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Effect of ultraturrax speed on properties of grapefruit peel essential oil microencapsulated with alginate gum

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ABSTRACT

In this study, the effect of different homogenization speed (10,000, 15,000 and 20,000 rpm) on the structural, rheological and antioxidant properties of nanoencapsulated grapefruit peel essential oil coated in alginate gum wall was investigated. Increasing the homogenization speed led to a decrease in particle size (from 264.0 to 226.9 nm) and PDI index (from 0.32 to 0.19) of nanoemulsion. All nanoemulsions had negative zeta potential (-23.6 to -21.65 mV). The nanoencapsulation efficiency of the essential oil decreased from 83.92 to 78.76% with increasing the speed of homogenization. Nanoemulsion of essential oil at 4 ° C had full stability (100%) for 5 days, while at 25 ° C over time, the stability of the nanoemulsion decreased and the highest stability was related to the prepared nanoemulsions at 15000 rpm because of a smaller zeta potential. The results of Fourier transform infrared spectroscopy and differential scanning calorimetry showed that the essential oil was encapsulated by alginate. The produced nanoemulsions had antioxidant activity and with increasing the concentration of nanoemulsions, the speed of DPPH free radical scavenging increased. The highest antioxidant activity was related to the prepared nanoemulsion at 10,000 rpm. All nanoemulsions showed pseudoplastic (shear thinning) behavior. Herschel–Bulkley and Power law models had higher R_2 than other fitted models. Homogenization had almost no effect on kp and kH. The results of this study introduce the use of 15,000 rpm to produce a nanoemulsion of grapefruit peel essential oil in the alginate gum wall.

1-Introduction

Increasing human awareness about the safety of food and the toxicity of chemical preservatives has led to an increase in the demand for finding natural alternatives for them. Plant essential oils have been receiving attention since ancient times, and now the tendency to use them has increased due to health promotion, therapeutic properties and prevention of various diseases [1]. In the past few decades, plant essential oils have attracted a lot of attention due to their antioxidant effects and inhibition of free radicals [2].

Citrus essential oils are the main aromatic by-products of the juicing industry and are widely used in the food, cosmetic, health and pharmaceutical industries. grapefruit (*Citrus paradisi*) is the second citrus fruit produced in the world with an annual production of more than 60 million tons. The yield of grapefruit and orange juice is almost half of the weight of the fruit, and thus a large amount of waste is produced every year [3]. Citrus peel is considered as a byproduct of citrus juice extraction, in which there are a group of cells in the form of sunken glands that contain essential oil [4].

Essential oils are liquids concentrated in molecules resulting from plant metabolism (terpenoids and aromatic molecules). They are very popular in perfumery and cosmetics and hygiene as well as food and pharmaceutical industries. The main components of essential oils extracted from the skin of some citrus fruits include α-pinene, sabinene, β-pinene, β-myrcene, D-limonene, linalool, m-cymene and 4-terpineol, of which D-limonene is the most important compound [5].

Essential oils contain heat-sensitive bioactive compounds that must be protected from environmental effects such as heat, light, oxygen, and pH, and one of the ways to protect them is microcoating. Microcoating is a technique in which the target compound is trapped and covered with the help of one or more materials called walls. It has found many applications in various industries such as pharmaceutical, chemical, food and printing industries. Walls with different materials can be used for the microcoating of food compounds, among which proteins, carbohydrates, lipids and their combination can be mentioned [6].

Hydrocolloids are complex carbohydrates that are used to improve consistency and textural properties (rheological properties) of liquid and semi-liquid foods. Their activity depends on the type and concentration of hydrocolloids, temperature and process conditions, as well as the solid content and chemical composition of food. Alginate is a linear polymer that includes connections b-D- Mannuronate and α-L- It is guluronate, and due to its biocompatibility, low toxicity and very high capacity in gel formation, it is studied in many food and medical applications [7]. Although many studies have been conducted in the field of microencapsulation of edible essential oils, but so far no study has been conducted on the use of nanomicroencapsulated grapefruit essential oil in an alginate wall. Therefore, in this study, the rheological and physicochemical properties of grapefruit peel essential oil coated with alginate gum have been investigated.

2- Materials and methods

1-2- Sample preparation and preparation

Sodium alginate and Tween 20 were obtained from Sigma. Grapefruit was procured from the local markets of Mazandaran province and after washing and peeling, the essential oil part was separated from the white part. Grapefruit skin was dried in a regular oven at 38 degrees Celsius until reaching a constant weight and completely powdered by a grinder. Then the powders were kept in nylon bags in the freezer at -18 degrees Celsius until the extraction of the essential oil.

2-2- Extraction of essential oil (Clonger)

In order to extract the essential oil in each time of essential oil extraction, 150 grams of fruit skin was poured into the balloon along with 1500 milliliters of distilled water and essential oil extraction was done by a Cloninger machine (at a temperature of 100 degrees Celsius and for 3 hours until all the essential oil is removed from the sample). Then the resulting essential oil was kept at a temperature of -18 degrees Celsius [5].

2-3- Nano coating of essential oil

Grapefruit skin essential oil was coated with alginate gum. An aqueous solution of sodium alginate (4%) was prepared under stirring at a temperature of 70 degrees Celsius. Then, while the solution was being stirred, M/m calcium chloride solution[3] 100 (5%) was added to it and kept for 24 hours at 4 degrees Celsius. After that, grapefruit peel essential oil at the rate of

4% by weight was added drop by drop to the emulsifier Tween 20 at the rate of 2% by weight and mixed well. The mentioned mixture was slowly and drop by drop added to the alginate solution and immediately after half an hour of stirring with a magnetic stirrer, it was homogenized using an ultrathorax at speeds of 10,000, 15,000 and 20,000 rpm and for 10 minutes at a temperature of 10 degrees Celsius. Then, to further reduce the particle size, an ultrasonic generator (HD3200, BANDELINE, Germany) was used with a control range of 45% and a frequency of 20 kHz at a temperature of 45 degrees Celsius with a number of 6 cycles (the time of each cycle is 30 seconds and the rest time is 15 seconds between cycles) [8, 9]. The prepared emulsions were frozen at -20°C for 24 hours and then dried at -57°C and 0.017 mPa pressure for 48 hours using a freeze dryer. After drying, the resulting nanoparticles were poured into a glass container and kept at -18 degrees Celsius.

4-2- Particle size, particle dispersion index and zeta potential of nanoemulsions

The particle size of the emulsion was examined using a laser light diffraction device (Zetasizer Nano ZS-90, Malvern, UK). Also, the particle dispersion index ([1]PDI) was calculated by the device software and reported as droplet size distribution. Zeta potential was measured using Zetasizer Nano ZS-90, Malvern, UK. Data analysis was done using Zetasizer Version 7.02 software [10].

5-2- Fine coverage efficiency of grapefruit peel essential oil ([2]EE)

To extract the surface oil, 15 ml of n-hexane was added to 1.5 g of the powder sample and was stirred with a shaker for 2 minutes at room temperature. Then the mixture was placed in a centrifuge for 20 minutes at a speed of 8000 rpm. Then it was passed through Whatman No. 1 filter and the collected powders were washed three times using 20 ml of n-hexane. The solvent was evaporated at room temperature and the amount of surface oil was calculated until constant dry weight was reached. To measure the total oil, 0.5 g of the resulting powder was added to 25 ml of isopropanol/n-hexane at a ratio of 3:1 and stirred for 5 minutes with a shaker. Then the resulting mixture was centrifuged for 15 minutes at 8000 rpm. After that, the clear organic phase was separated and the solvent was evaporated at a temperature of 60 degrees Celsius until reaching a constant weight [11].

$$(1)\quad EE\,(\%) = \frac{oil_{Total} - oil_{Surface}}{oil_{Total}} \times 100$$

oil_{Total} And $oil_{Surface}$ It is related to total oil and surface oil, respectively, based on the dry matter of the final emulsion.

6-2- Emulsion stability

To determine the stability of the emulsion, 10 ml of each emulsion was put into a test tube and kept at 4 and 25 degrees Celsius for 6 days. After storage, emulsion stability was calculated using the following equation [12].

$$(2)\quad Emulsion\ Stability\ Iindex(\%) = \frac{HE-HS}{HE} \times 100$$

HE and HS are the total emulsion and the height of the serum layer, respectively.

7-2- Fourier transform infrared spectrum [3]FTIR))

FT-IR measurement using a desktop FT-IR spectrometer (CARY 630, Agilent, USA) in the wavelength range of 400 to cm^{-1} 4000 was done.

2-8- Analysis of the differential scanning calorimeter ([4]DSC)

The thermal properties of grapefruit peel essential oil coated with alginate gum were investigated using a differential scanning calorimeter (DSC 822, Mettler Toledo, Switzerland) equipped with an automatic thermal analysis program. The starting temperature (T_{THE}), peak (T_P) and the end (T_{AND}) were determined using a DSC thermometer [13].

9-2- Antioxidant activity of finely coated grapefruit skin essence

In order to measure the antioxidant activity of nanoemulsions, first, 2.4 mg of DPPH was dissolved in 100 ml of methanol. Then, 975 microliters of DPPH methanolic solution was added to 25 microliters of each of the tested essential oil methanolic solutions [14]. This mixture was left at room temperature and in the dark for 30 minutes and the absorbance of the solution was read at 517 nm by a

1 - Poly Dispersity Index

2 - Encapsulation Efficiency

3 - Fourier-transform infrared

4 - Differential scanning calorimetry (DSC)

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spectrophotometer and measured according to the following equation:

(3) $Antiradical\ activity\ (\%) = [(\frac{A_{I0}-A_t)}{A_0}] \times 100$

10-2- Steady shear flow behavior

For this purpose, the flow behavior analysis test was performed using a rheometer device (Physica MCR-301, Anton Paar, Austria) equipped with a thermal circulator to control the temperature and using a parallel plate probe. Temperature regulation with peltier plate system with 0.01 sensitivity± It was equipped with a water circulator (Viscotherm VT2). Solutions with a concentration of 5% were prepared and placed on the stirrer for 5 minutes. Analysis of flow behavior was performed in the range of shear speed from 0.01 to 100/s. The following models were used to investigate the rheological properties [15]:

1- Powerla model

(4) $\tau = k_p \dot{\gamma}^{n_p}$

In this equation $K_p$consistency coefficient (Pa s^n) and n_p، It is an index of current behavior (without dimension). K shows the viscosity of the fluid and n shows the behavior of the fluid.

2- Herschel's model

(5) $t = K_{OR}(\dot{Y})^{n_{OR}}\ t_{0H}$

In this equation K_H ،, consistency coefficient (Pa s^n) and n_H، It is an index of current behavior (without dimension). K shows the viscosity of the fluid and n shows the behavior of the fluid.τ_{0H} The yield stress (Pa) for the Herschel model is absolute.

3-Bingham model

(6) $\tau = \eta_\beta \dot{Y} + \tau_{0b}$

In this equation η_B Bingham viscosity (Pa s) and τ_{0B} Yield stress (Pa) is the Bingham model.

4- Casson model

(7) $\tau^{0.5} = K_{0c}^{0.5} + k_c\ (\dot{c})^{0.5}$

In this equation k_{0c})^2)= τ_{0c}indicating the yield stress (Pa) and = (k_c^2) ηc Casson viscosity (Pa.s) is called.

2-11- Statistical analysis

The statistical analysis of the data obtained from this research was done using the ANOVA method based on a completely random statistical design using Duncan's test at the 95% confidence level. For this purpose, SPSS version 20 software was used and graphs were drawn with Excel version 2016 software.

3- Results and discussion

1-3- Particle size, particle dispersion index and zeta potential of nanoemulsion

The results related to particle size of fine grapefruit skin essence nanoemulsion coated with alginate gum are presented in Table 1. As can be seen, the size of nanoemulsion particles has decreased with the increase of homogenization speed and the difference is statistically significant. It is necessary that the size of the emulsion particles decreases with the increase of homogenization, so that the breaking force overcomes the interfacial forces and shear stress. In this way, the size of the particles decreases [16]. The high energy of high pressure homogenization leads to breaking the droplets and reducing their size. In a study, microencapsulation of orange essential oil was investigated using spray drying, and homogenization pressure had the greatest effect on particle size. When the homogenization pressure was below 500 bar, increasing the pressure led to a decrease in the droplet size [17]. These results are in accordance with the results obtained from the research of other researchers. They showed that the smallest particle size is obtained at the highest homogenization speed [16 and 18].

Table 1- Particle size, zeta potential and encapsulation efficiency encapsulated grapefruit peel essential oil

Ultraturrax Speed (rpm)	Particle size (nm)	PDI	Zeta potential	Encapsulation efficiency (%)
10000	264.0±2.8[a]	0.32±0.04 [a]	-22.6±7.1[b]	83.92±0.37[a]
15000	258.6±1.5[b]	0.29±0.03 [b]	-23.6±2.9[c]	81.25±0.25[b]
20000	226.9±2.4[c]	0.19±0.01 [c]	-21.65±5.7[a]	78.76±0.48[c]

* Different lower case letters in the same column indicate significantly different ($p < 0.05$)

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The dispersion index of nanoemulsion particles is shown in Table 1. As can be seen, this index is less than 0.33 in all samples. High speeds lead to particle dispersion index less than 0.5, which is desirable. The dispersion index of nanoemulsion particles decreased with increasing homogenization speed and a statistically significant difference was found. It seems that at high speeds of homogenization, the shear force increases, which leads to a decrease in particle size and homogeneity, both of which happened in this study. The researchers used the speeds of 6000, 8000, 10000, 12000, 14000, 16000 and 18000 rpm to cover the thyme essential oil in the alginate wall. Their results showed that there is an inverse relationship between the emulsion preparation speed and the particle dispersion index [16]. Homogenization by reducing the dispersion index of particles leads to the homogenization of the dispersed phase in the emulsion. In general, when the emulsion is formed at high speeds, a turbulent flow is formed, and these eddies apply a kind of shear pressure to the emulsion droplets, which lead to a decrease in the size of the particles and the dispersion index of the particles [12].

The researchers showed that when speeds lower than 8000 rpm are used for emulsion production, the amount of particle heterogeneity increases. While the use of speeds higher than 8000 rpm leads to the formation of stable emulsions, and with increasing speed, the dispersion index of particles decreases significantly [19]. These results are consistent with the results of the present study. Some other researchers showed that by increasing the particle size of shiraz thyme essential oil microcoated in alginate, the dispersion index of the particles decreases, which is in line with the results of the present study [4].

Zeta potential is a very good indicator for determining the electrostatic interactions between the surface of particles, so that it shows the charge accumulation in the stationary layer and the intensity of opposite ions on the surfaces [18]. The results of zeta potential of nanoemulsion particles are shown in Table 1. As can be seen, the zeta potential is negative in all the studied samples and there is no statistically significant difference between the samples. In other words, it can be said that the effect of homogenization on zeta potential was not significant. In general, emulsions with zeta potential in the range of -30 to +30 mV are unstable. Therefore, according to the obtained values, it can be said that all nanoemulsions are stable. In this case, the electric charge of the droplets is so strong that it is assumed that the repulsive forces between the droplets are dominant in the nanoemulsion system. The researchers reported the zeta potential of lemongrass essential oil microcoated in alginate gum in the range of -18 to -50 millivolts, which is consistent with the results of the present study [20].

2-3- Microcoating efficiency of grapefruit peel essential oil

The results of microcoating efficiency of grapefruit peel essential oil microcoated with alginate gum are shown in Table 1. As can be seen, the microcoating efficiency is more than 78%, which is a very favorable efficiency (≥50) is Also, by increasing the homogenization speed, the efficiency of essential oil microcoating has decreased and the difference is statistically significant. Emulsion droplet size and viscosity are the influencing factors on microcoating efficiency. The researchers declared the microcoating efficiency of thyme, clove and cinnamon essential oils in the alginate coating in the range of 70 to 90%, which is in accordance with the results of the present study [21]. In another study, they reported the efficiency of peppermint essential oil microcoated in alginate and carnauba wax in the range of 72.9% to 96.5%, which is within the range of the numbers reported in the present study [22]. Similar results have been reported for eugenol microcoated in alginate/gelatin wall [23] and sesame polyphenols in fenugreek and purslane gum wall [24].

3-3- Emulsion stability

Stability is a very important parameter for evaluating the properties of an emulsion. It is very difficult to produce a stable emulsion of micro-coated essential oil because the small drops of essential oil tend to be unstable and as a result the phases of the emulsion are separated [22]. In oil-in-water emulsions, various destabilization mechanisms such as gravity creaming, precipitation, flocque, and coagulation lead to a decrease in stability. Figure 1 shows the results related to the stability of fine grapefruit skin essential oil emulsion covered with alginate gum at 4 and 25

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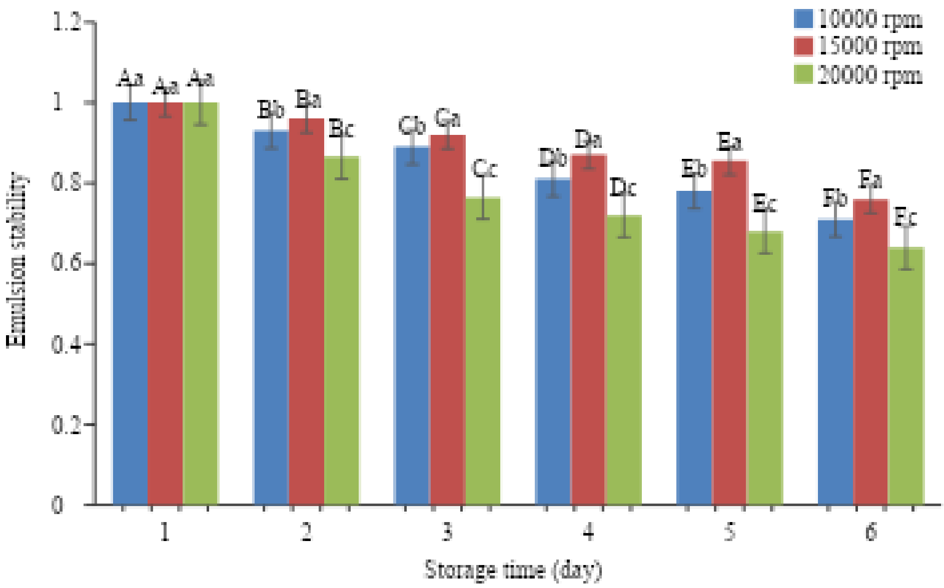
degrees Celsius. As can be seen, at the temperature of 4 degrees Celsius, the emulsion is stable until the 5th day of the storage period, and after that, from the 6th day, the stability decreases and there is a statistically significant difference. The trend of emulsion stability at the temperature of 25 degrees Celsius was decrease and significant statistical difference was observed between different days. On the first day, the samples had good stability, but in the following days, the stability decreased. As mentioned, increasing the speed of homogenization led to a decrease in the size of the particles, but the emulsion prepared at a speed of 15,000 rpm had the highest stability at both temperatures. A study by Garcia et al. (2012) found that although no phase separation was observed in any of the emulsions, the droplets may coagulate during the storage period, which can reduce the stability index of the emulsion [25]. After homogenization, when the emulsion remains at rest for a certain period of time, energy is dissipated and leads to the formation of larger droplets, which indicates droplet coagulation. According to the results of Garcia et al. (2012), increasing the homogenization pressure decreases the droplet size, but the emulsions produced under higher homogenization pressures were less stable, and this indicates a higher level of coagulation [25]. Other researchers showed that the speed of homogenization is effective on the stability of the emulsion. They announced the trend of decreasing stability for all clove essential oil emulsions in sodium alginate during the storage period. Also, emulsion stability decreased with increasing homogenization speed [26]. The stability of the emulsion corresponds to the results obtained from the zeta potential, and the emulsion prepared at a speed of 15000 rpm has more stability due to the lower zeta potential.



A)

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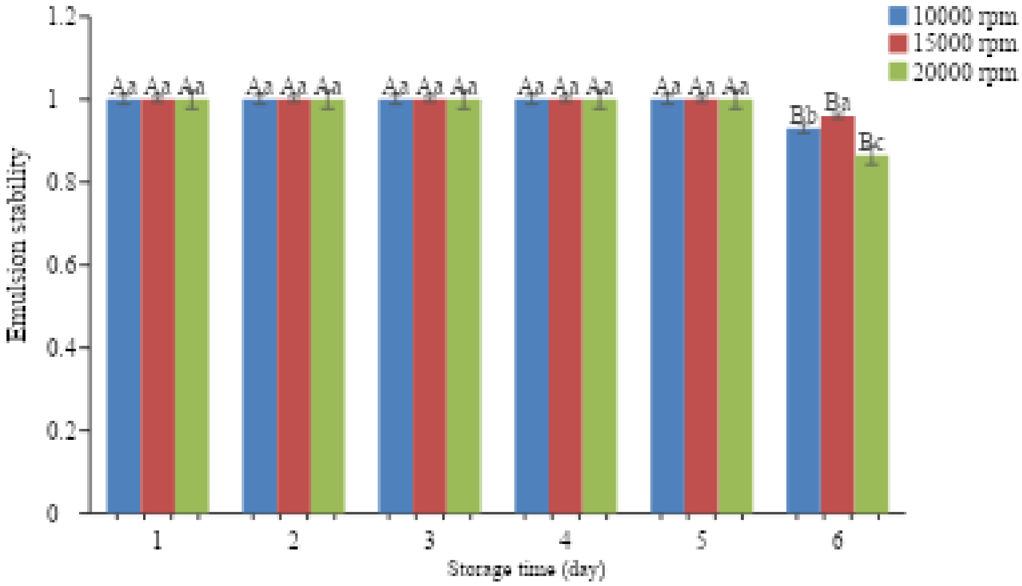

B)

Figure 1- Stability of emulsions in different storage conditions: A) At room temperature (25 ºC) B) At refrigerator temperature (4 ºC) (Uppercase and lowercase letters show a statistically significant difference between different storage times in the same sample and different samples at the same time, respectively.)

4-3- Fourier transform infrared spectrum

Fourier transform infrared spectrum of essential oil nanocoated in the wall of alginate gum which was prepared with different homogenization speeds and alginate gum alone is shown in Figure 2. cm range^{-1} 3000-3600 is related to O-H stretching bonds. As can be seen, all nano-coated essential oils in this range have a peak that is related to the bond between essential oil and alginate. The researchers measured the FTIR spectrum for lemon, orange, red and white grapefruit, and Darabi citrus peel essential oils and showed that all essential oils have peaks in the range of 3250 to 3600 and also in 3000 to 3250 [27] and it is consistent with the spectrum results of the present study. cm range^{-1} 2927 shows the presence of carboxylic acids in fine essential oil covered with alginate gum. Peaks cm^{-1} 3443، cm^{-1} 2928، cm^{-1} 1615 ، cm^{-1} 1096، cm^{-1} 1029 is related to the carboxylic group, the asymmetric stretch of the carboxylate salt and the C-O stretch of the ether. Essential oil coated with sodium alginate has a free carboxyl group, which has a negative charge [13]. Other researchers also reported similar results for peppermint essential oil coated in alginate [22].

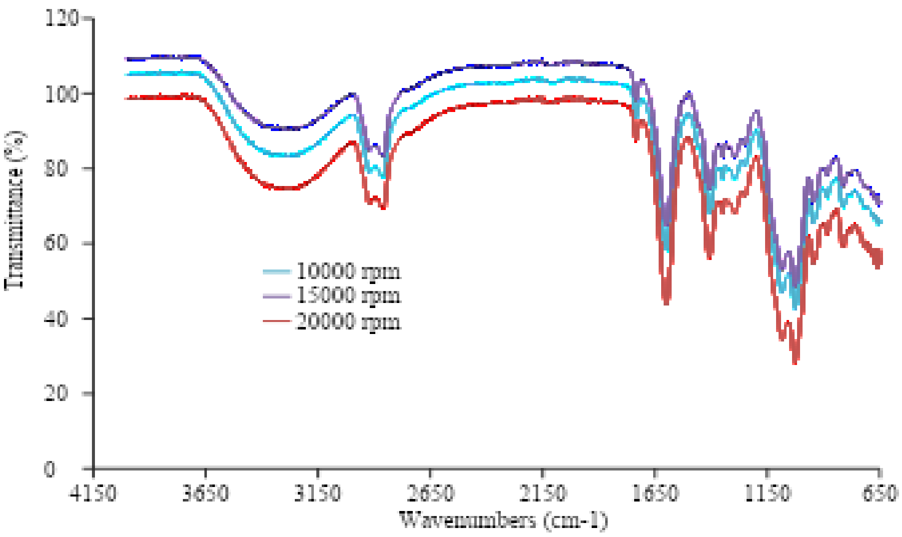

Figure 2. FTIR spectrum of encapsulated grapefruit peel essential oil with alginate.

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3-5- DSC differential scanning calorimetry analysis

Differential scanning calorimetry analysis DSC can be used to determine the thermal behavior of biomolecules, which is related to their structure, hydrophilic properties, and interaction mode. The DSC diagram of pure alginate and grapefruit peel essential oil nanocoated in alginate gum is shown in Figure 3. According to Figure 3, two ranges of 25-9-110 °C and 224-265-8 °C for the sample of nanofine essential oil covered in alginate gum correspond to endothermic (water evaporation) and endothermic peaks, respectively. According to the researchers' findings, the exothermic peaks at high temperature are related to the thermal decomposition of polysaccharides and the beginning of the random division of glycosidic bonds and proteins [28]. According to the results of Razmkhah et al. (2016), two heat-generating regions were observed at the temperature of 262.56 and 304.44 degrees Celsius, which is most likely related to the destruction of the sample [29]. In a study, researchers investigated the efficiency of nano-microcoating of orange and lemon peel extracts, and reported that after nano-microcoating, the melting point of the samples increased from 73.6 to 231.8 degrees Celsius [30]. Therefore, nano-microcoating can improve the stability of orange peel extract against high heat transfer during food processing. The researchers reported the reduction and increase peaks of mint essential oil microcoated in alginate gum and carnauba wax in the temperature range of 90 to 200 and 200 to 300 degrees Celsius, respectively [22]. As can be seen, the microcoated essential oil is closer to pure alginate in terms of DSC characteristics at a speed of 1000 rpm. Therefore, it can be concluded that at higher speeds of homogenization, some of the essential oil compounds have been decomposed and turned into newer compounds. Other researchers reported two peaks in the graph of essential oil coated with alginate, that the first peak is related to water evaporation from alginate and the second peak is related to essential oil [31].

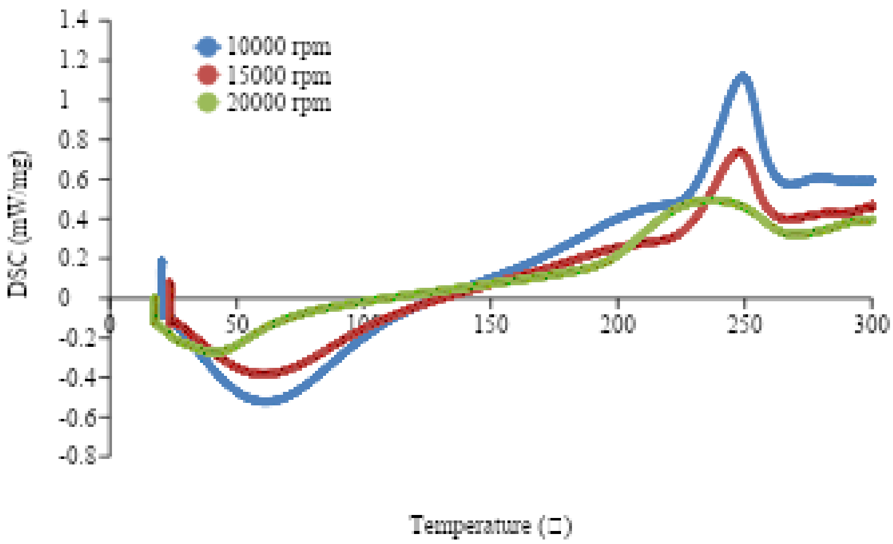

Figure 3- DSC diagram of encapsulated grapefruit peel essential oil with alginate gum.

6-3- Antioxidant activity of finely coated grapefruit peel essence (DPPH)

Grapefruit skin essential oil, like other plant essential oils, has antioxidant activity, because the phenolic and flavonoid compounds of these compounds can prevent lipid peroxidation by quenching proxy radicals and chelating iron [32]. Since the antioxidant activity of essential oils is related to their specific chemical structure, it seems that the antioxidant activity of produced nanoemulsions can be a good indicator for preserving their active compounds. Therefore, the effects of grapefruit peel essential oil nanoemulsion on DPPH radical activity were determined to evaluate the antioxidant activity of prepared nanoemulsions.

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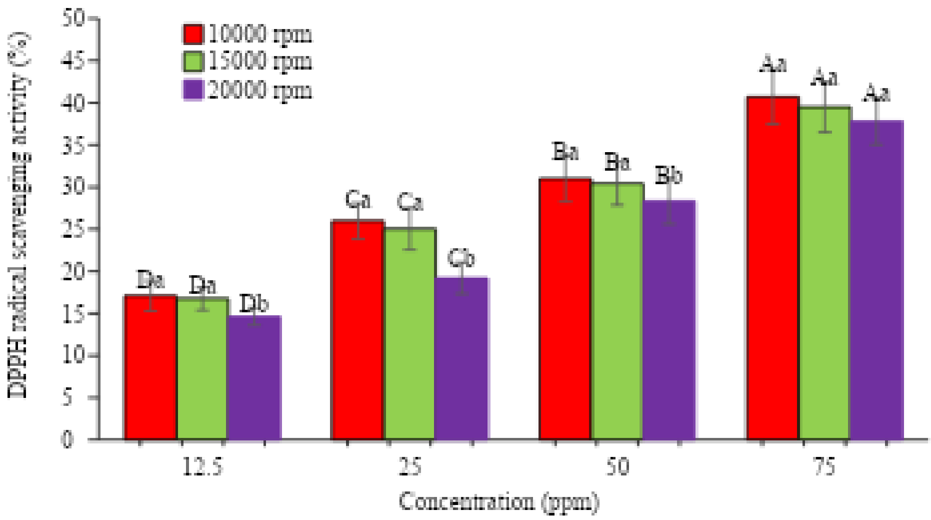

Figure 4 - The effect of Ultra-Turrax speed on the antioxidant properties of encapsulated grapefruit peel essential oil with alginate gum. (*Different lower case letters in the same concentration and upper case letters in the same rpm indicate significantly different ($p < 0.05$))

As shown in Figure 4, antioxidant activity has increased with increasing concentration of nanoemulsion and the difference is statistically significant. In all studied concentrations, the highest antioxidant activity is related to alginate 10. The reason for this can be related to the higher microcoating efficiency of the essential oil at a speed of 10,000 rpm. Our previous research showed that grapefruit peel essential oil coated in basil seed gum has antioxidant activity [33]. Caryophyllene and -α-pinene chemical compounds of grapefruit peel essential oil show weak and moderate DPPH inhibitory activity, respectively. Other researchers observed significant antioxidant activity in essential oils rich in monoterpene (di-limonene and -α-pinene). In general, essential oils rich in oxygenated compounds show a very important anti-radical activity. There is a direct relationship between antioxidant activity and the amount of α-pinene [34]. Likewise, Mimica-Dukich et al. (2004) reported that monoterpenes (limonene) and sesquiterpenes (caryophyllene) are responsible for DPPH radical neutralization [35]. In addition, Kellen and Tepe (2008) have recognized that monoterpenes (limonene and -α-pinene) alone do not have significant antioxidant activity in comparison with similar compounds [36].

7-3-Analysis of stable shear flow behavior

According to Figure 5, samples of essential oils coated with alginate gum at homogenization speeds of 10,000, 15,000 and 20,000 have shear thinning behavior. According to the fitted models (Powerla model, Herschel Balkli model, Casson and Bingham), Powerla model and Herschel Balkli model have R^2 are higher Also, the powerla model current index value (n_p) and Hershel Balkali (n_H) 0.66 - 0.68 have been obtained, which confirms the thinning behavior by cutting the samples. But the homogenization speed depends on the value of k_p and k_H It was not significant ($p>0.05$). In a research, researchers investigated the effect of homogenization pressure on the emulsion properties and shelf life of microcoated basil essential oil and concluded that homogenization had no effect on the viscosity of the emulsion [25]. According to Flory et al. (2000), although the homogenizer at a very high pressure reduces the viscosity of the emulsion to a limiting value. This means that in emulsions with low viscosity, such as emulsions based on gum arabic, the effect of high-pressure homogenization on viscosity is probably not significant [37]. Koflis et al. (2021) reported shear thinning behavior for lemon essential oil nanoemulsions in the wall of alginate gum [38]. In another study, da Silva Campello et al. (2021) reported the non-Newtonian behavior of shear thinning for clove essential oil nanoemulsions in alginate gum and carboxymethyl cellulose, which is in accordance with the results of the present study [39].

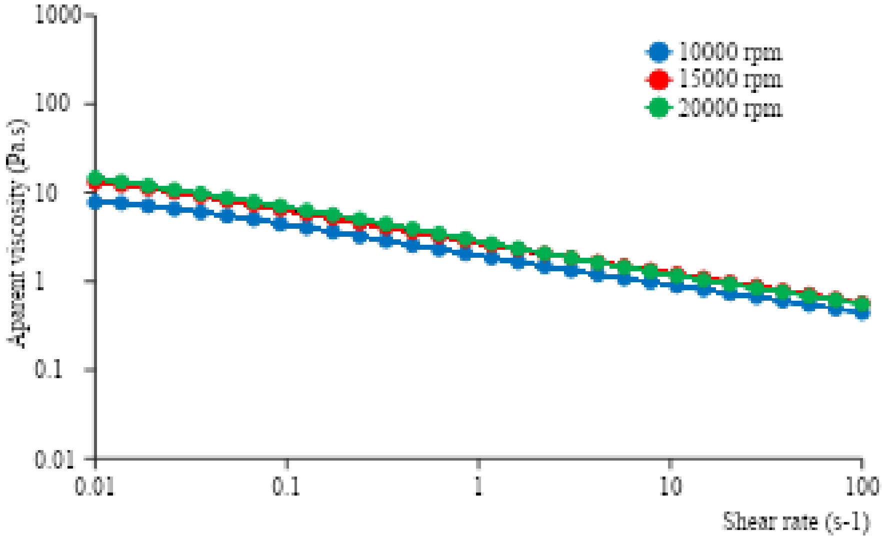

Figure 5- Flow behavior diagram encapsulated grapefruit peel essential oil with alginate gum.

Table 2 - Rheological parameters of Herschel–Bulkley and Power law model for encapsulated grapefruit peel essential oil with alginate gum

Ultraturrax Speed (rpm)	Power Law model			Herschel–Bulkley model			
	K_p(Bye s^n)	n_p	R^2	k_H (Pa×sn)	n_H	t_{0H} (well)	R^2
10000	1.91±0.05^{b}	0.68±0.01^{a}	1	1.90±0.04^{c}	0.68±0.01^{a}	0.02±0.01^{a}	1
15000	2.73±0.03^{a}	0.66±0.01^{b}	1	2.74±0.04^{a}	0.66±0.01^{a}	0.02±0.01^{a}	1
20000	2.60±0.17^{a}	0.66±0.00^{a}	1	2.51±0.13^{b}	0.67±0.00^{a}^{b}	0.03±0.00^{a}	1

* Different lower case letters in the same column indicate significantly different ($p < 0.05$)

Table 3- Rheological parameters of Bingham and Casson model for encapsulated grapefruit peel essential oil with alginate gum

Ultraturrax Speed (rpm)	Bingham model			Casson model		
	τ_{0b} (well)	η_β	R^2	τ_{0c} (well)	η_c	R^2
10000	1.79±0.06^{c}	0.47±0.00^{b}	0.97	3.58±0.11^{c}	0.21±0.01^{a}	0.97
15000	2.56±0.00^{a}	0.61±0.05^{a}	0.97	5.11±0.00^{a}	0.20±0.01^{a}	0.98
20000	2.42±0.03^{b}	0.58±0.00^{a}	0.98	4.84 ±0.07^{b}	0.20±0.00^{a}	0.97

* Different lower case letters in the same column indicate significantly different ($p < 0.05$)

4- Conclusion

Nowadays, the use of natural compounds as food preservatives has become increasingly popular among consumers because they are considered safe and healthy. In this study, grapefruit peel essential oil was nanocoated in alginate gum using different homogenization speeds (1000, 15000 and 20000 rpm). The results showed that the formed nanoemulsions had a nanometer size (less than 300 nm) and had a suitable PDI. Also, all nanoemulsions had negative zeta potential and nano-microcoating efficiency above 75%, which is ideal. Nanoemulsion of grapefruit peel essential oil showed antioxidant properties in the form of free radical inhibition due to the presence of bioactive compounds in the essential oil. The structural analysis of nanoemulsions showed the trapping of essential oil particles inside the alginate gum. In terms of rheological parameters, nano-coated grapefruit peel essential oils had shear thinning behavior. But different speeds of homogenization were not significant on the viscosity of the samples. Finally, considering the higher stability of nanoemulsions prepared at 15000 rpm and also taking into account other parameters that were studied in this study, the nano-microcoating of essential oil at 15000 rpm can be suitable for the nano-microcoating of grapefruit peel essential oil in the wall of alginate gum.

5- Reference

[1] Deng, W., Liu, K., Cao, S., Sun, J., Zhong, B., & Chun, J. (2020). Chemical composition, antimicrobial, antioxidant, and antiproliferative properties of grapefruit essential oil prepared by molecular distillation. *Molecules, 25*(1), 217.

[2] Sahay, S. (2015). A review on pharmacological uses of essential oil. *Int. J. Curr. Pharm. Rev. Res, 6*, 71-79.

[3] Okunowo, W. O., Oyedeji, O., Afolabi, L. O., & Matanmi, E. (2013). Essential oil of grape fruit (Citrus paradisi) peels and its antimicrobial activities.

[4] Kavoosi, G., Derakhshan, M., Salehi, M., & Rahmati, L. (2018). Microencapsulation of zataria essential oil in agar, alginate and carrageenan. *Innovative Food Science & Emerging Technologies, 45*, 418-425.

[5] Bozkurt, T., Gülnaz, O., & Kaçar, Y. A. (2017). Chemical composition of the essential oils from some citrus species and evaluation of the antimicrobial activity. *Journal of Environmental Science, Toxicology and Food Technology, 11*(10), 29-33.

[6] Đorđević, V., Balanč, B., Belščak-Cvitanović, A., Lević, S., Trifković, K., Kalušević, A. Nedović, V. (2015). Trends in encapsulation technologies for delivery of food bioactive compounds. *Food Engineering Reviews, 7*(4), 452-490.

[7] Choukaife, H., Doolanea, A. A., & Alfatama, M. (2020). Alginate nanoformulation: Influence of process and selected variables. *Pharmaceuticals, 13*(11), 335.

[8] Dehghan, B., Esmaeilzadeh Kenari, R., & Raftani Amiri, Z. (2020). Nano-encapsulation of orange peel essential oil in native gums (Lepidium sativum and Lepidium perfoliatum): Improving oxidative stability of soybean oil. *Journal of Food Processing and Preservation, 44*(11), e14889.

[9] Radunz, M., da Trindade, M. L. M., Camargo, T. M., Radunz, A. L., Borges, J. D., Gandra, E. A., & Helbig, E. (2019). Antimicrobial and antioxidant activity of unencapsulated and encapsulated clove (Syzygium aromaticum, L.) essential oil. *Food Chemistry, 276*, 180-186.

[10] Granata, G., Stracquadanio, S., Leonardi, M., Napoli, E., Consoli, G. M. L., Cafiso, V., . . . Geraci, C. (2018). Essential oils encapsulated in polymer-based nanocapsules as potential candidates for application in food preservation. *Food Chemistry, 269*, 286-292.

[11] Raeisi , S. , Ojagh , S. M. , Quek , S. Y. , Pourashouri , P. , & Salaun , F. (2019). Nano-encapsulation of fish oil and garlic essential oil by a novel composition of wall material: Persian gum-chitosan. *LWT, 116*, 108494.

[12] Razavi, R., Kenari, R. E., Farmani, J., & Jahanshahi, M. (2020). Fabrication of zein/alginate delivery system for nanofood model based on pumpkin. *International journal of biological macromolecules, 165*, 3123-3134.

[13] Naji-Tabasi, S., Razavi, S. M. A., & Mehditabar, H. (2017). Fabrication of basil seed gum nanoparticles as a novel oral delivery system of glutathione. *Carbohydrate Polymers, 157*, 1703-1713.

[14] Himed, L., Merniz, S., Monteagudo-Olivan, R., Barkat, M., & Coronas, J. (2019). Antioxidant activity of the essential oil of citrus limon before and after its encapsulation in amorphous SiO2. *Scientific African, 6*, e00181.

[15] Li, W., Wang, Y., Li, J., Jiao, Y., & Chen, J. (2019). Synergistic and competitive effects of monoglycerides on the encapsulation and interfacial shear rheological behavior of soy proteins. *Food Hydrocolloids, 89*, 631-636.

[16] Benavides, S., Cortés, P., Parada, J., & Franco, W. (2016). Development of alginate microspheres containing thyme essential oil using ionic gelation. *Food Chemistry, 204*, 77-83.

[17] Carmona, P. A., Tonon, R. V., da Cunha, R. L., & Hubinger, M. D. (2013). Influence of emulsion properties on the microencapsulation of orange essential oil by spray drying. *Journal of Colloid Science and Biotechnology, 2*(2), 130-139.

[18] Firoozi, M., Rezapour-Jahani, S., Shahvegharasl, Z., & Anarjan, N. (2020). Ginger essential oil nanoemulsions: Preparation and physicochemical characterization and antibacterial activities evaluation. *Journal of Food Process Engineering, 43*(8), e13434.

[19] Ruiz Márquez, D., Partal López, P., Franco Gómez, J. M., & Gallegos Montes, C. (2010). Oil-in-water food emulsions stabilized with tuna proteins.

[20] Salvia-Trujillo, L., Rojas-Graü, M. A., Soliva-Fortuny, R., & Martín-Belloso, O. (2013). Effect of processing parameters on physicochemical characteristics of microfluidized lemongrass essential oil-alginate nanoemulsions. *Food Hydrocolloids, 30*(1), 401-407.

[21] Soliman, E. A., El-Moghazy, A. Y., El-Din, M. M., & Massoud, M. A. (2013). Microencapsulation of essential oils within alginate: formulation and in vitro evaluation of antifungal activity.

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[22] Yilmaztekin, M., Lević, S., Kalušević, A., Cam, M., Bugarski, B., Rakić, V. Nedović, V. (2019). Characterization of peppermint (Mentha piperita L.) essential oil encapsulates. *Journal of microencapsulation, 36*(2), 109-119.
[23] Shinde, U., & Nagarsenker, M. (2011). Microencapsulation of eugenol by gelatin-sodium alginate complex coacervation. *Indian journal of pharmaceutical sciences, 73*(3), 311.
[24] Esmaeilzadeh Kenari, R., & Razavi, R. (2022). Phenolic profile and antioxidant activity of free/bound phenolic compounds of sesame and properties of encapsulated nanoparticles in different wall materials. *Food science & nutrition*, *10*(2), 525-535.
[25] Garcia, L. C., Tonon, R. V., & Hubinger, M. D. (2012). Effect of homogenization pressure and oil load on the emulsion properties and the oil retention of microencapsulated basil essential oil (Ocimum basilicum L.). *Drying Technology, 30*(13), 1413-1421.
[26] Purwanti, N., Zehn, A. S., Pusfitasari, E. D., Khalid, N., Febrianto, E. Y., Mardjan, S. S. Kobayashi, I. (2018). Emulsion stability of clove oil in chitosan and sodium alginate matrix. *International Journal of Food Properties, 21*(1), 566-581.
[27] Manaila, E., Berechet, M. D., Stelescu, M. D., Craciun, G., Mihaiescu, D. E., Purcareanu, B. Radu, M. (2016). Comparation between chemical compositions of some essential oils obtained by hydrodistillation from citrus peels. *Chemistry Review, 67*, 106.
[28] Krekora, M., Markiewicz, K. H., Wilczewska, A. Z., & Nawrocka, A. (2023). Raman and thermal (TGA and DSC) studies of gluten proteins supplemented with flavonoids and their glycosides. *Journal of Cereal Science*, 103672.
[29] Razmkhah, S., Mohammadifar, M. A., Razavi, S. M. A., & Ale, M. T. (2016). Purification of cress seed (Lepidium sativum) gum: Physicochemical characterization and functional properties. *Carbohydrate Polymers*, *141*, 166-174.
[30] Mahmoud, K. F., Ibrahim, M. A., Mervat, E. D., Shaaban, H. A., Kamil, M. M., & Hegazy, N. A. (2016). Nano-encapsulation efficiency of lemon and orange peels extracts on cake shelf life. *American Journal of Food Technology*, *11*(3), 63-75.
[31] Laurienzo, P., Malinconico, M., Motta, A., & Vicinanza, A. (2005). Synthesis and characterization of a novel alginate–poly (ethylene glycol) graft copolymer. *Carbohydrate Polymers, 62*(3), 274-282.
[32] Ahmed, S., Rattanpal, H., Gul, K., Dar, R. A., & Sharma, A. (2019). Chemical composition, antioxidant activity and GC-MS analysis of juice and peel oil of grapefruit varieties cultivated in India. *Journal of Integrative Agriculture, 18*(7), 1634-1642.
[33] Mohammad Kheshtchin, S., Farahmandfar, R., & Farmani, J. (2022). Effect of homogenization on encapsulation of grapefruit (Citrus paradisi) peel essential oil with basil seed gum. *Innovative Food Technologies*, *9*(3), 223-238 [In Persian].
[34] Wei, A., & Shibamoto, T. (2007). Antioxidant activities and volatile constituents of various essential oils. *Journal of Agricultural and Food Chemistry, 55*(5), 1737-1742.
[35] Mimica-Dukic, N., Bozin, B., Sokovic, M., & Simin, N. (2004). Antimicrobial and antioxidant activities of Melissa officinalis L.(*Lamiaceae*) essential oil. *Journal of Agricultural and Food Chemistry, 52*(9), 2485-2489.
[36] Kelen, M., & Tepe, B. (2008). Chemical composition, antioxidant and antimicrobial properties of the essential oils of three Salvia species from Turkish flora. *Bioresource technology*, *99*(10), 4096-4104.
[37] Floury, J., Desrumaux, A., & Lardières, J. (2000). Effect of high-pressure homogenization on droplet size distributions and rheological properties of model oil-in-water emulsions. *Innovative Food Science & Emerging Technologies, 1*(2), 127-134.
[38] Cofelice, M., Cinelli, G., Lopez, F., Di Renzo, T., Coppola, R., & Reale, A. (2021). Alginate-assisted lemongrass (Cymbopogon nardus) essential oil dispersions for antifungal activity. *Foods, 10*(7), 1528.
[39] Da Silva Campelo, M., Melo, E. O., Arrais, S. P., do Nascimento, F. B. S. A., Gramosa, N. V., de Aguiar Soares, S. Ricardo, N. M. P. S. (2021). Clove essential oil encapsulated on nanocarrier based on polysaccharide: A strategy for the treatment of vaginal candidiasis.*Colloids and Surfaces A: Physicochemical and Engineering Aspects, 610*, 125732.

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چکیده

در این پژوهش تاثیر سرعت‌های مختلف هموژنیزاسیون (۱۰۰۰۰، ۱۵۰۰۰ و ۲۰۰۰۰ دور بر دقیقه) بر ویژگی‌های ساختاری، رئولوژیکی و خاصیت آنتی اکسیدانی اسانس پوست گریپ فروت نانوریز پوشانی شده در دیواره صمغ آلژینات مورد بررسی قرار گرفت. افزایش سرعت هموژنیزاسیون منجر به کاهش اندازه ذرات (از ۲٦٤/۰ تا ۲۲٦/۹ نانومتر) و شاخص پراکندگی ذرات نانوامولسیون (از ۰/۳۲تا ۰/۱۹) شد. تمام نانوامولسیون‌ها پتانسیل زتای منفی (۲۳/٦– تا ۲۱/٦٥– میلی ولت) داشتند. راندمان نانوریزپوشانی اسانس با افزایش سرعت هموژنیزاسیون از ۸۳/۹۲ تا ۷۸/۷٦ درصد کاهش یافت. نانوامولسیون اسانس در دمای ٤ºC به مدت ٥ روز پایداری کامل داشت. در دمای ۲٥ºC با گذشت زمان، پایداری نانوامولسیون کاهش یافت و بیشترین پایداری مربوط به نانوامولسیون‌های تهیه شده با سرعت ۱٥۰۰۰ دور بر دقیقه بود که پتانسیل زتای کوچکتری داشت. نتایج مربوط به طیف بینی فروسرخ تبدیل فوریه و گرماسنجی روبشی تفاضلی نشان دهنده احاطه شدن اسانس توسط آلژینات بود. نانوامولسیون‌های تولید شده دارای فعالیت آنتی اکسیدانی بودند و با افزایش غلظت نانوامولسیون میزان مهار رادیکال آزاد DPPH افزایش یافت. بیشترین فعالیت آنتی اکسیدانی مربوط به نانوامولسیون تهیه شده در سرعت ۱۰۰۰۰ دور بر دقیقه بود. تمامی نانوامولسیون‌ها رفتار رقیق شونده با برش نشان دادند. مدل‌های هرشل بالکلی و پاورلا دارای R^2 بالاتری نسبت به سایر مدل‌های برازش شده بودند. هموژنیزاسیون بر روی مقدار k_p و k_H تقریبا بی اثر بوده است. نتایج این تحقیق استفاده از سرعت ۱٥۰۰۰ دور بر دقیقه را برای تولید نانوامولسیون اسانس پوست گریپ فروت در دیواره صمغ آلژینات مناسب معرفی می‌نماید.