is used. This result follows the statement of Yun et al. (2021) regarding the function of the coating material in the encapsulation system, namely being a barrier or barrier for active compounds and protecting from environmental influences.

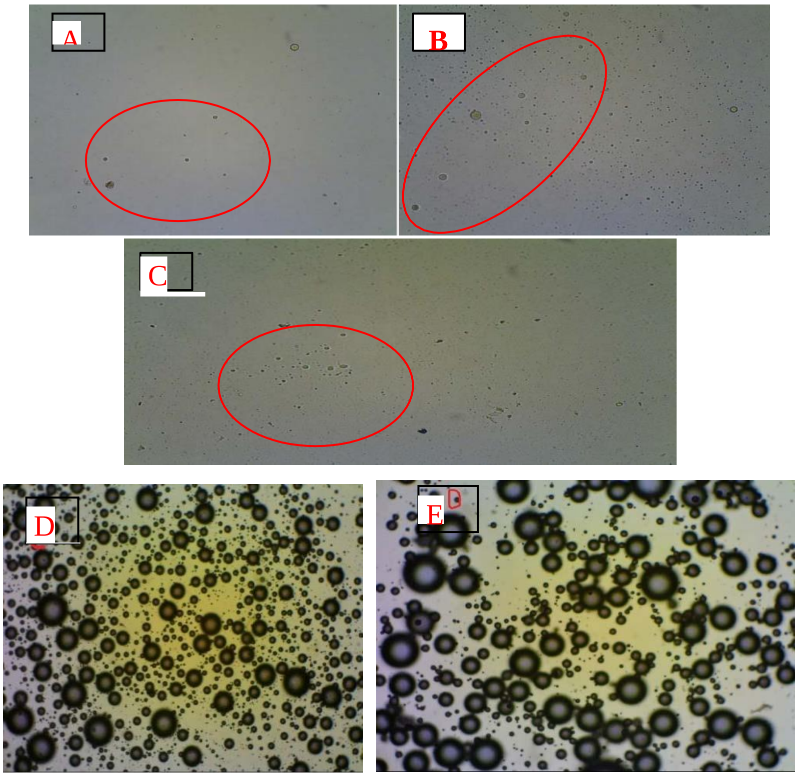

Figure 2 Staining of phenolic compounds with FeCl3, 400x magnification. (A)Liquid smoke nanoparticles gum arabic encapsulation only; (B) Maltodextrin encapsulated liquid smoke nanoparticles only; (C) Optimized nanoparticles; (D) Optimized nanocapsules (E) Maltodextrin encapsulated nanocapsules only

In liquid smoke nanoparticles using gum arabic and maltodextrin, it is suspected that gum arabic is near the core (in this case, a phenolic compound). It acts as a co-encapsulant, while the maltodextrin is in the outer layer. In contrast to encapsulation which only uses maltodextrin as an encapsulant, maltodextrin is located around the core (phenolic compound) so that the type of encapsulation is a matrix type. If the encapsulation type is described, it is included in the coated matrix type. An illustration of the encapsulation type is presented in Figure 2.

However, the purple-black color seen in the optilab observation (Figure 1 and Figure 2) has not been identified to distinguish the position of gum arabic and maltodextrin. As encapsulants, gum arabic and maltodextrin can trap phenolic compounds well because maltodextrin and gum arabic can form a matrix and bind to the O group of phenol through hydrogen bonds. According to Silva et al. (2013), the O group in phenol binds to the H group on the maltodextrin chain and gum arabic. The bond will form a particle/droplet with different sizes during homogenization. The gum arabic and Maltodextrin combination can also maintain the capsules' bioactive compounds or active substances.

Morphological Profile (SEM)

Observation of the morphological profile of nanocapsules was carried out using a Scanning Electron Microscope (SEM), which aims to see the shape and texture of the particles. This study made observations with SEM on optimized nanocapsules compared to control nanocapsules. Differences in the type of coating and the concentration of coating material will show different results in the morphological profile of the resulting nanocapsules, as shown in Figure 4.

Based on Figure 4, both the nanocapsules with maltodextrin coating and the optimized nanocapsules are spherical with creases on the surface. This result is related to the shrinkage process during the spray drying process due to rapid evaporation. Shrinkage also occurs due to the uneven swelling in the nanocapsules. According to Fu et al. (2021), if there is excessive evaporation, it can cause cracking of the membrane.

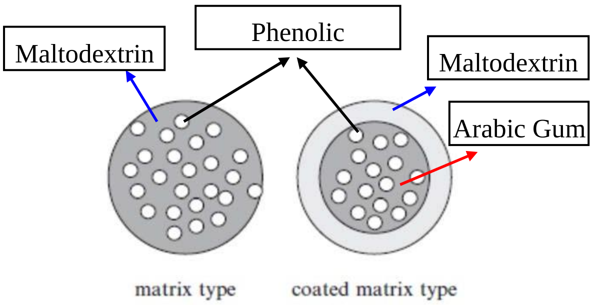

Figure 3 Illustration of encapsulation type

The cracks in the walls (shown by arrows) were only seen in nanocapsules with maltodextrin encapsulation (control). Cracks or cracks in the walls occur due to the brittleness and inability of the material to withstand pressure at high heating temperatures during the spray drying process. Moser et al. (2019)explained that the capsule walls could experience cracks and ruptures caused by the ballooning process, namely the bubble of the nanocapsule particles resulting from the formation of water vapor in it.

Cracking or rupture of the capsule wall can also be caused by high inlet temperatures, which usually causes the droplet surface to dry rapidly, resulting in excessive skin thickening or case hardening. These cracks can trigger the formation of an impermeable vapor layer, which is followed by the formation of a steam balloon and eventually causes the droplet to burst (Phisut, 2012). Cracks or breaks in the wall can trigger the release of the active ingredient, in this case, the total content of phenolic compounds. It was proved in this study that the total content of phenolic compounds in nanocapsules with maltodextrin coating alone (1.75 grams) was lower than the total phenolic compounds in optimized nanocapsules using a combination of Arabic gum coating and maltodextrin (2.98 grams). The total content of phenolic compounds in the optimized nanocapsules was higher because there were no cracks in the capsule walls. This increase indicates that gum arabic can strengthen the nanoparticle walls. The walls do not experience cracks during the spray drying process and can protect the active ingredient content. In addition, gum arabic is classified as a heat-resistant material to strengthen the capsule walls.

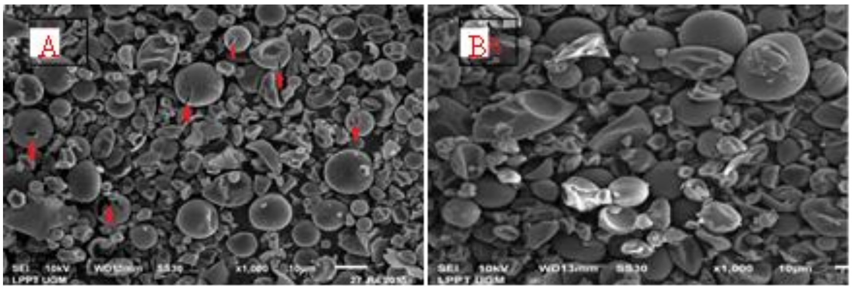

Figure 4 SEM Nanocapsule Morphology. (A) Control nanocapsules (maltodextrin encapsulation only) (1000x) (B) Optimized nanocapsules (1000x)

The use of gum arabic also causes an increase in molecular weight because gum arabic has a high molecular weight. According to Karrar et al. (2021), gum arabic with a high molecular weight causes gum arabic to have a high glass transition temperature (Tg). The resulting product is more compact during the spray drying process than products that use maltodextrin encapsulants alone. Based on observations of morphological profiles using SEM, it also appears that the optimized nanocapsules have various shapes, ranging from small to large (Fraj et al., 2019).

The results of observing the morphological profile of nanocapsules using a Scanning Electron Microscope (SEM) can only see the surface shape of the nanocapsules. The inside of the nanocapsule cannot be seen, so it is impossible to know whether the type of encapsulation is reservoir type (single-core) or matrix type (multi-core).

Therefore, it is necessary to observe the morphological profile using an Infrared Scanning Electron Microscope to find out the inside of the nanocapsule so that the type of encapsulation can be known.

Figure 5 Appearance of nanocapsules (A) Optimized nanocapsules (B) Control nanocapsules (maltodextrin encapsulation only)

Color

Color is crucial to analyze because it relates to consumer acceptance, interest, and product quality. The carbonyl component causes the color of liquid smoke. According to Rau and Glomb (2022), carbonyl compounds such as glycolaldehyde, metal glyoxal, and glyoxal indicate a browning potential. The higher the carbonyl compound, the higher the browning potential.

Testing of color characteristics is carried out objectively using a chromameter or photoelectric colorimeter, also known as a Hunter colorimeter. The Hunter color notation system is characterized by three parameters L, a, and b, each with a value ranging from 0 to ± 100. The L notation represents the brightness parameter (light). The parameter L has a value from 0 (black) to 100 (white). The L value represents the reflected light that produces achromatic white, gray, and black colors. The notation a indicates the mixed chromatic color of red-green if the value of +a (positive) is from 0 to ± 100 for red and the value of -a (negative) is from 0 to -80 for green. The notation b denotes the mixed chromatic color of blue-yellow, for -b (negative) values from 0 to -70 for blue (Kasajima, 2019).

From the analysis result, the optimized nanocapsule has a parameter value of L 85.17±1.13; a -0.45±0.11, and b 9.2±0.13. The L value of 85.17±1.13 indicates the achromatic color produced is white. A value of -0.45 ± 0.11 indicates a color that leads to green, while the value of b 9.2 ± 0.13 indicates a yellow color. Almost the same as the optimized nanocapsules, control nanocapsules had parameter values of L 84.78±0.22; a -0.58±0.03, and b 8.21±0.13. The L value of 84.78±0.22 indicates the achromatic color leads to white. The a value of -0.58±0.03 indicates the color leads to a green and the b value of 8.21±0.13 indicates the yellow color. If observed visually, the liquid smoke nanocapsules resulting from optimization and control have a yellowish-white appearance (ivory white). This encapsulation shows that it has a stable character against browning.

Total Phenolic Compounds and Recovery Efficiency of Phenolic Compounds in the Spray Drying Process

Phenolic compounds are one of the bioactive components in liquid smoke that act as antioxidants and antimicrobials. During making nanocapsules, especially during the spray drying process, phenolic compounds were damaged, which resulted in a decrease in the total content of phenolic compounds in the resulting product. To facilitate the understanding of the total phenolic compounds contained in the raw materials, nanoparticles, and nanocapsules of liquid smoke, they are presented in the form of material balance.

The calculation of the recovery efficiency of phenolic compounds in the spray drying process aims to determine how many were successfully sprayed in this study by comparing the total phenolic compounds in the nanocapsules (both inside and outside the encapsulation) with the total phenolic compounds in the nanoparticles. Thus, the level of effectiveness of the encapsulation process, in this case, is the effectiveness in the recovery of liquid smoke phenolic compounds in the spray drying process.

Based on the material balance in the optimized nanocapsules, the ratio of the total phenolic compounds after spray drying with the nanoparticles was 47.36%. Whereas in control nanocapsules (with maltodextrin coating only), the ratio of the total phenolic compounds after spray drying with the nanoparticles was 39.15%. This result shows a higher value.

Low compared to the optimized nanocapsules, which have a recovery efficiency of phenolic compounds in the spray drying process of 47.36%.

The decrease in the total content of phenolic compounds in nanocapsules was caused by heating during the spray drying process. It is suspected that low-boiling phenolic compounds were damaged due to easy evaporation during the spray drying process. According to Ahmad et al. (2019), phenolic compounds can react with carbonyl to reduce the total phenol content. In addition, the maltodextrin encapsulation used could not withstand the pressure at the heating temperature during the spray drying process. As a result, the capsule wall can crack and break due to the bubble event of the nanocapsule particles as a result of the formation of water vapor in it (Rezvankhah et al., 2020). During the spray drying process, The resulting sample or nanocapsule is attached to the wall of the spray dryer, thereby reducing the calculation of the total phenol content. In calculating total phenolic compounds, an analysis was carried out on the nanocapsules resulting from spray drying that were accommodated in the container, excluding the nanocapsules attached to the device wall.

Cracking or breaking of the coating wall impacts the release of the active ingredients, in this case, phenolic compounds. However, the combination of gum arabic as an encapsulant can provide better recovery efficiency of phenolic compounds in the spray drying process compared to maltodextrin alone, and is because gum arabic has a high molecular weight and can form a layer around the active substance, protecting it from oxidation, and prevent air absorption. Gum arabic has better stability(Navarro-Flores et al., 2020).

This study did not know what phenolic compounds were damaged during the spray drying process. Therefore, it is necessary to identify any volatile compounds in liquid smoke, nanoparticles, and liquid smoke nanocapsules using gas chromatography (GC) to determine which phenolic compounds were damaged during encapsulation.

In this study, the efficiency of recovery of phenolic compounds in the spray drying process on optimized nanocapsules was low because it was below 50%. It is due to the small concentration of gum arabic with maltodextrin used, namely gum arabic 12% while Maltodextrin 88% of the total encapsulant used. The low amount of gum arabic used impacts the fragility of the nanocapsule walls so that phenolic compounds are easily damaged. However, on the other hand, if large amounts of Arabic are used, or the comparison with maltodextrin is significant, the resulting particle size distribution (z-average) will be significant as well. Therefore,

Functional group analysis

Each material is composed of constituent chemical bonds involving functional groups. By testing using the Fourier Transform Infrared Spectrophotometer (FTIR), we can be seen the chemical bonds or functional groups that make up and divide the range of absorption wavenumbers. The functional groups of the optimized and control nanocapsules (with maltodextrin encapsulation only) are presented in Figure 6.

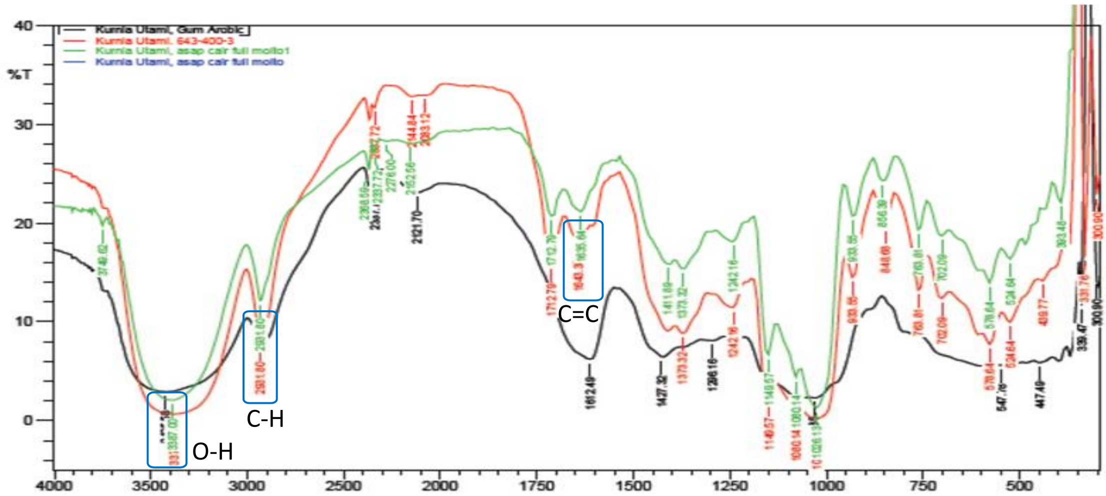

Figure 6 FTIR spectra of optimized nanocapsule (red line) and control nanocapsule (green line)

From the infrared spectrum in the functional group spectrum, it appears that the optimized nanocapsules and control nanocapsules contain OH, CH, and C=C bonds, respectively. The band indicates the CH bond. According to Nobari Azar et al. (2021), in research on cocoa polyphenols, the band indicated the presence of OH bonds at a wavenumber of 3200-3500 cm^{-1}.

At the wavenumber between 2960-2850 cm^{-1}, the C=C bond is indicated by the band at the wave number 600-1680 cm^{-1}.

The results indicate that both the optimized nanocapsules and the control nanocapsules contain OH bonds, which indicates that there are phenolic compounds in the liquid smoke that can be encapsulated or adsorbed by gum arabic and maltodextrin encapsulants. However, each product's percentage of transmittance (%T) and peak width showed different results. Based on Figure 6, it can be seen that the number of OH bonds in the optimized nanocapsule was higher than that of the control nanocapsules, which was indicated by a smaller transmittance percentage with a broader peak. This difference follows the calculation results of the total phenolic compounds in the optimized nanocapsules having higher values than the control.

The C=C bond in the optimized nanocapsule showed a lower transmittance percentage (%T) than in the control nanocapsule; in other words, the number of C=C bonds in the optimized nanocapsule was higher than in control. The decrease in the C=C bond was more common than in the control nanocapsules. According to Guengerich and Yoshimoto (2018), the decrease in the C=C bond results from oxidation by taking H atoms, resulting in the loss of the C=C bond. Nanocapsules with maltodextrin encapsulation alone (control) have no better ability than nanocapsules with Arabic gum encapsulation to protect against damage (oxidation).

CONCLUSION

The optimized nanocapsules had round and wrinkled morphology, and there were no cracks or breaks in the capsule walls. The optimization process can produce nanometer-dimensional particles. The use of maltodextrin combined with gum arabic as a co-encapsulant can increase the recovery efficiency of phenolic compounds in the spray drying process. The resulting efficiency is 47.36%.

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