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Encapsulating Agent Composite for NaFeEDTA Microencapsulation using Spray Drying Method

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Abstract

Iron microencapsulation is one of the solutions to protect iron from reacting with other substances that result in quality declines of fortified food products. The coating materials used should be appropriate for the specified encapsulation process. This study combined glucomannan and maltodextrin as coating materials and added HPMC as an additive to encapsulate NaFeEDTA. The results showed that such a combination could increase the encapsulation efficiency. The morphologies of the resulting microcapsules obtained from four formulations applied in this study were identical; numerous wrinkles appear on the particle's surfaces due to the presence of HPMC. All formulations had a narrow size distribution with an average particle size between 105 and 111 µm, thermal resistance up to a temperature of ± 200°C, and the same functional groups but with different intensities. The NaFeEDTA included in all formulations had a bioavailability value of 2.3 - 2.9%. The best formulation, based on the gastrointestinal digestion simulation, was shown by the formulation with a glucomannan:maltodextrin ratio of 2:3 (in 1% w/v coating material) with a release percentage of 64.28% in SGF solution and an increase of 2.27% in SIF solution. To prevent anemia, ± 400 mg of iron microcapsules resulting from the best formulation in this study are required to meet 75% of the daily intake.

Keywords: glucomannan; HPMC; maltodextrin; microencapsulation; NaFeEDTA

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INTRODUCTION

The World Health Organization (WHO) has declared anemia as a global health problem (Safiri *et al*., 2021). Anemia is defined as a decrease in hemoglobin concentration; the levels of erythrocytes or hematocrit below normal limits can result in a decrease in the oxygen-carrying capacity of the blood (Al-Attar *et al*., 2020). Some symptoms of anemia include fatigue, lethargy, and dizziness. Anemia can cause growth disorders and cognitive function, reduce body resistance, and slow down psychomotor development (Agustina *et al*., 2019). Its prevalence in the world reached 22.8% (1.74 billion people) in 2019 (Gardner & Kassebaum, 2021). Iron deficiency was claimed to be the most responsible cause of this figure (Gupta *et al*., 2015).

Iron plays an important role in the formation of hemoglobin, which functions to distribute oxygen throughout the body. Therefore, the absorption rate of iron into the body greatly affects the process of oxygen transfer (Gupta, 2014). To meet the daily need for iron to improve the nutritional quality of food and provide public health benefits with minimal risk, food fortification is considered the best approach (Saha & Roy, 2020) since it requires low costs, has long-term effectiveness, and is applicable to many types of food (Baldelli *et al*., 2022). Iron fortification can cause changes in the color, taste, texture, and aroma of foodstuffs so it may reduce consumer interest (Miller *et al*., 2022). Microencapsulation, in this case, can prevent contact between iron and other compounds in foodstuffs, thus preventing changes in food quality and bioavailability (Pratama, 2015).

Spray drying is the primary microencapsulation technique in which a liquid mixture containing particles is sprayed into a drying chamber. During this process, the particles dry and form spherical granules with adjustable morphology, size, and surface characteristics (Mardani *et al*., 2024). The stages generally include atomization of the liquid, droplet – hot air contact, evaporation of droplet water, separation and collection of the final product. When atomized droplets come into contact with hot air, a rapid equilibrium is established between the thermal vapor pressure and partial pressure of the liquid and gas phases. The temperature difference causes heat to flow from the air to the droplets, while the difference in partial water vapor pressure drives mass transfer in the form of water evaporation from the droplets (Pidalà *et al*., 2025). Microencapsulation using the spray drying process has many advantages, including the easy operation process, the possibility to perform scale-up in significant quantities, low water activity, low damage to heat-sensitive components, high production rates, and low operating costs (Pratap-Singh & Leiva, 2021).

The use of an appropriate coating material is one of the keys to the successfulness of a microencapsulation process (Mishra *et al*., 2013). Previous studies regarding the encapsulation of iron using gum arabic (Gupta *et al*., 2015), chitosan (Handayani *et al*., 2022), and glucomannan (Wardhani *et al*., 2020) faced tough operational challenge, which is mainly caused by their high viscosity, which required additional handling to ensure suitability of the microencapsulant solution viscosity with the operating conditions of the spray dryer. Glucomannan is an organic substance capable of forming a barrier between the internal and environmental elements, safe for consumption, easily degraded (Wardhani *et al*., 2020), and odorless when processed with other food ingredients (Anwar *et al*., 2016). It is also useful for improving digestive functions and the immune system, lowering cholesterol and blood sugar levels, and helping to lose weight (Urli *et al*., 2017). Another organic material that can also function as a good coating is maltodextrin, which can improve taste, replace fat, induce a mild sweet taste, increase glass transition temperatures, and reduce the hygroscopicity and tackiness of the microencapsulated products. The use of this organic sugar-based coating makes the excipients digestible at different times in the gastrointestinal system and reduces the effect of particular target compound deficiency (Baldelli *et al*., 2022). In addition, to increase iron absorption in the duodenum, mucoadhesive polymer compounds, such as HPMC (hydroxypropyl methylcellulose), can be employed. HPMC is used as an excipient in pharmaceutical preparations to increase the solubility of pharmaceutical active ingredients and to control the transfer of nutrients in the body (Zheng *et al*., 2021).

Based on research performed by Pandey et al. (2016), NaFeEDTA is a good type of iron to be used for fortification. The absorption of iron from NaFeEDTA is about two times higher than that observed for ferrous sulfate. NaFeEDTA is also stable during processing. At low pH, EDTA acts as a chelating agent, thus preventing iron from binding to phytic acid or phenolic compounds, which can inhibit iron absorption. Its addition enhances the absorption of dietary iron and soluble iron. Therefore, in this study, microencapsulation of iron with glucomannan and/or maltodextrin coatings was carried out for food fortification. The combination of two types of coating materials, namely glucomannan and maltodextrin is targeted to improve the physicochemical properties of the resulting microcapsules, specifically the thermal stability and resistance to oxidation. HPMC can also be a good additional encapsulating agent because it functions to control the delivery of iron into the body. The resulting microencapsulated iron can be used as an ingredient for food fortification, thus can be expected to lower the prevalence of anemia cases.

MATERIAL AND METHODS

Material

NaFeEDTA (in the form of a dietary supplement FeRRIZ, Nicholas, Jakarta, Indonesia), konjac glucomannan (100%, Now Foods, Blooming-dale, IL, USA), cellulase enzymes, maltodextrin, and HPMC (Sigma Aldrich, St. Louis, MO, USA), and other supporting reagents of pro-analytical quality.

Method

Preparation of Iron Microparticles

One hundred milliliters of coating agent solution was prepared by thoroughly mixing of hydrolyzed glucomannan, maltodextrin, and HPMC. The hydrolyzed glucomannan was prepared according to the method previous applied by Wardhani *et al*. (2020). It was carried out by homogenizing glucomannan with distilled water in a beaker glass using a hot plate magnetic stirrer (IKA C-MAG HS 10). After that, cellulase enzymes was carefully added to obtain a final concentration of 20 ppm with continuous stirring for 12 hours at 30 °C. The solution was then boiled for 10 minutes to inactivate the enzymes.

After equilibrating the solution to room temperature, maltodextrin and HPMC (0.5% w/v) were added successively while stirring. The glucomannan: maltodextrin ratios employed in this study were N-1 5:0, N-2 3:2, N-3 2:3, and N-4 0:5 (in 1% w/v total coating material). A thoroughly weighed 1.731 g of NaFeEDTA as the substrate was introduced after the coating material mixture had achieved its homogeneity.

The iron encapsulation process was performed using a spray dryer (Mini Buchi B-290) with a dry air flow rate of 667 L/hour, a feed pump flow rate of 0.18 L/hour, and an aspiration rate of 90%. The prepared sample solution was delivered at a dry inlet air temperature of 140°C to facilitate spray drying (Wardhani *et al*., 2020).

Encapsulation Efficiency and Iron Loading

Analysis of Fe content in microparticles began with dissolving 5 mg of microparticles in 2.5% v/v HCl solution accompanied by 250 rpm stirring at 85°C for 2 hours. Then, the solution was filtered using a Whatman 42 filter paper. Furthermore, the total iron content of the sample was determined by adding hydroxylamine hydrochloride (10% w/v) and followed by heating the mixture to achieve its boiling point. Sodium acetate 10% w/v, and 1.10 o-phenanthroline 0.2% w/v were then added until the color of the solution turned to orange. After being diluted with distilled water to a volume of 25 mL, this solution was then allowed to stand for at least 10 minutes and analyzed its total iron content using a UV-Vis Spectrophotometer (Genesys 10S) at a wavelength of 513 nm (Gutiérrez *et al*., 2016). The calculation of encapsulation efficiency and iron loading followed research by Handayani *et al*. (2022) as follows:

Encapsulation efficiency was defined as the ratio of the mass of iron microencapsulated to the initial mass of iron, as in Equation 1.

$$\text{Efficiency} = \frac{\text{the total mass of Fe microencapsulated}}{\text{the initial total mass of Fe}} \times 100\% \quad (1)$$

Iron loading capacity is described as the ratio of the total mass of iron in the microcapsules to 100 mg of dry microparticles, as in Equation 2.

$$\text{Iron Loading Capacity (\%)} = \frac{\text{the total mass of Fe in microcapsules (mg)}}{\text{100 g of dry microcapsules (mg)}} \times 100\% \quad (2)$$

Moisture Content (MC)

The resulting iron microparticle were tested for its moisture content by drying 2 grams of iron microcapsules powder spread on a petri dish in an electric oven at 105 ± 2°C until the achievement of a constant weight (AOAC, 2000). The moisture content was calculated using Equation 3.

$$\text{MC} = \frac{\text{wet sample weight-dry sample weight}}{\text{wet sample weight}} \times 100\% \quad (3)$$

Particle's Surface Morphology Analysis

Surface morphology of microcapsule particles was observed using a Scanning Electron Microscopy (JSM-6510 LV JEOL Ltd., Tokyo, Japan) at 20,000 V with a magnification of 10,000×.

Particle Size Distribution Analysis

The resulting microcapsules size distribution was quantified using a Laser Particle Sizer LLPA-C10. The average particle size was indicated by the volume-weighted average. The particle size distribution was determined using the SPAN factor (Jiang *et al*., 2017), which was calculated using Equation 4.

$$\text{SPAN Factor} = \frac{(D_{90}\text{-}D_{10})}{D_{50}} \times 100\% \quad (4)$$

Where D_x is the particle size that has x% particles smaller than the specific value.

Thermal Stability Analysis

Thermal stability testing was carried out using a Thermogravimetric Analyzer (NEXTA STA, Hitachi STA200RV). The resulting Fe microcapsules weighed 10 mg each were heated at 30°C to 550°C with a heating rate of 10°C/min in air. Changes in the samples' weights due to changes temperature and time were recorded.

Functional Group Analysis

Functional groups contained in the resulting Fe microcapsules were identified using Fourier-Transform Infrared Spectroscopy (Perkin-Elmer UATR Spectrum Two).

Iron Release Profile (*in vitro*)

The iron release profile assay was carried out using simulated gastric fluid (SGF, pH 1,2) and simulated intestinal fluid (SIF, pH 7.4) The composition of SGF and SIF followed the research by Gutiérrez *et al*. (2016). A carefully weighed 20 mg of iron microparticles were dispersed in 100 mL of SGF solution for 1 hour at 37°C. Parallelly, 150 mL of SIF solution was added to the mixture and the iron release was observed for the next 3 hours. The samples were withdrawn every 30 minutes and then filtered. Each time a sample was withdrawn, an equal volume of fresh SGF or SIF was added to the respective solutions. Total iron content was measured with a UV-Vis spectrophotometer (Gupta *et al.*, 2015). The iron release was calculated using Equation 5.

$$\text{Cumulative Fe release} = \frac{\text{mg of cumulative iron release}}{\text{mg of iron in microcapsules}} \times 100\% \quad (5)$$

Bioavailability of Iron (*in vitro*)

Iron bioavailability was analyzed using a simulation saliva-gastro-intestinal model. The liquid composition of simulated saliva fluid (SSF), simulated gastric fluid (SGF), and simulated intestinal fluid (SIF) was duplicated based on previous research (Gutiérrez et al., 2016). The SSF (10 mL, pH 6.5) was added to each flask containing the sample and stirred at 37ºC and 100 rpm for 5 minutes. Simultaneously, the SGF (15 mL) was then added to the sample. The pH of the mixture was adjusted to 1.2 by a careful addition of HCl and stirred at 37°C and 100 rpm for 1 hour. Accordingly, SIF liquid (25 mL) was added after pH neutralization (7.4) and stirred at 37 ºC and 100 rpm for 3 hours. The final mixture was then centrifuged at 3500 rpm for 60 minutes. The supernatant was withdrawn for iron content analysis using a UV-Vis Spectrophotometer (Handayani *et al*., 2022). The iron bioavailability was calculated using Equation 6.

$$\text{Bioavailability} = \frac{\text{mg of iron in the supernatant}}{\text{mg of iron in microparticles}} \times 100\% \quad (6)$$

Statistical Analysis

Research data are presented with the mean ± standard deviation of three repetitions. Statistical analysis was done using one-way analysis of variance (one-way ANOVA) to compare data groups by means of Ms. Excel 2019. Differences in data were considered significant at $p < 0.05$.

RESULTS AND DISCUSSION

Encapsulation Efficiency and Iron Loading Capacity

Figure 1.a shows the iron encapsulation efficiency, which is determined by the retention of the core material and the reduction of active chemicals on the surfaces of the powder particles quantified (Nguyen *et al*., 2022). In this study, the encapsulation efficiencies of Fe for single-coated microparticle with the glucomannan : maltodextrin ratio of 5:0 (N-1 sample) and 0:5 (N-4 sample) were almost the same. Meanwhile, microparticle with the glucomannan : maltodextrin ratio of 3:2 showed a higher encapsulation efficiency than that of N-1 and N-4. Although the encapsulation efficiency of N-3 sample with a glucomannan : maltodextrin ratio of 2:3 was higher than that of N-1, but it remains lower than that of N-4 sample. The higher concentration of glucomannan in the N-2 sample caused the viscosity of the solution to be higher, thus generating a thicker coating layer during the drying process, which led to exhibiting greater retention to the core material (Premi & Sharma, 2017). The results of this study were in line with previous research (Nguyen *et al*., 2022), which studied the encapsulation of anthocyanins using glucomannan and a glucomannan-maltodextrin combination as a coating using the freeze-drying method.

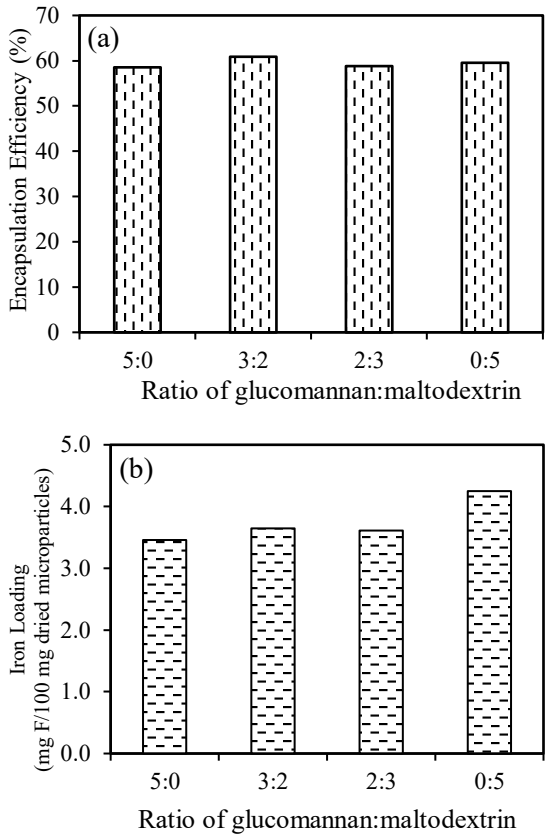

Figure 1. (a) Encapsulation efficiency and (b) iron loading of FE in varying ratio of glucomannan and maltodextrin

The encapsulation efficiency value obtained using glucomannan was 43.6%, while that of the combination of glucomannan coating with maltodextrin with a ratio of 1:1 was higher, namely 55.4% (Nguyen *et al*., 2022). Glucomannan as a coating material has high stability and relatively low oxidation resistance. On the contrary, maltodextrin has poor stability and high oxidation resistance. Therefore, the combination of glucomannan with maltodextrin can improve the physicochemical properties of the coating material in terms of thermal stability and resistance to oxidation (Sari *et al*., 2020; Yang *et al*., 2009).

Encapsulation efficiency depends on the physical and chemical properties of the coating materials used (Krisanti *et al*., 2019). In addition, process parameters, such as drying temperature, feed composition, and particle morphology also largely influence encapsulation performance. Excessively high drying temperatures can cause degradation of heat-sensitive compounds, while sticky materials with low glass transition temperatures can potentially stick to the walls of the drying chamber, resulting in considerable product loss. Furthermore, the formation of porous particle structures can accelerate the migration of oxygen and volatile compounds, which can cause degradation or breakage of encapsulated materials, ultimately reducing encapsulation efficiency (Pidalà *et al*., 2025).

The iron loading values obtained were inversely proportional to the glucomannan composition, as seen in Figure 1.b. This phenomenon occurred due to an increase in the total mass of dry microparticles. Besides, the higher viscosity of the glucomannan solution could cause the microparticles breakage as a result of the high osmotic pressure. Ultimately, the total iron trapped in the coating material declined, thus reducing the iron loading value (Krisanti *et al*., 2019). When consuming minerals such as iron, it is important to ensure adequate intake to prevent iron deficiency or iron overload. Iron in its free form can be toxic for humans because it plays a role in the formation of free radicals that damage cell membranes, proteins, and DNA, either directly or through hydrogen peroxide. Excessive iron accumulation can also have a negative impact on the brain and reduce cognitive function (Manckoundia *et al*., 2020). Based on the release test, the best formulation obtained in this study was the N-3 sample, which had an iron loading value of 3.61 mg Fe/100 mg dried microparticles. The iron microparticles produced in this study can be an intermediate product to be incorporated to the carrying food in the fortification process. As a food fortifier, to meet 75% of the daily need for iron: 18 – 20 mg per day, as recommended by WHO, ± 400 mg of iron microparticles are needed (Barragán-ibañeZ *et al*., 2016).

Moisture Content

The results of the moisture content test subjected to iron microcapsules obtained from four formulations are shown in Figure 2. According to Vanderbeek *et al*., (2007), a dietary fiber, glucomannan has a molecular structure that is composed of a specific combination of mannose and glucose. It has the highest molecular weight and viscosity values among other dietary fibers and will immediately form a highly viscous solution when dissolved in water. Moreover, it also has an incredible water-holding capacity that significantly affects its hygroscopicity. For that reason, glucomannan easily absorb water from the surrounding air, causing it to have a high moisture content.

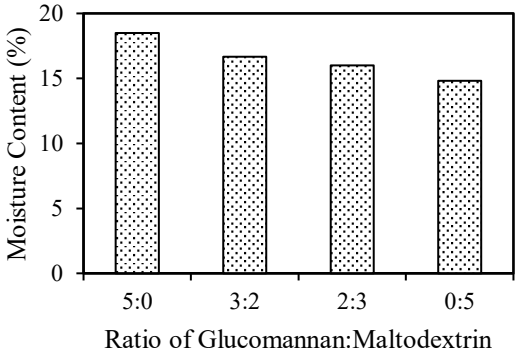

Figure 2. Moisture level content in varying glucomannan : maltodextrin ratio

Microparticle Morphology

The morphological characteristics of the iron microcapsules were observed using Scanning Electron Microscope (SEM) analysis with a magnification of 10,000×. Figure 3 displays some wrinkles on the outer surfaces of the resulting iron microcapsules. Research by Baldelli *et al*., (2022) used HPMC as an additional coating material for iron microcapsules and found that it was formed in the outer cells of the microcapsules which were characterized by a release force that reached its peak. Therefore, the outermost coating of the iron microcapsules was HPMC. No significant morphological differences were observed in the microcapsules derived from four formulations. The presence of wrinkles on the microcapsules's surface shown by the SEM images was caused by HPMC, which has a high molecular weight (Zheng *et al*., 2021). From a microstructural perspective, despite the wavy morphology, there were no surface gaps or microspherical gaps found, indicating a good structure and low gas permeability (Yonekura *et al*., 2014). Zheng *et al*., (2021) also found the same phenomenon where the outer layers of the four microparticles were confirmed as the HPMC.

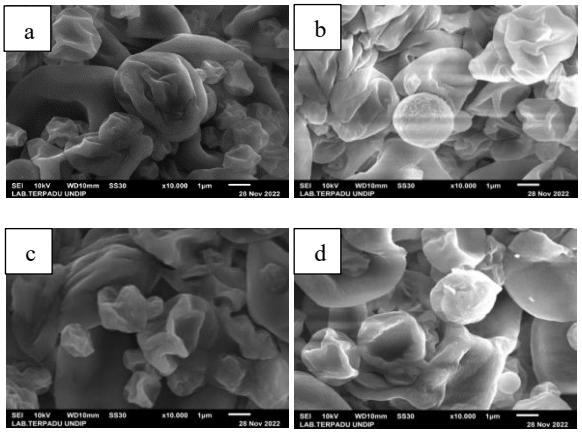

Figure 3. Morphology of iron microcapsules with SEM magnification of 10,000×(a) Formulation N-1(b) Formulation N-2(c) Formulation N-3 and (d) Formulation N-4

Particle Size Distribution

Particle size distribution is an important physical parameter because it affects the handling, stability, and storage conditions of the microcapsules (Wardhani *et al*., 2020). The results showed that the encapsulated iron microcapsules obtained in this study shown in Figure 4 varied in particle sizes, which were 111.412 µm for Formulation N-1, 107.615 µm for Formulation N-2, 108.955 µm for Formulation N-3, and 105.897 µm for Formulation N-4. Hence, it can be confirmed that the size of the resulting encapsulated iron was still in the micrometer range, namely 1-1000 µm. Previous research suggested that the resulting particle sizes were influenced by many factors, such as the type and concentration of coating material, feed flow rate, drying inlet air temperature, and atomization (Tontul & Topuz, 2017). As presented in Figure 4, the average

particle sizes were almost the same for all formulations. This could be due to the constant concentration of the total coating material (glucomannan-maltodextrin) despite the varying compositions. The results of this study were in accordance with those of a study by Lu *et al*. (2020) who encapsulated β-carotene emulsions with varying compositions of glucomannan-and-maltodextrin-based coatings at a constant total concentration and obtained particles, using the spray drying method, with almost the same average size for all formulations. Maltodextrin and glucomannan are polysaccharides that have a closer value of molecular weight and similar functional groups, so the difference between the two types of coatings does not affect the resulting particle sizes (Cahya *et al*., 2018; Lu *et al*., 2020). In addition, the operating conditions of the drying equipment used in this study were set to be the same for all formulations.

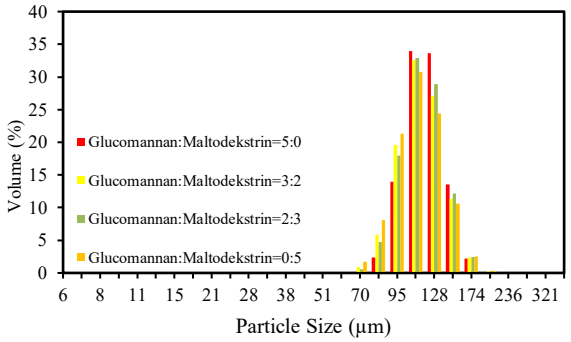

Figure 4. Particle size distribution on the four formulations of iron microparticles

FTIR Spectrum

Figure 5 shows peaks at a wavelength of 3.336 cm^{-1}, showing that all the formulations of iron microcapsules have O-H functional groups. The peaks at a wavelength of 2939 cm^{-1} indicate the presence of C-H functional groups in all the formulations of iron microparticles. Besides, peaks are also identified at wavelengths of 1,604 cm^{-1}, 1.370 cm^{-1}, 1.031 cm^{-1}, and 584 cm^{-1}, confirming the existence of C=O, C-O-C, C-O, and C-C-N functional groups (Farissi *et al*., 2017; Resende *et al*., 2014). Among the four formulations, only N-4 (glucomannan:maltodextrin = 0:5) had a lower transmission percentage.

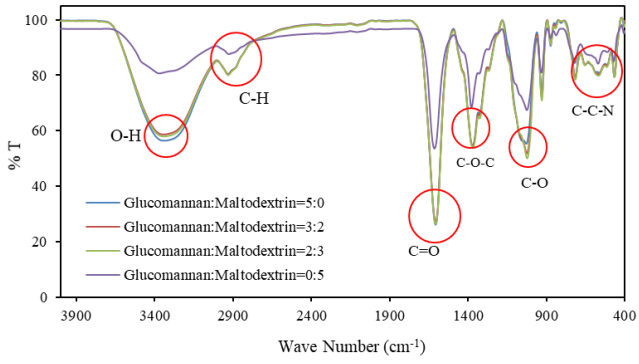

Figure 5. FTIR test results of the four formulations

The O-H, C-H, and C-O functional groups found in the four iron microparticles were obtained from glucomannan, maltodextrin, HPMC, and EDTA. The O-H group can be assigned to the stretching of glucose and mannose from glucomannan (Nurlela *et al*., 2021), while the C-O group can be assigned to the existence of the carbon bonded to the alcohol group (Farissi *et al*., 2017). The C-O-C functional group originated from the raw materials glucomannan, maltodextrin, and HPMC. Such group is abundant in polymers because oxygen tends to form cross-links in the carbon structure of precursor and polymer molecules (Jerigová *et al*., 2022). Meanwhile, C=O is found in glucomannan and EDTA. It is a carbonyl group attached to an acetyl group that binds to mannose in glucomannan (Zhang *et al*., 2014). In EDTA, it is a group that enters the carboxyl group (-COOH), while the identified C-C-N group is belongs to amine (Resende *et al*., 2014). Therefore, all functional groups contained in the iron microparticles obtained from this study correspond to those observed in the raw materials used for making iron microcapsules.

Thermal Stability

TGA analysis was carried out at a temperature range of 30 ─ 550°C. The results in Figure 6 show that the microcapsules experienced a first weight loss at 30-100°C due to water evaporation (Karaaslan *et al*., 2021) of 9.71% (N-1), 9.14% (N-2), 9.45% (N-3), and 6.59% (N-4). Furthermore, each formulation's weight decreased in a stable manner in the range of 100-200°C, namely 7.68% (N-1), 5.87% (N-2), 5.87% (N-3), and 2.94 % (N-4). The weight loss that occurred above 100°C was caused by residual water or moisture that was still trapped in the molecules that began to evaporate (Abdel-Aty *et al*., 2023). A drastic decrease in weight occurred in the range of 200 ─ 250°C, namely 19% (N-1), 16.9% (N-2), 14.22% (N-3), and 21.72% (N -4), probably caused by the start of the decomposition of the long chains of the coating material molecules, which were polysaccharide groups (Abdel-Aty *et al*., 2023), continuing up to 450°C. In the range of 250 ─ 450°C, the weight losses were 20.99% (N-1), 25.55% (N-2), 28.96% (N-3), and 33.03% (N-4). In this range, HPMC polymer degradation also began to slowly degrade (Soni *et al*., 2016).

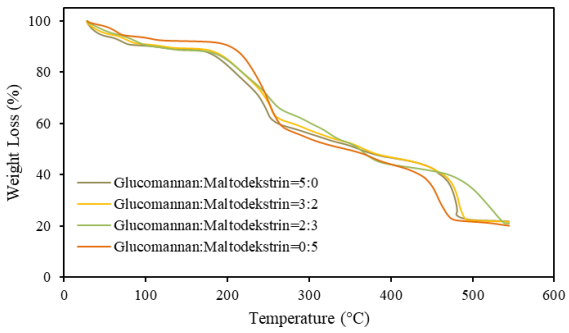

Figure 6. Thermogram of iron microparticles with variations in the coating composition

The second drastic weight loss occurred in the range of 450 – 550°C by 20.87% (N-1), 20.91% (N-2), 20.53% (N-3), and 15.64% (N-4), which were probably caused by the degradation of the HPMC polymer, which became an additional coating in iron microcapsules (Soni *et al*., 2016). The iron microcapsules produced in this study were intermediate products to be added to the carrying food in the fortification process. For this reason, TGA analysis became important to determine a suitable fortification method to use (Handayani *et al*., 2022). Based on the thermal resistance profiles, it is preferable for these iron microcapsules to be fortified in a system at a temperature below 200°C to prevent early decomposition of the microcapsules.

Iron Release Profile

The profile of iron release of the iron microparticles was tested in two different solutions, namely SIF and SGF. The results of the analysis displayed the amount of iron released against time, as shown in Figure 7. The four formulations had different iron release rates in the same time frame. All four formulations had a significant increase in iron release in the first half hour and then experienced a relatively constant increase the next time. As much as 64 – 79% of NaFeEDTA was released in the first hour in the gastrointestinal digestion and then continued to increase steadily until the 4th hour. The highest release rate of 79.89% in the SGF solution was shown by Formulation N-1, while the lowest one, of 64.28%, was of Formulation N-3. The most significant increases in release rates in the SIF solution were shown by almost the same figures for Formulations N-1, N-2, and N-3, namely 2.27%, 2.34%, and 2.27%, respectively. Formulation N-4 had the lowest increase in the SIF solution, namely 1.73%. According to Baldelli *et al*. (2022), poor encapsulation is characterized by a high release rate and low bioavailability. N-3 showed the lowest release rate among the other three formulations, and thus is considered the best. In addition, polymers have a large molecular weight which has a major influence on the distribution of iron compounds in microparticles by spray drying; choosing the most appropriate materials for coatings becomes necessary to get good encapsulation efficiency and release rate (Baldelli *et al*., 2022). Glucomannan and maltodextrin microencapsulation with the addition of HPMC showed a better release rate compared to other coatings that had been studied, such as chitosan (Handayani *et al*., 2022). The results showed that Formulation N-3 was the one with the best release rate of 64.28% in the SGF solution and an increase of 2.27% in the SIF solution.

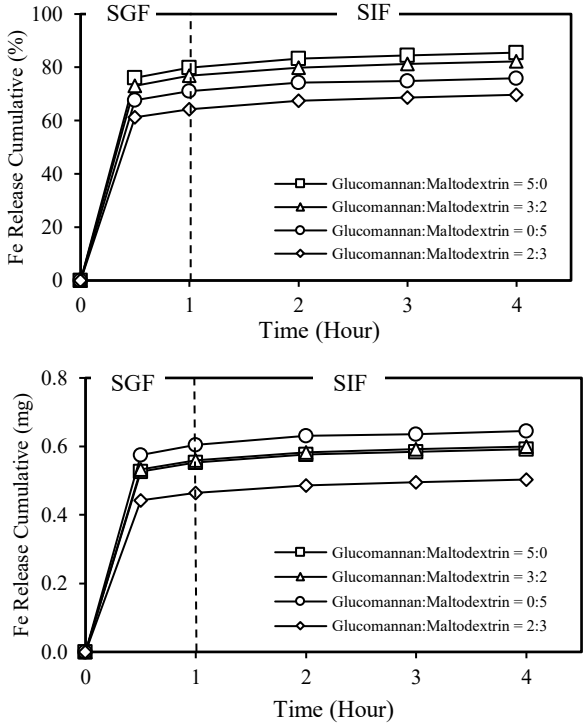

Figure 7. Fe release cumulative in various formulations

Iron Bioavailability

The iron bioavailability testing was carried out by in vitro simulation (saliva-gastro-intestinal model), in which bioavailability was expressed as the total amount of iron that could be dissolved in digestive juices (Shubham *et al*., 2020). Based on the test results shown in Figure 8, the bioavailability values of NaFeEDTA were 2.89%, 2.78%, 2.33%, and 2.57 % for Formulation N-1, Formulation N-2, Formulation N-3, and Formulation N-4, respectively. The actual amounts of iron dissolved in the digestive juices for Formulations N-1, N-2, N-3, and N-4 were 0.050, 0.051, 0.042, and 0.055 (mg of Fe in the supernatant), respectively.

The bioavailability of iron is influenced by several factors, including the types of iron source and the presence of inhibitors as well as enhancers (Piskin *et al*., 2022). In this study, the bioavailability of NaFeEDTA alone was tested without enhancer addition. Previous research by Handayani *et al*. (2022) tested specific ferrous gluconate with and without the supplementation of an enhancer (ascorbic acid) and found that its bioavailability values were 1.826% (without enhancer) and 2.199% (with enhancer). In this study, NaFeEDTA was better than ferrous gluconate at bioavailability, consistent with the statement of Yang *et al*. (2011) that NaFeEDTA is a suitable type of iron to be used in food fortification because of its higher bioavailability.

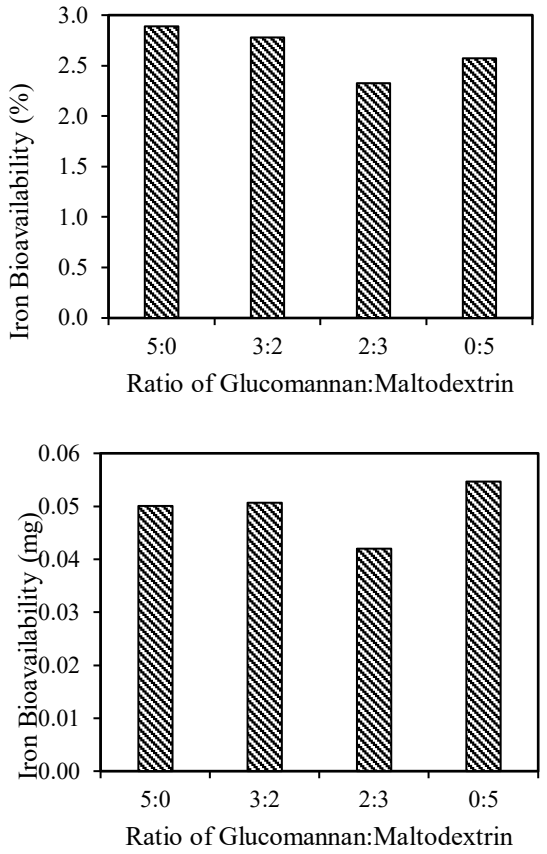

Figure 8. Bioavailability of iron in various formulations

Another study by Abizari et al. (2012) tested the bioavailability values of NaFeEDTA and FeSO4 with the existence of an inhibitor (phytic acid) and found that their bioavailability values were 2.9% and 1.8%, respectively. NaFeEDTA is an iron chelate with a similar bioavailability to FeSO4 when no inhibitor is employed. When an inhibitor like phytic acid is applied, the bioavailability of NaFeEDTA increases to about twice that of FeSO4 (Hurrell, 2021). So, NaFeEDTA can produce better results than other types of iron for iron fortification of food.

CONCLUSION

This study has successfully demonstrated iron encapsulation using glucomannan and maltodextrin as the coating material through a spray drying process. The results showed that the combination of glucomannan and maltodextrin as coating materials increased iron encapsulation efficiency, and higher glucomannan levels lead to reduced iron loading capacity. The physical and chemical characterizations of the microparticles showed that the morphologies of the iron microcapsules derived from the four formulations were identical. The iron microcapsules produced from formulations with higher glucomannan levels had higher moisture contents. The four formulations had a narrow size distribution (mono disperse) with nearly the same average particle sizes between 105 and 111 µm. The four formulations also showed good thermal resistance up to a temperature of ± 200 °C. The results of the FTIR analysis confirmed that the four formulations displayed functional groups specifically identified from the compounds that made up the microparticles. The results showed that the combination of glucomannan and maltodextrin as coating agents affected the iron release rate. The best release rate of 64.28% was shown by Formulation N-3 in SGF solution, with an increase of 2.27% in SIF solution. NaFeEDTA used in the four formulations had a high bioavailability value of 2.3–2.9%.

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REFERENCES

Abdel-Aty, A. M., Barakat, A. Z., & Mohamed, S. A. (2023). Garden cress gum and maltodextrin as microencapsulation coats for entrapment of garden cress phenolic-rich extract: improved thermal stability, storage stability, antioxidant, and antibacterial activities. *Food Science and Biotechnology*, 32(1), 47–58. https://doi.org/10.1007/s10068-022-01171-3

Abizari, A. R., Moretti, D., Schuth, S., Zimmermann, M. B., Armar-Klemesu, M., & Brouwer, I. D. (2012). Phytic acid-to-iron molar ratio rather than polyphenol concentration determines iron bioavailability in whole-cowpea meal among young women. *Journal of Nutrition*, 142(11), 1950–1955. https://doi.org/10.3945/jn.112.164095

Agustina, T., Indarto, D., & Sandy, Y. D. (2019). Relationship between menstrual duration, Fe intake, and protein with hemoglobin levels in female adolescents with more nutritional status. *National Symposium and Workshop Continuing Medical Education* XIII, 171–180.

Al-Attar, Z., Jassim, S., Hashim, I., & Jasim, S. (2020). The obesity research and therapy unit at AL-Kindy College of Medicine. *International Medical Journal*, 24(3), 435–448. https://www.researchgate.net/publication/343678565

Anwar, S. H., Maria Br Ginting, B. M. B., Aisyah, Y., & Safriani, N. (2016). Application of porang flour as stabilizer in O/W emulsion and coating material for fish oil microcapsules. *Journal of Agricultural Industry Technology*, 27(1), 76–88.

AOAC. (2000). *Official methods of analysis, 17th ed*. Association of Official Analytical Chemists. Gaithersburg, MD. USA.

Baldelli, A., Liang, D. Y., Guo, Y., & Pratap-Singh, A. (2022). Effect of the formulation on mucoadhesive

spray-dried microparticles containing iron for food fortification. *Food Hydrocolloids*, 134, 1–12. https://doi.org/10.1016/j.foodhyd.2022.107906

Barragán-ibañeZ, G., Santoyo-sánchez, A., & Ramos-Peñafel, C. (2016). Iron deficiency anemia. *Medical Magazine of the General Hospital of Mexico*, 79, 88–97.

Cahya, A. P., Syaflan, M., & Ngatirah, N. (2018). Probiotic (*Lactobacillus casei*) Encapsulation using the method of spray drying with combined prebiotic from Iles-Iles (*Amorphophallus oncophyllus*) and protectant agent (skim milk, gum arabic, maltodextrin). *Indonesian Food and Nutrition Progress*, 15(2), 61–68. https://doi.org/10.22146/ifnp.33423

Farissi, H. El, Lakhmiri, R., Albourine, A., Cherkaoui, O., & Safi, M. (2017). Removal of RR-23 dye from industrial textile wastewater by adsorption on *Cistus ladaniferus* seeds and their biochar. *Journal of Environment and Earth Science*, 7(11), 105–118. www.iiste.org

Gardner, W., & Kassebaum, N. (2020). Global, regional, and national prevalence of anemia and its causes in 204 countries and Territories, 1990 - 2019. *Current Developments in Nutrition*, *4* https://academic.oup.com/cdn/article/4/Supplement_2/830/5845051

Gupta, C., Chawla, P., Arora, S., Tomar, S. K., & Singh, A. K. (2015). Iron microencapsulation with blend of gum arabic, maltodextrin and modified starch using modified solvent evaporation method - Milk fortification. *Food Hydrocolloids*, 43, 622–628. https://doi.org/10.1016/j.foodhyd.2014.07.021

Gupta, C. P. (2014). Role of Iron (Fe) in Body. *IOSR Journal of Applied Chemistry*, 7(11), 38–46. www.iosrjournals.org

Gutiérrez, G., Matos, M., Barrero, P., Pando, D., Iglesias, O., & Pazos, C. (2016). Iron-entrapped niosomes and their potential application for yogurt fortification. *LWT - Food Science and Technology*, 74, 550–556. https://doi.org/10.1016/j.lwt.2016.08.025

Handayani, N. A., Mulia, K., Kartohardjono, S., & Krisanti, E. A. (2022). Fortifying jelly foods with microencapsulated anti-anaemic compounds, ferrous gluconate, ascorbic acid and folic acid. *Journal of Food Science and Technology*, 60(1), 147–159. https://doi.org/10.1007/s13197-022-05599-7

Hurrell, R. F. (2021). Iron fortification practices and implications for iron addition to salt. *Journal of Nutrition*, 151, 3S-14S. https://doi.org/10.1093/jn/nxaa175

Jerigová, M., Odziomek, M., & López-Salas, N. (2022). "We are here!" Oxygen functional groups in carbons for electrochemical applications. *ACS Omega*, 7(14), 11544–11554. https://doi.org/10.1021/acsomega.2c00639

Jiang, W.-Z., Cai, Y., & Li, H.-Y. (2017). Chitosan-based spray-dried mucoadhesive microspheres for sustained oromucosal drug delivery. *Powder Technology*, 312, 124–132.

Karaaslan, M., Şengün, F., Cansu, Ü., Başyiğit, B., Sağlam, H., & Karaaslan, A. (2021). Gum arabic/maltodextrin microencapsulation confers peroxidation stability and antimicrobial ability to pepper seed oil. *Food Chemistry*, 337, 1–9. https://doi.org/10.1016/j.foodchem.2020.127748

Krisanti, E. A., Naziha, G. M., Amany, N. S., Mulia, K., & Handayani, N. A. (2019). Effect of biopolymers composition on release profile of iron (II) fumarate from chitosan-alginate microparticles. *IOP Conference Series: Materials Science and Engineering*, 509(1), 1–9. https://doi.org/10.1088/1757-899X/509/1/012100

Lu, W., Maidannyk, V., Kelly, A. L., & Miao, S. (2020). Fabrication and characterization of highly re-dispersible dry emulsions. *Food Hydrocolloids*, 102. https://doi.org/10.1016/j.foodhyd.2019.105617

Manckoundia, P., Konaté, A., Hacquin, A., Nuss, V., Mihai, A. M., Vovelle, J., & Putot, A. (2020). Iron in the general population and specificities in older adults: Metabolism, causes and consequences of decrease or overload, and biological assessment. *Clinical Interventions in Aging*, 1927-1938. https://doi.org/10.2147/CIA.S269379

Mardani, M., Siahtiri, S., Besati, M., Baghani, M., Baniassadi, M., & Nejad, A. M. (2024). Microencapsulation of natural products using spray drying; an overview. *Journal of microencapsulation*, 41(7), 649-678. https://doi.org/10.1080/02652048.2024.2389136

Miller, C. H., Sheyholislami, H., Burns, J. L., & Connor, K. L. (2024). Sensory evaluations of a novel iron and zinc-enriched powder for the treatment and prevention of iron deficiency in women of reproductive age. *Maternal & Child Nutrition*, *20*(1), e13575. https://doi.org/10.1101/2022.12.10.22283294

Mishra, D. K., Jain, A. K., & Jain, P. K. (2013). A review on various techniques of microencapsulation. *International Journal of Pharmaceutical and Chemical Sciences*, 2(2), 962–977. www.ijpcsonline.com962

Mulligan, S., Ojeda, J. J., Kakonyi, G., Thornton, S. F., Moharamzadeh, K., & Martin, N. (2021).

Characterisation of microparticle waste from dental resin-based composites. *Materials*, 14(16), 1–17. https://doi.org/10.3390/ma14164440

Nguyen, Q. D., Dang, T. T., Nguyen, T. V. L., Nguyen, T. T. D., & Nguyen, N. N. (2022). Microencapsulation of roselle (*Hibiscus sabdariffa* L.) anthocyanins: Effects of different carriers on selected physicochemical properties and antioxidant activities of spray-dried and freeze-dried powder. *International Journal of Food Properties*, 25(1), 359–374. https://doi.org/10.1080/10942912.2022.2044846

Nurlela, Ariesta, N., Laksono, D. S., Santosa, E., & Muhandri, T. (2021). Characterization of glucomannan extracted from fresh porang tubers using ethanol technical grade. *Molecule*, 16(1), 1–8. https://doi.org/10.20884/1.jm.2021.16.1.632

Pandey, S., Asha, M. R., & Jayadep, A. (2016). Changes in physical, cooking, textural properties and crystallinity upon iron fortification of red rice (Jyothi). *Journal of Food Science and Technology*, 53(2), 1014–1024. https://doi.org/10.1007/s13197-015-2130-7

Pidalà, J., Lombardelli, C., Benucci, I., & Esti, M. (2025). Synergistic use of spray drying and fluidized bed in the microencapsulation of food ingredients. *Trends in Food Science & Technology*, 105323. https://doi.org/10.1016/j.tifs.2025.105323

Piskin, E., Cianciosi, D., Gulec, S., Tomas, M., & Capanoglu, E. (2022). Iron absorption: factors, limitations, and improvement methods. *ACS Omega*, 7 (24), 20441–20456. https://doi.org/10.1021/acsomega.2c01833

Pratama, D. Y. (2015). Karakterisasi dan Bioaksesibilitas Mikroenkapsulasi Zat Besi pada Formulasi Bahan Penyalut. Undergraduate Thesis, IPB University, Indonesia.

Pratap-Singh, A., & Leiva, A. (2021). Double fortified (iron and zinc) spray-dried microencapsulated premix for food fortification. *LWT - Food Science and Technology*, 151, 1–8. https://doi.org/10.1016/j.lwt.2021.112189

Premi, M., & Sharma, H. K. (2017). Effect of different combinations of maltodextrin, gum arabic and whey protein concentrate on the encapsulation behavior and oxidative stability of spray dried drumstick (Moringa oleifera) oil. *International Journal of Biological Macromolecules*, 105, 1232–1240. https://doi.org/10.1016/j.ijbiomac.2017.07.160

Resende, J. E., Gonçalves, M. A., Oliveira, L. C. A., Da Cunha, E. F. F., & Ramalho, T. C. (2014). Use of ethylenediaminetetraacetic acid as a scavenger for chromium from "wet Blue" leather waste: Thermodynamic and kinetics parameters. *Journal of Chemistry*, 2014, 1–8. https://doi.org/10.1155/2014/754526

Safiri, S., Kolahi, A. A., Noori, M., Nejadghaderi, S. A., Karamzad, N., Bragazzi, N. L., Sullman, M. J. M., Abdollahi, M., Collins, G. S., Kaufman, J. S., & Grieger, J. A. (2021). Burden of anemia and its underlying causes in 204 countries and territories, 1990–2019: Results from the global burden of disease study 2019. *Journal of Hematology and Oncology*, 14(1). https://doi.org/10.1186/s13045-021-01202-2

Sagiri, S. S., Pal, K., Basak, P., Rana, U. A., Shakir, I., & Anis, A. (2014). Encapsulation of sorbitan ester-based organogels in alginate microparticles. *AAPS PharmSciTech*, 15(5), 1197–1208. https://doi.org/10.1208/s12249-014-0147-2

Saha, S., & Roy, A. (2020). Whole grain rice fortification as a solution to micronutrient deficiency: Technologies and need for more viable alternatives. *Food Chemistry*, 326, 1–14). https://doi.org/10.1016/j.foodchem.2020.127049

Sari, D. K., Lestari, R. S. D., Khinanta, P., & Sahlan, M. (2020). Encapsulation bioactive compound propolis with carrageenan-gum arabic by spray drying. *Journal of Process Integration*, 9(1), 8–11.

Shubham, K., Anukiruthika, T., Dutta, S., Kashyap, A. V., Moses, J. A., & Anandharamakrishnan, C. (2020). Iron deficiency anemia: A comprehensive review on iron absorption, bioavailability and emerging food fortification approaches. *Trends in Food Science and Technology*, 99, 58–75). https://doi.org/10.1016/j.tifs.2020.02.021

Soni, S., Verma, A., & Ram, V. (2016). Evaluation of chitosan-hydroxy propyl methyl cellulose as a single unit hydrodynamically balanced sustained release matrices for stomach specific delivery of piroxicam. *MY Bioequivalence & Bioavailability*, 2(1), 1–14. https://doi.org/10.15406/mojbb.2016.02.00014

Tontul, I., & Topuz, A. (2017). Effects of different drying methods on the physicochemical properties of pomegranate leather (pestil). *LWT - Food Science and Technology*, 80, 294–303. https://doi.org/10.1016/j.lwt.2017.02.035

Urli, T. I., Hariyanto, T., Dewi, N., Program, M., Science, S., Faculty, K., Health, I., Tribhuwana, U., Malang, T., Program, D., Nursing, S., & Malang, P. K. (2017). Effect of porang flour (Amorphophallus muelleri Blume) on HDL levels in Wistar DM Type 2 rats (*Rattus novergicus*). *Nursing News*, 2(2), 652–664.

Vanderbeek, P. B., Fasano, C., O'Malley, G., & Hornstein, J. (2007). Esophageal obstruction from a

hygroscopic pharmacobezoar containing glucomannan. *Clinical Toxicology*, 45(1), 80–82. https://doi.org/10.1080/15563650601006215

Wardhani, D. H., Wardana, I. N., Ulya, H. N., Cahyono, H., Kumoro, A. C., & Aryanti, N. (2020). The effect of spray-drying inlet conditions on iron encapsulation using hydrolysed glucomannan as a matrix. *Food and Bioproducts Processing,* 123, 72–79. https://doi.org/10.1016/j.fbp.2020.05.013

Yang, J., Xiao, J. X., & Ding, L. Z. (2009). An investigation into the application of konjac glucomannan as a flavor encapsulant. *European Food Research and Technology*, 229 (3), 467–474. https://doi.org/10.1007/s00217-009-1084-2

Yang, Z., Siekmann, J., & Schofield, D. (2011). Fortifying complementary foods with NaFeEDTA - considerations for developing countries. *Maternal and Child Nutrition*, 7(SUPPL. 3), 123–128. https://doi.org/10.1111/j.1740-8709.2011.00355.x

Yonekura, L., Sun, H., Soukoulis, C., & Fisk, I. (2014). Microencapsulation of *Lactobacillus acidophilus* NCIMB 701748 in matrices containing soluble fibre by spray drying: Technological characterization, storage stability and survival after in vitro digestion. *Journal of Functional Foods,* 6(1), 205–214. https://doi.org/10.1016/j.jff.2013.10.008

Zhang, C., Chen, J. Da, & Yang, F. Q. (2014). Konjac glucomannan, a promising polysaccharide for OCDDS. *Carbohydrate Polymers*, 104 (1), 175–181. https://doi.org/10.1016/j.carbpol.2013.12.081

Zheng, J., Wang, B., Xiang, J., & Yu, Z. (2021). Controlled release of curcumin from HPMC (Hydroxypropyl Methyl Cellulose) co-spray-dried materials. *Bioinorganic Chemistry and Applications*, 2021, 1–6. https://doi.org/10.1155/2021/7625585