



## **The Effect of Fish Oil Concentration on the Microcoating Process with Coating Materials to Produce Microcapsules and to Investigate their Physicochemical Properties Using Wall Materials and Different Microcoating Methods**

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**Abstract:** Microcoating with coating materials is used to increase oil stability and control unpleasant taste and smell. The effect of fish oil concentration on the process of micro-coating with coating materials was investigated in order to emphasize the microcapsules and investigate In their physicochemical properties using wall materials and different micro-coating methods. order to check the physicochemical properties of the parameters and methods of measuring the moisture content, the total oil content, the surface oil content, the microcoating efficiency, the color and the morphology of the microcapsules are investigated using the research results. The results of the investigation of the physicochemical factors of the produced powders show that the coacervation method is also the most suitable microcoating method for fish oil. Microcoating with these materials and this method is able to produce powders with higher microcoating efficiency and lower surface oil content than other treatments. The results show that the microcapsules produced by the coacervation method are more spherical and larger than other treatments. The comparison of spray drying, freeze drying and coacervation and mixed mass methods confirms that wall composition, drying temperature, microcapsule morphology and process time are actually the most important factors that affect the stability of oily fish powder.

**Key words:** microencapsulation, fish oil, microcapsule, microencapsulation methods, composite agglomeration



## **1-Introduction**

In recent years, the trade of oils with long chains of unsaturated fatty acids from various sources such as canola, olive, linseed and fish oil has become common due to their importance for the health of consumers (1-2). There are many reports that these compounds are effective in protecting living systems against diseases such as breast and prostate cancer, heart diseases and Alzheimer's, preventing cancer, improving the functioning of the brain and nervous tissues. The effect of fish oil in creating health and its industrial applications in the food, cosmetics and paint industries bring very high applications for fish oil. In recent years, attention has been paid to the consumption of omega-3 fatty acids in the daily diet. The health benefits of eicosapentaenoic acid and docosahexaenoic acid found in fish and fish oil are well known and regular consumption of these fatty acids is suggested. (3) Different governments encourage their citizens to consume these fatty acids (2). The most recent proposal of the international community in the field of fatty acid studies is the consumption of EPA and DHA, 500 mg per day. These fatty acids in daily food are less than the recommended amount and this has caused producers in many countries to take steps towards the production of useful and practical foods containing these compounds. The health-enhancing effect of fish oil is well known and it is a source of food enrichment and nutritional supplements to maintain the normal function of the brain, reduce the possibility of cancer and prevent cardiovascular diseases (4). The biggest technical obstacle in adding effective amounts of EPA and DHA to foods is oxidation and the creation of fish aroma and smell, caused by lipid decomposition, which should be avoided. In addition, the unpleasant taste and smell of fish oil causes the quality of edible products to decrease and disappear (5). One of the suitable methods for protecting oily fish is micro-covering (6). Microcoating is a process in which very small particles, drops or bubbles are coated. In order to prevent the tongue from detecting the taste or changing the texture of food, the size of micro-coated oil particles should be less than 100 micrometers. Micro-coated omega-3 fatty acids in suitable wall materials can maintain the nutritional value and sensory characteristics of these materials (7). This technology is used in the food industry due to the reduction of the rate of evaporation and the transfer of core materials to the outside environment, the preservation of the flavor of the core, and the oxidation of materials during production and storage, the creation of



unpleasant odors and tastes, and the loss of nutritional value. And it becomes metabolic. The materials used to form capsules must have high emulsifying ability, low viscosity at high concentration and the ability to easily release core materials when dissolved in water, the relatively limited variety of these materials is one of the problems of this technique (8). Proteins, lipids and polysaccharides are among these materials, each of which has advantages and disadvantages and are often used together with other coating materials or modifying compounds, oxygen absorbers, antioxidants, chelating agents and surface active materials, in These polysaccharides are the most used because they have low viscosity and good solubility, and they have weak interfacial properties, which is a suitable feature for high efficiency in the microcoating process. Whey protein is widely used due to its good surface activity. A major part of whey proteins, beta-lactoglobulin, are capable of making thermal gels insoluble in water under acidic conditions and stability of anthocyanins, and are known as suitable matrices for the microencapsulation of water-soluble biological actives (8). The microcoating process usually includes two basic steps. First, create a suspension or emulsion of the core and wall materials, and then the obtained composition should be dried using a common drying technique or a microcoating method. In such a process, the preservation of the core constituents by the microcoating agent with the chemical nature of the core constituents (molecular weight, chemical function, solubility, polarity and stability), the physicochemical properties of the wall materials (solubility, stability, molecular weight, structure and physical state) and Also, the nature and effective parameters in the microcoating method are related to the one used (9). Microcoating techniques include: spray drying, freeze drying, spray cooling/cooling, fluid bed coating, extrusion, simultaneous crystallization, coacervation (phase separation), liposome entrapment, in situ polymerization (10). Materials used for lining in the food industry face limitations. These restrictions include allowing the use of these substances in food (11). The regulations for food additives are much stricter than for medicinal substances. Different compounds have been widely used for microcoating in the pharmaceutical industry, while they are not suitable for use in the food industry because many of these materials are not recognized as (GRAS) or generally safe for food applications (4). ). The most important criterion for choosing the wall material is the performance of micro-coating. (6). In general, the formation of the microcoating process requires incompatibility between the shell material and the active agents so that there will



be a coating on the surface of the active material. In order to microcoat hydrophobic active compounds, hydrophilic compounds should be used as wall materials, edible oil or fat is an example of a hydrophobic compound. Therefore, a wide range of proteins, polysaccharides and polymers are used for microcoating (5). The presented methods for microcoating can be divided into three physical groups (spray drying, freeze drying, spray cooling and cooling, fluid bed coating, simultaneous crystallization), chemical (addition complex and in situ polymerization), a combination of physical and chemically (coacervation and liposomal confinement) (12).

**Table 1: Conventional methods presented for microcoating**

| Description  | method                |   |
|--|-----------------------|---|
| Microcoating using a spray dryer has been used in the food industry since the 1950s. Spray drying is an economical, flexible and continuous method that produces particles of suitable quality. This technique is widely used in the food industry in the coating of dry and stable flavors and additives (3). During microcoating, the core materials are mixed and homogenized with the wall materials, then the resulting mixture enters the spray dryer and is atomized with a rotating nozzle or wheel. Due to the contact of hot air with the water particles in it, it evaporates, then the capsules are collected after falling in the lower part of the dryer (13).   | spray drying          | 1 |
| The technique of freeze drying or lyophilization is one of the most useful methods of drying heat-sensitive compounds that are unstable in aqueous solutions. This method is mainly used to produce high-quality dry ingredients that have heat-sensitive compounds such as aroma, vitamins, antibiotics, and microbial kills (14). Apart from drying heat-sensitive compounds, freeze drying is used for microencapsulation of water-soluble essential oils, as well as natural aromatic substances and medicinal substances. This process allows the frozen water in the material to directly change from the solid phase to the gas phase by freezing the material and then reducing the ambient pressure and increasing the temperature. | freeze drying         | 2 |
| Spray cooling/cooling is a low-cost microcoating process and is usually used for the microcoating of a number of organic and inorganic salts as well as texture-forming compounds, enzymes, flavors and other functional substances in order to improve thermal stability and delay release in humid environments. or converting hydrophilic liquid compounds into a flowable powder is used (14). Usually, the materials used as coating in these two techniques (spray cooling/cooling) are lipids. In the cooling and spray cooling method, the core material is dispersed in a washable coating liquid and sprayed from a heated nozzle into a controlled chamber (Bakan and Anderson, 1970). (lipid matrix) happens (14).               | Spray cooling/cooling | 3 |



|   |                              |   |
|---|------------------------------|---|
| Simultaneous crystallization is one of the processes of microencapsulation, which is carried out using sucrose as a background material for the inclusion of core materials. This process includes the spontaneous crystallization of sucrose at a temperature above 120 degrees Celsius, which leads to the production of accumulations of very fine sucrose crystals in the range of 3 to 30 micrometers, in which the core material is placed as a result of inclusion or inclusion in the structure of the crystals (15). During this process, many food compounds can be placed separately or in combination in the crystals (16). | Simultaneous crystallization | 4 |
|---|------------------------------|---|

In the meantime, the massification process is used as a technique to cover heat-sensitive compounds and solid or liquid compounds that are insoluble in water, such as omega-3. The lumping of the compound is defined as the separation of phase one or a number of hydrocolloids from the initial solution and the immediate deposition of the mass phase formed around the suspended or emulsified active compounds. A combination of gum arabic gelatin as wall-forming compounds In the composite agglomeration method, they have been known for a long time (8) the production of micro particles using composite agglomeration has merits such as micro coverage of a large amount of central materials, low mechanical force and thermal resistance due to the ionic nature of the reactions between polymers. The shaper of the wall and finally the formation of a strong wall. Generally, cross-linking compounds such as enzymes are used to harden the wall and strengthen the formed network (5). Among the researches carried out in this field, we can mention microencapsulation of rice bran oil (17), microencapsulation of fish oil in different levels of xanthan and alginate (18), microencapsulation of Kilka fish oil using maltodextrin, sodium caseinate, whey concentrate and starch (19). Linseed oil microencapsulation (20), fish oil microencapsulation in whey protein concentrate microcapsules (21), walnut oil microencapsulation using soy protein isolate and maltodextrin (22) and fish oil microencapsulation using soy protein (23). Various methods have been reported by researchers for the microcoating of compounds. Among these methods, spray drying is used to a greater extent in the food industry because it is cheap and with high precision.



## **2-Stages of producing microcapsules in conventional methods**

### **2-1- Preparation of emulsions**

In this method, solutions of wall material powders are prepared in distilled water (17). These solutions are prepared 1 day before the preparation of the emulsion and are placed in a shaker container overnight to ensure the complete absorption of the materials and their complete dissolution. The percentage of total dissolved solids is 40% by weight/volume for all emulsions. The final formulation of different treatments is determined after preliminary tests (emulsion cream stability) to determine the optimal ratio of wall materials and to achieve the best percentages for each treatment (13). Oil-in-water emulsions are prepared in two steps. At first, the core materials are gradually added to the wall materials with the stated ratio and mixed and stirred for 10 minutes. In the next step, the emulsions are completely homogenized with the help of the ultrathorax homogenizer for 5 minutes at 12,000 revolutions to obtain a homogeneous emulsion. In the continuation of the experiments, the stability of the emulsions was checked and then the emulsions were prepared for drying by freeze drying and spray drying (14).

#### **- Freeze drying of emulsions**

The prepared emulsions are first frozen for 24 hours at -80 degrees Celsius and then dried in a freeze dryer under reduced pressure (15). The process of drying frozen emulsions in a freeze dryer takes 45 hours. The resulting spongy masses are turned into uniform powder using a Chinese mortar. These powders are then stored in special and completely closed containers in a freezer at -20 degrees Celsius to perform the necessary physicochemical tests on them (14).

#### **- Spray drying of emulsions**

The operating conditions of the spray dryer are set after initial tests and pretreatment and reaching optimal conditions (inlet temperature 180 degrees Celsius and outlet temperature 80 degrees Celsius). Finally, the produced powders are stored in special and completely closed containers in the freezer at -20 degrees Celsius to perform the necessary physicochemical tests on them. Magnetic dissolves. Then this mixture is added



to the caparrageenan and gelatin solution. (14) Polyvinyl alcohol is used as a solvent and ionic strength enhancer (18). At this stage, the pH should be around 4.5-5. During the mixing process, the ionic strength of the mixture is adjusted by sodium salt with a concentration of 0.1 M. Because the addition of a large amount of salt stops the microcapsule process, glutaraldehyde is used in the next step to strengthen the end of the samples. They go around (18).

### **- Measurement of creaming index**

10 grams of each emulsion was transferred into a test tube (inner diameter = 15 mm and height = 125 mm) and kept for 24 hours at room temperature. (14) After storage, the emulsions were visually divided into 1 emulsions with The opaque and opaque creamy layer is separated at the top and the transparent (or opaque) "serum layer" at the bottom of the test tube. The total height of the emulsions and the height of the creamy layer are measured. The percentage of this index is calculated according to relation (1). All measurements are performed on at least 3 freshly prepared samples.

The relation (1 (

$$\text{Creaming index} = \frac{\text{height of creamy layer} \times 100}{\text{total height of emulsion}}$$

### **- Measurement of humidity and oil**

The moisture content of microcapsules is calculated by the weight difference of 1-2 grams of powder after drying for 16 hours in an oven at 105 degrees Celsius. The results are reported as moisture content per 100 grams of powder.

To measure surface oil, hexane-n milliliter 15 4 is added to 2.5 grams of powder and mixed for 2 minutes. This mixture is centrifuged at 8000 rpm ( $\square$ 12378 g) for 20 minutes. After passing the sample through filter paper and washing 2 times with n-hexane, the solvent is evaporated in Type KRvr TD at a temperature of 70 degrees Celsius. The solvent-free extract is dried at 105 degrees Celsius and the amount of surface oil of the powders is calculated. (16) To measure the total oil, 2 ml of acetate buffer (pH = 3) is added to 0.5 grams of powder and for 1 minute using They are mixed by vortex. The



resulting solution is extracted with 25 ml of n-hexane mixture (1/3) and the tubes are centrifuged for 15 minutes and the clear organic phase is collected and the aqueous phase is again mixed with the solvent. After filtration and passing through dry sodium sulfate, the solvent is evaporated in a rotary and the solvent-free extract is dried at 105 degrees Celsius, and the total oil is calculated to measure the microcoating efficiency using the amount of oil. The total and surface oil is calculated according to the relation (2). (16)

The relation (2)

Micro-coating efficiency =  $\frac{\text{micro-coated oil (grams per 100 grams of powder)} \times 100}{\text{total oil (grams per 100 grams of powder)}}$

### - Measuring the color of microcapsules

To measure the color of the microcapsules, some of the powders were placed in a container and then the amount of color is measured using a colorimeter and 3 factors \*L\*, a and \*b\* are measured in each sample. b\* \*L indicates the lightness of the microcapsules and \*a and \* indicate the color of the microcapsules (a = green, a+ = red, b- = blue, and b+ = yellow). (16)

### - Examining the morphology of microcapsules, the microstructure and morphology of microcapsules

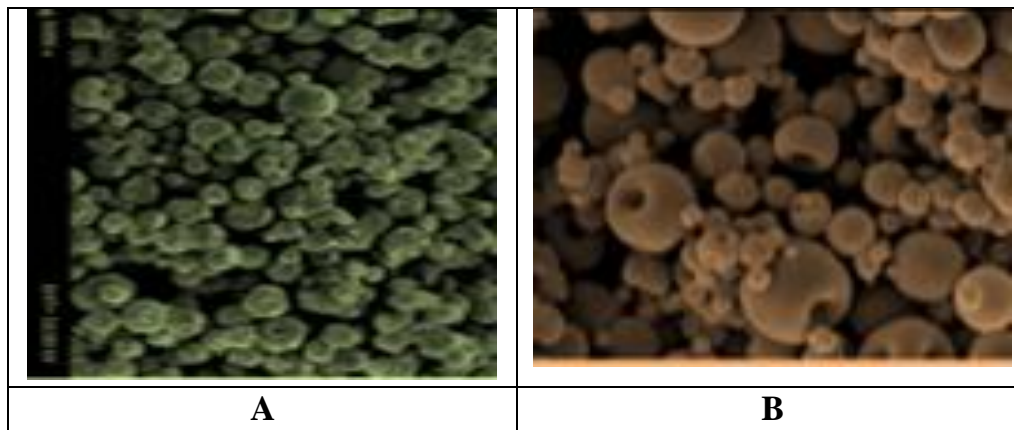
Production is checked by scanning electron microscope. The samples are fixed on the aluminum stubs of the device and then covered with gold (40): palladium (60). is prepared To measure the size of the particles, 0.10 grams of microcapsules are mixed with 0.5 milliliters of distilled water at room temperature, and then the size of the particles is measured with the help of a Zeta Sizer (19).

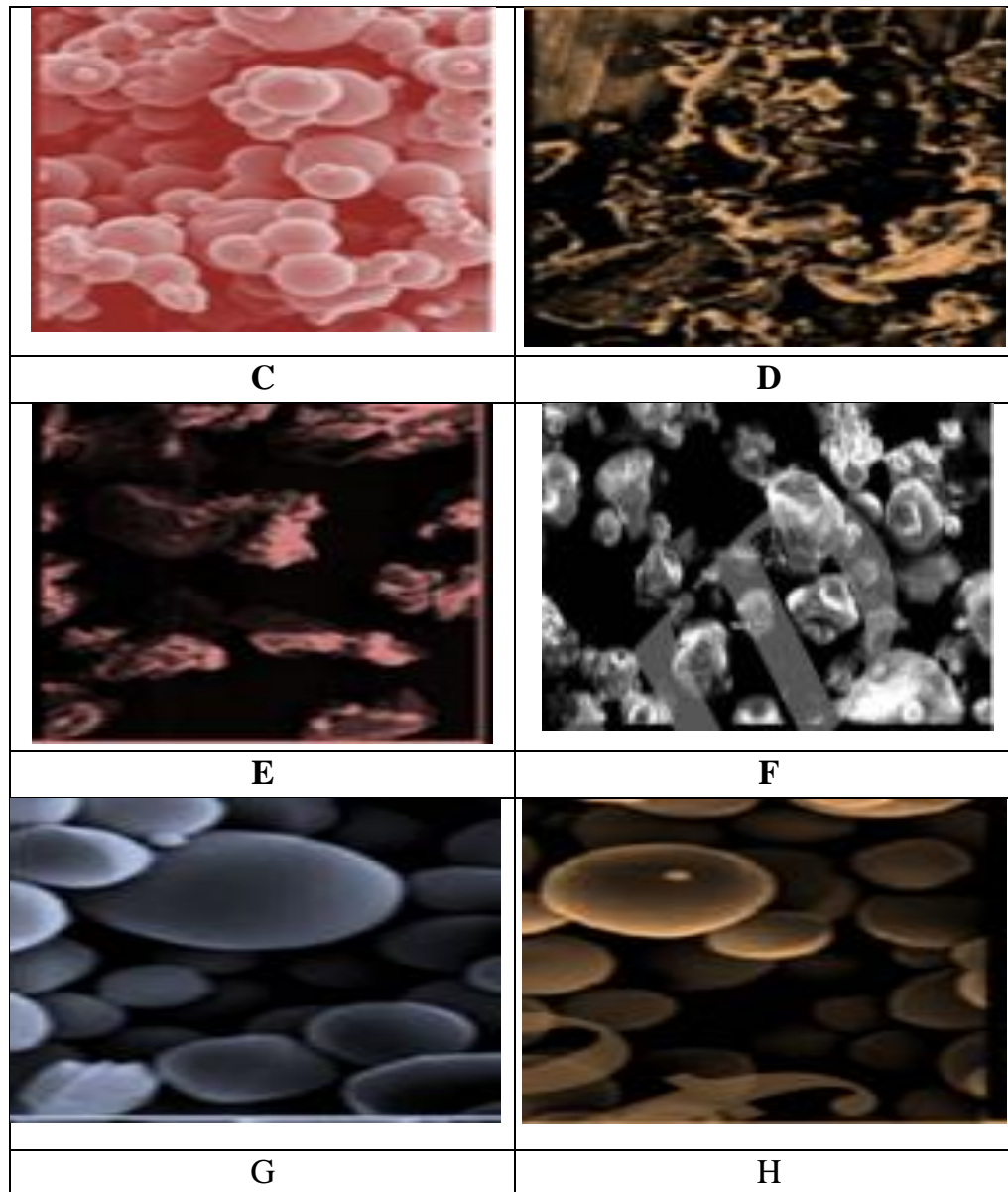
### 2-2- Discussing the morphology of microcapsules

Morphological studies of dried particles provide valuable information about the drying of particles and the chemical and physical factors affecting the structure of the particles. The surface morphology of the samples is checked by scanning electron microscope. Figure 1



shows the electron microscope images for each category of microcapsules. (18-21) In general, the produced microcapsules have spherical shapes and smooth surfaces, and in some samples, they have wrinkles, and in others, they have fractures and jagged edges. One of the characteristics of the external surfaces of microcapsules dried with a spray dryer is the presence of indentation (19). In a survey, a very small amount of wrinkling and indentation is observed on the surface of powders of spray drying treatments (Figures C-A). These powders are spherical, which is one of the characteristics The dried powders are in this way. Also, in some cases, the produced powders have pores, such a structure has also been observed in Rajam, (2004), Hakim & Stapley et al. (2012) and Swetank et al. (2015). The results of freeze drying treatments are fractured and jagged, and wrinkles can be seen on the surface, and the amount of wrinkles and indentation of the surface of the powders in these treatments is more than in other treatments, and many cracks and fissures can also be seen in these particles (Figs. (F-D1) The powders of these treatments are irregular, scale-like, have a lot of porosity and are crystal-like. Such a morphology was found for micro-coated powders through freeze drying in the study (2013) of Karthik & Swetank, Anandharamakrishnan (2015) and Errate et al. (2014). has also been reported.





**Figure 1 - Electron microscope images of microcapsules produced in different treatments (18,20,21,22)**



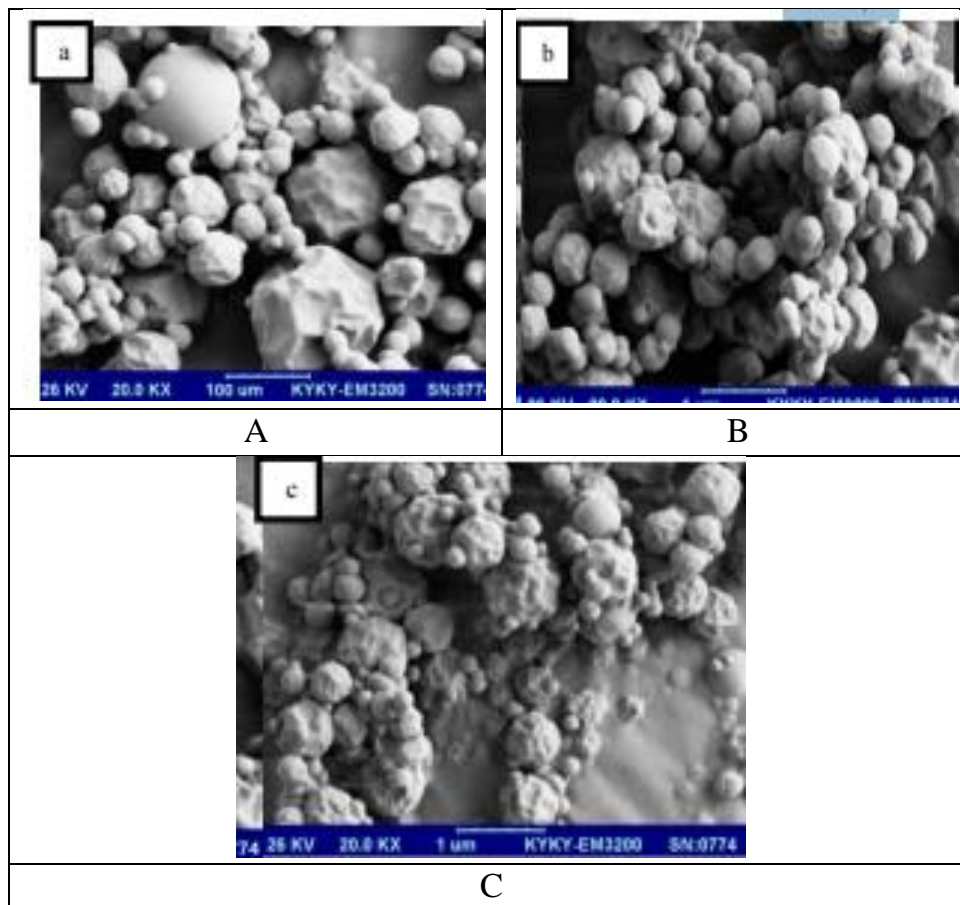
Images C-A of samples containing fish oil produced by the BetterTib spray drying method with fish gelatin walls, capaccarrageenan and a combined wall; F-D pictures of samples containing fish oil produced by the better-quality freeze-drying method with fish gelatin walls, capaccarrageenan and a combined wall; H-G images of samples containing fish oil produced by the BetterTib coacervation method with fish gelatin walls, capaccarrageenan and mixed walls

Based on these results, among the samples coated with gelatin, they have more wrinkles and uneven surface than other samples. In Cho et al.'s (2013) study, gelatin powders are spherical and very porous in shape, and their surfaces had wrinkles. The presence of wrinkles can be considered as a result of mechanical stresses caused by the non-drying of different parts of the liquid particles during the initial stages of drying, and The movement and movement of moisture causes the surface to swell and shrink (21). The holes are probably created in the final phase of drying and shrinking due to unevenness (20). caused by the expansion of microcapsules because in the final stage of drying, the temperature in the microcapsules increases and it is possible to cause cracks and cracks in the particles (25). Polymers with high molecular weight dry quickly and form surface films These films probably prevent the diffusion and diffusion of internal vapors and as a result of the creation of bubbles in the wall network, among the treatments, samples covered with gelatin have more wrinkles and uneven surface than other samples in the study of Cho et al. (2003). Gelatin powders are spherical in shape and very porous, and their surfaces had wrinkles. The presence of wrinkles can be considered as the result of mechanical stress, which is caused by the non-drying of different parts of the liquid particles during the initial stages of drying, and due to the movement and movement of moisture, it causes swelling and swelling or wrinkling of the surface. Holes are probably created in the final phase of drying and due to uneven wrinkling (16). Some researchers believe that the formation of central bubbles in particles dried with a spray dryer is caused by the expansion of microcapsules, because in the final stage of drying, the temperature in the microcapsules increases and it is possible to cause fractures and cracks in the particles (17). Polymers with high molecular weight dry quickly and create surface films, the creation of these films probably prevents the diffusion and diffusion of internal vapors, and as a result, the formation of bubbles in the wall network increases. Therefore,



it is expected that the rapid formation of the surface film expands the internal space of the microcapsules and causes more indentations in the treatments with mixed walls after wrinkling. Similar findings were reported by Peng et al. (2013) and Swetank et al. (2015) when they used carbohydrates in the wall composition.

One of the most important factors in the encapsulation of oils is the surface morphology of the capsules. In general, the more spherical the structure of the capsules and the smaller the gap in their surface, the better. This advantage shows that the penetration of gases and moisture into the capsule is less and the micro-coated oil is protected from oxidation to a greater extent. Figure 3 shows the scanning microscope image of 2 types of XA30, MIX40 nanocapsules, and 1 type of LG40 microcapsules. The efficiency of the microencapsulation process is dependent on creating a spherical structure and confirming the entrapment of the effective compounds of the core (24). The proper network and uniform coating of the wall around the capsules can be seen well. As can be seen in the figure, the obtained particles are all spherical in shape. No cracks or breaks were observed in the structure of nanocapsules. Various factors have an effect on the surface characteristics of the nanocapsules wall, among which we can mention the drying speed of the nanoemulsion, the composition of the wall and the production conditions of the nanoemulsion (when the drying temperature of the emulsions is high, the emulsions tend to remove moisture faster and As a result, a spherical structure is formed in the samples (25). Therefore, according to the obtained cell morphology, it can be ensured that all the moisture has been removed from the nanocapsules and microcapsules (27). Remaining inside the nanocapsules, their structure changes from smooth to rough (28 and 29), which is seen in the case of a partially sunken structure. (Similar morphology of nanocapsules showed that water melon extracts were coated with maltodextrin and gum arabic (30). Carneiro et al. It is smooth and has a shell without holes (24). The absence of holes in the structure of the capsules is considered a positive feature because it provides more protection to the encapsulated fish oil and increases the oxidative stability of the oil. The higher the permeability of the wall material, the faster the oxidation occurs (25).



Examining the surface morphology of nanocapsules shows that the nanocapsules prepared from the combined coating have a more uniform and smooth structure than the watercress gum alone, which indicates that the combined coating is more suitable for microcoating than the watercress gum alone. Therefore, it can be predicted that the combined nanocapsule will work better than other capsules in increasing the oxidative stability of fish oil. The results of the researchers' research have also reported an increase in the stability of safe capsules with smooth surfaces (29, 30). The creation of spherical structures with a uniform surface as a result of drying with a spray dryer has also been reported in the studies of Daly et al., (2021) and Karthik et al. (2013), Drasch et al. (2017), Gallardo et al. (2023) and Ferreira et al. (2016) observed similar structures for flaxseed oil, fish oil, flaxseed oil, and crude palm oil and reported that spherical



structures without Cracks have low permeability to gases, oxygen and unpleasant leakage of oil to the surface of particles (26,27,31,34).

### **3- Compound massification method**

#### **3-1-Method of preparation of microcapsules**

A 2% solution of gelatin and gum arabic biopolymer materials is prepared using deionized water. After preparing the aqueous gelatin solution at a temperature of 50 degrees Celsius, fish oil with material ratios of 2:1, 1:1, and 2:1 is added to the gelatin solution and homogenized at 9000 rpm for 3 minutes. Using a homogenizer, an oil-in-water emulsion is obtained. Then, solutions such as gum arabic are added drop by drop to the emulsion and its pH is adjusted to 4 using glacial acetic acid 50% by volume. After the formation of the complex, the prepared microcapsules were kept under rotation of 800 rpm and a temperature of 50 °C for 15 minutes. Next, the temperature of the emulsion is slowly reduced to 4-7°C and stirring is stopped. The formed microcapsules are separated using filter paper and frozen at 60°C for 24 hours and then made in a Hetoholten freeze dryer. Denmark is dried at a pressure of 0.08 millibar and a temperature of 38 degrees Celsius for 48 hours. The resulting powder is milled and sieved (mesh 100) and packed in moisture-proof plastic (39).

#### **- Shape and structure of microcapsules in emulsion**

The morphology of the particles in the emulsion before freeze-drying is observed using an Olympus CH optical microscope made in Japan (37).

Scanning Electron Microscopy (SEM) The powder is spread on one side of a double-sided adhesive tape and the other side of the adhesive tape is glued on a special metal plate. The fixed sample in the vacuum chamber is covered with gold atoms and its examination is done with a scanning electron microscope (Philips, made in the Netherlands) (38).



## **- Determination of surface oil of microcapsules**

2 grams of the produced microcapsules powder is mixed with 15 ml of hexane at room temperature for 2 minutes. The washed microcapsules are filtered between Whatman No. 1 filter paper and in order to minimize the surface oil on the microcapsules, the remaining powder is washed 3 times with 20 ml of hexane each time. The filtered solution containing the extracted surface oil was placed in an oven at a temperature of 70 degrees for 6 hours in order to completely evaporate the hexane (28).

## **- Determining the efficiency and oil of microcapsules**

The efficiency is calculated based on the difference between the surface oil and the total oil compared to the initial total oil in the emulsion, and the amount of oil in the microcapsules was obtained based on the ratio of the amount of microencapsulated oil to the weight of the microcapsules (10).

$$EE = \frac{W}{W_2} * 100$$

EE: microencapsulation efficiency

Oil content: The amount of oil in microcapsules

: W weight of microcapsules Oil content=  $\frac{W}{W_2} * 100$

W: The amount of finely coated oil

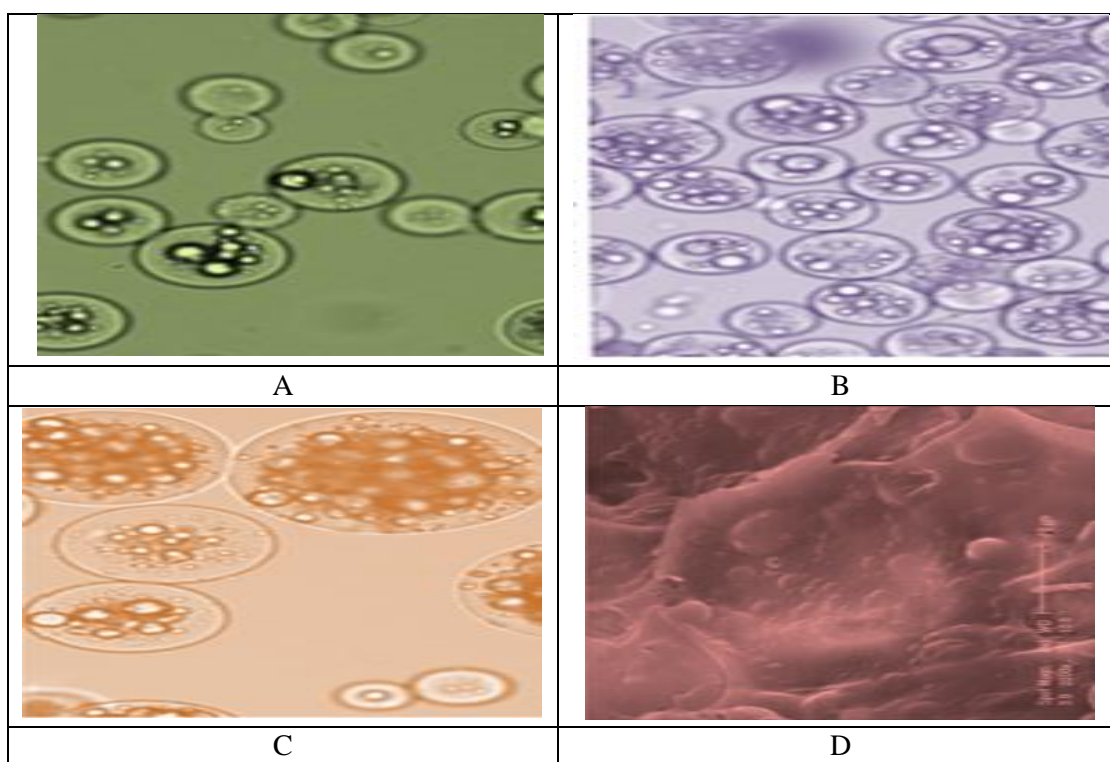
: W<sub>2</sub> amount of primary oil in the emulsion

## **2-3-The shape and structure of microcapsules in emulsion**

Based on the research and the photos taken with the light microscope, the shapes of the 2 microcapsule particles are observed as spherical and multi-nuclear. Fish oil is concentrated in the center of the microcapsules, and with the increase in the ratio of the wall material of the central material from 12 to 1:2, the number of multi-core microcapsules, as well as the size and amount of loaded oil, gradually increases (40). A



photograph taken with a scanning electron microscope (b) of microcapsules with a 1:1 ratio of wall material to central material after freeze-drying shows spherical shapes and a smooth surface of the microcapsules. These results show that when Dong et al. in 2011, peppermint essential oil It can be seen that they were covered by the method of compound Ishdan mass using gelatin and gum arabic (34).



**Figure 2- Examining the morphology of the particles in the emulsion and after freeze-drying (41-44)**

According to research (27), at lower concentrations of oil, less central material is available and the probability of producing empty microcapsules increases, so the microcoating efficiency decreased according to Duncan's test by changing the ratio of wall central material from 2:1 to 1:1. In terms of efficiency, there is no significant difference at the 0.05 level. While the ratio of 1:2 has a significant difference with other ratios in terms of efficiency at the level of 0.05. In this study, using the variance analysis method, 26 F was obtained for the efficiency variable of 23,840. In the ratio of wall



material to (central) material 1:1, enough biopolymer material is available in the system for micro coating of fish oil, and the wastage of oil is reduced and finally the efficiency of micro coating is increased. Therefore, the critical concentration of wall and central material in order to have maximum efficiency is 1:1 ratio. Cheng et al. (2006) coated camphor oil using this method. Based on their results, increasing the ratio of core to wall material decreases efficiency. Found .In these studies, the maximum amount of oil in the ratio of wall material to central material (2:1) and the value of 53.100.11 and the minimum amount of oil in the ratio of 1:2 with the value of 0.005 and 20.3 were obtained. All 3 groups of prepared microcapsules had a significant difference with each other (0.05) based on the method of variance analysis, the value of 26, 17.397 is determined with the increase of oil concentration, the amount of oil in the microcapsules increased. By increasing the percentage of oil in the formulation, the effect of the dispersing force of the stirrer decreased and larger microcapsules were formed. Maji et al. (2007) when they increased the amount of wall material, all gelatin and gum arabic were used to form the wall. Therefore, the wall thickness of microcapsules and the size of particles increased and the oil of microcapsules decreased. Yu et al. (2012) coated olive oil using this method. Based on their results, with the increase in the ratio of wall material to central material from 1:1 to 2:1, there is no significant change in the amount of oil, while in the ratio of 2: 3. The amount of oil in microcapsules decreased to 35.92%. The key conclusion of this research is that fish oil is coated using the method of agglomeration of gelatin and gum arabic. The prepared microcapsules are observed using a light microscope before drying. Microcapsules have spherical shapes and multi-core structure. With the increase of oil concentration, polynuclear particles increased. The surface of the dried microcapsules after freeze-drying have spherical shapes. The effect of the ratio of the wall material and the central material on the efficiency and oil amount is investigated. Based on the results, increasing the amount of oil from 11 to 2:1, the efficiency decreased and the amount of oil in the microcapsules increased...

#### **4-Discussion and conclusion**

The unpleasant taste and smell of fish oil reduces and destroys the quality of edible products. One of the suitable methods for protecting fish oil is micro-coating (11). Microcoating is a process in which very small particles, drops or bubbles are coated. In



order to prevent the tongue from detecting the taste or changing the texture of food, the size of finely coated oil particles should be less than 100 micrometers. Microcoating of omega-3 fatty acids in suitable wall materials can preserve the nutritional value and sensory characteristics of these materials. Spray drying, coacervation and extrusion are the main and common technologies in the microcoating of lipophilic compounds. One of the most important parameters in increasing the efficiency of micro-coating is paying attention to the wall material. According to the studies of microencapsulation efficiency, the amount of peroxide, size, density and moisture level of fish oil microcapsules are greatly influenced by the composition of the wall material, and the amount of solid material, viscosity and particle size are also affected by it. Therefore, the main step in the microencapsulation of food ingredients is to choose the appropriate composition of the wall material and it is from a wide range of natural and synthetic polymers. The use of chitosan, sugar beet pectin, sodium caseinate, gelatin, maltodextrin, starch, whey protein, gum of different plants, etc. are used alone or in combination for microencapsulation of fish oil. Despite the various studies that have been done on different combinations of wall material and different methods of micro-coating; Few studies have been done on the use of carrageenan in the microcoating of oily fish. Most of the studies have used carrageenan in the microcoating of probiotics and this microcoating causes the stability of probiotics. In another study, carrageenan is used as a wall in the microencapsulation of aroma, and it is clear that this wall has the potential of microencapsulation of aroma and its use in food formulations. There has been no study on the combination of carrageenan and fish gelatin. These two wall compositions of marine origin are selected for micro coating of oily fish. In this research, in addition to the study of the wall material, 3 different microcoating methods (spray drying, freeze drying and coacervation) have also been studied. The effect of using different wall materials and microcoating methods will be investigated by measuring the characteristics of the produced powders.

In this study, the physicochemical properties of microencapsulated fish oil were investigated using wall materials and different microencapsulation methods. Fish oil is examined by 3 different methods of coacervation, spray drying and freeze drying to produce powders, and to check the physicochemical properties, the parameters of moisture content, total oil content, surface oil content, microcoating efficiency, color and



morphology of microcapsules are examined and measured. The results of the investigation of the physicochemical factors of the produced powders show that fish gelatin + maltodextrin is the best wall composition and the coacervation method is the most suitable method for microcoating fish oil. Microcoating with these materials and this method is able to produce powders with higher microcoating efficiency and lower surface oil content than other treatments. The results show that the microcapsules produced by the coacervation method are more spherical and larger than other treatments. The comparison of spray drying, freeze drying and coacervation methods confirms that wall composition, drying temperature, microcapsule morphology and process time are actually the most important factors that affect the stability of oily fish powder.

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