

# Optimization of industrial shortening manufacturing procedure: The effect of cooling rate and kneading parameters on viscoelasticity and sensory properties

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## Research Article

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## Abstract

The main objective of this study was to optimize the industrial shortening manufacturing procedure and investigate the effect of cooling rate and kneading parameters on its viscoelasticity and sensory properties. The three selected main crystallization process parameters include the third cooling stage outlet temperature (28–31°C), kneading parameter (100–280 RPM), and filling temperature (30–36°C). The design type and model were Box-Behnken and quadratic, respectively. Rheological viscoelastic experiments on 17 samples with different crystallization factors were performed. Cooling and kneading intensities were manipulated in order to explore the resulting changes in frequency and strain sweep parameters. Additionally, sensory attributes related to the crystallization treatment samples were analyzed. The optimum crystallization parameters that led to the most desired functional attributes were crystallizer 3 outlet temperature of 31°C, kneading intensity of 280 RPM, and filling temperature of 33°C. All the storage modulus ( $G'$ ) and the loss modulus ( $G''$ ) model responses exhibited values in the minimum range at the optimum process conditions. This work's framework sets a new guideline for fine-tuning the functional properties of confectionary shortening through underlying the important role of the optimized crystallization process.

## 1. Introduction

Lipid shortenings are usually composed of a mixture of oils from a number of sources such as un-hydrogenated, partially, and fully hydrogenated oils, as well as emulsifiers, and other additives. Canola, cottonseed, lard, palm, soybean, and tallow are typical edible oils used for the production of shortenings. Because of the relatively high number of influencing factors, it is quite challenging to understand the effect of molecular composition, processing conditions, additives, and emulsifiers on physical functionality (Humphrey, Moquin, and Narine, 2003). One of the significant components in shortenings, margarine, and specialty products are plastic vegetable oils and fats. It is well known that the solid-like behavior of plastic fats is due to the presence of a fat crystal network (Litwinenko et al., 2002). In terms of the rheological analysis, shortenings are semi-plastic materials that are capable of creating the desirable textural properties in fatty-based foods (Hakimzadeh et al., 2020). Moriya et al. investigated the effect of solid fat content on the  $G'$  and  $G''$  of margarine and found that the  $G'$  and  $G''$  increased linearly with the increase in the solid fat content in a double-logarithmic plot, independent of the type of margarine (Moriya, Hasome, and Kawai, 2020). Bakery shortening has the ability to impart texture, tenderness, and taste to baked products. Shortening functionality depends on crystallization behavior, melting profile, and viscoelastic properties (Saghafi et al., 2018). Texturizing a shortening to improve its plasticity is usually achieved by a chilling process in which the melted fat is rapidly cooled in a chilling unit. Crystallization under shear not only improves the plasticity and texture of the mixture, it also removes the heat of crystallization from the sample (Ahmadi and Marangoni, 2009; Erickson, 1990). The nature of the crystal network, including its spatial distribution, and the number, size, and shape of its constituent microstructural elements can be dramatically altered by changes in crystallization conditions (Litwinenko et al., 2002). The effect of cooling in a surface scraping heat exchanger system on dynamic fat crystallization and physicochemical properties of puff pastry shortening was studied. The results revealed that crystallization temperature affected the rheological properties as well as the hardness of the produced shortenings (Nguyen et al., 2021). The scraped surface heat exchanger consists of a steel shaft rotating in a tube that is cooled externally by boiling ammonia. The rotating shaft is fitted with scraper blades which at high rotation speeds are pressed against the cooled inner surface by centrifugal force (Haighton, 1969). To minimize the post-crystallization phenomenon in order to ensure the development of the proper crystal structure and desired plasticity, the cooling and working stages must be prolonged (Alexandersen, Ghazani, and Marangoni, 2020; Masuchi et al., 2014). Accessing the desirable texture and functionality of shortenings could be accomplished by some modification processes, including hydrogenation, fractionation, blending, and chemical or enzymatic interesterification of fats and oils (Saghafi et al., 2018). Hydrogenation of unsaturated fatty acids in edible oils allows for the conversion of liquid oils into semi-solid or solid fats. These fats are characterized by altered melting and textural characteristics and a higher oxidative stability (Litwinenko et al., 2002). Ahmed et al. reported the physicochemical properties of a model shortening. Moreover, the rheological properties of soybean oil, as well as partially and fully hydrogenated soybean oil, were determined (Ahmed et al., 2020).

Rheological properties and texture in bakery shortening and margarine directly affect the quality aspects, physical appearance, sensory evaluation, and texture of baked goods as finished products (Saghafi et al., 2019). A comparison of lipid shortening application as a function of molecular ensemble and shear showed that changes in melting and crystallization behavior of

shortening systems could be significantly affected by manipulating the amounts of triacylglycerols containing specific fatty acids (Humphrey and Narine, 2004).

Investigating the effect of the crystallization regime on the macroscopic rheological and sensory properties of shortening was the aim of this study. Through working on a fixed formulation, it was attempted to determine the extent of the influence of crystallization process parameters on the final shortening texture, as well as storage and elastic attributes. The effects of the cooling rate, agitation, and filling temperature on the functional properties of shortenings including adhesiveness, hardness, plasticity, and homogeneity as well as the storage modulus ( $G'$ ) and loss modulus ( $G''$ ) were assessed. Although there have been few studies in which the fat mixture composition with or without considering an isolated crystallization factor has been investigated, the main focus of the current work was the crystallization process parameters and their interactions, which have never been investigated in similar research.

## 2. Materials And Methods

A multipurpose bakery shortening was selected in order to investigate the effect of the cooling rate and mixing on shortening sensory, storage, and elastic moduli. The concerned shortening contained a chemically interesterified fat blend, which included 4.2% canola oil, 71.8% palm oil, and 24% fully hydrogenated sunflower oil. The final shortening formulation was as follows: 33.25% chemically interesterified fat blend, 11.25% fully hydrogenated sunflower oil, 3.75% canola oil, and 51.75% palm oil. Design-Expert (version 12) software was employed to determine the experiment crystallization factor runs and to fit the response models through response surface methodology. The design type and model were Box-Behnken and quadratic, respectively. Because of some technical limitations, 17 runs were agreed on accordingly, all of which were related to one block. The experimental design with coded values and actual percentages and ranges of levels for independent variables of the crystallization process are mentioned in Table 1. In order to stay within a reasonable number of test runs, three process parameters were selected as the main indicators of the crystallization circumstances. The concerned crystallization arrangement contained four cooling units and four kneading units as shown in Fig. 1. Samples of the studied shortening were prepared in a Gerstenberg and Agger crystallizer (1 + 1 + 1 + 1 Perfector 250/NH<sub>3</sub>). The unit consists of a melting container, a chilling system (four separate chilling units), and a pin worker. Table 2 shows the parameters of the applied crystallization runs. The crystallization system fat inlet temperature was kept constant ( $65 \pm 1^\circ\text{C}$ ). The sensory analysis of shortening samples was carried out by a panelist team consisting of 15 experts who had been trained before the test for the expected performance. In this regard, four descriptive attributes including texture adhesiveness, hardness, plasticity, and homogeneity were assessed accordingly. The sensory evaluation test and analysis were carried out a week after packaging during which shortening samples were kept at  $16^\circ\text{C}$ .

Table 1  
Experimental ranges and levels of independent variables used in quadratic Box-Behnken response surface design

| Factor   | Symbol | Unit             | Type    | Range of Levels |           |             |            | Mean | Standard Deviation |
|--|--------|------------------|---------|-----------------|-----------|-------------|------------|------|--------------------|
|  |        |                  |         | Low actual      | Low coded | High actual | High coded |      |                    |
| Cooling Unit No. 3 Outlet Shortening Temperature | $T_3$  | $^\circ\text{C}$ | Numeric | 28              | -1        | 31          | +1         | 29.5 | 1.06               |
| Kneading Unit 2 Intensity                        | RPM    | Round per minute | Numeric | 100             | -1        | 280         | +1         | 190  | 63.64              |
| Filling Temperature                              | FT     | $^\circ\text{C}$ | Numeric | 30              | -1        | 36          | +1         | 33   | 2.12               |

Table 2  
Crystallization treatments

| Crystallization Treatments | Cooling Unit No. 3 Outlet Shortening Temperature °C | Kneading Unit 2 Intensity Round per minute | Filling Temperature °C |
|----------------------------|---|--|------------------------|
| T1                         | 29.5  | 280.0                                      | 30.0                   |
| T2                         | 31.0  | 190.0                                      | 36.0                   |
| T3                         | 31.0  | 280.0                                      | 33.0                   |
| T4                         | 29.5  | 100.0                                      | 30.0                   |
| T5                         | 28.0  | 280.0                                      | 33.0                   |
| T6                         | 29.5  | 280.0                                      | 36.0                   |
| T7                         | 28.0  | 190.0                                      | 36.0                   |
| T8                         | 31.0  | 100.0                                      | 33.0                   |
| T9                         | 31.0  | 190.0                                      | 30.0                   |
| T10                        | 28.0  | 190.0                                      | 30.0                   |
| T11                        | 28.0  | 100.0                                      | 33.0                   |
| T12                        | 29.5  | 190.0                                      | 33.0                   |
| T13                        | 29.5  | 100.0                                      | 36.0                   |
| T14                        | 29.5  | 190.0                                      | 33.0                   |
| T15                        | 29.5  | 190.0                                      | 33.0                   |
| T16                        | 29.5  | 190.0                                      | 33.0                   |
| T17                        | 29.5  | 190.0                                      | 33.0                   |

In this descriptive study, data were obtained in the rheology lab of the National Nutrition and Food Technology Research Institute, Tehran, Iran. Rheological measurements were performed with a Physica MCR 301 rheometer (Anton-Paar, GmbH, Graz, Austria). Temperature control was carried out using a Peltier system equipped with a fluid circulator. The authors decided to perform rheological tests at 25°C which is common as the confectionary workshop temperature.

For all the samples, parallel plate (PP40/S-SN 16891; d = 2 mm) geometry was utilized. Data analysis was carried out using Rheoplus software (version 3.21) (Anton Paar, Graz, Austria). Strain sweep tests were performed (0.001-500%, 6.28 rad/s) to determine the limiting value of the linear viscoelastic range (LVE). To provide a direct view of whether the samples behaved as liquids or solids, loss tangent (ratio of loss modulus to elastic modulus) was calculated at the LVE region. The crossover point ( $G' = G''$ ) was also obtained. Frequency sweep tests were performed using a frequency ramp from 0.0628 to 126 rad/s.  $G'$  and  $G''$  in three angular frequencies of 0.0628, 3.31, and 126 (rad/s) were calculated as representatives of low, moderate, and high angular frequencies, respectively.

### 3. Results And Discussion

#### 3.1. Sensory evaluation

According to Table 3, which reports the outcomes of four chosen qualitative attributes of the studied shortening samples, crystallization treatments number 3 and 7 possessed the most favorable overall functional sensory quality. Regarding the concerned shortening applications, the goal was to reach the highest possible plasticity and homogeneity while the adhesiveness needed to be minimum. The desired hardness for this shortening was recognized by expert assessors as favorable.

Table 3  
Sensory evaluation outcomes at 16°C

| Crystallization Run | Tempering Conditions | Adhesiveness | Hardness | Plasticity | Homogeneity | Overall Verdict         |
|---------------------|----------------------|--------------|----------|------------|-------------|-------------------------|
| 1                   | 16°C                 | 4 ± 1.2      | 3 ± 1.1  | 2 ± 1.1    | 2 ± 1.3     |                         |
| 2                   | More than One Week   | 4 ± 1.2      | 2 ± 1.3  | 3 ± 1.2    | 3 ± 1.1     |                         |
| 3                   |                      | 2 ± 1.1      | 3 ± 1.2  | 3 ± 1.1    | 3 ± 1.2     | Superior Quality (+++)  |
| 4                   |                      | 3 ± 1.3      | 4 ± 1.1  | 2 ± 1.3    | 2 ± 1.2     |                         |
| 5                   |                      | 5 ± 1.1      | 2 ± 1.3  | 2 ± 1.3    | 4 ± 1.1     |                         |
| 6                   |                      | 1 ± 1.3      | 4 ± 1.2  | 3 ± 1.3    | 3 ± 1.3     |                         |
| 7                   |                      | 4 ± 1.1      | 4 ± 1.2  | 3 ± 1.1    | 3 ± 1.3     | Overall Acceptable (++) |
| 8                   |                      | 3 ± 1.2      | 4 ± 1.3  | 2 ± 1.1    | 3 ± 1.3     |                         |
| 9                   |                      | 2 ± 1.3      | 4 ± 1.1  | 4 ± 1.3    | 2 ± 1.2     |                         |
| 10                  |                      | 2 ± 1.2      | 4 ± 1.3  | 3 ± 1.1    | 2 ± 1.1     | Overall Acceptable (++) |
| 11                  |                      | 3 ± 1.1      | 4 ± 1.2  | 2 ± 1.2    | 5 ± 1.3     | Overall Acceptable (++) |
| 12                  |                      | 4 ± 1.2      | 4 ± 1.1  | 2 ± 1.2    | 3 ± 1.3     |                         |
| 13                  |                      | 2 ± 1.3      | 4 ± 1.2  | 2 ± 1.3    | 5 ± 1.1     |                         |
| 14                  |                      | 4 ± 1.3      | 4 ± 1.2  | 2 ± 1.1    | 3 ± 1.3     |                         |
| 15                  |                      | 4 ± 1.2      | 4 ± 1.1  | 2 ± 1.2    | 3 ± 1.3     |                         |
| 16                  |                      | 4 ± 1.1      | 4 ± 1.2  | 2 ± 1.1    | 3 ± 1.3     |                         |
| 17                  |                      | 4 ± 1.3      | 4 ± 1.1  | 2 ± 1.2    | 3 ± 1.3     |                         |

Note: Mean Values ± SD (n = 3).

### 3.2. Rheological characterization

Figure 2 depicts the strain sweep rheograms related to the crystallization treatments. For the  $G'$  of 17 crystallization treatments, it was possible to discriminate two different regions, namely, a small linear viscoelastic region, in which  $G'$  was nearly constant and a bigger nonlinear region, in which  $G'$  started to diminish with increasing strain. The LVE region was determined in order to help distinguish between the strong and weak structures of the samples. As can be seen in Table 4, the LVE region was greater in sample 4. For most solid foods, LVE is between 0.1% and 2%; however, in this study, we chose the strain of 0.05% in the LVE region. In the case of  $G''$ , the behavior of samples in the strain sweep test was identical to the one mentioned for  $G'$ . In all the samples, both moduli finally tended to crossover; therefore, all 17 crystallization treatments showed flow points. As shown at lower strains,  $G'$  values were sensibly greater than  $G''$  values. While with increasing strain percentages,  $G''$  values were ultimately greater than  $G'$  values. Due to the fact that in all the cases, before the flow point,  $G'$  was higher than  $G''$  it can be concluded that all the samples in the linear viscoelastic region showed a solid-like behavior. However, at higher strains,  $G''$  was higher and the samples exhibited a more liquid-like behavior. The strain sweep parameters of 17 cases are listed in Table 4.  $G_f$  is the amount of the stress at the crossover point and indicates that exerting further stress resulted in the structure breakdown and thus the flow of the matter (Naeli et al., 2022). After this point,  $G''$  is more than  $G'$  ( $\tan(\delta) > 1$ ), which suggests a structure rupture and the start of the flow behavior. The highest amount of  $G_f$  was observed in sample 9.  $\tan(\delta)_{LVE}$  is the  $G'$  to  $G''$  ratio in the LVE region, and this parameter can be an indicator of the physical properties of the manufactured samples. According to the reported data in Table 4, samples 1 and 2 had the lowest amount of  $\tan(\delta)_{LVE}$ .

Table 4

Strain sweep parameters of 17 crystallization treatments at 25°C. Further rheological experiments were performed at the mentioned temperature, limiting value of linear viscoelastic range  $\gamma_1$  (%), damping factor for the end of LVE region ( $\tan \delta_{Lve}$ ), and crossover point  $G_f$  (pa) ( $G'=G''$ )

| Crystallization Treatments | $\gamma_1$ (%) | Tan $\delta_{Lve}$ | $G_f$ (Pa) |
|----------------------------|----------------|--------------------|------------|
| T1                         | 0.0513         | 0.1060             | 400.00     |
| T2                         | 0.0596         | 0.0703             | 1240.00    |
| T3                         | 0.0650         | 0.0833             | 2165.30    |
| T4                         | 0.2950         | 0.1932             | 1171.10    |
| T5                         | 0.0701         | 0.1920             | 741.28     |
| T6                         | 0.1060         | 0.1290             | 24714.00   |
| T7                         | 0.0932         | 0.1260             | 2459.70    |
| T8                         | 0.0799         | 0.1490             | 1740.40    |
| T9                         | 0.0972         | 0.1340             | 4398.90    |
| T10                        | 0.0595         | 0.1290             | 2760.20    |
| T11                        | 0.0619         | 0.1040             | 3578.70    |
| T12                        | 0.0598         | 0.0953             | 1965.70    |
| T13                        | 0.0605         | 0.1080             | 13051.00   |
| T14                        | 0.0701         | 0.192              | 741.28     |
| T15                        | 0.0701         | 0.192              | 741.28     |
| T16                        | 0.0701         | 0.192              | 741.28     |
| T17                        | 0.0701         | 0.192              | 741.28     |

Strain sweep curves for rheology parameters, such as  $G'$ ,  $G''$ , and the loss tangent of an all-purpose shortening crystallized with and without high-intensity ultrasound were studied in a similar investigation, which showed similar  $G'$  and  $G''$  profiles after 60 min of crystallization and during storage at 25°C (Lee, Marsh, and Martini, 2020). A comparable work reported that during crystallization, the rheology of the fats changed from a weak viscoelastic solid system to a weak viscoelastic liquid. In this examination, it appeared that, initially, the rheology was dominated by the few large crystals present, but after longer stirring, weak viscoelastic liquid behavior, which is characteristic of liquid slip planes between solid crystals, dominated the behavior (Bell et al., 2007). Additionally, storage and loss moduli changes of binary combinations of canola oil and palm stearin were assessed in a study intended for zero-trans cake shortening formulation and characterization. The rheological results of this work showed patterns compatible with the current study (Saghafi et al., 2018).

Considering the strain sweep, frequency sweep tests were performed, and the mechanical spectra of the cases are depicted in Fig. 3. Table 5 presents the viscous and elastic moduli of 17 samples in three angular frequencies of 0.0628, 3.31, and 126 rad/s. The results of the frequency sweep test revealed that  $G'$  modulus was greater than  $G''$  modulus in all frequency ranges and no crossover was observed. It is known that in gel systems,  $G'$  is greater than  $G''$  in the applied frequency range. According to the frequency sweep data, a solid-like behavior was observed in all frequency ranges. This behavior represents a strong network as a result of forming the corresponding network structure.

Table 5

Frequency sweep parameters of 17 crystallization treatments,  $G'$  (Pa), and  $G''$  (Pa) of each run in frequency sweep test was calculated in three angular frequencies, namely, high  $\omega = 126$  (rad/s), medium  $\omega = 3.31$  (rad/s) and low  $\omega = 0.628$  (rad/s) angular frequencies

| Crystallization Treatments | Storage Modulus, $G'$ (Pa)/100000 |                      |                     | Loss Modulus, $G''$ (Pa)/100000 |                      |                     | Tan $\delta$           |                      |                     |
|----------------------------|-----------------------------------|----------------------|---------------------|---------------------------------|----------------------|---------------------|------------------------|----------------------|---------------------|
|                            | $\omega =$<br>(0.0628)            | $\omega =$<br>(3.31) | $\omega =$<br>(126) | $\omega =$<br>(0.0628)          | $\omega =$<br>(3.31) | $\omega =$<br>(126) | $\omega =$<br>(0.0628) | $\omega =$<br>(3.31) | $\omega =$<br>(126) |
|                            | T1                                | 0.507                | 0.947               | 1.21                            | 0.359                | 0.24                | 0.265                  | 0.7                  | 0.3                 |
| T2                         | 8.64                              | 9.56                 | 11.1                | 2.1                             | 1.44                 | 1.22                | 0.2                    | 0.2                  | 0.1                 |
| T3                         | 9.48                              | 10.1                 | 11.6                | 1.85                            | 1.4                  | 1.17                | 0.2                    | 0.1                  | 0.1                 |
| T4                         | 9.77                              | 10.6                 | 12.1                | 1.97                            | 1.4                  | 1.22                | 0.2                    | 0.1                  | 0.1                 |
| T5                         | 1.89                              | 2.24                 | 2.69                | 0.644                           | 0.465                | 0.484               | 0.3                    | 0.2                  | 0.2                 |
| T6                         | 8.85                              | 10.2                 | 11.8                | 2.09                            | 1.36                 | 1.24                | 0.2                    | 0.1                  | 0.1                 |
| T7                         | 11.4                              | 12.4                 | 14.3                | 2.68                            | 1.89                 | 1.52                | 0.2                    | 0.2                  | 0.1                 |
| T8                         | 5.95                              | 7.0                  | 8.25                | 1.63                            | 1.21                 | 1.05                | 0.3                    | 0.2                  | 0.1                 |
| T9                         | 7.0                               | 8.27                 | 9.59                | 1.95                            | 1.1+                 | 1.01                | 0.3                    | 0.1                  | 0.1                 |
| T10                        | 5.24                              | 5.92                 | 6.91                | 1.3                             | 1.02                 | 0.911               | 0.2                    | 0.2                  | 0.1                 |
| T11                        | 10.9                              | 12.5                 | 14.4                | 2.86                            | 1.78                 | 1.45                | 0.3                    | 0.1                  | 0.1                 |
| T12                        | 11.4                              | 12.1                 | 13.9                | 2.46                            | 1.77                 | 1.36                | 0.2                    | 0.1                  | 0.1                 |
| T13                        | 8.26                              | 9.8                  | 11.5                | 2.18                            | 1.37                 | 1.25                | 0.3                    | 0.1                  | 0.1                 |
| T14                        | 11.4                              | 12.1                 | 13.9                | 2.46                            | 1.77                 | 1.36                | 0.2                    | 0.1                  | 0.1                 |
| T15                        | 11.4                              | 12.1                 | 13.9                | 2.46                            | 1.77                 | 1.36                | 0.2                    | 0.1                  | 0.1                 |
| T16                        | 11.4                              | 12.1                 | 13.9                | 2.46                            | 1.77                 | 1.36                | 0.2                    | 0.1                  | 0.1                 |
| T17                        | 11.4                              | 12.1                 | 13.9                | 2.46                            | 1.77                 | 1.36                | 0.2                    | 0.1                  | 0.1                 |

### 3.3. Fitting for the best model

Statistical analysis parameters of the Fisher test value (F-value), the p-value of model, coefficient of determination ( $R^2$ ), the adjusted coefficients of determination ( $R^2$ -adj), predicted  $R^2$ , the p-value of lack of fit (LOF), adequate precision, and percentage of the coefficient of variation (CV%) obtained from the analysis of variance (ANOVA) were used for evaluating the goodness of fit in models. Fitted quadratic equations were applied for achieving the optimized formulation. Statistical significance of the model parameters was set at 5% (p-value = 0.05). Tables 4 and 5 report the corresponding 17-run obtained responses. All of the responses were fitted to the quadratic model. According to Table 6, the p-value for the model indicates that the models are significant at less than a 0.05 level.

Table 6  
Parameter estimates and analysis of variance for responses model\*

| Response           | Model   |          | Fit Statistics |             |    |                |                         |                    |        |                    |       |
|--------------------|---------|----------|----------------|-------------|----|----------------|-------------------------|--------------------|--------|--------------------|-------|
|                    | F-value | p-value  | Sum of Squares | Mean Square | df | R <sup>2</sup> | Adjusted R <sup>2</sup> | Standard Deviation | Mean   | Adequate precision | CV %  |
| G' (0.0628 rad/s)  | 152.09  | < 0.0001 | 5401.87        | 600.21      | 9  | 0.9949         | 0.9884                  | 1.99               | 22.88  | 33.9401            | 8.68  |
| G' (3.31 rad/s)    | 34.92   | < 0.0001 | 278.97         | 31.00       | 9  | 0.9782         | 0.9502                  | 0.9421             | 7.09   | 15.4673            | 13.28 |
| G' (126 rad/s)     | 31.44   | < 0.0001 | 362.43         | 40.27       | 9  | 0.9759         | 0.9448                  | 1.13               | 8.24   | 14.7008            | 13.73 |
| G'' (0.0628 rad/s) | 23.88   | 0.0002   | 10.48          | 1.16        | 9  | 0.9685         | 0.9279                  | 0.2209             | 1.57   | 13.3543            | 14.09 |
| G''(3.31 rad/s)    | 29.23   | < 0.0001 | 4.62           | 0.5133      | 9  | 0.9741         | 0.9408                  | 0.1325             | 1.08   | 15.6810            | 12.27 |
| G'' (126 rad/s)    | 16.57   | 0.0006   | 2.56           | 0.2846      | 9  | 0.9552         | 0.8975                  | 0.1311             | 0.9462 | 11.3342            | 13.85 |

\*P < 0.05 is significant.

### 3.4. Storage and loss moduli

The equations of predicted models are presented in Table 7. The concerned regression equations predict the value of each response variable when the independent factors are varied; a positive sign in front of the terms suggests a synergistic effect, whereas a negative sign indicates an antagonistic effect.

Table 7  
Final equation of responses

| Response                  | Equations (X <sub>1</sub> : T3, X <sub>2</sub> : RPM, X <sub>3</sub> : FT)   |
|---------------------------|--|
| G' (0.0628 rad/s)/100000  | + 5886.56945-151.23892X <sub>1</sub> -0.848513X <sub>2</sub> -209.69818X <sub>3</sub> + 0.10492X <sub>1</sub> X <sub>2</sub> + 0.100633X <sub>1</sub> X <sub>3</sub> - 0.000013X <sub>2</sub> X <sub>3</sub> + 2.3769X <sub>1</sub> <sup>2</sup> + 0.001149X <sub>2</sub> <sup>2</sup> + 3.11504X <sub>3</sub> <sup>3</sup>      |
| G' (3.31 rad/s)/100000    | + 1394.30199-38.21195X <sub>1</sub> -0.003948X <sub>2</sub> -48.95159X <sub>3</sub> - 0.003469X <sub>1</sub> X <sub>2</sub> + 0.028333X <sub>1</sub> X <sub>3</sub> - 0.000741X <sub>2</sub> X <sub>3</sub> + 0.624278X <sub>1</sub> <sup>2</sup> + 0.00029X <sub>2</sub> <sup>2</sup> + 0.729208X <sub>3</sub> <sup>3</sup>     |
| G' (126 rad/s)/100000     | + 1589.50194-43.29593X <sub>1</sub> + 0.002218X <sub>2</sub> -56.09968X <sub>3</sub> - 0.004352X <sub>1</sub> X <sub>2</sub> + 0.038333X <sub>1</sub> X <sub>3</sub> - 0.000741X <sub>2</sub> X <sub>3</sub> + 0.705556X <sub>1</sub> <sup>2</sup> + 0.000335X <sub>2</sub> <sup>2</sup> + 0.832778X <sub>3</sub> <sup>3</sup>   |
| G'' (0.0628 rad/s)/100000 | + 269.99817-6.403457X <sub>1</sub> + 0.024624X <sub>2</sub> -10.57241X <sub>3</sub> - 0.001335X <sub>1</sub> X <sub>2</sub> + 0.031111X <sub>1</sub> X <sub>3</sub> - 0.000287X <sub>2</sub> X <sub>3</sub> + 0.092944X <sub>1</sub> <sup>2</sup> + 0.000053X <sub>2</sub> <sup>2</sup> + 0.146875X <sub>3</sub> <sup>3</sup>    |
| G''(3.31 rad/s)/100000    | + 188.44778-5.62574X <sub>1</sub> + 0.015144X <sub>2</sub> -6.27875X <sub>3</sub> - 0.001093X <sub>1</sub> X <sub>2</sub> + 0.011667X <sub>1</sub> X <sub>3</sub> + 2.56834 E (-18)X <sub>2</sub> X <sub>3</sub> + 0.0900X <sub>1</sub> <sup>2</sup> + 0.000037X <sub>2</sub> <sup>2</sup> + 0.089444X <sub>3</sub> <sup>3</sup> |
| G'' (126 rad/s)/100000    | + 133.80472-3.79015X <sub>1</sub> + 0.018788X <sub>2</sub> -4.67701X <sub>3</sub> - 0.000974X <sub>1</sub> X <sub>2</sub> + 0.005000X <sub>1</sub> X <sub>3</sub> - 0.000074X <sub>2</sub> X <sub>3</sub> + 0.062778X <sub>1</sub> <sup>2</sup> + 0.000028X <sub>2</sub> <sup>2</sup> + 0.068583X <sub>3</sub> <sup>3</sup>      |

Figure 4 demonstrates storage and loss moduli models in high angular frequency ( $\omega = 126$  rad/s) when the filling temperature is 33°C. Figure 5 indicates storage and loss moduli (at the angular velocity of 126 rad/s) when the concerned crystallization

parameters deviate from a reference point where all three crystallization factors are at their mean values (i.e.,  $T_3 = 29.5^\circ\text{C}$ , RPM = 190, and FT =  $33^\circ\text{C}$ ).

### 3.5. Optimized crystallization treatment

According to Table 3, the shortening sample with the most favorable sensory properties was related to crystallization treatment number 3 (i.e.,  $T_3 = 31^\circ\text{C}$ , RPM = 280, and FT =  $33^\circ\text{C}$ ). At the mentioned related parameters, all the modeled storage and loss moduli (i.e.,  $G'$  and  $G''$  at 0.0628, 3.31, and 126 rad/s) were in the minimum values range. As an example, Fig. 6 demonstrates the storage and loss moduli in the lower range of angular velocity (i.e.,  $G'$  and  $G''$  at 0.0628 rad/s).

As another illustrative example, Fig. 7 shows the storage and loss moduli changes at the middle angular velocity (i.e., 3.31 rad/s) when RPM is 280. In a similar fashion, the crystallization process conditions related to the most desirable functional properties correspond to the minimum storage and loss moduli values.

One of the current study's main objectives was to achieve a comprehensive model for consequent optimization activities regarding the determination of the proper crystallization parameters for different shortening functional requirements. As discussed earlier, the most favorable functional properties of the concerned shortening correspond to minimum storage and loss moduli values. The chosen optimization criteria and the achieved solutions are summarized in Table 8.

**Table 8** Optimization criteria and solution

| Optimization Criteria       |          |             |             | Solution<br>(Desirability=0.94) |      |                             |       |
|-----------------------------|----------|-------------|-------------|---------------------------------|------|-----------------------------|-------|
| Response                    | Goal     | Lower Limit | Upper Limit | Factor                          |      | Response value              |       |
| $G'$ (0.0628 rad/s)/100000  | minimize | 0.507       | 11.4        |                                 |      | $G'$ (0.0628 rad/s)/100000  | 0.845 |
| $G'$ (3.31 rad/s)/100000    | minimize | 0.947       | 12.5        |                                 |      | $G'$ (3.31 rad/s)/100000    | 1.383 |
| $G'$ (126 rad/s)/100000     | minimize | 1.21        | 14.4        |                                 |      | $G'$ (126 rad/s)/100000     | 1.749 |
| $G''$ (0.0628 rad/s)/100000 | minimize | 0.359       | 2.86        | T3                              | 30.4 | $G''$ (0.0628 rad/s)/100000 | 0.512 |
| $G''$ (3.31 rad/s)/100000   | minimize | 0.24        | 1.89        | RPM                             | 228  | $G''$ (3.31 rad/s)/100000   | 0.349 |
| $G''$ (126 rad/s)/100000    | minimize | 0.265       | 1.52        | FT                              | 33.1 | $G''$ (126 rad/s)/100000    | 0.406 |

Hence, these responses are considered in the range of the studied levels. Derringer's desirability function also calculated the optimum conditions:

$$D = \sqrt[m]{d_1 d_2 \dots d_m}$$

Equation 1. Derringer's desirability function

where  $m$  is the number of responses studied in the optimization process, and  $d$  is the individual desirability function of each response. Derringer's desirability function ( $D$ ) can take values from 0 to 1. A value greater than 0.7 indicates that the combination of the different criteria is matched in a global optimum (Bezerra et al., 2008; Ramsay et al., 2014).

To validate the optimization results, three replicates of the experiments for each response were performed under optimal conditions. When optimum circumstances were applied, the comparison of the experimental results with the predicted ones was carried out using paired t-test (Table 9). As indicated, insignificant differences between the experimental and predicted results imply the validity of the optimal outcomes.

Table 9  
Results of validity evaluation of optimal parameters

| Response                  | Predicted value | Experimental value | p-value<br>(paired-t test) |
|---------------------------|-----------------|--------------------|----------------------------|
| G' (0.0628 rad/s)/100000  | 0.845 ± 1.162   | 0.923 ± 0.765      | 0.62                       |
| G' (3.31 rad/s)/100000    | 1.382 ± 1.104   | 1.397 ± 0.665      | 0.96                       |
| G' (126 rad/s)/100000     | 1.748 ± 1.330   | 2.135 ± 0.804      | 0.25                       |
| G'' (0.0628 rad/s)/100000 | 0.512 ± 0.309   | 0.779 ± 0.435      | 0.62                       |
| G''(3.31 rad/s)/100000    | 0.349 ± 0.191   | 0.334 ± 0.245      | 0.84                       |
| G'' (126 rad/s)/100000    | 0.405 ± 0.179   | 0.335 ± 0.245      | 0.42                       |

## 4. Conclusion

There is growing interest regarding the relationship between crystallization parameters, and rheological and functional properties of different bakery and confectionary shortenings. There are several studies available in which rheological, functional, and sensory attributes of some fat and oil blends (including margarine and shortening product applications) have been reported. Rarely in these works, the effects of crystallization process parameters have been the main focus of the investigation. On the other hand, the current study's principal emphasis was to determine the impacts of crystallization arrangement and parameters on the resultant shortening. Here, it was shown that the storage and loss moduli of a fat mixture were altered considerably when the selected crystallization process parameters were manipulated. The storage and loss moduli values demonstrated similar patterns when crystallization parameters were altered. By applying the desired criteria, the optimum values of the third cooling stage outlet temperature, kneading intensity, and filling temperature were 30.4°C, 228, and 33.1°C, respectively. The following validity evaluation confirmed the emerged optimized factors which underlines the vital role of similar approaches for understanding, modeling, and predicting different shortening rheological behaviors.

## Declarations

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### Contributions

MA designed and performed all of the experiments. She also wrote the draft of the manuscript. LR designed the research and analyzed the results of the experiments. AZ designed the rheological tests and analyzed the results. FZ performed all of the

modeling procedures and their analyzes.

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### Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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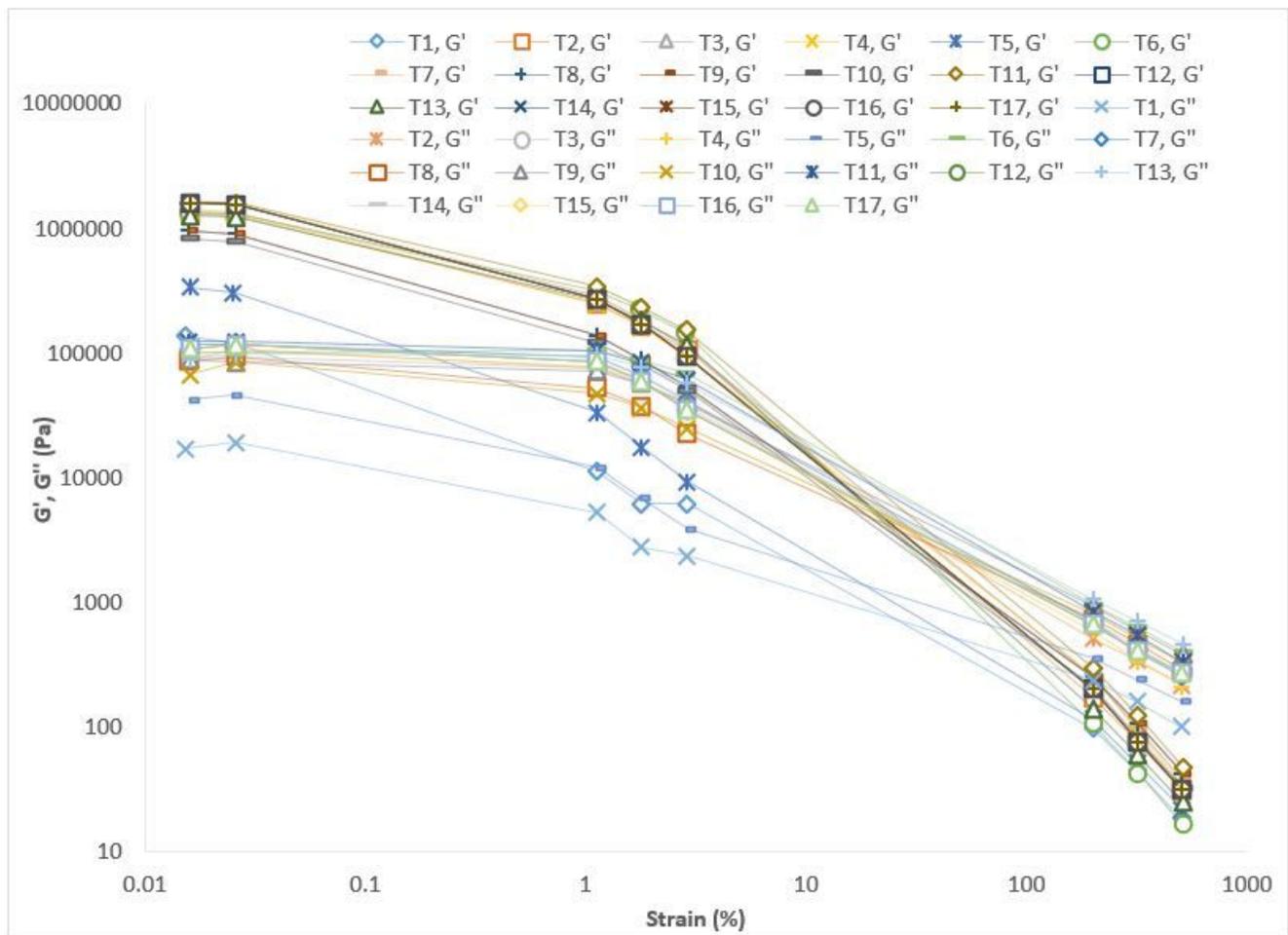
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## Figures



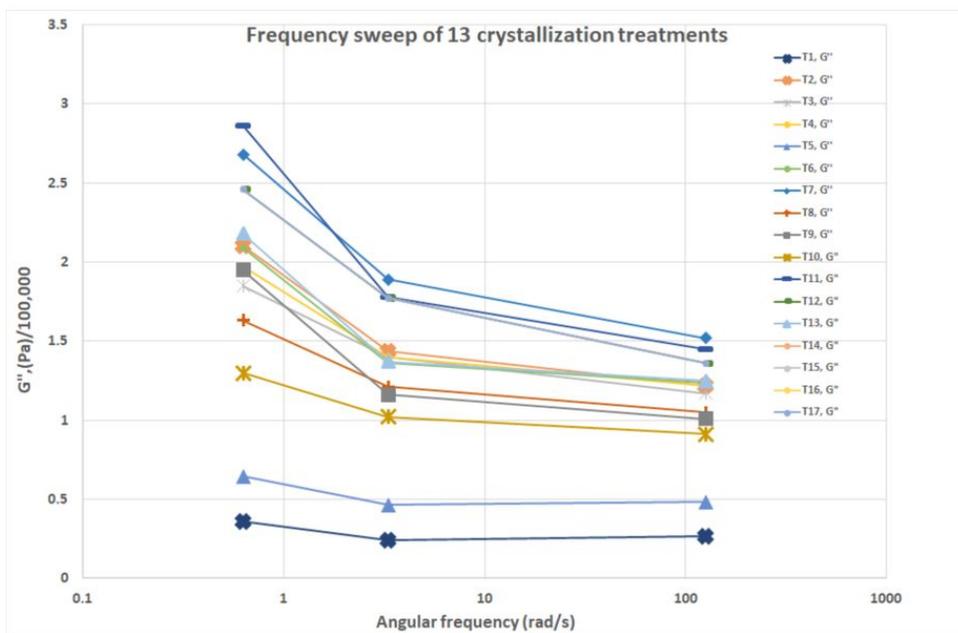
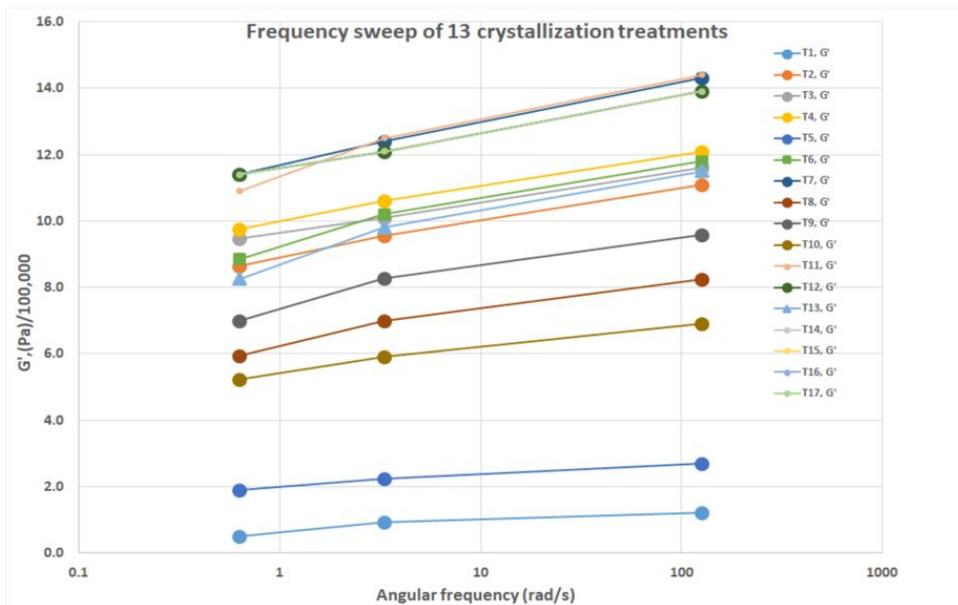
Figure 1

Crystallization process cooling and kneading units arrangement



**Figure 2**

Strain sweep of the 17 crystallization treatments



**Figure 3**

Frequency dependency of  $G'$  (a) and  $G''$  (b) of the 17 crystallization treatments

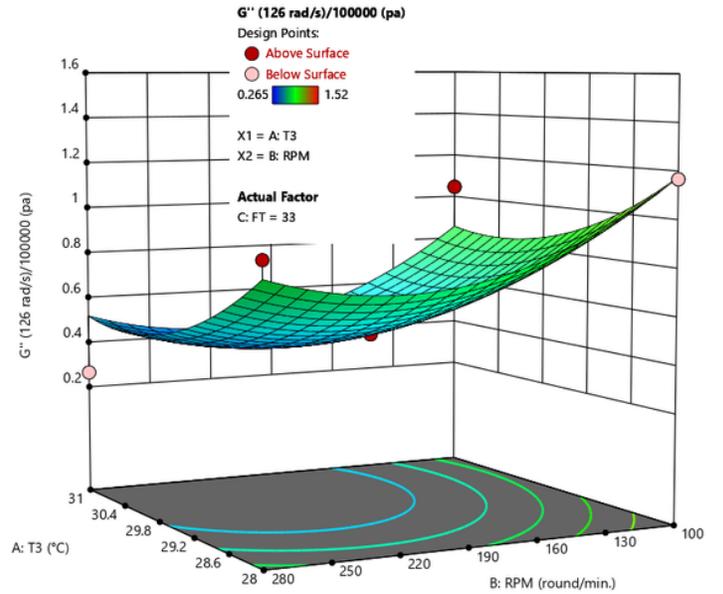
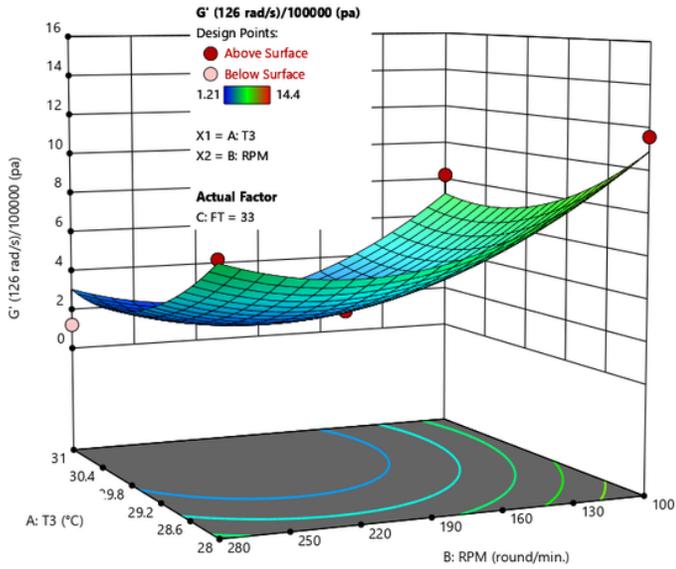


Figure 4

G' and G'' (126 rad/s) when filling temperature is 33°C

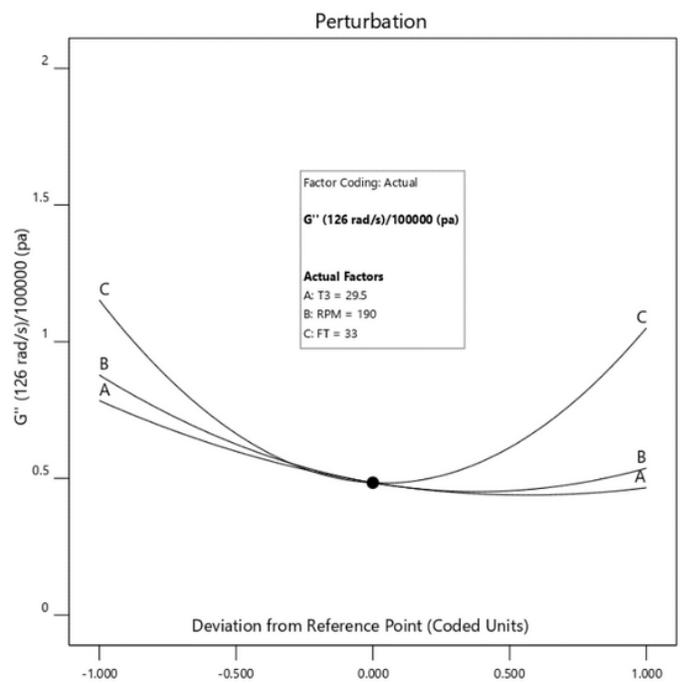
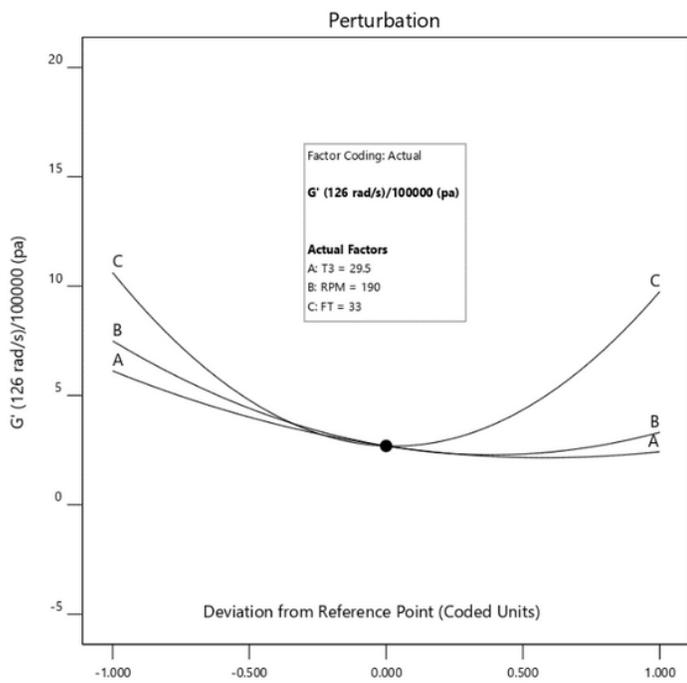


Figure 5

Perturbation diagram of G' and G'' (126 rad/s) when filling temperature is 33°C

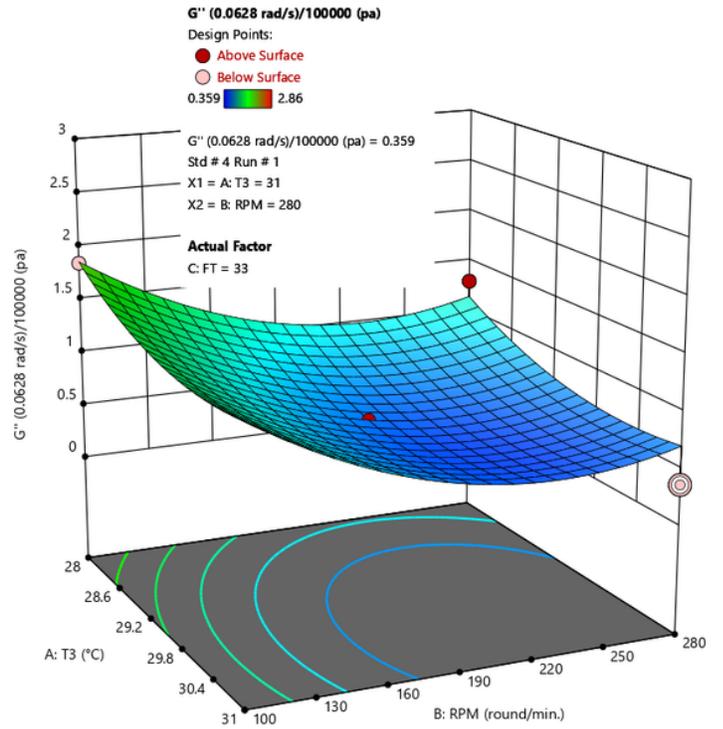
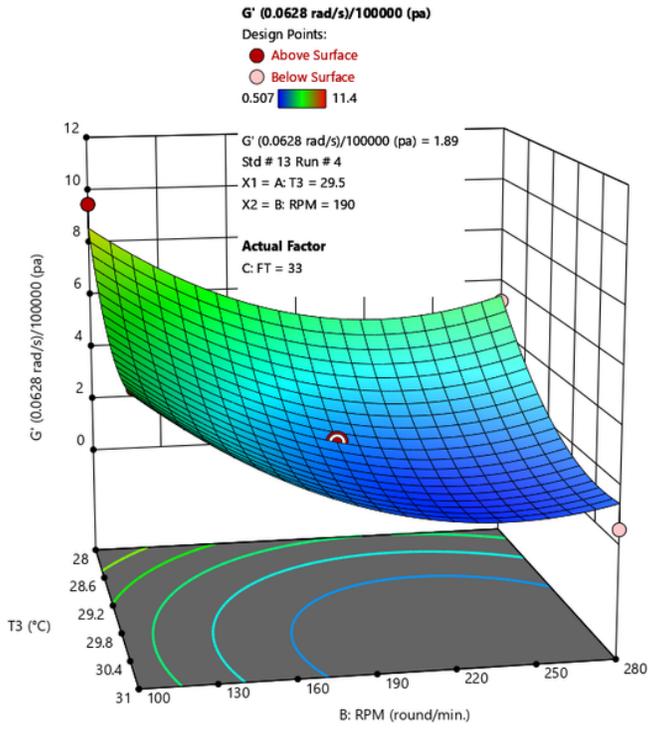


Figure 6

$G'$  and  $G''$  (0.0628 rad/s) when filling temperature is 33°C

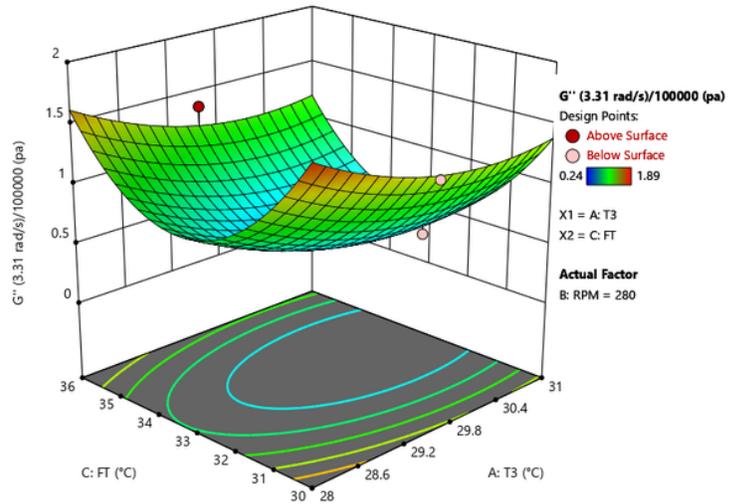
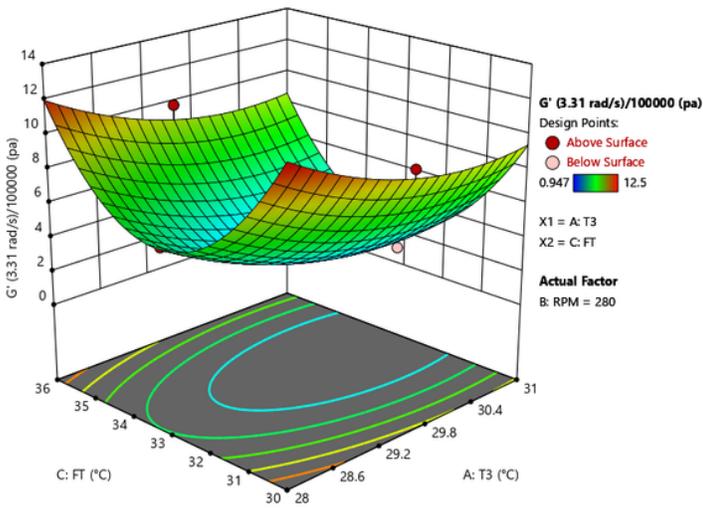


Figure 7

$G'$  and  $G''$  (3.31 rad/s) when RPM is 280