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Evaluation of quality properties of grape pomace and flaxseed oil microcapsules stabilized with different ratios of maltodextrin and gum tragacanth

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ABSTRACT

Encapsulation protects bioactive compounds against unfavorable environmental conditions and increases their stability and bioavailability. Regarding the existence of bioactive compounds in grape pomace and flaxseed oil, the aim of this research was to encapsulate of grape pomace (*Vitis viniferae cv. Rash*) and flaxseed oil with different ratios of maltodextrin and gum tragacanth (1:1, 1:2, 2:1) using a spray drier and evaluate of the quality properties of the obtained microcapsules. The produced microcapsules were analyzed in terms of microencapsulation yield, solubility, humidity contents, total phenolic contents, antioxidant activity, interaction between the wall and core materials, and microstructure. According to the results, there were no significant differences between the microcapsules in terms of humidity and solubility contents ($P<0.05$). The highest microencapsulation yield, total phenolic content and antioxidant activity were observed in microcapsules produced with equal ratios (1:1) of maltodextrin and gum tragacanth. While a spherical shape was observed by scanning electron microscopy for all microcapsule samples, the microcapsules produced with equal ratios (1:1) of maltodextrin and gum tragacanth had a more compact and homogeneous structure. The FT-IR analysis also confirmed the creation of new interactions between the wall and core materials of the microcapsules. The results of the present study showed that using maltodextrin and gum tragacanth as wall materials in an equal ratio (1:1) had a proper performance in the microencapsulation of grape pomace and flaxseed oil.

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1. Introduction

In today's societies, the tendency to produce and consume a variety of beneficial foods in order to improve the health of people in a society is one of the important indicators It is considered the progress of societies and refers to products containing some health-giving compounds that have nutritional properties beyond the basic foods that are commonly consumed, whose important role is to increase human health [1].

The addition of effective ingredients should not cause disturbances in food metabolism and the occurrence of side effects. These substances must be biologically obtainable from the food and can be consumed as a part of the daily diet and also obtained from natural substances, and the addition of effective substances should not change the quality characteristics of the food such as taste, color and smell or lead to a decrease in the shelf life of food [2]. Some of these substances are highly sensitive to heat, oxidation and other environmental factors, for this reason, in addition to controlling the added amounts, they should be evenly distributed throughout the food [3]. The most important of these compounds are essential fatty acids, vitamins, carotenoids, sterols and flavonoids [2].

hair with scientific name (*Vinifera L.*) is a plant from the genus*Ampeliaceae*, which most botanists call it*Vitaceae* have named The grape family has 14 genera, of which 12 are living and 2 are fossil and have 700 species. Among them gender*Vitis* It is the most important and the only genus that has edible fruit, 60 species and 10,000 varieties have been named [4].

Sardasht black grapes*Vinifera cv. Rash*) is one of the grape varieties that grows naturally in the mountainous areas of Sardasht city. This variety is late and dry, and the shape of the grape bunches is conical, the appearance color is blue to black, and it has a sweet and aromatic taste [5]. Harvested grapes are mainly used to produce grape juiceMeanwhile, a large amount of this valuable product is lost as waste. Grape pomace is one of the valuable by-products of juice and concentrate factories. Pectic substances are the main polymers forming the cell wall in grape pomace. Sardasht black grape pomace is a cheap source of relatively large amounts of sugar (mainly glucose, fructose and sucrose), tartrate, polyphenols (mainly anthocyanins, flavonols, flavanols, phenolic acids and resveratrol), vitaminC, beta-carotene, crude fiber, vitaminsAND and citric acid, which has anti-cancer, anti-inflammatory, antioxidant and antiviral properties, and their optimal extraction can bring economic benefits [7, 6].

Bezerk with a scientific name*The most common linen* From the flax family*Linaceae* It is a one-year plant that grows in the form of bushes, its seed morphology is oval, smooth and pointed at the end, and its origin is reported in the western Mediterranean [8,9]. Flaxseed oil contains triglycerides, phospholipids and amounts of free fatty acids. The extracted oil is a good source of essential fatty acids such as alpha-linolenic acid (omega-3), linoleic acid (omega-6) and oleic acid (omega-9) [10] and also a source of essential amino acids such as arginine, histidine, glutamine, cysteine, and lignan. phytoestrogenic components), dietary fiber[1], minerals and vitamins [11]. Flax seed oil is an alternative plant source of omega-3, which contains more than 50% of alpha linoleic acid. Among the health-giving properties of linseed oil, due to the presence of high amounts of omega-3 in this oil, it can be attributed to their positive role in improving heart diseases.– He mentioned vascular, breast, prostate, skin and colon cancers,

[1] Dietary fiber

osteoporosis joint problems, migraines and all kinds of headaches, stress and some other diseases [12].

Awareness of the potential benefits of omega-3 unsaturated fatty acids has strengthened the interest in food enrichment with these fats, however, the inclusion of omega-3 fatty acids in food products has caused major challenges in formulation. Many fats are sensitive to heat, light and oxygen and oxidize quickly. Oxidation of fatty acids is one of the main factors of food spoilage and can affect the taste, aroma, texture, shelf life and color of food, which limits the use of sources containing omega-3 fatty acids in the enrichment of food products.]13[. The main problem related to the consumption of omega-3 polyunsaturated fatty acids, both medicinally and nutritionally, is their high sensitivity to oxidation. The main substrates for the lipid oxidation reaction are unsaturated fatty acids with one or more double bonds]14[. Considering that phenolic compounds and omega-3 fatty acids, if used directly in food products, can have adverse taste and sensory effects on the product, therefore, the use ofMicrocoating technology It is necessary to protect these compounds inside the product.

Microencapsulation is a technique in which the core materials (food compounds, essential medicinal plants, enzymes, cells, nano-medicines, microorganisms, temperature-sensitive materials, etc.) are placed inside a thin membrane (biopolymer compounds such as carbohydrates, fats, and proteins). They find that this causes a decrease in the reactivity of the core with environmental factors, a reduction in the speed of transfer of materials inside the core to the external environment, increasing the possibility of easier transfer and transfer, controlled release of materials at the desired time, masking the taste of the core, uniform dispersion of the core throughout Mixing and preventing the reaction between core components and other food components [15]. Also, this method can be used to increase the stability of bioactive compounds and protect them against adverse environmental conditions in order to increase bioavailability [16].

There are various methods for the microcoating process, which fall into two main categories: mechanical methods and chemical methods. One of the mechanical methods is spray drying[2]Spray cooling[3]Freeze drying[4], coating by fluid bed method[5] and extrusion[6] And one of the chemical methods can be locked in a liposome[7], named emulsion or two-phase agglomeration system [17].

Spray drying usually involves dissolving, emulsifying or dispersing the core in an aqueous solution of carrier material and then grinding.[8] And the resulting mixture is sprayed into a chamber containing hot gas. Spray-dried capsules due to porosity and solubility. Above the wall, they release their active agent immediately after adding water. However, recently, by using hydrophobic coatings or substances that create cross-links (denatured proteins, proteins or biopolymers with cross-links), the possibility of gradual release of active substances in water has been provided [18].

[2]- Spray-drying
[3]- Spray-chilling/cooling
[4]- Freeze drying
[5]- Fluid bed coating
[6]- Extrusion
[7]- Liposome entrapment
[8]- Atomize

Choosing the right wall is essential to achieve a successful inlay. Carbohydrate wall is often used in the microcoating of sensitive materials, which is due to its high ability to absorb volatile compounds and also to firmly bind them during the process [19]. In this research, maltodextrin and katira were used as carriers. Maltodextrins that are oligomers or polymersDGlucose with 1 to 4 alpha bonds and usually have less than 20 dextrose equivalents. Also, they are completely soluble carbohydrates with low volume density, moisture absorbent and high viscosity, which has a very important role in their practical properties [20]. Among the common carrier materials used in spray dryers are maltodextrins. This combination has the ability to create a network in different microcoating processes and also the microcoating efficiency obtained by this combination has been reported above [21]. Maltodextrin has a low viscosity in high concentrations and has a low price. Their availability is one of the important factors in the use of these compounds in various microcoating processes, and the appearance of these compounds is white powder [22]. Maltodextrin lacks interfacial properties required for high encapsulation efficiency. Therefore, to solve this problem, it is usually used in combination with other biopolymer materials such as gums, modified starches and proteins such as sodium caseinate and whey concentrate [23]. Ketira is a high-quality hydrocolloid, which in terms of chemical structure is a non-uniform and highly branched hydrophilic carbohydrate. Ketira consists of two main components called tragacanthic acid[9] or Basorin[10] and tragacanth[11] Is. Basorin 70– It accounts for 60% of the total gum and is an insoluble component in water that has the ability to swell and form a gel. Another component, or tragacanthin, is also dissolved in water and leads to the creation of a colloidal solution. Basorin is an acidic component that produces sugars such as D– Xyloz, L– Fucose di-galacturonic acid and a small amount of L– Produces rhamnose. The acidic component of this molecule is related to calcium, magnesium and potassium cations. The properties of Katira gum are largely related to basorin. Tragacanthin is considered as a neutral polysaccharide whose structure consists of consecutive digalactose units and L-arabinose branched chains are attached to it, and methoxyl groups are also present in its structure in a significant amount [24]. There are several reports on the use of maltodextrin for the encapsulation of bioactive compounds [26, 25].

Faraq et al. (2018) studied the phenolic compounds of grapes using whey protein concentrate and maltodextrin as a carrier.**micro cover**they did Highest efficiency**background** In the ratio of 40/60 whey protein concentrate to maltodextrin was obtained and the smallest size was obtained**background**(82.26 nm) was observed in the sample in which only whey protein was used as a carrier. Also, capsules containing 10% of whey proteins released 83% of phenolic compounds [27]. In the research conducted by Lawley and Harsha (2018), hydrogen alginate for microencapsulation of grape skin phenolics as a release agent dependent onpH used. Calcium chloride was used as a hardening agent. At a pH equal to 1.4, microcoatings were healthy and 13% of total phenolics were released from microcoatings, and the release percentage of phenolic compounds depended on the type of chemical structure of the compound. ProcyanidinB174%, catechin and epicatechin had 50% release. While the release of anthocyanin and flavonol was less than 11%. At a pH of 7.4,

[9]- Tragacanthic acid
[10]- Bassorin
[11]-Tragacanthic

the microcapsules dissolved and created a viscous solution [28]. Mahdavi et al. (2016) investigated the optimization of microencapsulation of anthocyanins with maltodextrin, gum arabic and gelatin by freeze drying method. According to the results, the highest efficiency and the best quality of the powder was related to the combination of maltodextrin and gum arabic walls with a ratio of 35% of the core to the wall and a ratio of 3 to 1 of the walls and an efficiency of 94.39% [29]. Gallardo et al. (2013) investigated the microencapsulation of linseed oil using the spray drying process and different ratios of gum arabic, maltodextrin, methyl cellulose, whey protein isolate with the aim of obtaining different formulations with high microencapsulation efficiency. According to the results, the microcoatings formulated with 100% gum arabic and the microcoating containing a mixture of gum arabic, maltodextrin and whey protein isolate provided the highest protection against oxidation and the microcoating efficiency was over 90% [30].

Flaxseed oil contains high amounts of omega-3 fatty acid, which is highly sensitive to oxidation due to the unsaturated nature of the said compound. Therefore, the simultaneous use of linseed oil with Sardasht black grape pomace, which contains high amounts of natural antioxidants, can be effective in delaying the oxidation reaction of linseed oil. At the same time, microcapsules containing phenolic compounds found in grape pomace (epicatechin, catechin, gallic acid, procyanidins and phenolic acids) and essential omega-3 fatty acid found in linseed oil (alpha-linolenic acid) can be useful in the formulation of food products. be used The purpose of this research is to evaluate and compare the ability of different ratios of two types of wall materials (Katira gum and maltodextrin) for the simultaneous microcoating of Sardasht black grape pomace and linseed oil using the spray drying method. Structural and morphological properties of microcoatings by means of analyses FT-IR AndWHICHwere studied.

2- Materials and methods

2-1- Preparation of grape pomace and linseed oil

In order to carry out this research, Sardasht black grapes of Khushnav variety were obtained from West Azerbaijan Agricultural Research Center and transferred to the laboratory of Herbarium Department, Faculty of Pharmaceutical Sciences, Tehran Islamic Azad University of Medical Sciences for further studies. After dewatering the samples, the resulting sludge is dried using a freeze dryer (Dorsa-DFR-02) was dried and subjected to chemical analysis. Flaxseed oil was obtained from the specialized plant oil production center of Adonis Gol Daro Company.

2-1-1- Preparation of extract

In order to perform chemical tests, an extract was prepared from grape pomace. One gram of the samples was mixed with 100 ml of 60% ethanol and kept under constant stirring with a magnetic stirrer for 30 minutes in a light-free environment.Heidolph, Germany) it placed. The resulting mixture was centrifuged (4500×, 20 minutes) (Pole ideal tajhiz, Iran) was placed and the upper phase was used for analysis [31].

2-1-2- Analysis of phenolic compounds of grape pomace

The extract was filtered with a nylon filter of 0.45 micron and injected into HPLC (Waters, Alliance system, USA) and used for analysis by reverse phase column RP18 and UV detector at 210 nm. The mobile phase includes solvent A (water completely acidified with 1% phosphoric acid) and solvent B (acetonitrile) using the gradient method and starting with 90% of solvent A and then adjusted as follows:

60% of solvent A in 5 minutes, then 90% of solvent B in 45 minutes, and the injection volume is 10 microliters and the flow rate is 0.5 milliliters per minute. Room temperature to do The test was considered 25 degrees Celsius and also from the softwareChrome gate was used for analysis [32].

2-1-3- Measurement of the total amount of phenolic compounds of the extract

300 microliters of the extract was mixed with 100 microliters Folin Ciocaltio reagent for 5 minutes. Next, 300 microliters of saturated sodium carbonate was added to it and the mixture was kept at room temperature for 30 minutes. Then at 750 nm by spectrophotometer (Uinico, UV/Vis 4802, United states(was read and the result was reported in terms of mg equivalent of gallic acid per 100 grams of dry matter) [33].

2-1-4-Measurement of radical scavenging capacityDPPH extract

100 µl of extract with 3 ml of methanolic solutionDPPH It was mixed and kept for 30 minutes in the dark at room temperature. After the desired time, absorbance at 515 nm by spectrophotometer (Uinico, UV/Vis 4802, United states) read. The amount of extract (mg/ml) required to inhibit 50% ofDPPH(IC50) It was determined by using the graph of concentration against inhibition percentage and from 0.1 ml of methanol and 3.9 ml of methanolic solution.DPPH was used as a witness [34].

2-2- production of micro cover

Aqueous solutions of maltodextrin and katira gum in different proportions were prepared separately by mixing with distilled water using a magnetic stirrer. Mixing and preparation of solutions was done in 30 minutes with the help of a magnetic stirrer. Then, to ensure the complete saturation of biopolymers, the walls were prepared one day before coating and kept in the refrigerator for 24 hours. The production steps were carried out as described in figure 1 and according to table 1 [35].

Figure 1- Microcapsules production steps.

Table 1 - Microcapsule composition

Formula	1	2	3
Grape extract (g)	0.125	0.125	0.125
Flaxseed oil (g)	0.125	0.125	0.125
Citric acid (g)	3.5	3.5	3.5
Gum tragacanth (g)	0.5	0.5	1
Maltodextrin (g)	0.5	1	0.5
Branched chain amino acid (g)	4.7	4.7	4.7
Tween 20 (µl)	20	20	20

2-4- quality control tests of micro-covering

2-4-1- Measuring the total amount of phenolic compounds of the microcoating

For this purpose, first, 0.3 grams of each sample was mixed with 30 ml of deionized water and brought to pH 4.3, 5.5 and 7 using 0.1 sodium bicarbonate and lactic acid, and to separate the supernatant phase in a centrifuge. (7000x, 17 minutes) (Uinico, UV/Vis 4802, United states) Was placed. Next, 500 microliters of the supernatant was mixed with 500 microliters of Folin Ciocaltio reagent and 300 microliters of saturated sodium carbonate, and after 30 minutes, the absorbance at 760 nm was read in terms of gallic acid equivalents in 100 grams of dry matter [36].

2-4-2-Measuring the antioxidant activity of microcoating

10 microliters of the supernatant obtained from the previous step was mixed with 3.9 ml of methanolic DPPH solution and after 30 minutes of storage at room temperature and darkness, the absorbance was read at 515 nm. The amount of sample (mg/ml) required to inhibit 50% of DPPH (IC50) was determined using the graph of concentration versus inhibition percentage, and 0.1 ml of methanol and 3.9 ml of DPPH methanolic solution were used as controls.]34[.

2-4-3- Efficiency of micro-coatings

Measuring the efficiency of microcoatings based onThe total weight of the product is the weight of the feed given to the spray dryer system (Buchi mini spray dryer B290, Switzerland, with a double atomizer nozzle with a diameter of 0.7 mm, inlet air temperature 140 °C, outlet air 70 °C, air flow rate 40 cubic meters per hour and feeding flow rate was 5 ml/min) and the following equation was used for calculations]35[.

$$y = \frac{warm\ to\ mass\ micro - encapsulation}{grams\ to\ the\ device\ to\ the\ entrance\ of\ the\ solution\ of\ the\ total\ mass} \times 100$$

2-4-4- Humidity level

The samples of micro-coatings prepared in an oven (Shimaz co, Iran) were kept at a temperature of 105 °C until reaching a constant weight and the moisture content was calculated [37].

2-4-5- The amount of solubility

0.5 grams of each sampleThe microcapsule was placed with 5 ml of distilled water at room temperature on a magnetic stirrer (Heidolph, Germany) at 220 rpm for 30 minutes, then centrifuged at 1400 g for 5 minutes.(Pole ideal tajhiz, Iran) became. The resulting sediment was transferred to pre-weighed containers and reached a constant weight and was placed in an oven (Shimaz co, Iran) at 105 °C until reaching a constant weight [38].

$$Solubility\ percent = \frac{It\ is\ dried\ and\ dissolved\ in\ microcoating\ weight}{Microcoating\ of\ primary\ weight\ powder} \times 100$$

2-4-6- Material interactionwall, core and core with wall by FTIR[12]

To determine the mutual effect and interaction between the walls and the core from FTIR (Bruker Tensor 27) in the wavenumber domain.$^{-1}$ cm 4000-400 use became [39].

2-4-7- Examination of the microstructureMicroscopes with SEM[13]:

In order to examine the microstructure of the samples and the distribution of the particles, the samples were covered with a thin conductive layer of gold and then using a scanning electron microscope (EM-8000, KYKY, China) in an accelerating voltage from 10 to 20 kW observations were made [40].

[12] - Fourier transform infrared spectrometry

[13] - Scanning Electron Microscope

5-2- Statistical analysis

In this study, the effect of different proportions of maltodextrin and gum arabic on the qualitative properties of microcoatings was investigated based on a completely randomized design with three replications. Variance analysis and comparison of averages respectively by methodANOVA and Duncan's multiple range test by softwareIBM SPSS Statistics (Version 23) was done. To compare the mean, the least square mean method was used at the 5% probability level.

3. Results and Discussion

3-1- Identifying the phenolic compounds of Sardasht black grape pomace by high-performance liquid chromatography

CFigure 2 shows the phenolic compounds present in Sardasht black grape pomace. The highest amount was related to gallic acid, followed by caffeic acid, ferulic acid and salicylic acid. Cui et al. (2023) investigated the effect of steam explosion on phenolic compounds and dietary fiber of grape pomace, and the highest amount of these compounds was related to gallic acid, syringic acid, vanillic acid and caffeic acid [41]. Rodrigues et al. (2023) investigated the phenolic compounds of aqueous, ethanolic, methanolic, acetone and butanol extracts of grape pomace. , chlorogenic acid, vanillic acid, caffeic acid and synergic acid were reported [42]. Borin et al. (2014) extracted the bioactive compounds of red and white sorghum under different conditions using different solvents. The results showed that extraction with ethanol had the highest antioxidant activity compared to other solvents. HPLC results showed that gallic acid and cinnamic acid were the main phenolic compounds in grape pomace [43]. Metini et al. (2017) identified the highest amount of phenolic compounds in the skin of Sardasht red grapes as gallic acid, catechin, vanillic acid and salicylic acid [44].

Table 2-Phenolic compounds present in Sardasht black grape pomace extract

Phenolic compounds	Retention Time (min)	Concentration (mg/100 Dry g GP)

gallic acid	21.387	278.72 ± 0.01
Caffeic acid	34.674	144 ± 0.04
Ferulic acid	42.326	52.3 ± 0.08
Salicylic acid	47.336	35.45 ± 0.03

2-3- Investigating the characteristics of the finely coated powder of Sardasht black grape pomace and linseed oil

The results related to the analysis of some physical properties of microcoated powder including moisture, solubility, microcoating efficiency are given in Table 3.

3 samples of finely coated Sardasht black grape powder and linseed oil were analyzed for moisture content. The ratio of maltodextrin walls to Katira gum was 1:1, 2:1 and 1:2 respectively. According to the table, although the highest moisture content was related to the microcoated sample of Formula 2 (4.86%), it was not statistically significantly different from the other two samples. Considering that according to the results obtained from the related researches that determine the acceptable moisture content for products dried with a spray dryer is less than 5% (weight/weight), it can be concluded that all the samples produced in This study has suitable levels of humidity [45]. In the research conducted by Mahdavi et al. (2016) they also showed that the extracted anthocyanin extract of barberry coated with maltodextrin wall (3.07 percent) and maltodextrin and gum arabic (3.47 percent) had no significant difference in terms of moisture content. [46]. Also, according to the presented results, different proportions of maltodextrin and kathira gum had no significant effect on the solubility and efficiency of microcoatings. Sharifi et al. (2015) also pointed out the lack of significant effect of increasing the percentage of maltodextrin on increasing the solubility of powders [47]. The results of this research showed that the efficiency of covering grape pomace and linseed oil is in the range of 69-78% and it is significantly dependent on different proportions of maltodextrin and gum arabic. So that the microcoating is prepared with a 1:1 ratio of maltodextrin and gum Ketira had the highest efficiency in covering. The operating conditions of the spray dryer, the type of carrier and their ratio, as well as the wall-to-core ratio are among the factors affecting the efficiency of the coating [45].

Table 3 - Properties of microencapsulated powder of Sardasht black grape pomace and flaxseed oil

Formula	Moisture content (%)	Water solubility index (%)	Encapsulation yield (%)
1	4.17±0.83[a]	66.58±2.1[a]	78.56±0.13[a]
2	4.86±0.35[a]	67.32±2.1[a]	74.70±0.12[b]
3	4.75±0.95[a]	67.18±2.7[a]	69.66±0.11[c]

1- Values are reported as mean ± SD
2- Different lower case the significant difference (P<0.05) between samples

3-3- The content of phenolic compounds

The table shows the total phenol content of microcoatings prepared with different wall materials at pH 4.3, 5.5 and 7. The initial total phenolic content of Sardasht black grape pomace before encapsulation in microcapsules was equal to 1637.08 mg of gallic acid per 100 grams of dry matter. The total phenolic content of all samples is lower than the initial amount in unencapsulated grape pomace. This means that even spray drying as a drying method leads to the destruction of a small fraction of phenolic compounds. According to Table 4, the investigation of the effect of pH on the content of phenolic compounds showed that at pH 7, 5.5 and 4.3, grape pomace and linseed oil coated with a 1:1 ratio of maltodextrin and kathira gum had the highest stability. This means that the 1:1 ratio of maltodextrin to kathira gum protects the contents inside the microcapsules against pH reduction. A decrease in pH from 7 to 5.5 and 4.3 leads to a significant decrease in phenolic compounds in all microcoating formulas, so that the highest content of phenolic compounds was observed at pH 7 and in samples with formulas 1, 2 and 3, respectively. The possible reason for this can be related to the effect of changing hydrogen ion concentration on changing the mechanism of DPPH radical inhibition process by phenolic compounds [48]. Tolun (2020) investigated the effect of different compounds in the microcoating process and relative humidity on the stability of microcoated grape pomace extract. The aim of this work was to increase the storage stability of polyphenols obtained from grape pomace using microcoating method based on spray drying. The most stable microcoatings were obtained with maltodextrin DE4-7 prepared by adding gum arabic to the wall material at a ratio of 8:2. The loss rate of phenolic content was in the range of 0.93-5.42% depending on the phenolic composition [35]. In the research conducted by Lawley and Harsha (2018), hydrogen alginate was used to encapsulate the phenolic compounds of grape skin as a pH-dependent release agent and calcium chloride was used as a hardening agent. At a pH of 1.4, the microcoatings were healthy and 13% of total phenolics were released from the microcoatings, and the release percentage of phenolic compounds depended on the type of chemical structure of the compound. Procyanidin B was released by 174%, catechin and epicatechin by 50%. while the release of anthocyanin and flavonol was less than 11% [28].

3-4- Antioxidant activity

Table 4 shows the results related to changes in antioxidant activity and the comparison of average data related to changes in antioxidant activity of grape pomace and linseed oil coated with spray drying method, as well as two substances carrying maltodextrin and gum kathira with different ratios. Antioxidant activity based on the DPPH radical scavenging power of microcoatings was similar to the trend of total phenol content. The antioxidant activity of Sardasht black grape pomace was reported to be 25.26% in the unencapsulated state, which was higher than the antioxidant activity of the microcapsules. Based on the results of the analysis of variance obtained in this research, it was found that the antioxidant activity of microcoated grape pomace and linseed oil was significantly dependent on the type and ratio of the carrier used for coating. According to Table 4, respectively, the highest and lowest antioxidant activity belongs to the microcoated sample with a ratio of 1:1 at pH 7 (3.21%) and the microcoated sample with a ratio of 1:2 maltodextrin to ketiradar gum at pH 4.3 (92 /10 percent). Also, the effect of reducing pH from 7 to 5.5 and 4.3 caused a significant decrease in antioxidant activity in all formulas. In other words, the encapsulated phenolic compounds that were attached to the surface of the microcoatings are sensitive to evaporation and physical or chemical degradation, and as a result, they are easily decomposed and lead to a decrease in the antioxidant activity of the microcoatings [49]. In the study done by Cruz-Molina et al. (2021), the stability of green tea extract microcoated with maltodextrin was investigated at different pH and temperatures. According to the obtained results, increasing the pH from 6 to 7 increases the DPPH radical inhibitory activity [50].

Table 4- Total phenolic contents and antioxidant activity of different microcapsule formulas at different pH values

Formula	pH 4.3	pH 5.5	pH 7
	Total phenol content (mg GAE/100g pow)		
1	907.91±0.04^{That}	949.58±0.01^{Not}	1241.25±0.02^{Aa}
2	803.75±0.01^{Cb}	866.25±0.05^{Bb}	991.25±0.02^{Ab}
3	616.25±0.01^{Cc}	749.58±0.04^{Bc}	824.58±0.03^{And}
	Antioxidant activity (IC50%,mg/ml)		
1	8.18±0.51^{And}	7.09±0.89^{Bc}	3.21±0.70^{Cc}
2	9.62±1.03^{Ab}	7.60±0.57^{Bb}	3.93±0.47^{Cb}
3	10.92±1.83^{Aa}	7.94±0.47^{Not}	4.31±1.00^{That}

1- Values are reported as mean ± SD

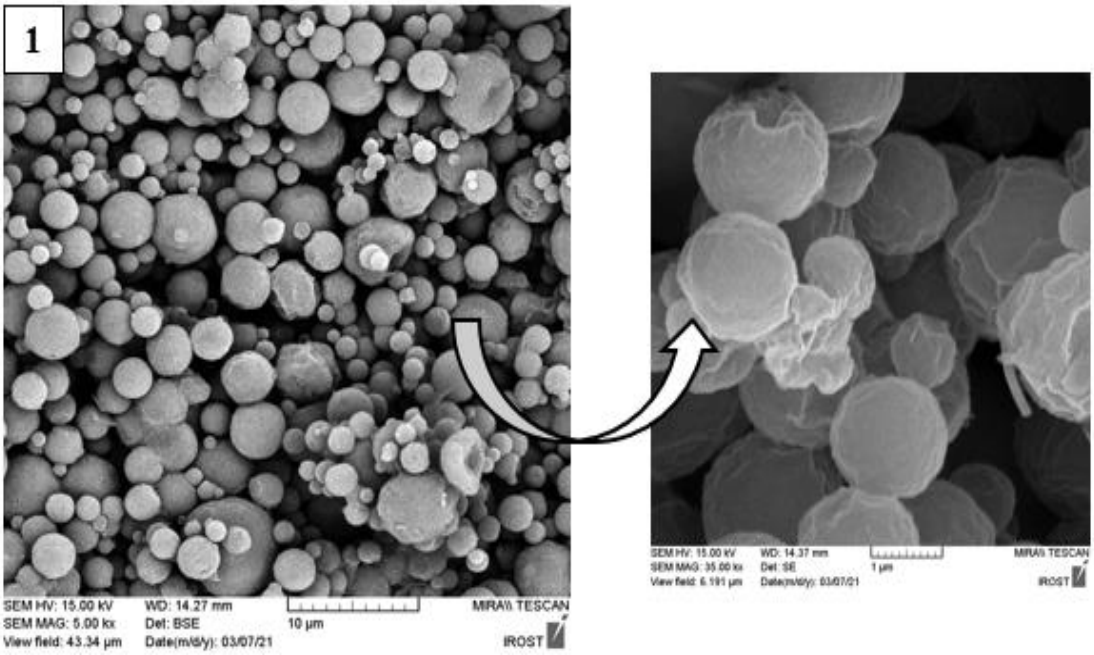
2- Different lower case and upper case the significant difference (P<0.05) between samples and pH, respectively

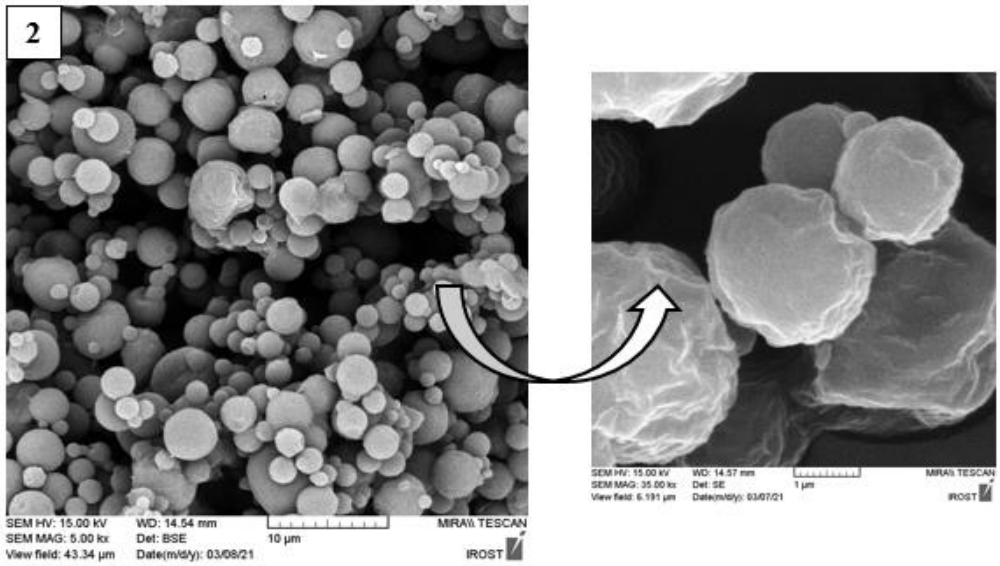
3-5- Morphology study with SEM microscope

The size, shape and morphology of the microcapsules can be seen in the scanning electron microscope images presented in Figure 2. The absence of porosity and cracks on the surface of the particles shows the complete coverage of the core material by the wall material and the success of the coating process by the spray dryer. As can be seen, the microcoatings stabilized with a 1:1 ratio of maltodextrin to kathira gum exhibited the lowest particle size (less than 1 micron) with a uniform distribution. Also, the absence of pitting and a smoother surface on the surface of formula 1 microcoatings indicate the good efficiency of the ratio of 1:1 maltodextrin to catira gum on the coating of grape pomace and linseed oil compared to other formulas. The average diameter for the microcoatings of formulas 2 and 3 was more than 1 micron, and this parameter decreased with the equal ratio of maltodextrin to castor gum, and with the increase in the ratio of maltodextrin and castor gum, the particle size became larger due to the increase in the content of total solids. The possible cause of this can be attributed to the increase in the viscosity of the feed, which increases with the increase in the concentration of solids [45]. Hogan et al. (2001) and Jinapong et al. (2008) also indicated an increase in the average diameter of particles following an increase in solid content and an increase in feed viscosity [52 and 51]. In relation to the surface, the smaller the particle size, the more interaction with the environment and chemical reactions can be done. Size is also related to the effectiveness and availability of stabilized phenolic compounds in the microcoating [50].

High temperature in the dryer chamber (inlet temperature140 °C and outlet temperature 70 °C, nozzle diameter 0.7 mm, feeding flow rate 5 ml/min) and the size of the atomized droplets which are very small lead to the rapid exit of water from it and as a result creating wrinkles and depressions in The surfaces are powdered, as it has been partially shown in the samples of the present study [53]. Such a structure can be mainly attributed to maltodextrin and gum arabic as an immobilizer. In the research conducted byCruz-Molina et al. (2021) green tea extract was covered by a wall made of maltodextrin and according to the results presented in this research, microcoatings covered with polysaccharide

showed depressions in their structure, which is in line with the present study [50]. Ameri and Maa (2006) showed that the rate of water removal from droplets, the type and concentration of wall materials, as well as the wall-to-core ratio are among the main factors that determine the shape and morphology of particles dried by the spray dryer method [54].





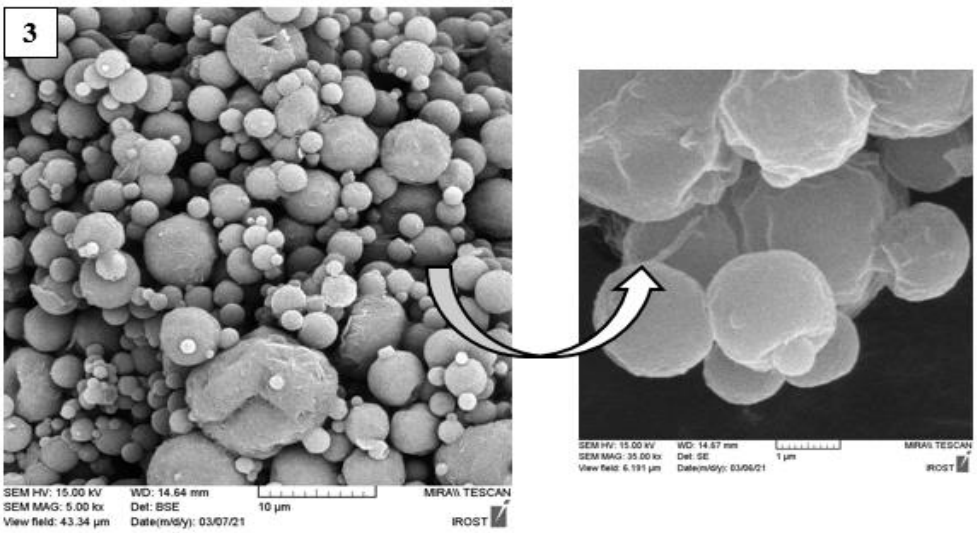


Figure 2- SEM images of Sardasht black grape pomace and flaxseed oil microcapsules [(1): Formula 1, (2): Formula 2 and (3): Formula 3].

3-6- FT-IR analysis

Figure 3 shows the FT-IR spectrum for grape pomace, linseed oil, citric acid, leucine amino acid, maltodextrin and kathira gum. FT-IR analysis of microcoatings with wall materials is also shown in Figure 3. Band 3335 for grape pomace is related to the O-H stretch of the hydroxyl group in the carboxylic acid group (-COOH) on the benzene ring of phenolic acid. The 1655 band is attributed to the C-C stretching vibrations of the aromatic ring, which are common functional groups in phenolic compounds. 3413 is for the O-H stretching band in maltodextrin. The spectra of grape pomace and linseed oil have the same absorption peak in the range of 2925-2974 for the C-H stretching band. Peak 1751 corresponds to C-O stretching band in Katira gum and peak 1512 in leucine amino acid indicates the stretching band of -COO groups. Grape pomace, leucine amino acid, maltodextrin and catira gum have the same absorption peak in the range of 1050-1030 for C=O or C=C double bands [55].

Figure 4 shows the FT-IR spectrum for microcoatings. The peak in the range of 2950-2800 indicates the chain carbon groups in the 3 formulas of microcapsules, and since this group is present in large amounts in linseed oil, it can be concluded that the microcapsules contain linseed oil. From peak 3000 onward, it is related to O-H and N-H2 groups, which, because these two peaks are present in grape pomace and leucine amino acid, it is a proof of the presence of the above compounds in the core of microcapsules. The index bond in citric acid is the carbonyl bond in 1740, which appeared in 1728-1724 in 3 microcoating formulas, and the reason for the decrease towards the lower field is due to the new interactions between citric acid, linseed oil, grape pomace, maltodextrin, and gum. Compared to their free compounds, the most important changes of the microcoatings were related to the change of the O-H group towards higher frequencies. This change can indicate the formation of hydrogen bonds between the components of the wall and the core, and these bonds can be directly related to the stability of the particles [50]. For the microcoating produced with formula 1, the intensity of the O-H peak is higher compared to the other two formulas, which can indicate its higher antioxidant property, which is consistent

with the results of the antioxidant activity of the microcoatings.

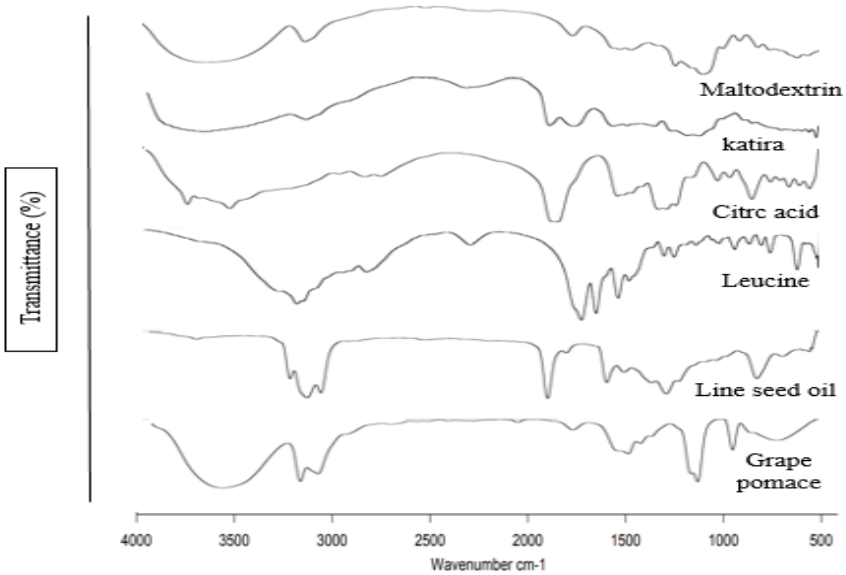


Figure 3- FT-IR spectra of neat powder of wall materials

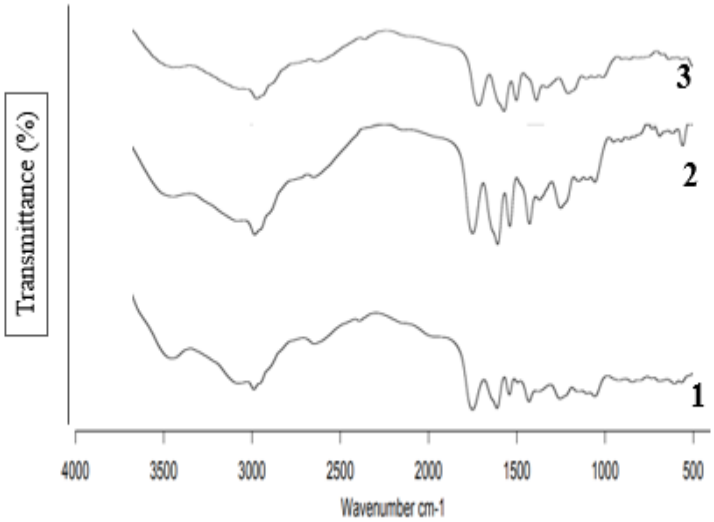


Figure 4- FT-IR spectra of Sardasht black grape pomace and flaxseed oil microcapsules [(1): Formula 1, (2): Formula 2 and (3): Formula 3]

4 - Conclusion

In this research, the effectiveness of different wall ratios in the covering of Sardasht black grape pomace and linseed oil was evaluated. In terms of moisture content and solubility, the sample coated with a ratio of 2:1 had the highest values compared to other formulations, but this amount was not statistically significant (0.05).> *P*).. Also, the highest efficiency of microcoating was observed in the formula 1 sample, the effect of reducing pH on the content of phenolic

compounds and DPPH radical scavenging activity was significant in all samples and led to the reduction of the above parameters, so that the highest total phenolic content belongs to the microcoated sample with a ratio of 1 1: The pH of the walls was equal to 7. In the investigation of total phenol content and DPPH radical scavenging activity, the microcoated sample with a 1:1 ratio of maltodextrin to catira gum performed better in protecting the phenolic compounds of grape pomace, which led to an increase in the antioxidant activity of the microcoatings. SEM images showed that it is possible to successfully produce microcoatings with maltodextrin and castor gum, but it is better to use their ratio of 1:1 in order to obtain particles with a smaller size and a more uniform appearance. The formation of new interactions between grape pomace components and linseed oil with maltodextrin and gum arabic was confirmed by FT-IR analysis. In general, by comparing different ratios of wall materials, it can be concluded that the ratio of 1:1 maltodextrin to katira gum is the best factor for coating and using it in food.

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مقاله علمی_پژوهشی

ارزیابی خواص کیفی ریزپوشینه‌های تفاله انگور و روغن بزرک پایدار شده با نسبت‌های مختلف مالتودکسترین و صمغ کتیرا

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چکیده

ریزپوشانی فرایندی جهت محافظت از ترکیبات زیست فعال در برابر شرایط نامساعد محیطی به منظور افزایش پایداری و دسترسی زیستی آن‌ها می‌باشد. با توجه به حضور ترکیبات زیست فعال در تفاله انگور و روغن بزرک، هدف از این پژوهش درون پوشانی روغن بزرک و تفاله انگور سیاه سردشت با نسبت‌های مختلف مالتودکسترین و صمغ کتیرا (۱:۱، ۲:۱ و ۱:۲) با استفاده از خشک‌کن پاششی و بررسی ویژگی‌های کیفی ریزپوشینه‌های تولید شده بود. بر این اساس، ریزپوشینه‌های تولیدی از لحاظ بازده ریزپوشینه، میزان حلالیت و رطوبت، محتوای ترکیبات فنولی، فعالیت آنتی اکسیدانی، برهم کنش مواد دیواره و هسته و ریزساختار مورد ارزیابی قرار گرفتند. مطابق با نتایج، از لحاظ محتوای رطوبت و میزان حلالیت تفاوت معنی‌داری بین ریزپوشینه‌ها وجود نداشت. بالاترین بازده ریزپوشانی، بیشترین محتوای ترکیبات فنولی کل و بیشترین ظرفیت مهار رادیکال DPPH در ریزپوشینه‌ی دارای نسبت ۱:۱ مالتودکسترین و صمغ کتیرا مشاهده شد. بررسی شکل کروی ریزپوشینه‌ها به وسیله میکروسکوپ الکترونی روبشی نشان داد که ریزپوشینه‌های دارای دیواره مالتودکسترین و صمغ کتیرا با نسبت ۱:۱ ساختار فشرده‌تر و یکنواخت‌تری داشتند. آنالیز FT-IR نیز شکل‌گیری برهمکنش‌های جدید بین مواد دیواره و هسته ریزپوشینه‌ها را تایید کرد. نتایج این پژوهش نشان داد که استفاده از مالتودکسترین و صمغ کتیرا به عنوان ماده دیواره با نسبت ۱:۱، کارآیی مناسبی جهت ریزپوشانی تفاله انگور و روغن بزرک دارد.