



## Optimization of permeate-based orange beverage foam production by response surface methodology

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### ABSTRACT

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In recent years, the foam mat drying has been used in food processing. Due to the fact that the stability of the foam plays an essential role in improving this process, in this study, the factors affecting the structure of the permeate-based orange beverage foam and the physical characteristics of the foam have been investigated. For this purpose, the egg white (3-5%) and basil gum solution (0.1- 0.3%) was added to the orange beverage according to the experimental design and the desired mixture was mixed with an electric mixer with the highest speed was for (4, 6, 8) minutes. Then, initial tests of beverage foam including foam density, foaming ability, foam stability, rheological test, viscosity, and foam microstructure were performed. Then, the optimal treatment was selected using the RSM and Design Expert software. The optimal treatment introduced by the sample model had a concentration of 4% egg white powder and 0.2% basil gum and the mixing time was 6 minutes, which showed the highest quality in the experiments.

## 1- Introduction

The foam has a two-phase system consists of a dispersed phase, usually air and a continuous phase, in which the dispersed phase is wider than the continuous phase. Based on the dispersed phase - continuous phase ratio, the foams are divided into two categories of multi-layer foams and single-layer foams. In multi-layered foams, this ratio is high, which leads to an increase in the number of bubbles. As the number of bubbles increases, the pressure of the bubbles to each other also increases, which leads to the formation of a honeycomb structure. In single-layer foams this ratio is low, so that in single bubbles, the spherical shape is maintained.

Foams are gas bubbles surrounded by interface. The thin-walled bubble is called a lamella. The mechanical strength of the lamella determines the stability of the foam. If viscous liquids are used to make the foam, and then the foam produced is stable due to the increase in lamella elasticity, and because of the curvature of the boundary layer, the pressure at the bubble boundary is lower. On the other hand, foam stability is restored by surfactants or by protein. Foam is the dispersion of gas in a liquid and is known as solid foam if the liquid form turns into a gel or solid after dispersing the gas. The average size of the bubbles is between 0.1 and 3 mm and there are  $10^3$  bubbles per milliliter, but this number reaches  $10^{11}$  bubbles per milliliter in the emulsion [1-3].

In each system, a different distribution of bubble size is observed based on the foam. In some systems, the same bubble size is desirable. While in some products a wider range improves oral sensation. In general, the size distribution in dispersion depends on operating conditions such as pressure, agitator speed, temperature and chemical composition of the gas. In the production process, it is necessary to control the size and distribution of air bubble particles. Bubble size is used not only to determine the area of the interfacial area, but also in the connection of gas phase bubbles. Bubble size is an important parameter in determining the stability of the foam [4,5].

The formation of foam and its properties are controlled by colloidal forces, surface forces and the interaction between them, and the films separate the gas bubbles. These foams have thin and flat layers between the bubbles. The mechanical strength of the lamella controls the stability of the foam at the air-water interface [6,7]. Most stabilizers are natural polysaccharides with high molecular weight. The hydrophobic nature of these compounds prevents them from being absorbed on the interface. This helps to strengthen the bubble wall and ultimately improve the stability of the foam. Polysaccharides are more common than other types of stabilizers. Proteins can also be used as stabilizers. The stabilizing concentration of the foam must be optimized because the foam is unstable at concentrations below the critical level, while a very high dose of stabilizer prevents air from entering the structure of the liquid food. Foam stabilizers substantially increase the interfacial viscosity in the foam lamella, which in turn increases the foam stability. Various types of stabilizers are used in the stability of foams and emulsions, including gums.

Numerous functional properties of hydrocolloids lead to their widespread use in food products. These properties, which cover a wide range from adhesion to flowability, are due to the common basic property between all gums, such as viscosity and increased consistency. Stability of emulsions, suspensions and dispersions, foaming, film formation, coating and preventing the formation of crystals are other important functional properties of the gums. Gum from seeds is an important food additive in the food industry. Seed mucilages and plant polysaccharides are readily available and are of particular importance due to their reasonable price, and most of them can be used in food formulations. Recently, the demand for hydrocolloids with special functional properties has increased, therefore, finding new sources of plant gums with suitable properties for use in industry is of particular importance and food industry researchers are always looking for new

sources of polysaccharides [2,3]. Basil with the scientific name (*Ocimum basilicum*) is from the genus of Mint. This plant is found in many parts of the world, especially in the tropics of Asia, Africa and South America. Basil seeds contain large amounts of hydrocolloids with remarkable rheological properties that make it comparable to other commercial hydrocolloids. The seeds of this plant contain a heteropolysaccharide structure including glucomannan, xylan and glucan. Basil seed gum is classified as anionic gum with  $\text{pH} = 7.78$ . This gum contains 63.79% carbohydrates and 32.1% protein. Glucose, galactose and mannose are 6.29, 1.16, 9.8% of the major constituent sugars respectively and potassium with 64.2% is the major ions in this gum [8].

Albumin is the major component of egg white protein (about 54%) and its molecular weight is about 42.7 kDa. This protein is widely used in the food industry due to its emulsifying properties and foam production [9]. Albumin is a heterogeneous protein system in which protein-protein interactions occur during the foaming process. The mechanism of egg white foam production is due to the surface denaturation of albumin proteins at the liquid-air surface and the foam stability mechanism is related to the insolubility of albumin. The most important performance of albumin in the foam is to increase the storage time of a large volume of air in the system [10].

Egg albumin contains 40 different and complex proteins. The main foaming proteins in egg whites include ovalbumin, globulin and ovomucoid. Ovalbumin forms a monolayer film between the air-water interfaces. Lysozyme forms a much thicker film than monolayer film. The foaming properties of egg whites are increased by adding sucrose and sodium chloride [11]. In 2017, Affandi et al. studied beverage powder (*Nigella Sativa*) by foam mat method. For this purpose, different amounts of egg white (2.5, 8.75 and 15% w / w) and methyl cellulose (0, 0.5 and 1% w / w) and stirring times of 2, 5 and 8 minutes were examined. The results showed that 15% egg white and 0.69% methylcellulose and stirring time of 8 minutes is the optimal process

conditions for the foam sample prepared [12]. In 2015, Abbasi et al. investigated the physicochemical properties of sour cherry powder by foam mat method, and for this purpose, different amounts of egg white powder (1, 2 and 3 g / 100 g) and methyl cellulose (1, 1.5, and 2 g / 100 g) were examined on the characteristics of sour cherry foam. The results showed that with increasing the concentration of methyl cellulose and egg white, drainage volume and foam density decreased [13]. The purpose of this study was to investigate the performance of different levels of basil gum and egg white powder in creating and stabilizing the foam of orange beverage.

## 2- Materials and methods

### 2-1- Materials

The raw materials used in this research including orange concentrate (Noush Company Mazandaran), permeate powder (Kaleh factory of Amol), basil seeds, egg white powder and sugar were prepared from a store in Sari, Iran.

Equipment used includes home electric mixer (GOSONIC, GHM-818 model, China), digital scale (HS-300S model, Germany) with an accuracy of 0.001, pH meter (CP-511 model, Poland), Pars Khazar juicer (JC-700P model, Iran) and a magnetic stirrer (MS300HS model, Korea).

### 2-2- Gum extraction

Basil seeds were prepared and removed by screening of seed impurities. To extract the gum, basil seeds were first placed in distilled water with a pH of 7 and the water-to-grain ratio of 30 to 1(w/w). The mixture of water and seeds is preheated for 10 to 20 minutes. Separation of gum from seeds was done using the juicer and after filtering, it was kept in the freezer at  $-20^{\circ}\text{C}$  and dried by the freeze dryer (VaCo 5, Zirbus technology, Germany) at  $-50^{\circ}\text{C}$  for 24 hours and after grinding and sieving, it was used for experiments. The gum solution was made by adding the required amounts of gum powder by weight to distilled water to create a concentration of 2% and was mixed with a magnetic stirrer. The gum

solution was then kept in the refrigerator temperature (4 ° C) for 18-24 hours to fully hydrate the gum molecules [14].

### 2-3- Beverage preparation

The formulation of the beverage used in the study, including permeate and orange concentrate, sugar and water were each weighed in terms of brix and specified (w/w) ratio until a final brix of 12 was weighed with a certain percentage and the compounds were mixed by magnetic stirrer until they were completely dissolved [15].

### 2-4- Preparation of foam samples

According to the design of the study, the desired amounts of egg white (3-5%) and basil gum solution (0.1-0.3%) were added to the orange beverage and then the desired mixture was stirred with an electric mixer at the highest speed at room temperature for (4, 6 and 8) minutes. At the end of the stirring time, the foam properties were immediately investigated. Then, the optimal treatment was selected using the response surface method and design-expert software.

### 2-5- Evaluating the characteristics of the foam

#### 2-5-1- Foam density

In order to determine the density of the foam, the weight of 50 ml of the foam sample was determined by using a graduated cylinder at ambient temperature. In order to prevent the destruction of the foam structure and to prevent the formation of air cavities inside the graduated cylinder, the foam was transferred to the cylinder carefully [13].

$$\text{Eq (1) foam density} = M(g) / V(\text{ml})$$

#### 2-5-2- Foam stability

Measuring the foam stability is the most important step in evaluating foam systems. The amount of liquid separated from the foam is the most common method of measuring the stability of the foam. In order to determine the stability of the foam, immediately after the stirring operation, the volume of the produced foam was stated and a certain amount of foam was transferred directly to a glass funnel with the least stress and its weight was measured by a scale with an accuracy of 0.001. The funnel

containing the foam was attached to a clamp and a 10 ml cylinder was placed under it to check the process and measure the amount of water coming out of the foam. The amount of water output at intervals of 5 minutes to 30 minutes from the foam sample, due to gravity at ambient temperature that was collected inside the graduated cylinder, was expressed as the volume of drainage in millimeters [13].

$$\text{Eq (2) Foam stability index} = V_f - V_d / V_1 \times 100$$

#### 2-5-3- Foaming ability

In order to investigate the increase in volume, 50 g of sample after stirring and foaming, was immediately transferred into a graduated cylinder and its volume was recorded and calculated with the following formula [16].

$$\text{Eq (3) volume increase} = V_f - V_1 / V_1 \times 100$$

$$\text{Eq (4) gas phase} = OR / OR + 100$$

$$\text{Eq (5) mixing index} = V_f / V_1 \times 100$$

In Eq (1) to (5):  $V_1$ : initial liquid volume,  $V_f$ : the volume of the foam formed,  $V_d$ : volume of water discharged, OR: volume increase,  $m_d$  is weight of water discharged in 30 minutes,  $m_f$ : weight of foam,  $V_f$ : volume of foam.

#### 2-5-4- Viscosity

In order to measure the viscosity of the samples, 15 ml of each sample was taken, and with the help of Brookfield rotational viscometer and cylindrical spindle number 63, their viscosity at a constant temperature of 4 ° C in the range of shear stress from zero to 96 was investigated and reported per second [17].

#### 2-5-5- Examining the microstructure of the foam

In order to study the morphology of the foams, a light microscope equipped with a camera at a temperature of 25 ° C was used. For this purpose, the foam samples were fixed 2 mm apart immediately after preparation between two lamellae and the bubble size was photographed immediately using 40 X magnification light microscope [18].

#### 2-5-6- Rheological examination

Flow behavior analysis was performed using a rheometer MCR302 (Anton Paar GmbH, Germany) equipped with a thermal circulator

at a temperature of 20 ° C in the linear viscoelastic range. The distance between the plates is considered to be almost 10 times more than the average size of the bubbles, and the scan, strain, frequency scan, temperature scan, and flow behavior tests were investigated. Continuous phase rheological behavior was measured at shear rate (0.02-80 S) at 20 ° C and the data analysis was performed using flow behavior models [19].

### 2-6- Statistical analysis of data

Designing the experiment, analysis of the results and determining the optimal foam production conditions were performed using the response surface method and Design Expert software version 10. In this study, a centralized composite design with three independent variables at three levels and six repetitions at the central point of the design was used and the total number of treatments was 20.

## 3-Results and discussion

### 3-1- Density

One of the important parameters in foam systems is the amount of density. The density of the foam affects the drying process. Lower density leads to shorter drying time. The high amount of trapped air during stirring leads to foam expansion, which indicates the foaming ability. The results obtained from the study of the effect of different concentrations of egg white powder and basil gum on the foam density are shown in Figure (1). The results of analysis of variance were significant at the level of 0.05%. As the stirring time increased, the density of the samples decreased due to more air entering the foam systems. According to the figure, with increasing the concentration of egg white powder from 3 to 5%, due to the increase in the number of bubbles produced in the foam and its increasing volume, the density of the foam showed a significant downward trend. As the concentration of gum increases, the density of the foam increases. When gum is added to a liquid, it increases its viscosity. This increase in viscosity prevents air from entering and reduces the maximum amount of air trapped in the mixture, thus

reducing expansion and increasing the density of the foam. Similar results were reported by Azizpour et al. 2013 [20, 21].

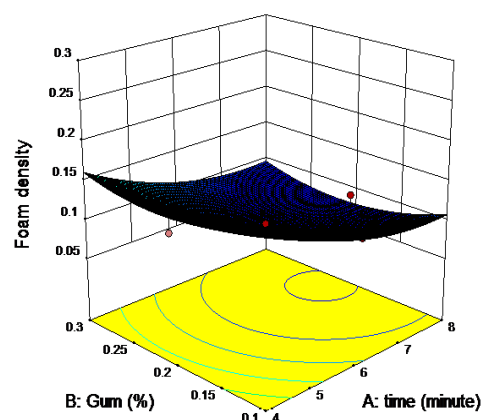


Fig 1 Effect of time and amount of basil gum on foam density.

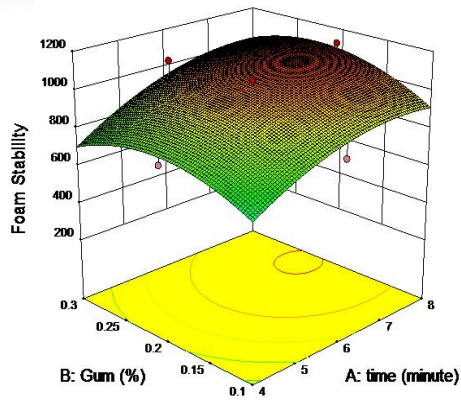
The researchers said that polysaccharide compounds such as gums reduce the accumulation and deposition of proteins and increase their performance in foam systems by increasing the continuous phase viscosity, reducing protein-protein interactions and preserving charged groups. Liquid viscosity plays an important role in the amount of density and a certain level of viscosity is required to create the appropriate amount of density [22].

### 3-2- Foam stability

Determining the stability of the foam is a key and important step in studying the properties of the foam, which is usually determined by measuring the amount of liquid separated from the foam over a specified period of time. The stability of the foam is affected by its physical and rheological properties at the air-liquid interface and continuous phase. The characteristics of the interface, the size distribution of the foam bubbles, the permeability of the interface and the surface tension affect the amount of drainage volume. Liquid flow through the foam structure is due to capillary force or external force such as gravity, which occurs as a result of thinning and rupture of the bubble wall [1, 23].

The results of the effect of basil gum and egg white powder on foam stability are shown in

Figure (2).



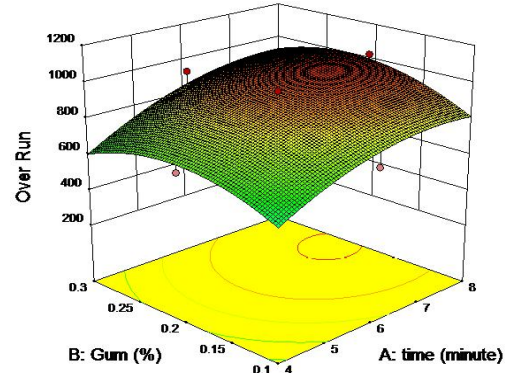
**Fig 2** Effect of time and amount of basil gum on foam stability.

The results show that increasing the amount of gum increases the continuous phase viscosity and viscoelastic and elastic properties of the lamella, in which case the bubble wall strength increases, leading to increased strength of the foam structure and prevention of the bubble collapse. Increasing the viscosity of the liquid phase creates a lattice structure in the continuous phase that protects the interface wall from collapsing and improves the stability of the foam. Initially, with the increase of basil gum and egg white powder, an increasing trend in foam stability is observed, and the viscosity of the liquid phase prevents the retention of the air bubbles during the stirring process, and the foam stability at higher concentrations of gum shows a decreasing trend. Flade et al. (2003) reported that by increasing the total solid content of the nutrient, the stability of the foam increases, which in turn reduces the volume of drainage [24]. The high energy at the air-liquid interface and the density difference between the two phases cause the foam to be thermodynamically unstable [25]. In 2011, Bag et al. reported in their study that increasing the viscosity of the liquid phase creates a lattice structure in the continuous phase, by which the interface of the bubbles is protected from collapsing, and thus the conditions for improving the foam stability are provided [16]. If the size of the air bubbles in the suspension becomes smaller than usual, the foam stability decreases significantly

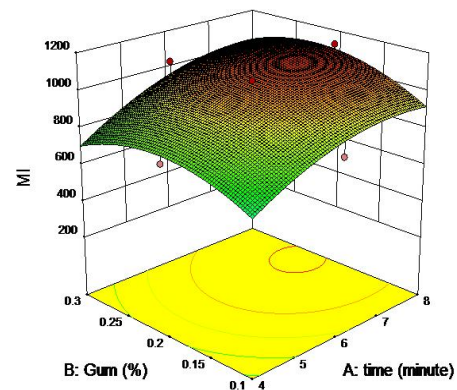
compared to when larger bubbles are in the sample [29].

### 3-3- Foaming ability

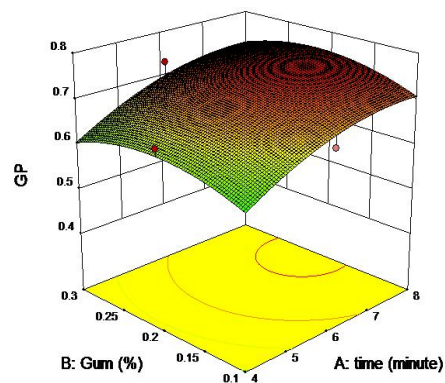
The results of the effect of basil gum and stirring time on foaming ability are shown in Figures (3).



(a)



(b)



(c)

**Fig 3** Effect of time and amount of basil gum on (a) over run, (b) mixing index, and (c) gas phase

The results of analysis of variance were significant at the level of 0.05%. As the egg

white concentration increases, the foam density decreases and its volume increases. High viscosity of the liquid phase prevents the retention of air bubbles during the stirring process [26]. When gum is added to a liquid, it increases its viscosity. This excessive increase of viscosity prevents air from entering and reduces the maximum amount of air trapped in the mixture. Therefore, it reduces expansion and consequently reduces the volume of the foam [13]. Increasing the foaming agent increases the viscosity and yield tensile strength of the continuous phase, and increases the strength of the films in the air-water interface. Egg whites reduce foam density because of their high foaming proteins due to their denaturation and increase in hydrophilic groups on the surface [27].

### 3-4- Viscosity

Hydrocolloids are used to create stability and constant viscosity and prevent food hydration during heat treatment. Hydrocolloids increase viscosity by dissolving or dispersing in water. Increased basil gum played an important role in foam stability, so that its increase could increase the strength of air bubble walls by increasing the continuous phase viscosity and

prevent their collapse. The effect of basil gum concentration and stirring time on the foam viscosity of orange beverage is shown in Figure (4).

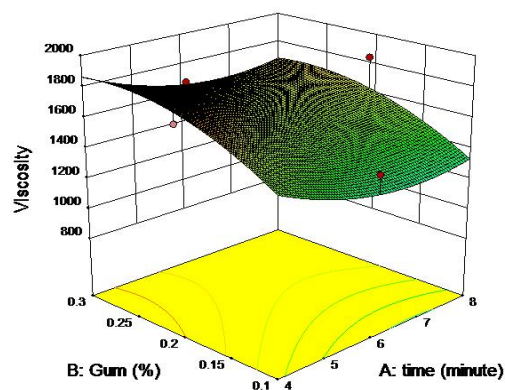


Fig 4 Effect of time and amount of basil gum on viscosity.

The results of analysis of variance were significant at the level of 0.05%. With increasing of the amount of basil gum, an increasing trend was observed in the viscosity of the samples.

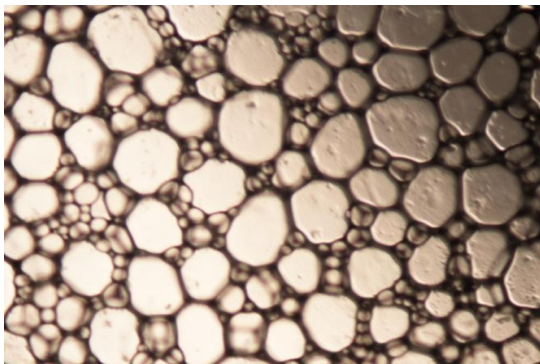
**Table 1** Quadratic equation and coefficient of determination ( $R^2$ ) of foaming properties.

Foaming properties	Equation	$R^2$
Foam density	$= 0.092 - 0.034 \times A - 7.100E-003 \times B - 0.017 \times C + 1.625E-003 \times AB + 0.038 \times AC + 0.011 \times BC + 0.022 \times A^2 + 0.023 \times B^2 + 0.026 \times C^2$	0.966
Foam stability	$= 1045.14 + 141.18 \times A + 37.35 \times B + 14.73 \times C + 21.99 \times AB - 133.19 \times AC - 48.09 \times BC - 91.81 \times A^2 - 122.96 \times B^2 - 157.24 \times C^2$	0.976
Over run	$= 946.62 + 141.67 \times A + 36.58 \times B + 14.62 \times C + 22.02 \times AB - 133.23 \times AC - 48.02 \times BC - 90.24 \times A^2 - 124.78 \times B^2 - 156.63 \times C^2$	0.975
Mixing index	$= 1047.14 + 141.72 \times A + 35.90 \times B + 14.58 \times C + 22.08 \times AB - 133.29 \times AC - 48.08 \times BC - 91.55 \times A^2 - 122.43 \times B^2 - 157.94 \times C^2$	0.976
Gas phase	$= 0.73 + 0.058 \times A + 0.0900E-003 \times B + 0.035 \times C + 2.125E-003 \times AB - 0.061 \times AC - 0.031 \times BC + 0.033 \times A^2 + 0.039 \times B^2 - 0.044 \times C^2$	0.959
Viscosity	$= 1604.29 - 91.10 \times A + 172.8 \times B + 53.80 \times C + 1 \times AB - 76.50 \times AC + 86.50 \times BC + 117.77 \times A^2 - 120.73 \times B^2 - 235.73 \times C^2$	0.894

A-time (min), B- Gum (%), C- Egg white(%).

### 3-5- Foam structure

Figure (5) shows the optimal sample structure of the beverage foam. Increasing the concentration of gum and egg white powder had a significant effect on the size of the bubbles, so that with the increase of each of these values, the diameter of the bubbles decreased.



**Fig 5** The optimal sample (with a concentration of 4% egg white powder and 0.2% basil gum and stirring time of 6 minutes) structure of the beverage foam.

Due to their dual nature, proteins can be located in the between surfaces, while reducing surface tension; they can more easily decompose foam bubbles and eventually create smaller bubbles [28].

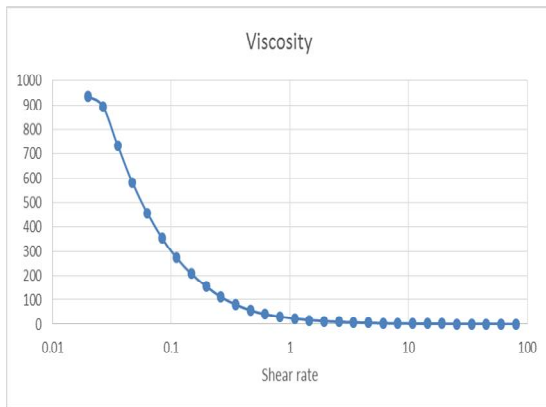
Increasing the concentration of gums reduced the size of the bubbles. As the viscosity of the liquid phase increases, the connection of the bubbles to each other decreases, this causes the increase of the bubbles stability and consequently their smaller size [29]. Increasing the viscosity of the continuous phase prevents the lamellae from becoming too thin between the bubbles, and as a result the shape of the bubbles remains spherical in the environment instead of becoming multifaceted. As the percentage of basil gum and egg white powder increases, the size of the foam pores decreases due to the increase in density. Bubble size is a very important parameter to understand the effect of the process parameters on the foam structure. The size of the foam bubbles is affected by the properties of the liquid and the air-liquid interface, the pH of the solution, the protein concentration, the surface viscosity and the surface tension.

### 3-6-Foam rheology

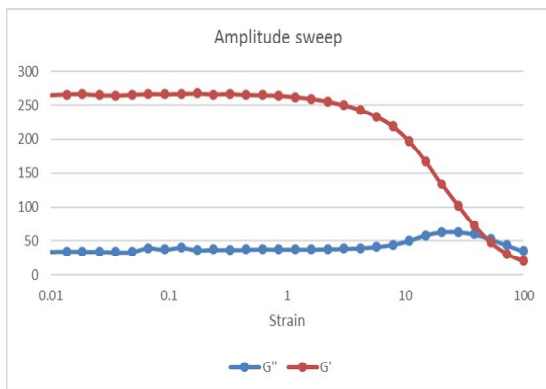
Under the influence of frequency, elastic modulus and viscous modulus showed a constant trend at first and then an increasing trend. The elastic modulus showed higher values compared to the viscous modulus. The results also showed that under the influence of shear stress, the viscous modulus shows a decreasing trend. The results of strain sweep showed that under the influence of the applied stress, first the elastic modulus and viscous modulus showed a constant trend and then a decreasing trend was shown in each of the values. The elastic modulus showed higher values, indicating the elastic behavior and resistance of the foam under stress. Factors such as concentration, molecular weight, dispersion, structure and properties of the solvent affect the frequency sweep. Increasing the loss modulus by increasing the frequency can be interpreted as when the low frequencies are applied to the material, it has enough time to repair the broken bonds in the frequency cycle, but when high frequencies are applied, the material does not have the opportunity to repair the broken bonds, and when the bonds are broken, the viscous modulus increases. Higher storage modulus than the loss modulus indicates the presence of a elastic structure in the system. When elastic modulus crossed viscous modulus indicates the structural changes in beverage foam that indicates a structure rupture and start of the flow behavior. We choose the strain of 1% in linear viscoelastic region.

The structure of the foam is effective in the rate of moisture transfer from the foam sample during the foam mat drying process and finally the quality of the final powder. The optimized foam should be stable for up to one hour after preparation during the drying process. In this case, it can be stated that the powder prepared by the foam mat drying method has a good quality [1, 30]. Figures (6) show the flow behavior, amplitude sweep and frequency sweep of the optimal foam sample.

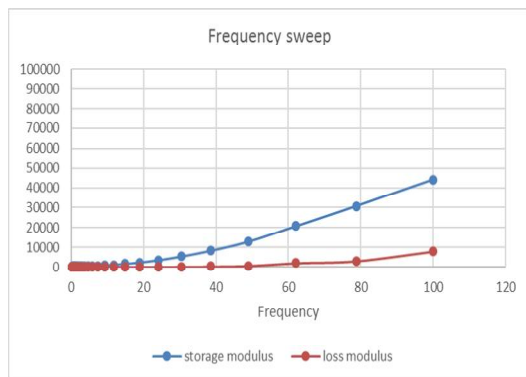




(a)



(b)



(c)

**Fig 6** The (a) flow behavior, (b) amplitude sweep and (c) frequency sweep of the optimal foam sample.

### 3-7- Determining the optimal conditions of foam production

In order to produce the desired foam in terms of density and drainage volume of the foam, the optimal ratio of orange beverage and basil gum and egg white powder and stirring time were determined using the design technique of design expert software. In this study, the purpose of optimization was to produce orange

beverage foam with minimum density and drainage volume in the concentration ratio of 4 g of egg white powder, 0.2% of basil gum and stirring time of 6 minutes.

## 4- Conclusion

Preparing stable foam with optimal density and high stability is one of the most important factors in the process of foam mat drying and the quality of the final powder. Therefore, the structure of the foam is effective in the rate of moisture transfer from the foam sample during the foam mat drying process and finally the quality of the final powder. Based on this, the optimal conditions for producing orange beverage foam were the optimal sample introduced by the sample model with a concentration of 4% egg white powder and 0.2% basil gum and stirring time of 6 minutes, which showed the highest quality in the experiments.

## CONFLICT OF INTEREST

The authors declare no conflict of interest.

## ETHICAL APPROVAL

The human and animal testing was unnecessary in the current study.

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## بهینه سازی تولید کف نوشیدنی پرتقالی به روش سطح پاسخ

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<p>تاریخ های مقاله :</p> <p>تاریخ دریافت: ۱۴۰۰/۱۲/۱۱</p> <p>تاریخ پذیرش: ۱۴۰۱/۰۶/۳۰</p>	<p>در سال های اخیر برای خشک کردن مواد غذایی از روش کف پوشی استفاده می شود. با توجه به این که پایداری کف حاصل در بهبود این فرآیند نقش اساسی دارد لذا در این پژوهش به بررسی عوامل مؤثر بر ساختار کف نوشیدنی و ویژگی های فیزیکی کف حاصل پرداخته شده است به این منظور مقدار مورد نظر از سفیده تخم مرغ (۵-۳٪) و محلول صمغ ریحان (۳-۱/۰) درصد بر اساس طرح آزمایش، به نوشیدنی پرتقالی اضافه شد و مخلوط مورد نظر با هم زن برقی با بیشترین سرعت به مدت (۴، ۶، ۸) دقیقه همزده شد. سپس آزمون های اولیه کف نوشیدنی شامل دانسیته کف، قابلیت کف زایی، پایداری کف، بررسی رئولوژیکی، ویسکوزیته، بررسی ریز ساختار کف انجام شد. سپس با استفاده از روش سطح پاسخ و نرم افزار دیزاین اکسپرت تیمار بهینه انتخاب گردید. تیمار بهینه معرفی شده توسط مدل نمونه با غلظت ۴٪ پودر سفیده تخم مرغ و ۲/۰٪ صمغ ریحان و زمان هم زدن ۶ دقیقه بود که در آزمایشات صورت گرفته بالاترین کیفیت را نشان داد.</p>
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