Katalin Zay[1], László Somogyi[1], Anita Soós[1]

Received: January 2018 – Accepted: April 2018

The effect of ground poppy seeds with different properties on the rheological properties of white chocolate

Keywords: poppy seed, white chocolate, strength research, rheological properties, product development

1. Summary

In the coarse of our research, white chocolate samples prepared using poppy seeds and poppy seed flour of different grain size and in different amounts were compared in order to be able to determine what kind of effect they had on the rheological properties of the finished products, i.e., how the penetration and rotational viscosity properties of the finished products were influenced. Based on the results of rotational viscometry, there was a significant difference between the samples that contained poppy seeds, depending on the poppy seed grain size. However, on the basis of the penetration measurement results, there was a significant difference between the samples containing lower oil content poppy seed flour, correlating to the grain size. During grinding, two opposite effects occur: on the one hand, removal of the oil content, and on the other hand, an increase in the number of particles in a given volume unit. In the technology, these two effects prevail together, but to varying degrees. There was a significant difference in the average particle size of the paste samples containing poppy seeds or poppy seed flour. Based on the results obtained using main component analysis and discriminant analysis it can be said that the main factor when classifying chocolates containing poppy seeds/poppy seed flour was the form of the poppy seeds/poppy seed flour added to the chocolate. The effect of the amount of the added poppy seeds/poppy seed flour was only secondary.

Based on the results of the consumer sensory evaluation, samples that contained whole poppy seeds in smaller amounts were most preferred by the consumers interviewed, while chocolates that contained larger amounts of whole poppy seed flour were less preferred.

2. Introduction

The poppy chocolate produced in the course of the research belongs to the group of single layer products, more specifically, to the complex composition suspensions. The dispersion medium in the molten chocolate is cocoa butter, and finely ground sucrose and the other components are suspended in this **[12]**. Chocolate is a unique food that is solid at room temperature, while it easily melts in the mouth at around 37 °C during consumption. This property is due to cocoa butter **[2]**. The cocoa butter content of chocolate predominantly composed of saturated fatty acids. Its components include stearic acid (S, 34%), palmitic acid (P, 27%), and the monounsaturated oleic acid (O, 34%). Thus, the most characteristic triglycerides in cocoa butter are the esters of the above-mentioned fatty acids (POP,

[1] Szent István University, Faculty of Food Science, Department of Grain and Industrial Plant Processing

POS, SOS) **[1]**. A specialty of this type of products is texture strength, which means that the molten fat is frozen, during which triglyceride molecules crystallize in the proper arrangement, and these crystal forms are responsible for the shell fracture and surface sheen of the chocolate **[12]**.

Based on the vast majority of foods and the above-mentioned characteristics, chocolate paste displays a property characteristic of non-Newtonian liquids, meaning that its viscosity changes with shear stress **[17]**. The flow of non-Newtonian materials can be characterized by the Bingham test yield point equation **[9]**. The most common behavior is called pseudoplastic behavior, which means that viscosity decreases with increasing shear stress. This type of behavior is common in liquids such as molten chocolate. The viscosity of the chocolate paste is higher than that of cocoa butter, and it depends on the shear rate. Based on its rheogram it can be stated that its flow only starts after reaching the limit stress, ad after passing the yield point, the starting viscosity of the paste is the highest, and the slope of it decreases with the gradual increase of the shear rate.

The rheological behavior of chocolate is described by Casson by the following equation:

$$\tau^{1/2} = \tau_{Ca}^{1/2} + (\eta_{Ca} \cdot \gamma)^{1/2} \quad (1)$$

The advantage of the Casson equation (1) is that the paste can be characterized by an easily linearizable formula, the Casson yield point and the Casson viscosity in a very wide shear stress and shear rate range.

There are several measuring instruments available for industrial viscosity measurements, but in the production of chocolate, mainly the use of rotational viscometers has gained acceptance, such as the MacMichael single-speed instrument, or the Brookfield and Haake multiple-speed instruments **[11]**.

Sensory tests are usually carried out by trained and/or lay (consumer) judges. During the tests carried out with a trained panel, analytical questioning is applied, i.e., questions are aimed at the intensity values of the different organoleptic properties (e.g.: how intense the sweet taste of the sample is). Members of the trained panel are regularly trained, as detailed in standard MSZ EN ISO 8586:2014 **[13]**. The standard includes, among other things, instructions for the continuous evaluation of the performance of the judges. On the other hand, during consumer sensory tests, lay judges with no prior training are used. During the evaluations, only opinions regarding the popularity or some other characteristics of the product are elicited, no analytical questioning is used. Results of consumer evaluations can be used to describe consumer preference, together with either the data of trained judges or the results of instrumental analyses **[4]**.

A distinct group of consumer sensory evaluations are the evaluations with optimum scales used in product development. Optimum scales are bimodal scales, which, in conjunction with the popularity scales, identify the organoleptic properties that affect popularity the most **[5]**. A number of methods for the evaluation of optimum scales are described in the literature. Examples include penalty analysis, opportunity analysis, PRIMO analysis **[15]**, bootstrapped penalty analysis **[10]**, linear regression **[3]**, chi-squared test, Spearman rank correlation, multivariate regression **[15]**, multivariate adaptive regression spline (MARS) **[18]**, partial least squares regression **[18]**, the generalization of pair correlation method **[6]** and the sum of ranking differences method **[8]**. The benefits of using optimum scales have been demonstrated in a recent publication using kefirs as an example **[7]**.

3. Materials and methods

3.1. Materials used

At the beginning of our work, a number of preliminary experiments were carried out, during which different amounts of poppy paste, and poppy seeds or poppy seed flour ground in various ways were added to commercially available milk, dark or white chocolates. Chocolate bars were evaluated by a group of 13 lay judges in a popularity test, during which mainly the preference for the poppy taste was examined, and then the results were valuated using variance analysis.

Based on the results of these preliminary experiments, the most popular white chocolate was selected, which was a product containing 29.5% cocoa butter. The samples used in the tests were produced by adding whole poppy seeds or poppy seed flour to this product.

Poppy seeds were added to the paste whole, ground and conched in amounts of 2, 4 or 6%, while poppy seed flour was added whole and conched in amounts of 6, 8 or 10%. Poppy seeds were ground using a hand grinder for 2 minutes. Conching was carried out for 24 hours in a laboratory grinder and refiner conche. Based on the results of the preliminary experiments, the samples listed in ***Table 1*** were produced.

The samples prepared were evaluated by a consumer panel of 42 judges using optimum scales. Results of the optimum scales were evaluated using the penalty analysis method in order to determine which organoleptic properties affected product popularity the most. Based on the results, the most and least preferred chocolates were selected, and it was determined the tuning of which parameters would be useful during product development.

3.2. Tools used

- Thermo Scientific HAAK Viscometer and iQ Rheometer,
- Stable Micro System (SMS) texture analyzer,
- hand-operated particle size analyzer,
- Red Jade sensory software;

3.3. Particle size determination

The particle sizes of the samples listed in ***Table 1*** and of the raw materials were determined using a hand-operated particle size analyzer. The individual raw materials were mixed in oil and applied to the surface of the probe. Particle sizes were given in micrometers.

3.4. Rheological testing of samples

White chocolate purchased as a standard and the samples were analyzed using a Thermo Scientific HAAKE-type viscometer and an iQ rheometer as functions of γ vs η and γ vs τ, and the following parameters were determined:

- apparent viscosity = Casson viscosity (Pas) Apparent viscosity of the value of shear stress extrapolated to 0 shear rate.
- viscosity (Pas)
- shear stress (Pa)
- shear rate (1/s)

Viscosity and yield point readouts were plotted against the shear rate.

3.5. Texture analysis

For the determination of the texture of the chocolates, a Stable Micro Systems Texture Analyser was used with a 2 mm diameter needle-shaped probe. 9 parallel measurements were carried out for each chocolate sample. Results of the experiment were obtained as penetration values as a function of force (maximum force (N), force required to break the chocolate layer (N), compression work (Nmm) and gradient force (N/mm). Of the results obtained, the Fmax (N) maximum force describing the hardness was found to be the most characteristic. Its values are summarized in ***Table 4***. For penetration testing, certified weights were used, but the results were given by the instrument in N, of course.

From the results obtained, mean and standard deviation values were calculated and these were compared.

The differences between the tested parameters of the samples were evaluated at a 5% significance level using variance analysis, based on the added amount and form of the poppy seeds. For the mathematical analysis of the data, the *least significant differences* (LSD) method was used, a condition for which was standard deviation homogeneity **[16]**. Sample grouping was carried out by SPSS-based *principal component analysis* (PCA) and *canonical discriminant analysis* (CDA), in order to determine the group-forming effect of the amount and particle size of the added poppy seeds and poppy seed flour.

3.6. Sensory tests

During the consumer sensory analysis of the samples, using penalty analysis, product properties were sought which affect consumer acceptance the most. With the help of the evaluation method it is possible to specify how much the individual properties, in this case the intensity values of poppy seeds/poppy seed flour differ from the optimum for consumers. Sensory tests were carried out in the Sensory Analysis Laboratory of the Faculty of Food Science of Szent István University using the Red Jade® sensory software. Samples were encoded with 4-digit random numbers so that the judges could not identify the individual chocolates. Neutral tasting non-carbonated mineral water was provided to the judges during the evaluation for taste neutralization. The evaluation room complied with the prescriptions of standard MSZ ISO 8589:2015 **[14]**. 42 consumer judges participated in the sensory evaluation. During the evaluation, optimum scales and a popularity scale were used to determine which product properties affected popularity significantly.

4. Results

4.1. Particle size determination

Figure 1 and ***Figure 2*** show the particle sizes of chocolates with poppy seeds and poppy seed flour, respectively, based on which it can be stated that in the case of samples containing either poppy seeds or poppy seed flour, conching resulted in significant particle size reduction , compared to the product containing whole poppy seeds. In the case of samples containing poppy seeds, grinding also resulted in particle size reduction.

In the case of samples mixed with poppy seeds, particle size increased with increasing amounts of whole or ground poppy seeds. No significant increase was observed with increasing the amount of poppy seeds in the case of co-grinding. For the samples containing poppy seed flour, no particle size increase was observed either in the case of samples containing whole flour or conched samples, with increasing amounts of added flour.

4.2. Rheological analysis of the samples

In order to determine the rheological properties, both qualitative and quantitative evaluations were performed. Qualitative results are shown on the viscosity curves, while quantitative results are presented on the flow curves.

4.2.1. Viscosity results

Figure 3 shows the viscosity curves of chocolates mixed with poppy seeds. Based on the curves it can be stated that the highest viscosity values were measured at the initial shear stress, and the viscosity values decreased with increasing shear rates. Based on these, the chocolate samples exhibit a thinning characteristic as a result of shearing. In the case of samples with poppy seeds, viscosity curves showed viscosity values of around 2 Pas.

Figure 4 shows the viscosity curves of the samples containing poppy seed flour, also exhibiting the thinning characteristic as a result of shearing, similarly to samples mixed with poppy seeds. Higher shear rate curves of the samples with poppy seed flour, as opposed to samples with poppy seeds, showed a less pronounced tendency and had viscosity values around 5 Pas.

4.2.2. Shear stress results

Flow curves were similar to viscosity curves, but shear stress values increased with increasing rates, significant differences were found especially in the case of samples with poppy seed flour.

The Casson model, most widely used in industrial practice, was fitted to the results with a correlation coefficient of $r > 0.9$, which can be considered as accurate. From the flow curve, τ_0 (yield point) and η_∞ (apparent viscosity) were calculated.

4.2.3. Apparent viscosity results

Based on ***Table 2*** it can be stated that, in the case of samples prepared with whole poppy seeds, apparent viscosity values decreased as a result of co-grinding, while an increase in viscosity was caused by conching, compared to samples with added whole poppy seed flour. In the case of samples with ground poppy seeds, no trend in viscosity values could be observed.

Based on the results of variance analysis, there was a significant difference between samples prepared with poppy seeds. Apparent viscosity has a role in material transport, it is an important factor in adding the proper amounts of chocolate.

4.2.4. Yield point measurement results

Yield points increased in the case of both samples with poppy seeds and poppy seed flour, compared to the control white chocolate. This increase, as a result of conching, was smaller in the case of samples containing whole poppy seeds, and larger in the case of samples with poppy seed flour. In the case of samples with ground poppy seeds, no trend in yield point values could be observed. Based on the results of variance analysis, a significant difference was found between samples prepared with poppy seeds. (***Table 3***)

4.3. Results of texture analysis

Generally, it can be stated that the same phenomenon was supported by all 4 factors investigated during texture analysis, therefore, the modifying effect on the structure of chocolate is presented using the most widely accepted Fmax results.

Based on the maximum force values shown in ***Table 4*** it can be stated that the texture of the chocolate sample containing 2% whole poppy seeds was identical to that of the control sample. Analytical results of all the other samples showed higher values compared to white chocolate, so the texture of the products became harder as a result of the added poppy products. The increase was smaller compared to the sample containing whole poppy seeds, and larger compared to the sample containing whole poppy seed flour, as a result of conching. In the case of samples with ground poppy seeds, no clear trend could be observed during the texture analysis. Based on the results of variance analysis, significant differences were observed between the samples prepared with poppy seed flour for all four parameters investigated.

4.4. Results of the discriminant analysis

In order to be able to interpret the rheological and textural properties of the experimental samples together, multivariate data processing (Principal Component Analysis, PCA) was also carried out. Contrary to variance analysis, groups were formed by taking into account non-significant samples, so that the total standard deviation squares of the results were broken down into factors with decreasing weights. In this way, a more clear picture of the separation of the samples could be obtained, compared to variance analysis.

In ***Figure 5***, samples are positioned as a function of the first two factors. The first principal component describes 77%, samples are arranged and separated from the control according to the quantities added.

The second principal component, describing 19% of the total variance, can be correlated to the particle size.

Figure 6 shows the results of canonical discriminant analysis, which is similar to the results of principal component analysis. In the figure, samples are positioned in the system of two grouping functions. It can be stated that the first grouping function is in accordance with the amount of poppy seeds added, while the other one is in accordance with poppy seed particle size.

Overall, multivariate analysis showed that not only the particle size of the added poppy seeds, but also the amount of added poppy seeds has a grouping nature.

4.5. Results of the sensory tests

In the preference map shown in ***Figure 7***, the set of experimental samples is divided into four parts. The quarter marked with a 1 in the upper right hand corner designates the group of most favored samples, 2 designates less favored, 3 the even less favored and 4 the least favored samples.

Based on the preference map drawn from the results of consumer sensory tests it can be stated that the samples most preferred by the 42-person judging panel were the ones prepared with 2% or 4% whole or ground poppy seeds, while the one with 10% poppy seed flour was the least favored sample.

Our results showed that samples containing whole poppy seeds in smaller amounts were found to be most acceptable by consumers. In our opinion, these samples can serve as models for further product development. In quarter 2 of the preference map, conched samples were separated, regardless of the quantity and type of the poppy seed product. In popularity tests, this is the set of second most popular products, also in terms of product development. Quarter 3 contains products enriched with larger amounts of poppy seeds or poppy seed flour, either whole or in the ground form. Quarter 4 contains the sample with 10% whole poppy seed flour, which was least accepted by consumers.

Preparation of the most organoleptically accepted product requires additional sensory tests. Based on our results so far, samples added with whole poppy seed products and having lower poppy seed contents were most preferred by consumers. This result should be taken into consideration when planning further product development.

5. Conclusions

During grinding, two opposite effects are present together in the technology, that is, as the oil content is removed, the continuous phase of the chocolate increases, facilitating the sliding of the layers on each other, while grinding increases the number of particles per given volume, making the movement of the layers more difficult.

The above-mentioned effects prevail to different extents:

- significant differences are caused in the characteristics obtained by rotational viscometry in the case of poppy seed containing samples with higher oil content,
- and in the penetration effect in the case of poppy seed flour containing samples with lower oil content.

Among the characteristics of the samples, there was a significant difference in the average particle sizes of pastes prepared both with poppy seeds and poppy seed flour. Based on the results of PCA and CDA it can be stated that in the grouping of chocolates containing poppy seeds/poppy seed flour the main factor was the form of the poppy seeds/poppy seed flour, and the second factor was the effect of the amount.

Based on our results it can be stated that the effect of none of the factors investigated by us should be overlooked in a given technological design because, based on the analyses presented, the results obtained by revealing the two effects contribute to the determination of the optimal rheological properties of chocolates together.

As far as technological use is concerned, it can be concluded from the results that high oil content samples mixed with poppy seeds are suitable for producing more plastic materials that are easier to measure out and spread, such as stuffed bonbons.

Lower oil content samples with poppy seed flour are more suitable for the preparation of crunchier texture, harder and more solid samples, as opposed to bar products.

According to our test results, the behavior of the system was most predictable during co-grinding. Knowing this could be helpful in technological design aimed at improving product manufacture.

Based on the consumer sensory test, preparation of organoleptically accepted products should be based on samples containing whole poppy seeds in smaller amounts, but to determine optimal characteristics, further sensory tests are required.

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