Received: 28 October 2021 | Revised: 15 January 2022 | Accepted: 13 March 2022

DOI: 10.1111/jfpp.16553

Journal of Food Processing and Preservation | Institute of Food Science + Technology | ifst | WILEY

ORIGINAL ARTICLE

Multi-response optimization of process parameters of saponin-based model foam using Taguchi method and gray relational analysis coupled with principal component analysis

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Abstract

Foam is a two-phase system in which continuous liquid and discontinuous gas phases interact. Maintaining equilibrium between these phases can only be achieved by optimizing the properties of the foam. The aim of the current research is to optimize process parameters (PPs) such as protein type, hydrocolloid concentration, hydrocolloid type, and mixing temperature in the preparation of model foam using the Taguchi method (TM) and gray relational analysis (GRA) in conjunction with principal component analysis (PCA). The experiments were performed using the Taguchi orthogonal array (L16) and then the effects of PPs on the overrun (OR), bubble size (BS), and loss tangent (LT) were investigated. The results showed that GRA-PCA performed better than TM in optimizing the multiple responses. Consequently, the foam optimized for OR, LT, and BS could be prepared by whipping a sugar-containing solution with saponin (0.096%), whey protein concentrate (0.5%) and pectin (0.05%) at 80°C.

Practical applications

There is a need for alternative foaming agents for foam production in the food industry. For this purpose, optimization methods such as Taguchi method (TM) and gray relational analysis (GRA)- principal component analysis (PCA) were used to determine better foaming properties (higher foamability and more elasticity) of the test sample with a combination of *Gypsophila* saponin, milk proteins, and hydrocolloids. As a result, it was found that GRA-PCA is a more effective optimization method than TM in multi-response optimization of food foams.

1 | INTRODUCTION

Foam is a thermodynamically unstable colloidal system in which the low-density gas phase is dispersed in the high-density liquid phase. It is usually obtained by whipping or spraying the aqueous system with surfactants (Campbell & Mougeot, 1999; Lazidis et al., 2014). Aerated foods such as cakes, breads, ice creams, and some confectionery products are obtained by the foaming process (Campbell & Mougeot, 1999; Foegeding et al., 2006), which is directly associated with rheological, sensory, and textural properties (Ptaszek et al., 2015). These aerated foods are stabilized by proteins, which are mostly used in foam systems to stabilize the air–liquid interface (Campbell & Mougeot, 1999). When the proteins are denatured, the hydrophobic and hydrophilic parts orient toward the air phase and the liquid phase, respectively. Moreover, the hydrophilic parts of the proteins cause a higher bulk viscosity. Therefore, the properties of a foam system can be improved by maintaining the equilibrium between the air and liquid phases as much as possible (Abascal & Gracia-Fadrique, 2009). Egg white protein (EWP) is the most preferred protein in the production of food foam, and there is no other

protein source that could satisfy the demand of foam manufacturers. In recent years, alternative foaming agents have been demanded by foam manufacturers to improve foam properties as some consumers are allergic to egg white (Asghari et al., 2015).

Milk proteins, flexible sodium caseinate (Na-CAS), and globular whey proteins have been used as alternative proteins (Marinova et al., 2009). Although these proteins have excellent foam ability, they have lower foam stability than EWP (Davis & Foegeding, 2007). Therefore, these proteins alone are not suitable for use as foaming agents. However, saponins, which are alternative foaming agents, are also used in foam production (Böttcher et al., 2016; Böttcher & Drusch, 2016; Wojciechowski et al., 2014). They have been reported to be superior to many proteins in terms of interfacial rheological properties (Góral & Wojciechowski, 2020). Compared to proteins, they are stable under acidic conditions and at high temperatures (Gonzalez & Sörensen, 2020). In addition, the synergistic associations between globular proteins and saponins have been found to improve foam quality (Çelik et al., 2007), by increasing the heat stability of the protein (Guclu-Ustundag & Mazza, 2007) or improving the interfacial properties (Böttcher et al., 2016).

The saponin-containing extract obtained from *Gypsophila* root by water extraction is traditionally used in confectionery to produce stable foams. For example, the whitening effect of this extract has been widely used in the manufacture of Tahini halva (Özdikicierler et al., 2019). *Gypsophila* extract contains ionic and non-ionic saponins consisting of hydrophilic sugar residues and hydrophobic aglycone moieties (Böttcher & Drusch, 2016). In an aqueous solution, they can significantly increase foamability by rapidly reducing the air/liquid interfacial tension (Canto et al., 2010).

Stability is critical in aerated foods to maintain the foam structure during further processing and storage (Foegeding et al., 2006; Sadahira et al., 2016). A foam cannot maintain its stable form when milk proteins are used alone in the formulation (Narchi et al., 2009). A linear relationship between foam stability and continuous phase viscosity has been observed for whey protein isolate foams (Lau & Dickinson, 2005; Yang & Foegeding, 2011). Sugar is commonly used in confectionery products to increase the consistency of the continuous phase (Campbell & Mougeot, 1999). Thus, the structure of these products has been improved by the addition of sugar (Ptaszek et al., 2015). However, sugar interacts with components such as proteins and polysaccharides in aerated foods and affects the foam structure (Neves et al., 2018). Moreover, hydrocolloids such as locust bean gum (LBG), carrageenan (Carr), pectin (Pec), and xanthan gum (E415) can be used to obtain a stable foam structure by reducing the fluidity of the liquid phase and/or forming protein-hydrocolloid complexes at the interface (Dachmann et al., 2018; Ibanoglu & Erçelebi, 2007; Mohanan et al., 2020; Murray et al., 2006; Narchi et al., 2009; Neves et al., 2018; O'Chiu & Vardhanabhuti, 2017; Oduse et al., 2018; Ptaszek et al., 2014, 2015; Sadahira et al., 2014, 2016; Wang et al., 2015; Zmudziński et al., 2014). Intermolecular interactions (electrostatic, covalent, and hydrogen bonding) between these biopolymers lead to improved functionality of the proteins and have a great importance in deciding the process variables for foam production. Electrostatic polymer interactions mainly arise from oppositely charged regions of the biomolecules and lead to the formation of soluble and/or insoluble complexes (Mohanan et al., 2020). On the other hand, heat-induced covalent interactions also affect foam properties through the formation of complexes between proteins and hydrocolloids of different sizes (Ibanoglu & Erçelebi, 2007). However, foam properties are positively or negatively affected by protein–saponin interactions. While an improvement in foam quality was observed at low *Quillaja* bark saponin (QBS)/β-Cas ratios (<1.5), an antagonistic effect was found at higher ratios (Wojciechowski et al., 2014). Similar results were obtained with the QBS/β-lactoglobulin system (Piotrowski et al., 2012).

The quality of a food foam is directly related to the effectiveness of the stabilizer in the bulk and at the air/water interface (Sadahira et al., 2018). Overrun (OR), also called foam capacity, can be used as a reliable indicator for evaluating foam quality. OR is related to the ability of stabilizers to reduce the interfacial tension between two adjacent foam bubbles (Zhan et al., 2020) and is also used to express the amount of air dispersed in the liquid (Walstra, 2003). Foam rheology is another way to evaluate the effect of stabilizing agents on foam properties (Foegeding et al., 2006). It is used not only as a crucial factor in evaluating the quality of food foams, but also in the design of engineering operations and the development of new products (Dogan et al., 2014). In a foam system, the dispersed and dissolved proteins diffuse at the air–liquid interface. However, denaturation and coagulation of these substances can create a strong viscoelastic interface (K. Lau & Dickinson, 2006). The storage modulus (G') and loss modulus (G'') show the elastic and viscous contribution of the applied shear stress, the loss tangent (LT), while tan (δ)) symbolizes the ratio of G'' to G' (Steffe, 1996). Foam is an unstable system; therefore, the size of the bubbles changes over time depending on the efficiency of the stabilizers. However, both physicochemical properties and foam production methods can affect the average bubble size (BS), which is highly correlated with foam formation and stabilization. Therefore, the control of BS is of great importance in industrial applications (Nicorescu et al., 2010). It has been reported that BS can be used as a quality indicator in complex foam systems made of potato protein isolate/maltodextrin/pectin (Dachmann et al., 2018), EWP/hydroxypropyl methylcellulose (Sadahira et al., 2018), EWP/Pec (Ptaszek et al., 2015), Na-CAS/carboxymethyl cellulose (Zhu et al., 2020), and whey protein concentrate (WPC)/Pec (Oduse et al., 2018).

In traditional experimental design methods, the number of experiments to be performed increases substantially with increasing independent variables. The Taguchi method (TM) can be proposed to solve this problem, as it provides a simple, efficient, and systematic strategy to optimize experimental designs in terms of quality, manufacturing process, and design stage (Dimou et al., 2009). The Taguchi technique has been used to optimize the rheological properties of ice cream (Aslan Türker & Dogan, 2021) and semi-liquid syrup (Molina-Rubio et al., 2010). Gray relational analysis (GRA) is an effective approach to determine the optimal process parameters in a

multivariate problem. The goal of GRA is to identify a gray relational grade (GRG) that can be used to optimize from a multi-objective problem to a single-objective problem. GRG is used to estimate the effects of parameters on overall performance (Sankar et al., 2015). However, principal component analysis (PCA) is an analytical method that can be used to optimize a system with numerous performance characteristics. This analysis management significantly reduces the complexity of the solution by reducing the interrelated variables to independent PCs while preserving the primary data with linear combinations (Raju et al., 2017).

From the literature review, it is clear that many studies have already been conducted to optimize food process parameters using TM. But only one parameter which is affected by process parameters can be optimized using the Taguchi technique (Arunachalam et al., 2020). Therefore, alternative optimization methods such as GRA, PCA, Response Surface Methodology (RSM), and GRA-PCA can be used for multi-response optimization (Pandey & Yadav, 2020). No studies were found on GRA-PCA optimization of food foam production parameters. Therefore, the current study focused on the application of the TM and GRA-PCA technique to optimize selected parameters in foam production to obtain more elastic foams with greater foamability and smaller foam bubbles.

2 | MATERIALS AND METHODS

2.1 | Materials

Commercial samples of WPC (WPC-80) and WPI (WPI-95) were obtained from Lactalis (France). Both D-WP and Na-CAS were supplied by Maybi (Istanbul, Turkey) and Alfasol (Istanbul, Turkey), respectively. A commercial *Gypsophila* extract, locust bean gum, κ-carrageenan, high methoxyl pectin, xanthan gum, and citric acid were obtained from Tito (Izmir, Turkey). The saponin content of the *Gypsophila* extract was determined using the gravimetric method described by Battal et al. (2003) and calculated with an average value of 6.4%. All commercial samples were stored at laboratory conditions (22 ±2°C). Granulated sugar was purchased from a local company (Kayseri, Turkey).

2.2 | Methods

2.2.1 | Preparation of protein and hydrocolloid solutions

Whey protein and Na-CAS solutions (10%) were prepared with deionized water and phosphate buffer (pH 7.4), respectively. They were stirred for 2 hours at room temperature (1500 rpm). The solutions were stored at 4°C and allowed to stand overnight for complete hydration. All samples were equilibrated to ambient temperature before use (Shen et al., 2017).

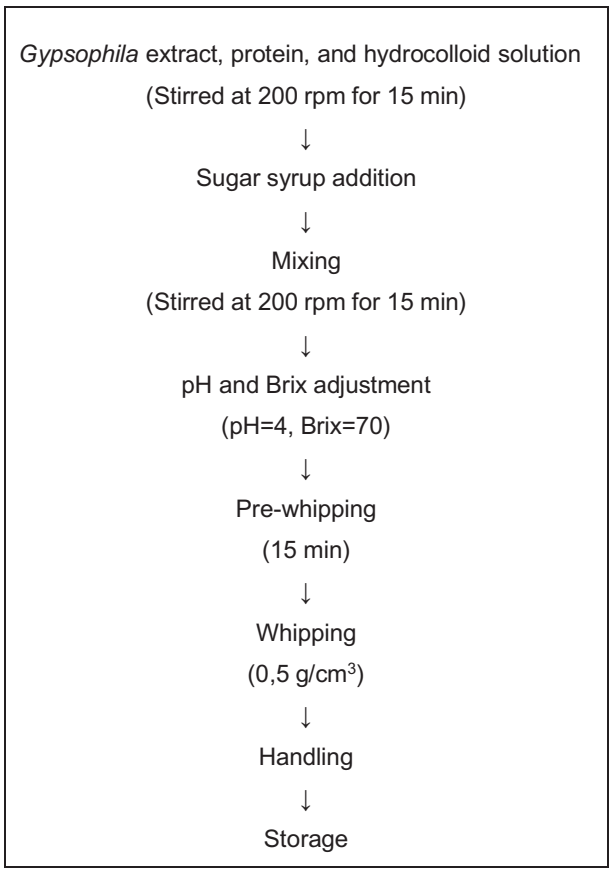

FIGURE 1 Production recipe of saponin-based foam model

LBG, Carr, Pec, and E415 solutions (1%) were mixed with the powders, dispersed in distilled water, and shaken vigorously at 300 rpm for 10 min. All hydrocolloid solutions were gently (200 rpm) and continuously stirred overnight at room temperature.

2.2.2 | Preparation of prefoam solution

A halva recipe obtained from a local company (Camlica Halva, Kutahya, Turkey) was used to prepare the saponin-based model foam (Figure 1). The preliminary tests were used to determine the Brix and pH values of the prefoam solution, the impeller type of the mixer, the speed/time of mixing, the type/amount of saponin, milk proteins, and hydrocolloids.

The quality parameters of the model foam and their corresponding levels are shown in Table 1. In the preparation of the foam model, the *Gypsophila* extract (3 ml), protein solution (10 ml), and hydrocolloid solution were mixed in a beaker (250 ml) according to the orthogonal design order in Table 2. The resulting mixture was gently shaken with a magnetic stirrer (200 rpm) for 15 min to allow surfactant–hydrocolloid interactions. The final weight was fixed at 200 g by adding sugar syrup (<60°C) and then continuously stirring for 15 min. After adjusting the pH to 4 using a pH meter (Hanna HI 12211) by adding citric acid solution (10%), the total soluble solids

TABLE 1 Process parameters and their levels for the foam model

Factor	Symbol	Level 1	Level 2	Level 3	Level 4
PT	A	WPC	WPI	Na-CAS	D-WP
HC (%)	B	0.05	0.10	0.15	0.20
HT	C	LBG	Carrageenan	Pectin	Xanthan gum
MT (°C)	D	40	60	80	100

Abbreviations: HC (%), hydrocolloid concentration; HT, hydrocolloid type; LBG, locust bean gum; MT (°C), mixing temperature; PT, protein type.

TABLE 2 Taguchi L16 orthogonal array, average results, and S/N ratios for the foam model

	Process parameters				Average results			S/N ratios, dB		
Order	A[a]	B[b]	C[c]	D[d]	Overrun (OR) (%)	Loss tangent (LT) (–)	Bubble size (BS) (μm)	Overrun (OR) (%)	Loss tangent (LT) (–)	Bubble size (BS) (μm)
1	1	1	1	1	454.5 ± 1.68	1.234 ± 0.00	36 ± 0.00	53.15	−1.83	−31.13
2	1	2	2	2	441.6 ± 1.54	2.091 ± 0.00	36 ± 0.00	52.90	−6.41	−31.13
3	1	3	3	3	500.0 ± 3.67	1.337 ± 0.01	35 ± 0.25	53.98	−2.52	−30.88
4	1	4	4	4	263.0 ± 0.42	1.187 ± 0.01	38 ± 0.25	48.40	−1.49	−31.60
5	2	1	2	3	500.6 ± 1.17	2.014 ± 0.01	38 ± 0.50	53.99	−6.08	−31.60
6	2	2	1	4	412.4 ± 0.56	2.144 ± 0.00	44 ± 0.25	52.31	−6.62	−32.87
7	2	3	4	1	363.5 ± 0.76	0.917 ± 0.00	55 ± 0.50	51.21	0.76	−34.81
8	2	4	3	2	423.9 ± 1.69	1.230 ± 0.02	48 ± 0.50	52.54	−1.80	−33.62
9	3	1	3	4	249.5 ± 0.93	1.467 ± 0.02	40 ± 0.25	47.94	−3.33	−32.04
10	3	2	4	3	242.5 ± 0.83	2.204 ± 0.00	45 ± 0.50	47.70	−6.86	−33.06
11	3	3	1	2	96.10 ± 0.57	2.128 ± 0.00	49 ± 0.25	39.65	−6.56	−33.80
12	3	4	2	1	52.69 ± 0.34	2.070 ± 0.01	44 ± 0.00	34.43	−6.32	−32.87
13	4	1	4	2	44.03 ± 1.17	1.573 ± 0.00	40 ± 0.25	32.87	−3.93	−32.04
14	4	2	3	1	46.88 ± 0.25	1.240 ± 0.01	39 ± 0.50	33.42	−1.86	−31.82
15	4	3	2	4	90.71 ± 0.16	1.963 ± 0.02	43 ± 0.25	39.15	−5.86	−32.67
16	4	4	1	3	94.86 ± 0.08	2.205 ± 0.00	42 ± 0.50	39.54	−6.87	−32.46

[a]A = protein type.
[b]B = hydrocolloid concentration.
[c]C = hydrocolloid type.
[d]D = mixing temperature.

content in the prefoam samples was adjusted to 70 Brix using an automatic refractometer (Reichert AR700).

2.2.3 | Model foam production

A laboratory-scale, high-shear planetary mixer (KM070, Kenwood, UK) with induction heating was used to produce the foam models. After the prefoam solution was transferred to a mixing bowl (6.7 L), the mixing speed of the mixer was set to "min" and the solution was then heated to the mixing temperature given in Table 2. After setting the mixing speed to 158 rpm and selecting the mixer impeller as a K-mixer, the prefoam solution was whipped for 15 min. The OR measurements were taken immediately after the first whipping. Finally, the foam was mixed to a specific density (0.5 g/cm^3) after setting the heating function of the mixer to "0." Rheological analysis was carried out to study the LT immediately after the resulting foams were filled into polypropylene bowls (280 ml). Finally, microscopic observations were also made to determine the size of the bubbles (BS) after 50 h of preparation.

2.2.4 | Foam analysis

Overrun (%) measurement

The OR was measured on a gravimetric basis. After the prefoam solution was stirred for 15 minutes, the mixing device (K-mixer) was carefully removed from the sample. The foam was then carefully poured into a weighing pan and leveled with a metal spatula before recording the total weight, but this procedure was limited to 2 minutes. Thus, OR (%) was calculated according to Equation (1) (Wang et al., 2015). Finally, the foam sample was poured into the mixing

bowl and all foams were mixed until they reached a specific density (0.5 g/ml).

$$\text{Overrun } (\%) = \left(\frac{((\text{wt. } x \text{ cm}^3 \text{ prefoam solution}) - (\text{wt. } x \text{ cm}^3 \text{ foam}))}{\text{wt. } x \text{ cm}^3 \text{ foam}} \right) * 100 \quad (1)$$

Rheological analysis

Stress sweep and frequency sweep tests were conducted using a probe with plate-plate geometry (upper plate diameter: 35 mm, space between lower and upper plates: 1 mm) with a shear-controlled rheometer (Thermo HAAKE, Mars III, Karlsruhe, Germany) at 25°C. Stress sweep tests were performed in the stress range of 0.1–10 Pa and a constant frequency (0.1 Hz) to identify the linear viscoelastic region (LVR). Using the data from the frequency sweep test performed from 0.1 to 10 Hz at a constant stress of 0.2 Pa (within the LVR), the average values for the storage modulus (G'), viscous modulus (G''), and LT (tan δ) of the foams were automatically calculated by RheoWin Data Manager (RheoWin Pro V 4.0, HAAKE, Karlsruhe, Germany) using the following equations: (Ozgur et al., 2017).

$$G' = \frac{\sigma_0}{\epsilon_0} * \cos\delta \quad (2)$$

$$G'' = \frac{\sigma_0}{\epsilon_0} * \sin\delta \quad (3)$$

$$\tan\delta = \frac{G''}{G'} \quad (4)$$

Microscopic measurement

A confocal laser scanning microscope (Zeiss LSM 510, Germany) equipped with a 20X objective lens was used to image the foam samples. As a labeling dye, a solution of sodium fluorescein (0.01 mM) was mixed with the prefoam solution before whipping. First, a foam sample was placed on the slide to obtain images with the microscope. Then, the sample was excited with an argon laser at 488 nm and the rays in the wavelength range from 458 to 633 nm were collected. Images recorded with microscope software with a resolution of 1024 x 1024 pixels were analyzed using Image J software (Sadahira et al., 2018). An average of 500 to 1000 bubbles were detected in each foam sample, which were utilized to calculate the BS. Bubbles with a circle value of less than 0.5 were not considered. The average BS of the model foam samples was estimated using the following equation:

$$d_{32} = \left(\sum_i d_i^3 \right) / \left(\sum_i d_i^2 \right) \quad (5)$$

2.2.5 | Analysis method

Taguchi method (TM)

To optimize the individual properties, the TM was initially performed. In this respect, an orthogonal design L_{16} with four factors and four levels was chosen (Table 2). The PT, HC, HT, and MT were chosen as control parameters. Literature data and the results of preliminary experiments were used to determine the foam production parameters and their corresponding levels (Table 1). The Taguchi orthogonal array was used to determine the differences between the signal-to-noise (S/N) ratio. The highest S/N ratio was considered to determine the optimal levels for each production parameter. They were determined by converting the response variable to a constant value (decibels, dB) using the criteria "smaller the better" or "larger the better." The S/N ratios for the LT and BS and OR responses were calculated using the criteria "smaller the better" (Equation 6) and "larger the better" (Equation 7), respectively.

$$\frac{S}{N} = -10\log\left[\frac{1}{n} \sum_{i=1}^{n} y_i^2 \right] \quad (6)$$

$$\frac{S}{N} = -10\log\left[\frac{1}{n} \sum_{i=1}^{n} \frac{1}{y_i^2} \right] \quad (7)$$

where i is the experimental order, y_i is the experimental result, and n is the total number of trials.

Gray relational analysis (GRA)-principal component analysis (PCA)

GRA-PCA was used to optimize the optimal process parameter levels corresponding to each response (OR, LT, and BS), considering their importance (weighting) in foam production. In this study, GRA was used to analyze complex relationships in a multi-response system, while PCA was used to determine the corresponding weight value reflecting the relative importance of each response in the GRA. In this regard, the following stages of GRA were applied in combination with PCA to optimize multiple responses:

1. Normalization of s/n results for each response: the process of normalizing the s/n values obtained from the Taguchi analysis was performed using Equation (8)

$$N_{y_i}(k) = \frac{x_i(k) - \min x_i(k)}{\max x_i(k) - \min x_i(k)} \quad (8)$$

where $N_{y_i}(k)$ was the normalized value, $x_i(k)$ was the S/N ratio, and $\min x_i(k)$ and $\max x_i(k)$ were the minimum and maximum S/N ratio values of the test results, respectively.

2. Determination of the gray relational coefficient (GRC) and gray relational grade (GRG): to explain the relationship between the ideal and the actual test results, the GRCs were calculated according to Equation (9). In this equation, the value of coefficient (φ) was assumed to be 0.5. In studies in literature, this coefficient was found to not effectively change the gray relational ranking (Üstüntağ et al., 2020). Δ_{0i} was the difference between the referential ($y_0(k)$) and comparison ($y_i(k)$) values (Equation 10). However, $\Delta_{\min}$ (Equation 11) and $\Delta_{\max}$ (Equation 12) represent the lowest and highest values of Δ_{0i}, respectively (Canbolat et al., 2019)

$$GRC\left(X_0(k) X_i(k)\right) = \frac{\Delta_{\min} + \varphi\Delta_{\max}}{\Delta_{0i} + \varphi\Delta_{\max}} \quad (9)$$

$$\Delta_{0i}(k) = \left|X_0(k) - X_j(k)\right| \tag{10}$$

$$\Delta_{\min} = \min_j \min_k \mid X_0(k) - X_j(k) \mid \tag{11}$$

$$\Delta_{\max} = \max_j \max_k \left|X_0(k) - X_j(k)\right| \tag{12}$$

The GRG showed a correlation between the reference sequence and the compared sequence. In this respect, higher GRG values (closer to 1) were expected for the analyzed responses. The lower GRG values indicated differences between the data sequences. The weighted GRG was determined using the different weighted factors in Equation (13):

$$GRG\ (X_0X_i) = \frac{1}{n}\sum_{k=1}^{n} w_k \varphi_i(k) \tag{13}$$

where n was the number of experiments and w_k was the weighting factor for the kth performance (Arce et al., 2015). In this study, the w_k value for each response was determined by principal component analysis.

3. Principal component analysis (PCA): In GRA, the weighting values for multiple responses were objectively determined using PCA (Oliver Nesa Raj & Prabhu, 2017). The purpose of this multivariate data analysis is to reduce the dimensionality of the study as much as possible (Sehgal, 2018). PCA is an orthogonal transformation technique that converts the observations of likely correlated factors into the values of uncorrelated factors (principal components [PCs]) (Vasudevan et al., 2018). The following mathematical procedure was used to evaluate the targeted PCs.

(i) The variance–covariance array X was formulated by the GRCs, as shown in Equation (14):

$$X = \begin{bmatrix} X_1(1) & \dots & X_1(n) \\ \vdots & \vdots & \vdots \\ X_m(1) & \dots & X_m(n) \end{bmatrix} \tag{14}$$

where X was the GRC for each response, m was the number of tests, and n was the number of responses.

(ii) The correlation coefficient array was estimated using Equation (15):

$$R_{jl} = \left(\frac{cov\left(x_i(j)\, x_i(l)\right)}{\sigma_{xi}(j)\, x \sigma_{xi}(l)}\right), j = 1, 2, 3, \dots, n \;\; l = 1, 2, 3, \dots, n \tag{15}$$

where $cov\left(x_i(j)\, x_i(l)\right)$ was the covariance of sequence $x_i(j)$ and $x_i(l)$; $\sigma_{x_i(j)}$ and $\sigma_{x_i(l)}$ were the standard deviation of the sequence $x_i(j)$ and $x_i(l)$, respectively.

(iii) The eigenvector and eigenvalues obtained using the correlation coefficient array are shown in Equation (16):

$$\left(R - \lambda_k I_m\right) V_{ik} = 0 \tag{16}$$

where λ_k was Eigenvalues; $V_{ik} = \left[\alpha_{k1}\alpha_{k2}\dots\alpha_{km}\right]^T$ were Eigenvectors corresponding to Eigenvalues λ_k and $\sum_{k=1}^{n}\lambda_k = n,\ k = 1, 2, \dots, n$.

(iv) The PCs of the model responses were estimated by Equation (17):

$$Y_{mk} = \sum_{i=1}^{n} Y_m(i) \times V_{ik} \tag{17}$$

where Y_{m1}, Y_{m2}, ..., Y_{mn} were called the first principal component, second principal component, and so on (descending order of the variance).

4. Analysis of variance: The effect ratio of the model factors on the response variables was determined by ANOVA (analysis of variance). In terms of output results, differences in control parameters could be indicated by this statistical approach. The most effective parameter for the characteristics of the process could be identified so that the development of the process was made possible by controlling this factor (Deepanraj et al., 2017).

5. Confirmatory experiments: The final phase of the optimization process was to validate the results. Once the optimum process parameters were determined, the predicted and experimental results of the optimal factor levels derived from GRA were used to prove the accuracy of the optimization process. The results of the experiment conducted under optimal conditions were used to determine the improvement in the output variables. The predicted S/N ratio was calculated by Equation (18) using the optimal levels of the process parameters:

$$\eta_o = \eta_m + \sum_{i=1}^{j}\left(\eta_i - \eta_m\right) \tag{18}$$

where η_m was the overall average of the S/N ratio, η_i was the average of the S/N ratio corresponding to optimal levels, j was the number of process factors (Celik et al., 2018).

3 | RESULTS AND DISCUSSION

3.1 | Taguchi optimization

In the first part of the study, Taguchi's optimization method was used to determine the optimal process parameters for selected responses. It was assumed that the interaction effect of production variables was negligible. The average experimental results and S/N ratio values are given in Table 2. The average S/N ratios of each process parameter for the OR, LT, and BS responses are displayed in Table 3. The S/N ratio of the OR response ranged from 32.87 dB to 53.99 dB (Table 2). Table 3 shows that the foam capacity values of WPC-Pec (0.15%) and WPI-Carr (0.05) foams were higher than those of D-WP/E415 and D-WP/Pec foams.

TABLE 3 The S/N ratio table for OR, LT, and BS responses

	Overrun (OR)				Loss tangent (LT)				Bubble size (BS)			
Level	PT	HC	HT	MT	PT	HC	HT	MT	PT	HC	HT	MT
1	52.11	**46.99**	46.16	43.06	**−3.07**	−3.78	−5.46	**−2.31**	**−31.18**	**−31.70**	−32.357	−32.66
2	**52.51**	46.58	45.12	44.49	−3.43	−5.43	−6.16	−4.68	−33.22	−32.22	**−32.07**	−32.65
3	42.43	46.00	**46.97**	**48.80**	−5.76	**−3.55**	**−2.38**	−5.58	−32.94	−33.04	−32.09	**−32.00**
4	36.25	43.73	45.04	46.95	−4.62	−4.11	−2.88	−4.33	−32.25	−32.64	−32.88	−32.29
Delta	16.27	3.26	1.93	5.75	2.69	1.88	3.78	3.27	2.04	1.34	0.81	0.65
Rank	1	3	4	2	3	4	1	2	1	2	3	4

Note: The bold values show the optimal values.

Abbreviations: HC, hydrocolloid concentration; HT, hydrocolloid type; MT, mixing temperature; PT, protein type.

TABLE 4 Gray relational analysis results for the model foam

	Normalized data			Deviation sequence			GRC[d]				
Test No.	OR	LT	BS	OR	LT	BS	OR	LT	BS	GRG	Rank
1	0.90	0.75	0.95	0.10	0.25	0.05	0.83	0.67	0.91	0.804	2
2	0.87	0.09	0.95	0.13	0.91	0.05	0.79	0.35	0.91	0.698	4
3	1.00	0.67	1.00	0.00	0.33	0.00	1.00	0.61	1.00	0.890	1
4	0.48	0.79	0.85	0.52	0.21	0.15	0.49	0.70	0.77	0.607	7
5	1.00	0.15	0.85	0.00	0.85	0.15	1.00	0.37	0.77	0.779	3
6	0.81	0.05	0.55	0.19	0.95	0.45	0.72	0.34	0.53	0.577	8
7	0.70	1.00	0.00	0.30	0.00	1.00	0.62	1.00	0.33	0.666	5
8	0.83	0.76	0.35	0.17	0.24	0.65	0.75	0.67	0.43	0.661	6
9	0.45	0.57	0.75	0.55	0.43	0.25	0.48	0.54	0.67	0.534	9
10	0.43	0.00	0.50	0.57	1.00	0.50	0.47	0.33	0.50	0.439	12
11	0.11	0.06	0.30	0.89	0.94	0.70	0.36	0.35	0.42	0.369	16
12	0.02	0.10	0.55	0.98	0.90	0.45	0.34	0.36	0.53	0.383	15
13	0.00	0.49	0.75	1.00	0.51	0.25	0.33	0.50	0.67	0.448	11
14	0.01	0.75	0.80	0.99	0.25	0.20	0.33	0.67	0.71	0.505	10
15	0.10	0.19	0.60	0.90	0.81	0.40	0.36	0.38	0.56	0.406	13
16	0.11	0.00	0.65	0.89	1.00	0.35	0.36	0.33	0.59	0.401	14

Note: Average gray relational grade = 0.573.

Abbreviations: BS, bubble size; GRC, gray relational coefficient; GRG, Gray relational grade; LT, loss tangent; OR, overrun.

However, WPC and WPI foams with better foaming properties have higher OR values than those of Na-CAS and D-WP foams. In particular, the lower OR values (less than 100) were obtained when D-WP was used as milk protein. According to milk protein powder manufacturers, D-WP (11%) had a comparatively lower protein content than WPC, WPI, and Na-CAS (>80%, >94%, and 86.5%, respectively). Therefore, the lower protein content resulted in lower foamability. From Table 3, it can be seen that the foam prepared by mixing a prefoam solution with saponin (0.096%), WPI (0.5%), and Pec (0.05%) at 80°C was found to be the optimum sample ($A_2B_1C_3D_3$). The value of S/N ratio, which was determined according to the "larger-the-better" criteria revealed that PT with the highest delta value (δ = 16.27) was the most important parameter that affected the foamability of the saponin-milk protein-hydrocolloid system (Table 3).

The viscoelastic properties used to identify the technological properties of protein foams are closely associated with physicochemical properties such as foam capacity and density (Ptaszek, 2013). More comprehensive results could be obtained using tan (δ) values to estimate the rheological properties of food foams (Steffe, 1996). The S/N ratios for the LT response varied from −6.87 to 0.76 dB. It was found that the foam with WPI and E415 (0.15%) had a more elastic structure compared to the other samples (Table 2). Under optimal conditions, a more elastic (ideal) foam structure could be obtained by whipping a prefoam solution with saponin (0.096%), WPC (0.5%), and Pec (0.15%) at 40°C. According to the analysis of S/N

ratio, it is clear that HT is the most important criterion ($\delta = 3.78$) for LT (Table 3).

The differences in BS in protein foams provide information about the stability of protein films in foam bubbles (Lau & Dickinson, 2005). The literature emphasizes that foams with smaller bubbles have more stable and elastic interfacial layers than those with larger bubbles (Marinova et al., 2009; Martínez-Padilla et al., 2015; Yang & Foegeding, 2011). The S/N ratio of the BS results in the model foam varies between −30.88 and −34.81 dB. It can also be inferred from Table 2 that the foam prepared with *Gypsophila* extract, WPC, and Pec (0.15%) by whipping at 80°C had bubbles with a smaller size than other samples. The optimum levels of BS are shown in Table 3. It can be suggested that the foam with the smallest BS could be obtained by mixing acidic sugar medium (pH 4, 70 Brix) with saponin (0.096%), WPC (0.5%), and Carr (0.05%) at 80°C ($A_1B_1C_2D_3$). Table 3 also shows the average S/N ratio for the process parameters obtained according to the criterion "smaller the better." The results indicate that PT has the most significant effect ($\delta = 2.04$) on the BS of the model foam. Finally, comparing the optimal factor levels of the responses, it can be deduced that TM was not sufficient for the simultaneous optimization of OR, LT, and BS responses of the model foam.

3.2 | GRA-PCA optimization

In the second section of the study, GRA was used in conjuction with PCA to optimize the combined effects on selected responses. Table 4 displays the GRA-PCA data for the responses (OR, LT, and BS). Briefly, the S/N ratio values were first converted into normalized data. Then, the deviation sequence of the test samples was calculated, and then the GRCs, which play an important role in GRA and PCA, were estimated using PCA.

The GRGs were calculated using PCA. The correlation coefficient matrix, created using the variance–covariance matrix, was used to determine the eigenvalues and associated eigenvectors for each response of the model. In Figure 2, it can be seen that the variance in the response data was better explained by the first principal component (the contribution of variance>40%).

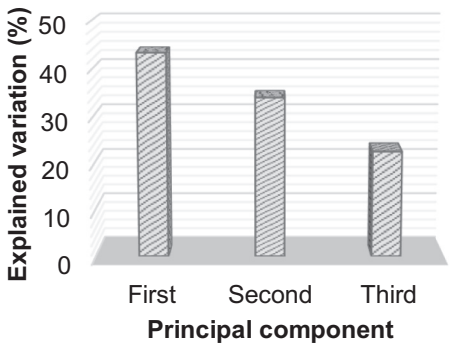

FIGURE 2 Contribution of principal components to eigenvalues

The eigenvectors corresponding to the principal components and the weight contribution of each process parameter, estimated for the first principal component, are shown in Table 5. The weight contribution of OR was found to be the highest for foam characteristics (51.55%). The quality of milk protein foam has been reported to be closely related to foamability (Oduse et al., 2018). The weight contribution for LT and BS was 27.36% and 21.07%, respectively. After PCA was completed, the GRCs for the responses were recalculated on the basis of weight factors. Finally, the OR, LT, and BS responses were converted into an individual response as a GRG value (Table 4). The factor levels in the experiment with higher GRG values were closely related to the optimal conditions for foam production in the responses. In this study, the highest value for OR and the lowest value for LT and BS were obtained in the third test of the orthogonal design. This means that the test sample "3" with the highest GRG value (0.890) has a maximum of OR and a minimum of LT and BS among the 16 trials.

The average gray relational levels for the process parameters are presented in Table 6 and Figure 3. The highest S/N ratio for the level of each control factor was accepted as the optimal level for the foam model. The bold values for each process factor symbolize the optimal values for the production of model foam (Table 6). The most efficient process parameters in the production of the model foam were the PT, while the MT was the least dominant factor for the foam properties. The order of the rate of influence of the parameters was as follows: MT < HT < HC < PT. Similar findings also revealed by ANOVA, that PT is the most important parameter with a contribution of 77.79%, followed by HC (8.52%) and HT (7.87%). The influence of MT (5.75%) on the optimum properties of the model foam is very small compared to the other foaming factors (Table 7).

The most important main effect for each foaming parameter was also considered as the optimal production condition (Figure 3). Foam with maximum OR and minimum LT and BS can be produced by mixing the sugar solution with saponin (0.096%), WPC (0.5%), and Pec (0.05%) at 80°C ($A_1B_1C_3D_3$). As can be seen in Figure 3, the effect of WPC and WPI on foam properties was greater than that of Na-CAS and D-WP. Marinova et al. (2009) observed that the maximum foamability of the WPC and WPI solutions was found at pH 4, while the minimum OR value in the Na-CAS solution was found at isoelectric pH ($PI_{Na\text{-}CAS}$ = 4.6). Similar results were reported by Morr (1985) and Lee et al. (1992). The foam properties improved with a decrease in the amount of hydrocolloids added. The OR value of the milk protein foams was increased by decreasing the amount of the polysaccharide. The high viscosity of the prefoam solution resulted in a reduction of the air phase in the foam system. Our findings are consistent with those previously reported by Ibanoglu and Erçelebi (2007) and Mohammadian and Alavi (2016). Pec appeared to be the best hydrocolloid to obtain the specific optimized value for the responses of OR, LT, and BS. Milk proteins formed foams with higher OR and lower LT and BS at lower hydrocolloid concentrations. The OR value of the foam system has been reported to be strongly influenced by the HT used in the formulation (Karasu et al., 2014).

TABLE 5 Eigenvectors for PCs and the contribution of each process parameters

Process parameters	Eigenvectors			Weight contribution[a]
	First PC	Second PC	Third PC	
Overrun	0.718	0.007	0.696	0.5155
Loss tangent	0.523	0.665	0.533	0.2736
Bubble size	0.459	0.746	0.481	0.2107

[a]Calculating for the first principal component (PC).

TABLE 6 Response table for weighted GRG

Symbol	Process parameters	Level 1	Level 2	Level 3	Level 4	Max-min	Rank
PT	Protein type	**0.750**	0.670	0.431	0.440	0.319	1
HC	Hydrocolloid concentration	**0.642**	0.555	0.583	0.513	0.128	2
HT	Hydrocolloid type	0.538	0.566	**0.648**	0.540	0.110	3
MT	Mixing temperature	0.589	0.544	**0.627**	0.531	0.096	4

Note: The bold values show the optimal process levels.

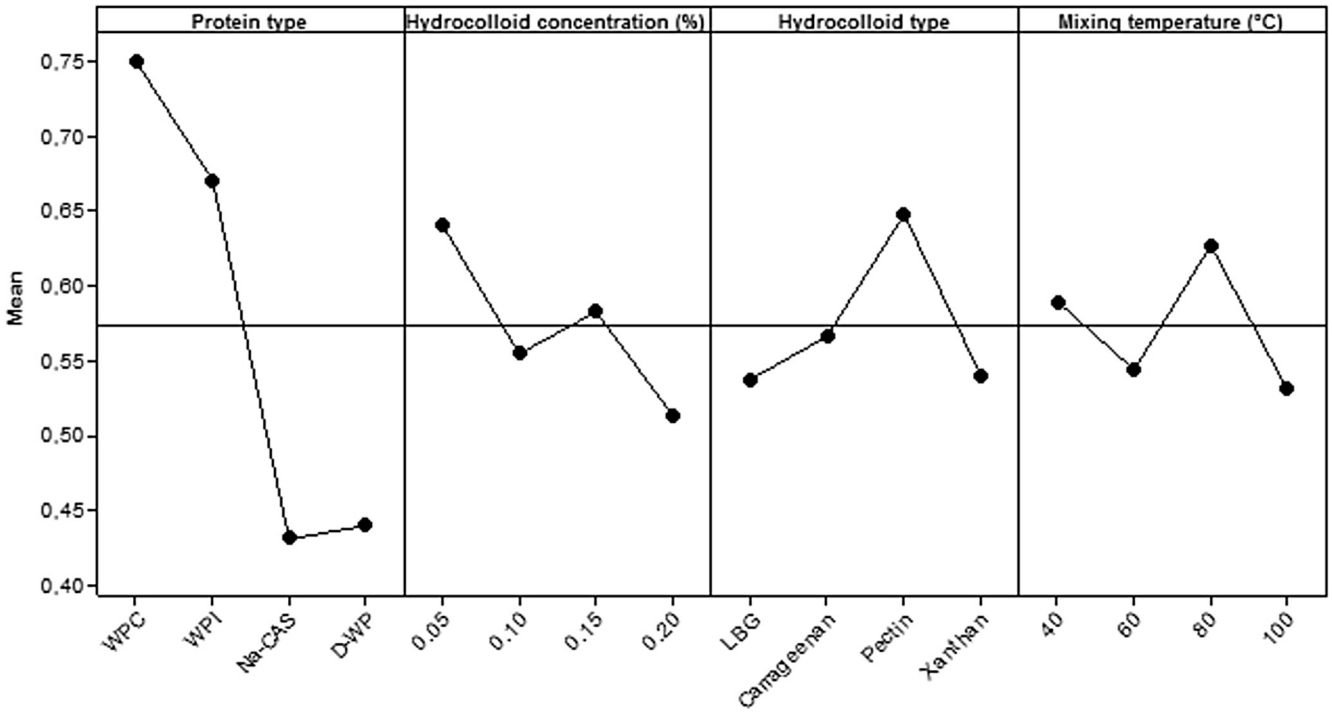

FIGURE 3 Main effects plot for weighted GRGs

In this study, Pec and E415-containing foams exhibited a firmer structure than other samples. More elastic foams were obtained because xanthan leads to higher viscosity than other hydrocolloids at the same concentration, pectin forms a stronger gel in sugar and acidic environments, and ionic interactions of these two hydrocolloids with biomolecules lead to the formation of soluble/insoluble complexes (Güldane & Doğan, 2020). The other interactions (electrostatic, hydrophobic, physical, etc.) also play a crucial role in the formation of more elastic foams (Liszka-Skoczylas et al., 2014; Zmudziński et al., 2014). There was a competition between saponins and proteins for adsorption at the air/liquid interface. Aggregation of surfactants in different regions of the foam film resulted in larger bubbles, depending on local pressure differences in the foam bubbles (Wojciechowski et al., 2014).

Sadahira et al., 2016 reported that the Pec/EWP ratio had a significant effect on foam properties. The EWP foams had lower OR and higher BS at a ratio of 1:7. In contrast, at a ratio of 1:49, the foams had a higher OR and a lower BS. In our study, the properties

TABLE 7 ANOVA results for weighted GRG

Process parameters	DF	SS	MS	F-value	p-value	Contribution (%)
Protein type	3	0.3151	0.1050	907.83	0.000	77.79
Hydrocolloid concentration	3	0.0345	0.0115	99.45	0.002	8.52
Hydrocolloid type	3	0.0319	0.0106	91.81	0.002	7.87
Mixing temperature	3	0.0233	0.0078	67.00	0.003	5.75
Error	3	0.0003	0.0001			0.07
Total	15	0.4050				100

Note: S = 0.0108, R^2 = 99.91%, R^2(adj) = 99.75, R^2(pred) = 97.56.

TABLE 8 Results of confirmation experiments

	Initial process parameters	Optimal process parameters	
		Prediction	Experiment
Factors levels	$A_1B_1C_1D_1$	$A_1B_1C_3D_3$	$A_1B_1C_3D_3$
Overrun (%)	454.5		574.5
Loss tangent (tan δ)	1.234		1.280
Bubble size (μm)	36.0		32.5
GRG	0.804	0.889	0.901
Improvement in GRG	0.097		

Abbreviation: GRG, gray relational grade.

of the model foam were also strongly influenced by the protein-to-hydrocolloid ratio. The formation of soluble complexes between proteins and other components (proteins, hydrocolloids, saponins, sugars, etc.) led to an increase in OR value and a decrease in BS value under certain conditions. Dabestani and Yeganehzad (2019) reported that the structural properties of globular proteins changed due to heat treatment. In our study, it was found that the quality characteristics of the model foam deteriorated when the MT increased from 80 to 100°C (Figure 3). Thus, excessive heating resulted in irreversible denaturation of proteins. The denaturation temperature of β-Lactoglobulin and α-Lactalbumin at pH 7 was reported to be 78°C and 64°C, respectively (Lajnaf et al., 2018). Therefore, the foamability of whey protein foams decreased when the heating temperature exceeded the denaturation point (Nicorescu et al., 2010).

Due to differences in the temperature stability of milk proteins, the OR value for whey proteins decreased when the temperature exceeded 80°C, but the opposite trend was observed for heat-stable Na-CAS. We concluded that the presence of cross-linking agents such as proteins, saponins, hydrocolloids, etc., resulted in improved heat stability. Similar findings were reported by Schmitt et al. (1998). In addition, a more concentrated medium formed as a result of an increase in MT. Similarly, Clarkson et al. (2000) informed that the denaturation rate of whey proteins decreased as the viscosity of the solution increased. Moreover, Bals and Kulozik (2003) found that the improvement in foam properties, especially BS, of protein foams depended on an increase in the viscosity of the liquid phase. However, in this study, no similar relationship was found between increased viscosity (an increase in added hydrocolloid) and BS (Figure 3).

The order of importance of the process parameters for the OR, LT, and BS responses of the model foam is shown in Table 6. PT and HC were found to be the most important parameters for foam quality. However, since the max-min difference (0.096) is less, MT was found to be the least efficient control parameter. These findings were further supported by the ANOVA presented in Table 7. The effect of process parameters on model foam production was found to be significant at a 95% confidence level. Table 7 shows that PT, which had the highest F-ratio, was found to be the most important factor in the production of the model foam, since the contribution of PT to the foam quality parameters was 77.79%, followed by HC, HT, and MT with a contribution of 8.52%, 7.87%, and 5.75%, respectively.

3.3 | Confirmation experiments

Confirmatory tests were carried out to confirm the optimal foaming parameters obtained from the optimization process and to verify the improvement in foam quality properties. The results are presented in Table 8. The experimental results of OR, LT, and BS responses at optimum factor levels were used to estimate the actual GRG. Furthermore, the predicted GRG was calculated using the maximum values of the S/N ratio of the foam parameters according to Equation 18. As a result, the actual GRG value (0.901) was found to be in agreement with the predicted GRG value (0.889). However, compared to the original process parameters ($A_1B_1C_1D_1$), an improvement of about 12% in the GRG value was obtained. As shown in Table 8, the OR value was also improved from 454.5% to 574.5%

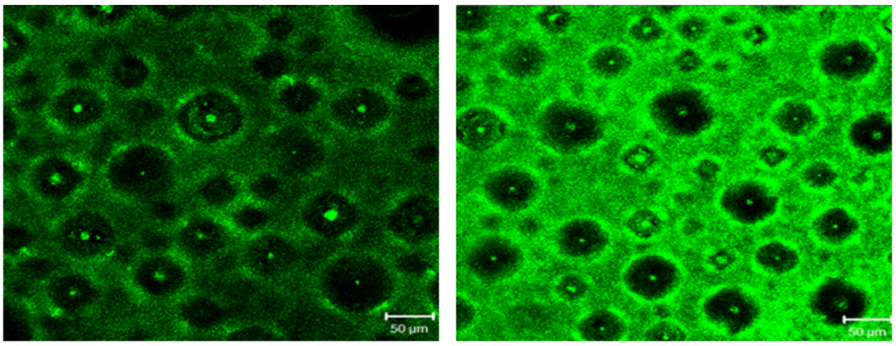

FIGURE 4 Microscopic images and average bubble sizes for foams produced with initial (left) and optimal (right) process parameters

and the BS value was reduced from 36 µm to 32.5 µm (Figure 4), while the LT value was not improved.

4 | CONCLUSIONS

The article reports the optimization of parameters of saponin-based food foam using the Taguchi method (TM) and gray relational analysis (GRA)–principal component analysis (PCA). In this context, the effects of the process parameters (protein type (PT), hydrocolloid concentration (HC), hydrocolloid type (HT), and mixing temperature (MT)) on the response variables (overrun (OR), loss tangent (LT), and average bubble size (BS)) were investigated. GRA-PCA was found to be more effective than TM in optimizing multiple responses. As a result of GRA-PCA analysis, it was found that the most important parameter for foam production is protein type. Experimental results have shown that the properties of food foams such as OR and BS are improved by using GRA-PCA. The optimal combination of foam parameters based on multiple response variables is $A_1B_1C_3D_3$, that is, WPC PT, 0.05% HC, pectin HT, and 80°C MT.

ACKNOWLEDGMENTS

This research has been not supported by any organization.

CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

AUTHOR CONTRIBUTIONS

Mehmet Guldane: Conceptualization; data curation; investigation; methodology; writing – original draft. **Mahmut Doğan:** Supervision.

DATA AVAILABILITY STATEMENT

No data are available.

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How to cite this article: Guldane, M. & Dogan, M. (2022). Multi-response optimization of process parameters of saponin-based model foam using Taguchi method and gray relational analysis coupled with principal component analysis. *Journal of Food Processing and Preservation*, 46, e16553. https://doi.org/10.1111/jfpp.16553