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Influence of different hydrocolloids on the rheological behaviour of low caloric food deserts

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Abstract

Background: This study is focused on the steady rheological properties of the a-caloric dairy dessert containing carrageenans, agar-agar and xanthan gums characterization. The flow behaviour of the samples was fitted to Power Law, Herschel- Bulckely, Bingam, Sisko and Moore models to see which one is describing better the rheological parameters evolution. The Power Law model is the suitable one for predicting the rheological parameters. All the samples exhibited a pseudoplastic behaviour. The rheological parameters modelling based on the mixture design was made using a 3rd grade polynomial equation with variables, all the models have a regression coefficients greater than 0.99. The optimization of the rheological parameters, in function of the proportion of the hydrocolloids, so the parameters be maximum have lead to 2 combinations.

Keywords: food desert, hydrocolloids, mix design, polynomial model, optimization

1. Introduction

Now days, the dairy desert are very popular for children, young and older people too worldwide. However due to the highly interest of people to consume low caloric products, for maintain their weight or for diabetes people which could not consume sugar based products, is an increasing demand on this type of products. These products are based on hydrocolloids, skimmed milk and artificial sweetener. The rheology of food products is very important because flow properties define food structure during industrial manufacturing or preparation in the kitchen and this is physiologically important in the mouth, stomach and intestine where food structure is perceived and digested [1]. The rheological characteristics of dairy desserts mainly depend on the fat content of milk, type and concentration of starch, type and concentration of hydrocolloids and their interactions [2,3]. The use of two or more gums in the formulation of the product is very widespread in the food industry due essentially to the synergistic effect of combined use [4].

However, no other studies have been made on low caloric gels using inulin as sugar replacer in combination with hydrocolloids and skimmed milk.

Hydrocolloids are bio-polimers which strongly interacts with water. Agar-agar is a natural produce which is find in marine algae from *Galidiaceae, Sphaerococcaceae* families. Agar-agar has an important industrial application due to it great gel formation and thickening abilities [5]. Xanthan gum is a fermentation gum and is used because of its pseudoplastic behaviour and for the pseudoplastic solutions forming at low concentration [6]. Guar gum is extracted from *Cyamopsis tetragonalobus*, is used in food industry because is generating strong gels at low concentrations.

The aim of this paper is to study the influence of three hydrocolloids (agar-agar, guar gum and xanthan gum) on the rheological properties of desert gels, to model the rheological parameters in function of the hydrocolloids portions and to optimize the hydrocolloids mix so the rheological parameters to be maximum.

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2. Materials and methods

2.1 Materials. In this study were prepared a-caloric desert gels using inulin, skimmed powder milk, starch and a hydrocolloids mix made using agar-agar, xanthan gum and guar gum (purchased from Enzymes and derivates, Piatra Neamt, Romania). The food gels were prepared as: 30 g inulin, 22.5 skimmed milk powder, 10 g starch and 0.75 g hydrocolloids mix. 19 hydrocolloids mixes were prepared as it is shown in table 1. The food gel was prepared by mixing 63.25 g of the mix in a Erlenmeyer flask which contains 250 ml distilled water. The mix was shaken on a water bath at °C using an agitator with an anchor device at 200 rpm for 15 min. The gels were cooled at the room temperature for 2 h and then were kept for 24 h at 5 °C.

During the experiment, the hydrocolloid quantity added is kept constant and the measured parameters are modifying with the hydrocolloids portion. The level of the portion is ranged between 0 – 1 (the concentration ranges between 0 – 100%). The hydrocolloids combinations are presented in table 6.1.. The sum of the portion is equal to 1, representing the total mix, as:

$$0 \le x_i \le 1,\ i = 1, 2, \ldots, q \text{ şi } \sum_{i=1}^{q} X_i = 1 \qquad (1)$$

where q represents the number of ingredients and X_i represents the portion of i ingredient in the mix.

pH, concentration and refractive index measurement

The pH measurement was made using a portable pH-meter from Lange, the concentration (°Brix) and refractive index measurements were made using a Leica Mark II refractometer. The parameters were measured at 20 °C. All the measurements were made in triplicate.

2.2 Rheological properties determination.The rheological parameters have been measured using RV-DV II Pro (Brookfield Engineering Inc.) using RV spindles (RV2). The spindle nose was used in accordance with the sample nature to get all readings within the scale. The samples in 300 ml of beaker with a 8.56 cm diameter (according to the Brookfield requests) were kept in a thermostatically controlled bath for about 10 min before measurements in order to attain the desirable temperature of 5 °C. The viscosity measurement was realized at 1 – 100 rpm.

All measurements were taken in triplicate.

The flow curves of the food desert were described by Power-Law, Bingham, Herschel- Bulckley, Sisko and Moore models.

The Power Law model gives a good description of fluid flow behavior in the shear rate range available by most rheological instruments; therefore it has been extremely used in studies on food operations. The model equation is:

$$\eta = K_P \cdot \dot{\gamma}^{n_P - 1} \qquad (2)$$

Where η – is apparent viscosity (Pa·s), $\dot{\gamma}$ - shear rate (1/s), K_P – consistency index (N·s^n/m^2), n_P – flow index (dimensionless).

Herschel-Bulckley model is presented in the next equation:

$$\tau = \tau_0 + K_H \dot{\gamma}^{n_H} \qquad (3)$$

Where τ – shear stress (Pa), τ_0 – yield stress (Pa), $\dot{\gamma}$ - shear rate (1/s), K_H – consistency index (N·s^n/m^2), n_H – flow index (dimensionless).

Bingham model is presented in the next equation:

$$\tau = \tau_{0B} + \mu_B \dot{\gamma} \qquad (4)$$

Where τ – shear stress (Pa), τ_{0B} – Bingham yield stress (Pa), μ_B- Bingham viscosity (Pa·s), $\dot{\gamma}$ - shear rate (1/s).

Sisko model is presented in the newt equation:

$$\eta = \eta_\infty + K_S \cdot \dot{\gamma}^{n_s - 1} \qquad (5)$$

Where K_S and n_s are the consistency index and the flow index of the Sisko model, and η_∞ stands for the so-called high shear rate limiting viscosity.

Moore model is presented in the newt equation:

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + \tau \dot{\gamma}} \qquad (6)$$

Where $\eta_0$and τ are shear rate viscosity and relaxation time respectively.

2.3. Statistical analysis. Statistical analysis was performed using version 5.1 of the Statgraphics Plus software system. The data corresponding to each variable were analyzed by one-factor analysis of variance (ANOVA). Multiple comparisons were performed using the least significant difference test (LSD), and statistical significance was set at α00.05. In addition to this, the data were analyzed by using multivariate techniques, applying the software SPSS 16.0 (IBM, USA) and Design Expert 6.0 (Stat-Ease

Inc. USA). The Principal Components Analysis (PCA) was performed using Unscramble X 10.1 (CAMO Process AS, Oslo, Norway), it was applied to describe the relationship among the physicochemical and rheological parameters.

3.Results and discussions

In table 2 are presented the values of pH, concentration and refractive index of the food gel analysed. The pH ranged between 6.98 –7.06; the values are closed to the one reported in the case of other food gels [7, 8]. The relative values closed of the 19 formulations can result from the molecular structure of the hydrocolloids used and from the interaction with others ingredients from the mix [9]. The refractive index ranged between 1.3584-1.3613.

The concentration ranged between 16.75-18.55; this difference is due to the different absorption capacity of the hydrocolloids molecules and from the interaction with others ingredients from the mix [9]. The highest concentration was reached in the case of the sample 9 (a mix formed using 0.50 g xanthan gum and 0.25 guar gum and in the case of sample 18 (a mix of 0.3 g xanthan gum, 0.3 g agar-agar and 0.15 g guar gum). The lowest concentration was observed in the case of sample 3 (a mix of 0.75 g guar gum). The concentration are closed to others food gels [10].

a. Rheological properties

The flow curves of the deserts were analysed using Power-Law, Bingham, Herschel-Bulkley, Sisko and Moore models to see which one is suitable for the rheological parameters modelling. In table 3 are presented the regression coefficients for the fifth models. It can be observed that Power Law model is the suitable one for the rheological parameters modelling. The regression coefficients are decreasing in the next way: Power Law > Sisko model > Herschel-Bulkley model > Moore model > Bingham model. The Bingham model is reaching the smallest regression coefficients. The parameters used below are corresponding to the Power Law model.

In table 4 are presented the rheological parameters analysed (consistency index, flow index, dynamic viscosity, shear stress and phase angle) of the desert samples using the Power Law model.

The consistency index, K, ranged between 13.592 $N{\cdot}s^n/m^2$ (S1) to 71.791 $N{\cdot}s^n/m^2$ (S17).

The dynamic viscosity ranged between 0.944 Pa·s (S1) to 3.336 Pa·s (S9). The sample S9 was made mixing 0.25 g xanthan gum with 0.5 g guar gum leading to the highest dynamic viscosity. The synergism between xanthan gum and guar gum was observed and by others authors [11]; they observed the appearance of intermolecular interactions between guar gum and xanthan gum which are increasing the viscosity. This mechanism was explained by Mannion et al. [12]; they consider that gelatinization of xanthan and galactomannans (in our case guar gum) is explained by two mechanisms. The first one appears at room temperature, where the xanthan molecules are interacting and forming weak gels, which are influenced by the galactose content from galactomannans. The second one is assuming that by heating the sugars and by the interaction between the disorderly molecules of xanthan gum is generating strong gels which depend on the structural form of the galactomannans.

The flow index, n, ranged between 0.213 – 0.513; all the values were under 1, this fact confirms the pseudoplastic nature of the desert samples analysed.

In table 6 is presented the Pearson correlation matrix of desert samples. The pH is negative influenced by consistency index (r = -0.581), dynamic viscosity (r=-0.542) and shear stress (r=0.980). The °Brix is strongly correlation with refractive index (r = 0.980). The dynamic viscosity is positively correlated with consistency index (r = 0.957) and shear stress (r=0.945).

b.Rheological parameters modelling

The data model regarding the prediction of rheological parameters (K, n, η_{50}, τ_{50}, δ) of food gels in function of the proportion of the hydrocolloid used in their making (agar-agar, xanthan gum and guar gum) was made using a 3rd grade polynomial model with variables (3). The measured and predicted values have been compared to see the suitability of the model. The equation of the model is as given (eq. 7.):

$$A = b_0 + \sum_{i=0}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n} b_{iii} x_i^3 + \sum_{i<j<k}^{n} b_{ijk} x_i x_j x_k + \sum_{i<j}^{n} b_{ij} x_i x_j + \sum_{i<j}^{n} b_{ij} x_i^2 x_j + \sum_{i<j}^{n} b_{ij} x_i x_j^2 \quad (7)$$

where A is the parameter predicted (K, n, η_{50}, τ_{50}), b_0 is a constant that fixes the response at the central point of the experiments, bi – regression coefficient for the linear effect terms, b_{ij} – interaction effect terms, b_{ii} – quadratic effect terms and b_{iii} – cubic effect terms.

In table 6 and 7 are presented the correspondence between actual and coded values of design variables.

The design parameters (X_1-X_3) have been modeled in order to achieve the model. The model summary is presented in tabel 8. In table 8 are presented the equations form the rheological parameters modelling; in all the cases the coefficients of regression was greater than 0.90.

In figures 1-4 are plotted the predicted values versus measured values of the rheological parameters. It can be seen that the pairs of values are closed to the line with the equation x = y.

3.4. Parameter optimization

The optimization of parameters simultaneously is very important for product quality. The desirability function approach is used to optimize the multiple characteristics concurrently.

In the desirability function approach, first each characteristic, y_i, is converted into an individual desirability function, d_i, that varies over the range,

$$0 \le d_i \le 1 \quad (8)$$

If the characteristic yi is at its target, then d_i =1. If the characteristic is outside an acceptable region, then d_i = 0. Finally, the design variables can be chosen to maximize the overall desirability

$$D = (d_1 x d_2 x \dots x d_n)^{1/n} \quad (9)$$

where n is the number of characteristics.

When the target (T) for the characteristic y is a maximum value and the lower limit is denoted by:

$$L,\ d_i = \begin{cases} 0 & y < L \\ \left(\frac{y-L}{T-L}\right)^r & L \le y \le T \\ 1 & y > T \end{cases} \quad (10)$$

When the target (T) for the characteristic y is a minimum value and the upper limit is denoted by U, [13].

$$d_i = \begin{cases} 0 & y < T \\ \left(\frac{U-y}{U-T}\right)^r & T \le y \le U \\ 1 & y > U \end{cases} \quad (11)$$

In this study, the desirability value of n was calculated by Eq. 11, while desirability values of other characteristics were calculated by Eq. 10. The exponent r is referred to weight and specified as 1. Overall desirability, which represents the desirability of all characteristics simultaneously (D), was calculated by Eq. 9. Two of the gum combinations yielding the largest D values and predicted characteristic values are given in Table 9. Making tradeoffs between these parameters were possible.

3.5.Principal component analysis

A PCA was conducted to evaluate the global effect of physicochemical and rheological properties on the sample type from a descriptive point of view. Figure 5 and 6 are presenting the sample scores and compound loadings of the PCA analysis performed. It was found, that two principal components (PCs) explained 100% of the variations in the data set. The PC1 explains 92% of variability, and the PC2 explains 8% of the variability.

In figure 5 is observing very well two different groups. The PC2 is dividing the samples in two groups: one group, situated in the left part, is formed by the gels obtained using the three hydrocolloids mixed in different proportions, while the food gels situated in the right part are gels obtained using one of two hydrocolloids.

In figure 6 are presented the parameters loadings of food gels. The closeness of flow index (n) to the centre of the two coordinates shows that these parameters do not influence the descriptive view. The refractive index and concentration are closed one to the other one having the same influence to the variation. The rheological parameters which have the greater influence on the variance are situated in the outline ellipse (shear stress (T) and consistency index (K)).

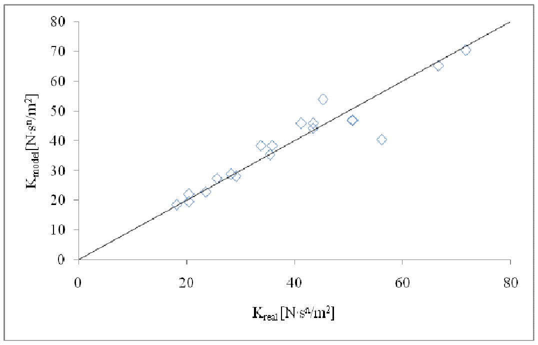

Figure 1. Graphical representation of measured values versus model values for consistency index

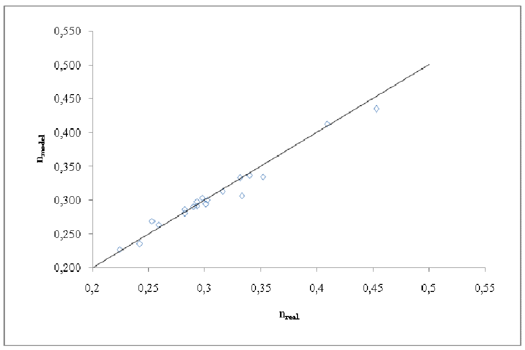

Figure 2. Graphical representation of measured values versus model values for flow index

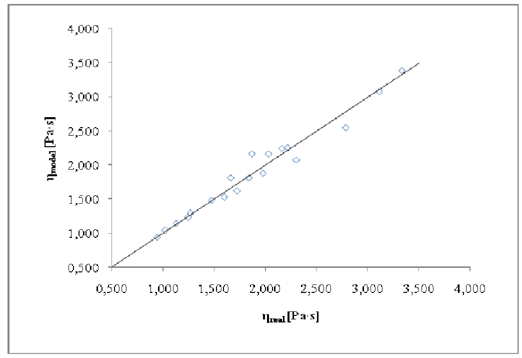

Figure 3. Graphical representation of measured values versus model values for dynamic viscosity

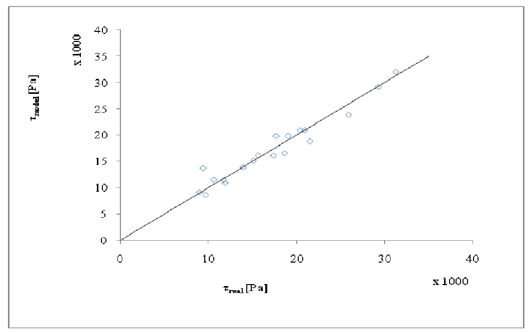

Figure 4. Graphical representation of measured values versus model values for shear stress

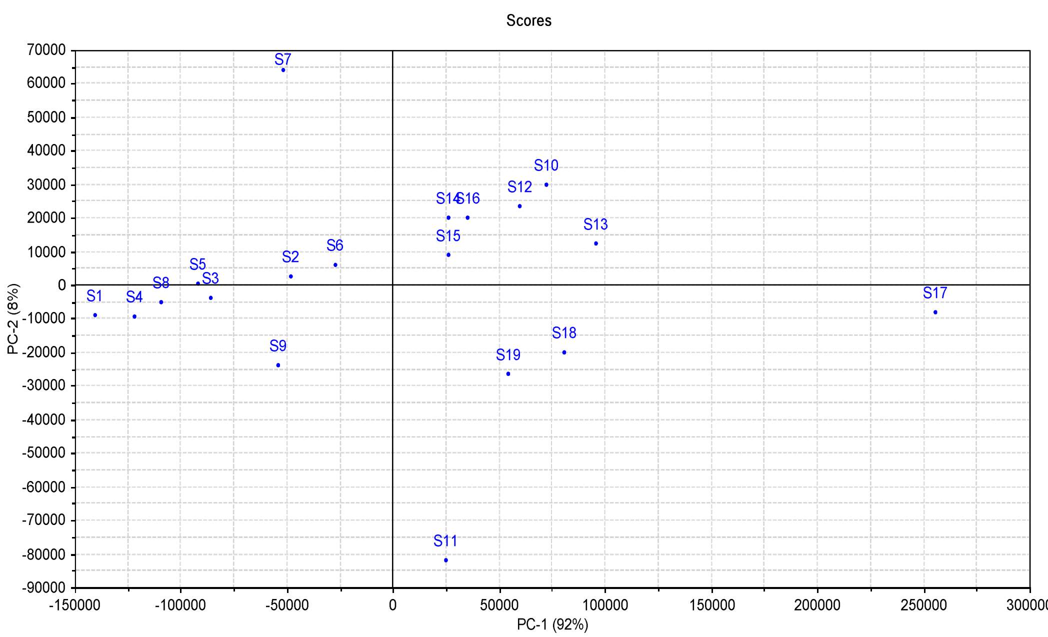

Figure 5. Principal component analysis of the physicochemical and rheological parameters of food gels

Table 1. Hydrocolloids mix composition used in the food product

Sample	Hydrocolloid (proportion)			Hydrocolloid (g/250 g)		
	Agar-agar	Xanthan gum	Guar gum	Agar-agar	Xanthan gum	Guar gum
1	1	0	0	0,75	0	0
2	0	1	0	0	0,75	0
3	0	0	1	0	0	0,75
4	0,667	0,333	0	0,50	0,25	0
5	0,667	0	0,333	0,50	0	0,25
6	0,333	0,667	0	0,25	0,50	0
7	0	0,667	0,333	0	0,50	0,25
8	0,333	0	0,667	0,25	0	0,50
9	0	0,333	0,667	0	0,25	0,50
10	0,334	0,333	0,333	0,25	0,25	0,25
11	0,5	0,25	0,25	0,375	0,1875	0,1875
12	0,25	0,5	0,25	0,1875	0,375	0,1875
13	0,25	0,25	0,5	0,1875	0,1875	0,375
14	0,4	0,3	0,3	0,3	0,225	0,225
15	0,3	0,4	0,3	0,225	0,3	0,225
16	0,3	0,3	0,4	0,225	0,225	0,3
17	0,2	0,4	0,4	0,15	0,3	0,3
18	0,4	0,2	0,4	0,3	0,15	0,3
19	0,4	0,4	0,2	0,3	0,3	0,15

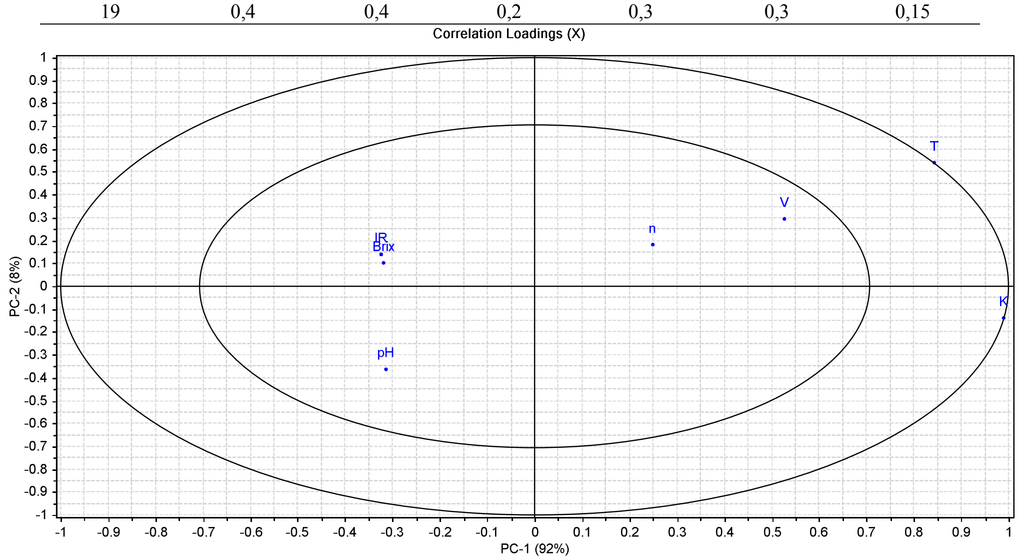

Figure 6. Principal component analysis of the physicochemical parameters loadings of food gels

Table 2. pH, concentration (°Brix), refractive index of food gel

Sample	pH	°Brix	Refractive index
1	7.06	18.35	1.3610
2	6.99	17.95	1.3604
3	7.02	16.75	1.3584
4	7.00	18.15	1.3606
5	7.04	18.25	1.3601
6	6.99	17.55	1.3597
7	6.99	17.95	1.3604
8	7.05	17.15	1.3590
9	6.99	18.55	1.3613
10	7.02	17.45	1.3595
11	7.05	16.95	1.3587
12	7.01	17.35	1.3594
13	7.03	17.45	1.3596
14	7.03	17.75	1.3601
15	7.02	17.15	1.3590
16	6.98	17.85	1.3602
17	6.98	17.15	1.3590
18	7.01	18.55	1.3614
19	7.03	17.45	1.3595

Table 3. The regression coefficients for the Power Law, Herschel Bulckely, Bigham, Moore and Sisko models

Sample	Power Law	Herschel Bulckely model	Bingham model	Sisko model	Moore model
S1	1	0.995	0.842	0.997	0.983
S2	0.998	0.996	0.901	0.995	0.917
S3	0.999	0.999	0.902	0.994	0.894
S4	1	0.997	0.917	0.996	0.95
S5	1	0.999	0.824	0.997	0.941
S6	1	0.999	0.969	0.995	0.983
S7	0.994	0.985	0.943	0.992	0.955
S8	1	0.999	0.900	0.996	0.943
S9	0.999	0.948	0.766	0.994	0.983
S10	0.998	0.992	0.831	0.992	0.922
S11	0.999	0.965	0.638	0.993	0.93
S12	0.998	0.993	0.860	0.994	0.929
S13	0.999	0.998	0.968	0.995	0.987
S14	0.997	0.991	0.928	0.997	0.995
S15	0.999	0.983	0.855	0.993	0.946
S16	0.998	0.995	0.873	0.995	0.948
S17	0.998	0.998	0.963	0.996	0.965
S18	1	0.983	0.84	0.997	0.976
S19	0.997	0.992	0.782	0.996	0.904
Media	***0.998***	***0.989***	***0.868***	***0.994***	***0.950***

Table 4. Power Law model parameters

Sample	K	n	η	τ	R^2
S1	13.592	0.282	0.944	8904.693	1
S2	28.356	0.316	1.472	13885.28	0.998
S3	18.186	0.331	1.248	11696.84	0.999
S4	20.512	0.259	1.024	9659.328	1
S5	25.648	0.224	1.264	11847.77	1
S6	29.180	0.293	1.601	15092.7	1
S7	66.673	0.302	3.112	29279.84	0.994
S8	20.469	0.293	1.128	10564.89	1
S9	71.791	0.301	3.336	31241.89	0.999
S10	43.454	0.242	2.304	21507.1	0.998
S11	35.441	0.282	1.720	9357.474	0.999
S12	50.799	0.409	2.160	20375.15	0.998
S13	50.718	0.253	2.216	20903.39	0.999
S14	56.146	0.453	1.976	18639.48	0.997
S15	43.448	0.29	1.872	17658.46	0.999
S16	41.258	0.298	2.032	19016.8	0.998
S17	45.316	0.34	2.790	25883.98	0.998
S18	35.942	0.352	1.840	17356.61	1
S19	33.802	0.333	1.664	15620.94	0.997

Table 5. Pearson correlation of desert samples

Variabila	pH	Brix	Refractive index	Consistency index	Flow index	Dynamic viscosity	Shear stress
pH	1						
Brix	-0,179	1					
Refractive index	-0,233	0,980**	1				
Consistency index	-0,581**	-0,015	0,035	1			
Flow index	-0,394	-0,380	-0,376	0,215	1		
Dynamic viscosity	-0,542*	0,084	0,138	0,957**	-0,002	1	
Shear stress	-0,599**	0,161	0,214	0,945**	0,050	0,971**	1

Table 6. Correspondence between actual and coded values of design variables

Design variables	Symbol	Actual values of coded levels	
		-1	+1
Agar-Agar (g/250 g)	X_1	0	0,75
Gumă Xanthan (g/ 250 g)	X_2	0	0,75
Gumă Guar (g/250 g)	X_3	0	0,75

Table 7. Real values and reduced values of hydrocolloids mix

Sample	Agar-agar (g/250 g)	Xanthan gum (g/ 250 g)	Guar gum (g/250 g)	X_1	X_2	X_3
1	0.75	0	0	1	-1	-1
2	0	0.75	0	-1	1	-1
3	0	0	0.75	-1	-1	1
4	0.5	0.25	0	0.333333	-0.33333	-1
5	0.5	0	0.25	0.333333	-1	-0.33333
6	0.25	0.5	0	-0.33333	0.333333	-1
7	0	0.5	0.25	-1	0.333333	-0.33333
8	0.25	0	0.5	-0.33333	-1	0.333333
9	0	0.25	0.5	-1	-0.33333	0.333333
10	0.25	0.25	0.25	-0.33333	-0.33333	-0.33333
11	0.375	0.1875	0.1875	0	-0.5	-0.5
12	0.1875	0.375	0.1875	-0.5	0	-0.5
13	0.1875	0.1875	0.375	-0.5	-0.5	0
14	0.3	0.225	0.225	-0.2	-0.4	-0.4
15	0.225	0.3	0.225	-0.4	-0.2	-0.4
16	0.225	0.225	0.3	-0.4	-0.4	-0.2
17	0.15	0.3	0.3	-0.6	-0.2	-0.2
18	0.3	0.15	0.3	-0.2	-0.6	-0.2
19	0.3	0.3	0.15	-0.2	-0.2	-0.6

Table 8. Equations and regression coefficients of the rheological parameters

Parameter	Equation	R^2
K	$K = 58.66 + 12.84 \cdot X_1 + 22.54 \cdot X_2 + 7.78 \cdot X_1^2 - 6.88 \cdot X_2^2 - 42.91 \cdot X_3^2 -$ $+ 5.32 \cdot X_2^3 - 9.26 \cdot X_3^3 - 32.00 \cdot X_1^2 \cdot X_2$	**0,901**
n	$n = 0{,}111 - 0{,}287 \cdot X_1 - 0{,}235 \cdot X_2 + 0{,}115 \cdot X_1^2 - 0{,}157 \cdot X_2^2 + 0{,}248 \cdot X_3^2$ $\cdot X_1^3 - 0{,}133 \cdot X_2^3 + 0{,}037 \cdot X_3^3 + 0{,}354 \cdot X_1^2 \cdot X_2$	**0,972**
η_{50}	$\eta = 2{,}615 + 0{,}259 \cdot X_1 + 1{,}042 \cdot X_2 + 0{,}151 \cdot X_1^2 - 0{,}229 \cdot X_2^2 - 1{,}877 \cdot X_3^2 \cdot$ $\cdot X_1^3 + 0{,}110 \cdot X_2^3 - 0{,}555 \cdot X_3^3 - 1{,}579 \cdot X_1^2 \cdot X_2$	**0,965**
τ_{50}	$\tau = 22652.96 - 890.114 \cdot X_1 + 9776.126 \cdot X_2 + 2068.155 \cdot X_1^2 - 2487.73 \cdot$ $- 17754.3 \cdot X_3^2 - 7260.86 \cdot X_1^3 - 913.951 \cdot X_2^3 - 7015.54 \cdot X$ $- 14644.6 \cdot X_1^2 \cdot X_2$	**0,941**

Table 9. Optimal values and the rheological parameters for 2 combinations

	X_1	X_2	X_3	K	n	η	τ	D
1	-0.04	1.00	0.00	79.1322	0.427451	3.52678	29043.4	0.974
2	-1.00	-1.00	0.00	70.7343	0.271267	3.66718	36166.8	0.933

4.Conclusions

The desert samples studied had a pseudoplastic behaviour (n < 1). The hydrocolloids influence is a positive one, the adding of hydrocolloids are leading to the increasing of the rheological parameters (dynamic viscosity, consistency index and flow index). The mix of xanthan gum and guar gum have the greatest influence on the dynamic viscosity, consistency index and flow index because of the structural bonds formed between this types of gums. pH, refractive index and concentration have not ranged in a great domain, the differences are because of the different solubility capacity of the hydrocolloids. The PCA conducted have revealed two different groups: one formed by the samples made using all the three hydrocolloids and the other one formed by the samples made using one or two hydrocolloids. The cubic modelling of the rheological parameters have obtained 3rd grade polynomial models with a regression coefficients greater than 0.90. The optimization of the rheological parameters, in function of the proportion of the hydrocolloids, so the parameters be maximum have lead to 2 combinations.

Compliance with Ethics Requirements

Authors declare that they respect the journal's ethics requirements. Authors declare that they have no conflict of interest and all procedures involving human and/or animal subjects (if exists) respect the specific regulations and standards.

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