Nano-Emulsion and Nano-Encapsulation of Fruit Flavor: Review

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Abstract. Nano-emulsion and nano-encapsulation of fruit flavor aim to make flavor stabilization which is generally labile in the form of liquid (emulsion) and powder (encapsulation) preparations. Nanotechnology in food is a technology to reduce food particle size to 20-200 nm which is generally followed by new properties both physical, chemical and sensory. In nano-emulsion products such as transparent color and products stability are characterized by: absence of sedimentation (creaming), unification of two particles (flocculation), unification of particles to enlarge (coalescent) and exchange of internal and external phases (inversion). In nano-encapsulation products flavor stability is maintained by reduced aroma volatility which is bound by the carrier matrix while controlling the release of aroma. The method of forming nano-emulsion uses two approaches, namely high energy such as high-pressure homogenization and ultra-sonication methods, and low energy such as spontaneous emulsification. Making an emulsion is by mixing two different compounds of polarity stabilized by the emulsifier. In the emulsion flavor system for making nano-encapsulation a biopolymer is needed which acts as a core coating. The encapsulation was made by making an emulsion first, then one of the drying techniques was carried out such as spray drying, drum drying and freeze drying. This literature review describes nano-emulsion techniques and nano-encapsulation of fruit flavor.

Keywords: nano-emulsion, nano-encapsulation, fruit flavor, emulsifier, coating matrix

1. Introduction

1.1 Flavor Nano-Emulsion

Fruits have the preferred flavor, where different types of flavoring components for each different type of fruit. ‘Kawista’ fruit, for example, which is classified as a tropical plant, identified using Gas Chromatography-Mass Spectrometry (GC-MS) has 75 flavor components as volatile compounds, which consist of esters, alcohols, aldehydes, ketones, lactones, heterocyclic aromatic, hydrocarbons, acetal, furan and carboxylic acids [1]. Flavor compounds consist mostly of volatile components so that they are labile by environmental conditions. Emulsion and encapsulation of flavor is one way to maintain flavor stability.

Nanotechnology can be applied in food processing technology in the form of nano-emulsion and nano-encapsulation. The definition of nanotechnology in food processing is the manufacture of food materials that are nano-scale, i.e. 20-200 nm. Nano-sized material designed in the order of the smallest atom or molecule, has special properties compared to the origin of the material.

Nano-emulsion is often referred to as mini-emulsion, nano-emulsion, ultrafine emulsion, and submicron emulsion. Nano-emulsion looks transparent and translucent which is seen with ordinary eyes and...
does not occur sedimentation (creaming) due to Brownian. One other hand, it is also resistant to flocculation because efficiently of steric stability. The unique properties of the nano-emulsion can be categorized as nano-emulsion having thermodynamic stability. Most nano-emulsions are stable by synthetic surfactants which have long hydrophilic groups [2].

Emulsions consist of conventional emulsion (O/W and W/O), multiple emulsions (W/O/W), multilayer emulsions (MO/W), solid lipid particles (SLP-O/W), and filled hydrogel particles (O/W/W). Double emulsion of water in oil in water (W/O/W), where small water droplets contained in larger oil droplets, will then be dispersed in a continuous water phase [3].

![Figure 1](image)

**Figure 1.** Emulsions type: Nano-emulsion (A), Lipid droplets (B), Filled Lipid droplets (C), solid lipid nanoparticle (D), Multilayer emulsions (E), colloidomes (F)

Compounds in the form of nano-emulsion have stability to environmental influences. A stable material is still within acceptable limits during the period of storage and use, which has the same characteristics which are made. Physical instability of the material is characterized by the bleaching of colors or the appearance of colors, odor arising, changes or separation of phases, rupture of the emulsion, deposition of suspensions or caking, changes in consistency, crystal growth, gas formation and other physical changes. Whereas, the stability of nano-emulsion is influenced by changes in physics and chemistry. Indicator that becomes of the instability of nano-emulsions includes creaming, flocculation, coalescent and ostwald ripening, and inversion.
Creaming is the emulsion phase separation based on the difference in density between the dispersed phase and the continuous phase. Creaming is an undesirable process, but this condition can be dispersed again with agitation. Creaming speed is influenced by several factors, namely based on the depositional velocity theory stated by the Stokes law [5]. Flocculation is the incorporation of globules depending on the electrostatic force (potential Zeta). This instability can still be improved by shuffling because the film between the surfaces is still there [6].

Coalescent is the process by which the droplets of a phased approach and combine to form larger particles and become a layer. This happens not only because of the surface free energy but also because not all globules are coated by interface films [5]. This instability is more damage than creaming. Attempts to stabilize this instability cannot be done by shuffling, usually additional emulsifiers and reprocessing are needed [7].

Ostwald Ripening is the process by which small droplets turn large and form new droplets. This phenomenon is related to systems that have varying droplet sizes. The phenomenon of coalescence and Ostwald Ripening causes the separation of the system into three phases, namely the internal, external and emulsifier phases. Inversion is an event where the external phase becomes an internal phase, and vice versa [5].

There are two methods for forming nano-emulsions, namely the formation of using high energy and low energy approach. High energy formation uses mechanical equipment capable of breaking oil and water phases such as high-pressure homogenizers, micro-fluidizers, and sonication methods. The formation of low energy nano-emulsion is produced as a result of phase transitions during the emulsification process where there is a change in composition with constant temperature. The method often used is membrane emulsification, spontaneous emulsification, emulsion inversion point, phase inversion point, solvent displacement [8].

Emulsion formation can be done directly and indirectly, where the quality of the emulsion is better in indirect emulsions [9]. The emulsion technique directly by mixing becomes one of all ingredient emulsions, while the indirect emulsion is carried out in two stages, namely pre-emulsion and emulsion. The minimum grain size that can be produced using each approach depends on several different factors. Reducing particle size using a high-energy approach depends on the type and operating conditions of the homogenizer (eg energy intensity, time and temperature), sample composition (eg oil type and concentration and emulsifier) and physicochemical properties of component phases (eg interface voltage and viscosity).
Making fruit flavor nano-emulsion is expected to improve its color characteristics, particle size, viscosity, pH, solubility and availability so that it will increase acceptance by consumers. The combination of emulsifying materials and stabilizing fruit nano-emulsions will produce smaller emulsion grain sizes. Malto-dextrin or inulin as a biopolymer is added as a thickener which aims to increase viscosity and slow down the deposition process, so that the nano-emulsion produced will be more stable.

| Fruits (Flavor) | Emulsion type and emulsifier | Method | Parameters | References |
|----------------|-----------------------------|--------|------------|------------|
| Orange (D-limonene) | O/W. Sorbitan trioleate and poly oxy-ethylene (20) oleyl ether (HLB 1.8 and 15.0), co-surfactant Ethylene glycol | Emulsification two steps, homogenization and sonication 20 kHz | Droplet size, morphology (TEM) stability 14 weeks room temperature, electrophoretic (zeta potential), | [10] |
| Grape berries | W/O continuous phase lemongrass oil. Tween 80 | Vortex and homogenizer ultra-thorax 10000 rpm continued dynamic high pressure (5000 rpm) | Droplet size and stability 7 days at room temperature. Sensory analysis. Anti-microbial (disk), Phenol total, anthocyanin, antioxidant activity, degree color | [11] |
| Medium-chain triglyceride (MCT) | O/W. Gelatin. Tween 80 | Spontaneous homogenization (magnetic stirrer 20°C and 60°C) | Droplet size (nano zetazier ZS), turbidity, degree color, stability, rheology | [12] |
| ‘Temulawak’ extract | O/W. Malto-dextrin. Tween 80 | Homogenization 24000 rpm (High-Speed Homogenizer - Virtis 23) | Curcumin Bio-availability, solubility, stability, index bias, pH, viscosity, droplet size | [13] |
| Zingerrol extract | O/W. Tween 80 | Low-temperature homogenization 22000 rpm | Droplet size, stability, bioactive compound, solubility, bioviability | [14] |

1.2 Flavor Nano-encapsulation

Encapsulation technology is able to convert scents into stable compounds in the form of liquids, pastes, powders, granules, capsules, microcapsules or nano-capsules. The form of encapsulation of the aroma will be able to maintain, protect, reduce the power of volatility, stabilize, and control the release of aroma. Variations in components that can be used as encapsulation matrices include starch, gelatin, or cyclo-dextrin [15].

Encapsulation is a technique of trapping food components with a polymer coating, forming round nano-capsules measuring between tens of nano to several microns. Encapsulation of flavor in nano-capsules can be released at controlled speeds under certain conditions. Nano or micro-encapsulation techniques that are widely used commercially are spray drying, air suspension coating, accreditation, spray cooling and spray chilling, centrifugal extrusion, rotational suspension separation and inclusion complexion. Spray drying or spray dryer is a widely used encapsulation technique [16].
In general, flavor encapsulation techniques can be carried out chemically such as conservation, co-crystallization, molecular complex, and interfacial polymerization; physically such as Spray drying, spray chilling / cooling, extrusion, and fluidized beds [17, 18]. The advantages and disadvantages of each flavor encapsulation technique are presented in Table 2.

**Table 2. Advantages and disadvantages of flavor encapsulation method [19].**

| Encapsulation method | Advantages | Disadvantages |
|----------------------|------------|---------------|
| Conservation         | Encapsulation efficiency of 40-90%, Using organic solvents, Applied to heat sensitive components | Expensive, complex mechanisms (advance technology), sensitive to the environment, the presence of toxic compounds such as glutaraldehyde |
| Co-Crystallization   | the method of entrapping (using sugar), the orange peel is quite good, but it is recommended to add antioxidants | Flavor sensitivity is quite high, rarely used |
| Spray drying         | Encapsulation efficiency of 10-90%, Easy, low cost, high reproducibility, high productivity | Not suitable for heat sensitive components, it is difficult to control particle size |
| Spray cooling/spray chilling | Encapsulation efficiency 10-100%, The cost is cheaper than spray drying, suitable for heat sensitive components | It is not easy to control particle size, Moderate yield on small scale production |
| Freeze drying        | The quality of flavor is better than hot drying | Expensive production costs, expensive handling and storage in the long period |
| Fluidized bed coating | Encapsulation efficiency 5-50%, Can control particle size, low cost, can dry encapsulate multi-layer | Heat temperature will integrate the flavor component |
| Extrusion            | Encapsulation efficiency of 20-50%, Can be used in aerobic and un-aerobic conditions | Difficult in increasing scale, It is difficult for high viscosity polymers |

Encapsulation makes flavor protected from adverse environmental influences such as damage due to oxidation, hydrolysis, evaporation or heat degradation. Thus, flavor will have a longer shelf life and better process stability. In addition, the release of material from the capsule can also be controlled so that its effectiveness can be designed as desired. The challenge of the application of nano-encapsulation technology lies in the selection of the nano-encapsulation technique and the appropriate encapsulating material (coating wall) so that the capsule can function according to its purpose.

Some reasons for implementing nano-encapsulation in the food industry are: to reduce core reactivity with environmental factors, reduce the transfer rate of core materials with the outside environment, to promote easier handling, to control the release of core material, to cover the core taste, and melt core material when
must be used in small quantities [20]. In its simplest form, nano-capsules are small balls with uniform walls around them. The material in nano-capsules is called the core, internal phase, or filler, while the wall is also called shell, layer, material wall, or membrane. Nano-capsule can have several coating walls [21].

Nano-capsule coatings can use carbohydrate ingredients such as dextrin, sugar or starch. Other ingredients can also come from proteins such as gelatin and soy protein. While one method commonly used in industry is using a spray dryer. Coating materials commonly used for the spray drying process include carbohydrates, Arabic gum, semi-synthesized cellulose derivatives and synthesis polymers [22]. Each coating material (carrier / coating) has advantages and disadvantages to the properties, price and encapsulation efficiency. At present malto-dextrin, a modified starch derivative that can be dissolved, used individually or in combination with other ingredients in the process of making food and medicines from plant extracts, aromatic preservatives and vitamins [23].

Malto-dextrin has various aspects of function including enlargement and the properties of film formation, the ability to bind taste and fat, and reduce the permeability of oxygen to the wall matrix. Some of the reasons underlying malto-dextrin for microencapsulation materials, according to Aakash [24] that malto-dextrin can reduce the reactivity of core material with an environment, controlled release which is suitable for core material. Some microencapsulation studies using malto-dextrin as coating wall material have been carried out including the microencapsulation process of ginger oleoresin [25]. The highest microencapsulation efficiency of ginger oleoresin with a ratio of 1: 16.7 (oleoresin: malto-dextrin) with particle sizes of 1.05-12.90 µm.

| Material          | Encapsulation method               | Wall matrix                        | parameter                                                                 | References |
|-------------------|------------------------------------|------------------------------------|---------------------------------------------------------------------------|------------|
| Vanillin extract  | Spray drying                       | Malto-dextrin, modified tapioca    | Yield, vanilla content, vanillin recovery, Aw, Solubility, Morphology (SEM) | [26]       |
| Flavors (d-limonene, ethyl hexanoate, citral and ethyl propionate) | Spray drying | Saccharomyces cerevisiae | Component of flavor (GC-MS), encapsulation efficiency, particle size, moisture content, morphology (SEM) | [27]       |
| Red pepper        | coacervation (ionic gelation system) | Alginate substituted with Tapioca is photooxidated | yield, color, particle size, water content, texture, capcaicin content, encapsulation efficiency and loading capacity | [28]       |
| Temulawak extract | Preparation by ultra-sonication, spray drying | TPP and chitosan | The yield, crystallinity (XRD), a complex group (FTIR), size and morphology (SEM) | [29]       |
| PUFA (Sunflower)  | Preparation by 2-stage homogenization and continued | Malto-dextrin and agave inulin | Particle size, viscosity, % surface oil, |
The application of the spray drying process to microencapsulation involves three basic steps [30], namely preparation of dispersions or emulsions for processing, homogenization of dispersions, and atomization of masses into the drying chamber. The first step is the formation of a good and stable emulsion from the core material in a solution of the wall. The dispersion must be heated and homogenized, with or without the addition of emulsifiers depending on the emulsifying properties of the coating material because some have their own interfacial activity. Before the spray drying step, the emulsion formed must be stable for a certain period of time, oil droplets must be rather small and the viscosity must be low enough to prevent the entry of air in the particles [31].

In food products that do not heat stable such as flavor, the use of spray drying techniques needs to be considered, considering the drying operating temperature can reach 180°C. In flavor products the freeze-drying technique in the encapsulation process is an alternative. The advantages of freeze-drying techniques are that they can produce higher quality products because the products produced have a rigid structure due to the sublimation process. Rigid structures make the product porous and do not contract at dry conditions. Other advantages reduce the degradation of substances that are not heat resistant [32, 18].

2. Emulsifier
Emulsifiers and surfactants are two terms that have the same meaning. An emulsifier is an active ingredient at the interface between two phases, a hydrophilic and hydrophobic phase. Emulsifiers have two groups in one molecule, namely polar groups which remember polar compounds such as water and non-polar groups which remember non-polar compounds such as oil. The unique nature of this emulsifier will be able to reduce the surface tension of two compounds that cannot mix together to be able to mix.

The use of excessive emulsifiers will cause low solubility so that emulsifying crystals are formed. The formation of these crystals will reduce their ability to form emulsions between fat and water. Imperfect emulsion formation will cause fat to separate during the processing so that the resulting fat content decreases [33].

The HLB (hydrophile-lipophile balance) value of an emulsifier is a number that shows a measure of balance and strain of a hydrophilic group (like water or polar) and a lipophilic group (like oil or non-polar), which is an emulsified two-phase system. HLB numbers are used to characterize emulsifiers. If the HLB emulsifier number is high, the emulsifier will be more soluble in water, whereas if the HLB number is low, the emulsifier will dissolve easily in oil.

| Nilai HLB | Penggunaan       |
|-----------|-----------------|
| 1-3       | Anti foaming agent |
| 3-6       | W/O Emulgator    |
| 7-9       | wetting agent    |
| 8-18      | O/W Emulgator    |
| 13-15     | Detergent        |
| 10-18     | solubilizing agent |
The W/O/W emulsion consisting of W/O and O/W emulsions requires two emulsifiers to form two systems, which must contain a low HLB (Hydrophilic Lipophilic Balance) value to stabilize the W/O emulsion and one must contain an HLB value high to stabilize the O/W emulsion.

| Kind of Emulsifier | HLB  |
|--------------------|------|
| Tween 20           | 16.7 |
| Tween 40           | 15.6 |
| Tween 80           | 15.0 |
| Tween 60           | 14.9 |
| Tween 85           | 11.0 |
| Tween 65           | 10.5 |

Tween 20 is a non-ionic hydrophilic surfactant which functions as an emulsifier, solvent, wetting agent. Tween 20 is widely used as an emulsifier in the preparation of oil-in-water emulsions. Tween 20 has a distinctive and warm odor, tastes rather bitter, yellow. Tween 20 is soluble in ethanol and water, and is insoluble in mineral oil and vegetable oil. Tween 20 must be stored in a tightly closed container, protected from light in a cool, dry place. Tween 80 emulsifier (polysorbate 80) in an oil in water (O/W) emulsion has the chemical name poly-oxyethylene 20-sorbitan mono-oleate with formula formula C\textsubscript{64}H\textsubscript{124}O\textsubscript{26}. Tween 80 has a distinctive and warm odor, tastes rather bitter, yellow. Tween 80 is soluble in ethanol and water and is insoluble in mineral oil and vegetable oil. Tween 80 is a group of nonionic surfactants wherein the ingredients are hexanhydrous alcohols, alkaline oxide and fatty acid hydrophilic properties are given by the oxyethylene free hydroxyl group [35].

| Kind of Emulsifier | HLB  |
|--------------------|------|
| Span 20            | 8.6  |
| Span 60            | 4.7  |
| Span 80            | 4.3  |
| Arlacel 83         | 3.7  |
| Gom                | 8.0  |
| Trietanolamine     | 12.0 |

| Dispersibility                        | Range of HLB |
|---------------------------------------|--------------|
| Not dispersed                         | 1-4          |
| A little dispersed                    | 3-6          |
| Dispersed like milk with gouging      | 6-8          |
| Dispersed like milk with stable conditions | 8-10        |
| Dispersed combines a translucent solution to clear | 10-13     |
| Dispersed into a clear solution       | 13+          |

Surfactants are divided into four groups i.e. anionic surfactants, cationic surfactants, nonionic surfactants, and amphoteric surfactants [33]. Anionic surfactant is a type of surfactant that has a negatively charged molecule in the hydrophilic part due to the presence of a very large ionic group. Anionic surfactants are surfactants whose alkyl parts are bound to an anion such as alkane sulfonate salt, olefin sulfonate salt, a long chain fatty acid sulfonate salt.

Cationic surfactant is a type of positively charged surfactant in its hydrophilic group due to the presence of ammonium salts, such as Quaternary Ammonium Salt (QUAT). Cationic surfactants are surfactants in which the alkyl portion is bound to a cation, such as alkyl trimethyl ammonium, dialcyl-dimethyl ammonium salt and dimethyl benzyl ammonium alkyl salt.

Nonionic surfactant is a type of surfactant that is not charged or does not occur molecular ionization due to the presence of an ether or hydroxyl oxygen group. Nonionic surfactants are surfactants which are not charged alcylic parts, such as fatty acid glycerin esters, fatty acid sorbitan esters, fatty acid sucrose esters, polyethylene alkyl amines, glucamines, alkyl poly-glucosides, mono alkanol amines, dialkanol amines and alkyl amine oxides.
Amphoteric surfactant is a type of surfactant that is positively and negatively charged on its molecule, where the charge depends on pH, at a low pH it will be negatively charged and at a high pH positively charged. For example surfactants that contain amino acids, betaine, phosphobetain.

3. Emulsion stabilizer
In the flavor emulsion system a biopolymer is needed as a core coating. The encapsulation process begins with making the emulsion first, then drying it. In microencapsulation synthetic flavor to reduce flavor loss and improve stability using biopolymers from native starch and starch modified succinylated and octenyl succinylated. Corn and barley succinylated starch is more effective than native starch and oktenyl starch. Succinylated starch shows the ability of flavor resistance compared to β-cyclodextrin which is widely used as a Coating material in microencapsulation of essential oils or flavor Succinate starch shows the ability of flavor resistance compared to β-cyclodextrin which is widely used as a coating material in essential microencapsulation of oil or flavor [36].
Microencapsulation of cumin extract with modified starch as a coating is more tolerant of heat than those using β-cyclodextrin [37]. During heating, β-cyclodextrin protects volatile compounds from evaporation to 100°C while modified starch to 140°C. Protection of volatile compounds from heat by maltodextrin depends on encapsulated material (160°C for limonene and 120°C for carvone).

**Figure 3.** Polymer coatings in nano-encapsulation [19]

**Table 7.** Types of coating matrices and their characteristics in flavor encapsulation [17]

| Wall Matrix       | Characteristics                                                                 | Products                               |
|-------------------|--------------------------------------------------------------------------------|----------------------------------------|
| Carbohydrates     | Low cost and good retention                                                    | Tuna oil; liquid tomato flavor         |
| Malto-dextrin     |                                                                                  |                                        |
| Porous starches   | Effective absorption and sustained release, simple production technology, and low cost | Coffee oil; limonene                   |
| Hydrolyzed starches| Excellent oxygen-barrier, low viscosity at high solid level                     | Meat flavor; wasabi flavor             |
| Protein           | Excellent functional and nutraceutical properties                               | β-Pinene; citronella                   |
| Milk proteins     |                                                                                  |                                        |
| Gelatin           | Early formation of surface crust, high emulsifying and stabilizing activities    | Lime oil; lavender oil                 |
| Arabic gum        | Excellent emulsifying properties and provides good volatile retention           | Rosemary oil; mint oil                 |
| Cyclo-dextrin     | Good resistance to oxidation, excellent inclusion of volatiles                  | Shiitake flavor; pine flavor           |
| Liposomes         | Lipid bilayer that increases emulsifying and emollient effects                  | Resveratrol; sesame                    |
| Inulin            | Prebiotic effects, dietary fiber action, and good solubility in water           | Rosemary oil; olive oil                |
Microencapsulation of the aroma components of d-limonene, ethyl hexanoate, octanal and 1-hexano used native corn starch, acetylation modified corn starch and pre-gelatinization and maltodextrin. Malto-dextrin is the most efficient coating material for flavor retention [38]. Gum Arabic is very effective as a coating material because it can protect colloids well [39]. Gum Arabic can stabilize emulsions in a wide pH range. Gum Arabic can be used in conjunction with other gum, carbohydrates and proteins.

4. Method of Nano-emulsion and nano-encapsulation

4.1 Procedure of Fruit Flavor Nano-emulsion

Making nano-emulsion refer to Sanchez with modification [9]. The ingredients used are fruit flavor extract, fruit flavor standard (Maltol, furaneol, benzyl acetate, hexyl acetate, Cis-3- Hexenol, trans-2-hexenal), citral, lime, C8 aldehyde, acetyl pyridine), internal standard (D4 Pyrazin and 1,4-dichlorobenzene), nitrogen gas, Tween 20, Pure water, whey protein concentrate, Malto-dextrin, β-cyclodextrin, Anhydrous Na₂SO₄, Antimicrobial compounds, Acidity regulators.

4.1.1 Procedure of micro-emulsions

Micro-emulsions O/W are made by high-pressure homogenization techniques. Mixing of fruit flavor extract, Tween 20, and pure water with 16000 rpm homogenizer (Ultra-Turrax T25, IKA, FR) for 3 minutes, resulting in an emulsion of 30% w/w of oil in water.

4.1.2 Procedure of biopolymer solution

1. Prepare a solution of whey protein concentrate, β-cyclodextrin, Malto-dextrin and third mixture according to the formula with pure water.
2. Add 0.02% sodium azide as an antimicrobial agent.
3. Stirring for 30 minutes using a magnetic stirrer at room temperature.
4. Standardize the solution at pH 6.0 using 0.1 M HCl then stored at room temperature.

4.1.3 Procedure of double W/O/W emulsions

W/O/W double emulsion is made gradually by adding O/W micro-emulsion into the aqueous biopolymer phase with high-pressure homogenizer with different pressures of 35, 50 and 300 bar (high-pressure valve homogenizer - 1001 L PANDA, Soavi NIRO, FR) at 16000 rpm for 5 minutes at 10°C, then emulsify again with homogenizer 15000 rpm for 8 minutes at 10°C.

![Figure 4. Procedure of Flavor nano-emulsion Fruit extract [9]](image-url)
4.2 Procedure of Flavor Fruit Nano-encapsulation
To manufacture fruit flavor nano-encapsulation using spray drying. Encapsulation of double emulsions into powders uses a spray drier (Model SP1500, Fanyuan Instrument Co., Shanghai, China) at a pressure of 2.5 bar, inlet temperature 140-150°C, outlet temperature 70-80°C, and average speed of 450 mL/h. Powder stored in closed bottles was stored at 4°C for further analysis. The encapsulation method can be done by freeze-drying technique. The mechanism of the encapsulation freeze-drying in flavor includes freezing the emulsion, followed by drying by conditioning the ultra-high vacuum at 0.036 psi or about 0.0025 Bar so it will sublime to produce a solid product. Optimization of freeze-drying by setting the freezing temperature (-10°C to -40°C) and processing time (18-30 hours).

![Diagram of procedure of flavor nano-encapsulation](image)

Figure 5. Procedure of flavor nano-encapsulation Fruit extract

4.3 Characteristics of nano-emulsion and nano-encapsulation
4.3.1 Droplet size, Poly-dispersity Index and stability
The size of the droplets from the nano-emulsion is determined by photon correlation spectroscopy. Droplet measurements were carried out using the Zetasizer 1000 HS (Zetasizer Nano Zs, Malvern Instrument, Malvern, UK). The data obtained include nano-emulsion droplet and poly-dispersity index (PdI) sizes. The poly-dispersity index value (PdI) (0-1) provides information about the uniform size of an emulsion droplet. The smaller the poly-dispersity index value shows the droplet size distribution is getting narrower, which means the size of the droplet diameter is increasingly homogeneous. The stability of nano-emulsion size was observed in three storage temperature conditions, then the size and dispersion of size measurements were carried out using the PSA at the time of observation, which was day 0 to ... i.

4.3.2 Viscosity
The nature of viscosity and flow of liquid food products can be measured using an instrument called a viscometer. The nano-emulsion viscosity was measured by Brookfield viscometer (LVDV Pro II, Brookfield Engineering Laboratories, USA) with S34 spindles.

4.3.3 Color analysis
Nano-emulsions color evaluation using a spectrophotometer while observing nano-encapsulation surface color using the image processing system, the results of the shooting are analyzed by software (Image j 1.47v).
4.3.4 FTIR Analysis of Biopolymers
The structure of emulsifying biopolymers such as β-cyclo-dextrin and inulin was tested by Fourier transform infrared spectroscopy (FT-IR) using FT-IR spectrophotometer (Shimadzu, Japan) with reflection techniques. The spectrum is observed in transmissions from 400 to 450 nm. Samples are mixed with KBr powder and pressed into disk.

4.3.5 Scanning Electron Microscopy (SEM)
Nano-emulsion and nano-encapsulation flavor were placed on both sides of the plate then coated with a thin layer of gold. Particle morphology was observed through emissions emitted by scanning electron microscope (S-4160 Cold Field-Emission SEM, Hitachi, Tokyo, Japan) at 320 kV and photographed at a magnification of 6000 times.

4.3.6 Analysis of Transmission Electron Microscope (TEM)
Nano-encapsulation morphology was visualized using transmission electron microscope (TEM). The sample (50µl) was added to the 200 mesh copper-coated TEM holder sample (EM Sciences, Hatfield, PA, USA) after being given a negative staining then observed with JEOL electron microscope JSM-1200EX II (Peabody, MA, USA).

4.3.7 Measurement of encapsulation efficiency
Nano-encapsulation analysis of fruit flavor include total content and surface content. Encapsulation efficiency is calculated using formula

\[
\text{Encapsulation efficiency (\%)} = \frac{\text{Total extract of fruits flavor - extract free flavor in the supernatant}}{\text{Total extract of fruits flavor}}
\]

5. Conclusion
Nano-emulsion is a nano-sized disperse (<100nm) with two different of polarity stabilized by the emulsifier. Fruit flavors which are dominated by volatile compounds are labile so they need to be stabilized in the emulsion system. The characteristics of nano-emulsion are very good for maintaining physical and chemical stability such as being transparent and preventing the separation of the emulsion phase. The formulation of the dispersing phase composition, dispersed phase, type and quantity of emulsifiers, homogenization process conditions greatly influenced the physical and chemical characteristics of fruit nano-emulsion flavor. Nano-encapsulation is the binding of core compounds by coating both bio and synthetic polymer, from the emulsion system formed and then dried to obtain nano-sized powder (<200nm). The formulation of the dispersing phase composition, dispersed phase, type and number of emulsifiers, polymer group coating material, homogenization process conditions, and type of drying technique greatly influenced the physical and chemical characteristics of the nano-encapsulation of fruit flavor.

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References
[1] Apriyantono A dan Kumara B. 2004. Identifikasi Character Impact Odorants Buah Kawista (Feronia limonia). Jurnal Teknologi dan Industri Pangan, Vol. XV, No.1 Th. 2004
[2] Wooster T. J., Golding M., Sanguansri P. 2008. Impact of Oil Type on Nano-emulsion Formation and Ostwald Ripening Stability. Langmuir 24: 12758-12765.
[3] McClements DJ. 2004. Food Emulsion Principles, Practices, and Techniques. New York: CRC Pr.
[4] Gupta A, Eral HB, Hattona TA and Doyle PS. 2016. Nanoemulsions: formation, properties and applications. The Royal Society of Chemistry 2016 DOI: 10.1039/c5sm02958a
[5] Abdulkarim, MF et al. 2010. Stability Studies of Nano-Cream Containing Piroxicam. International Journal of Drug Delivery 2, 333-339.
[6] Ansel HC. 1989. Pengantar Bentuk Sediaan Farmasi. Ibrahim F, penerjemah. Jakarta: UI-Press.Terjemahan dari Introduction to Pharmaceutical Dosage Forms.
[7] Sanchez MRH , Cuvelier ME, Turchiuli C. 2015. Design of liquid emulsions to structure spray dried particles. Journal of Food Engineering.
[8] Burgos N, Mellinas AC, García-Serna E., Jiménez A. 2017. Nano-encapsulation Of Flavor And Aromas In Food Packaging. http://dx.doi.org/10.1016/B978-0-12-809740-3.00007-6
[9] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[10] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[11] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[12] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[13] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[14] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[15] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[16] Shahidi F and Han XQ. 1993. Encapsulation of food ingredients. Critical Review in Food Science and Nutrition, 33, 501–547.
[23] Bae EK, dan Lee SJ. 2008. Microencapsulation of avocado oil by spray drying using whey protein and maltodextrin. *Journal of Microencapsulation, 25*(8): 549-560.

[24] Aakash P, Siddharth A, Kirtesh R. 2014. A Review on applications of Maltodextrin in Pharmaceutical Industry. *Department of pharmaceutical science and technology, page 67, volume 4, 67-74*

[25] Fatchul A N, Purnama D, Yudi P. 2014. Mikroenkapulasi olesrinsk ampas jahe dengan penyalut maltodekstrin. *Jurusan Teknologi Pangan Universitas Gajah Mada, vol.34, No.1.*

[26] Setyaningsih D, Rahmalia R, Sugiyono. 2010. Kajian Mikroenkapulasi ekstrak vanili. *Jurnal Teknologi Industri Pertanian Vol 19(2) 64-70*

[27] Sultana A, Miyamoto A, Hy QL, Tanaka Y, Fushimi Y, Yoshii H. Microencapsulation of flavors by spray drying using Saccharomyces cerevisiae. *Journal of Food Engineering 199* (2017) 36-41

[28] Palupi N W, Pandu Khrisna Juang Setiadi, Sih Yuwanti. 2014. Enkapsulasi Cabai Merah dengan Teknik Coacervation Menggunakan Alginat yang disubstitusi dengan Tapioka Terfotooksidasi. *Jurnal Aplikasi Teknologi Pangan 3 (3) 2014*

[29] Sidqi T. 2011. Pembuatan dan Karakterisasi Nanopartikel Ekstrak Temulawak dengan Metode Ultrasonikasi. *Skripsi departemen biokimia MIPA IPB*

[30] Dziezak J D. 1988. Microencapsulation and encapsulated ingredients. Food Technology (April), 136–151

[31] Drusch S, Serfert Y, Heuvel AVD, & Schwarz K. 2006. Physicochemical characterization and oxidative stability of fish oil encapsulated in an amorphous matrix containing trehalose. *Food Research International, 39*,807–815.

[32] Eshtiaghi M, Stuture N, and Knoor D. 1994. High pressure and freezing pretreatment effect on drying, rehydration texture and colour of green beans, carrots and potatoes. *J. Food Sci. 59*(6): 1.168-1.170.

[33] Rossen, J. M. 2004. Surfactant and Interfacial Phenomena. Edisi ke-3. John Wiley & Sons, Inc. New York.

[34] Voight. 1995. Buku Pelajaran Teknologi Farmasi edisi 5. *Gajahmada university press. Yogjakarta*

[35] Wade, Ainley and Paul Wellen J. 1994. Hand Book of Pharmaceutical Excipients. Second Edition. Pharmaceutical Press. London

[36] Jeon YJ, Vasanthan T, Temelli F, Song BK. 2002. The suitability of barley and corn starches in their asli and chemically modified forms for volatile meat flavour encapsulation. *Food Research International 36 : 349–355*

[37] Partanen R, Ahro M, Hakala M, Kallo H, Forssell P. 2002. Microencapsulation of caraway extract in beta cyclodextrin and modified starches. *European Food Research and Tech. 214 (3) : 242-247.*

[38] Boutboul A, Giampaoli P, Feigenbaum P, Ducrue V. 2002. Influence of the nature and treatment of strach on aroma retention. *Carbohydrate Polymers 47 : 73-82.*

[39] Krishnan S, Bhosale R., Singhal RS. 2005. Microencapsulation of cardamom oleoresin: Evaluation of blends of gum arabic, maltodextrin and a modified starch as wall materials. *Carbohydrate Polymers 61 : 95–102*