# A Rheological Study of Lemon Curd Made of Frozen-thawed Liquid Egg Yolk

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Abstract—In this study, we examined the industrial usability of frozen and thawed liquid egg yolk (LEY) by preparing lemon curd samples. Therefore, LEY frozen at-18 °C was used to make lemon curd samples. LEY was stored for 90 days at -18 °C and it was thawed with two different methods (in 2 hours, 35 °C and in 24 hours, 5 °C) on measurement days (day 1, 7, 14, 30, 60 and 90). After thawing of LEY samples, lemon curd samples were made by adding liquid egg white, butter, lemon juice and sugar. Ingredients were mixed by constant whisking over the steam of hot water. After that, butter was also added to the cream and it was cooled to 20 °C. Rheological parameters of the lemon curd samples were examined with rotational rheometer. Shear stress data were recorded by increasing, than by decreasing shear rate values in the range of 10-1000 1/s. Herschel-Bulkley model was fitted to the flow curves of decreasing shear rate. Based on our measurements, we found that the thawing method of frozen liquid egg yolk does not affect the rheological properties of lemon curd made of them, but the length of the frozen storage does.

*Index Terms*—rheological properties, lemon curd, freezing, liquid egg yolk, thawing method, flow curve

## I. INTRODUCTION

Egg is a popular ingredient in many branches of the food industry. Besides its excellent nutrition content, egg has numerous prosperous functional properties. In the last few decades, processed egg products, such as liquid egg products are marketed in huge amounts for industrial purposes. These products are made by the separation, homogenization and pasteurization of hen eggs.

Liquid egg yolk (LEY) is sought ingredient in the food industry because of its excellent gelling, emulsifying, colouring and coagulating properties, high nutritional value and unique sensory characteristics. It is often used in confectionery products such as lemon curd, ice cream, cr éme brul ée and pudding [1], [2].

Because its excellent nutritive values, LEY serves as an excellent medium for microorganisms. Its shelf life is limited to only a few weeks [3]. Pasteurization is used in general to decrease the number of microorganisms and to increase the shelf life of these products. Because of the sensitivity of LEY compound, especially the proteins, a relatively mild time-temperature combination can be used. Heat processing above  $60 \,^{\circ}$ C leads to transitions that cause changes in the microstructure, appearance, in the calorimetric and rheological properties of egg yolk [4].

By freezing and frozen storage, shelf life of different liquid egg products can even reach 1 year. By determination of the right freezing procedure, minimal changes can be achieved for whole egg and protein juice. However, egg yolk undergoes an irreversible fluid loss, when it is frozen at  $-6 \ C$  or a lower temperature. This leads also to an unwanted phenomenon, a gelation process, which affects the functionality of egg yolk. In terms of freezing, the egg yolk has a much more complex texture than other foods. It contains particles of different stability and solubility in the form of protein solutions. Unstable lipoprotein micelles are damaged by freezing and thawing and lipoproteins dissolve and denature under the process. [5]

Freezing of liquid egg products is a time-temperaturerelated process [6]. By increased freezing and thawing rates, smaller ice crystals are formed, that results in lower drip loss and better texture. In addition, proteins are less dehydrated [7], [8] Due to the gelation, the usability of frozen and thawed egg yolk changes.

Lemon curd belongs to the group of traditional English spreads. Fruit curds are made by the addition of fruit juice, butter, egg, sugar and flavouring. Each ingredient has its special role in the texture and flavor of the product [9]. Homemade lemon curd can be stored for one week in the refrigerator [10].

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Lemon curd has a unique texture. The rheological behaviour of this cream was examined by several authors [11], [12]. Lemon curd produced industrially was examined between planar and spherical surfaces. The authors stated that its behaviour can be described by Herschel-Bulkley equation [13].

In our study, we examine the rheological characteristics of homemade lemon curd samples. We examine the effect of using frozen-thawed LEY to the lemon curd on the rheological properties. Effect of frozen storage and thawing method will be discussed, as well. Frozen storage of LEY of 90 days is performed.

#### II. MATERIALS AND METHODS

# A. Materials

In this study, raw egg yolk was used, which was provided by Capriovus Ltd. (Szigetcs  $\phi$ , Hungary). It was produced by the separation and homogenization of fresh hen eggs. LEY was cooled to  $3 \,^{\circ}$ C and packaged in PET bottles under industrial circumstances. Besides that, freshly made liquid egg white was also provided by Capriovus Ltd. The rest of the ingredients of the lemon curd were butter, sugar and, which were bought commercially.

# B. Freezing and Frozen Storage of Liquid Egg Yolk

The PET bottles of LEY were opened sterile and their content was dosed into sterile polypropylene sample containers of 100.0 ml volume. Sample containers were put into laboratory freezer, where samples were frozen at -18 °C. A three months long storage experiment was carried out. Days of sampling were the following: day 1, 7, 14, 30, 60 and 90. On the measurement days, two sample containers were taken out of the freezer. One sample was thawed in tap water in two hours, one of them in a laboratory refrigerator at 5 °C in 24 hours.

#### C. Preparation of the Lemon Curd

As a first step, we prepared the ingredients, which included squeezing the lemon, warming the butter to room temperature and weighing the ingredients. 15 g of thawed LEY, 28.5 g of LEW, 37.5 g sugar and 20 g freshly pressed lemon juice were pre-measured and mixed in a metal mixing bowl.

Two cups of water were poured into a metal saucepan and it was heated. When water began to boil, gas burner was switched the to the lowest level and the mixing bowl containing the ingredients placed over the saucepan and the mixture was cooked for three minutes. Meanwhile, it was constantly stirred with a metal mixer. After removing the bowl from the steam, 14 g of butter was added to the mixture and the lemon curd was blended. Thereafter, lemon curd was cooled to  $20 \,^{\circ}$  in ice water. The addition of lemon zest was neglected because rheological parameters would have been difficult to measure with solid pieces.

# D. Examination of the Rheological Properties

Rheological parameters were examined by MCR 92 rheometer (Anton Paar, Les Ulis, France) by using a

concentric cylinder measuring head in rotational mode. Properties of the probe are the following: cup diameter 28.920 mm, bob diameter 26.651 mm, bob length 40.003 mm, active length 120.2 mm, positioning length 72.5 mm.

Anton Paar RheoCompass software was used to control the equipment and carry out the measurements. The set temperature during the measurements was 20 °C. Shear rate was increased from 10 to 1000 1/s, then decreased from 1000 to 10 1/s. Three parallels were measured in each case. As control sample, lemon curd made by adding raw liquid egg yolk was examined.

Slowing phase of the flow curves were evaluated by fitting the rheological model of Herschel-Bulkley (1) to shear rate-shear stress diagrams [13]:

$$\tau = \tau_0 + K \left(\frac{d\gamma}{dt}\right)^n \tag{1}$$

where  $\tau$  is the shear stress [Pa],  $\tau 0$  is the yield stress [Pa],  $\gamma$  is the shear rate [1/s], K is the consistency coefficient [Pa·s<sup>n</sup>] and n is the flow behaviour index (dimensionsless).

Fitting of Herschel-Bulkley model was performed by using the least square fit method of Excel Solver, where  $\tau_0$ , K and n were the changeable values. To check proper fitting, correlation coefficient (R<sup>2</sup>) was calculated.

## E. Statistical Evaluation

Statistical analysis was carried out using IBM Statistics 24 software. The significance level was 5% (p<0.05) Kolmogorov-Smirnov test was used to examine the normality of the error terms. Levene's test was used to determine whether variances are equal or not. Statistical analysis of variance was performed using two-way ANOVA test. In case of significant ANOVA test, post hoc test were used to decide which groups differ. In case of equal variances, Tukey-test was used. If variances were assumed not equal, Games-Howell test was used in case of not equal variances.

#### III. RESULTS

# A. Examination of the Rheological Behavior of the Lemon Cream

Two selected flow curves of lemon curd samples can be seen in Fig. 1. One is the flow curve of the control sample, which was made by adding raw egg yolk. The flow curve belongs to lemon curd made by adding LEY, which was stored in the freezer for 14 days and thawed in refrigerator 24 hours long.

The lemon curd samples tested showed in all cases non-Newtonian rheological behaviour, since their flow curves are not linear. All of the examined flow curve has a yield point and the shear stress decreases with increasing shear rate less and less. When the applied shear stress is bigger than the yield stress, lemon curd begins to flow. Herschel-Bulkley model fitted the flow curves well,  $R^2$  was bigger than 0.99 in each cases. Different high-fat foods, like margarine, butter and fats show such rheological behaviour. Lemon curd also contains a high amount of fat in form of butter and LEY. Previous studies also found, that Herschel-Bukley model describes the rheological behaviour of lemon curd the most properly [11], [12], [14]. A hysteresis loop can be seen on the examined samples flow curves, which means that the samples undergo structural transformation due to shear. Besides that, difference between the first and the last measuring point increases by increasing the storage time.

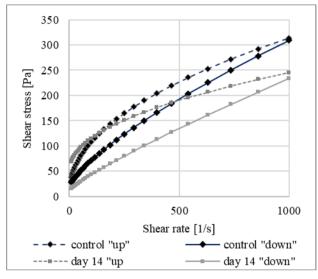
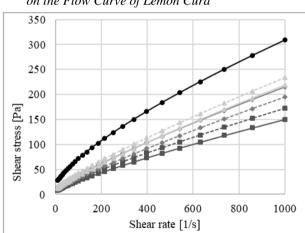


Figure 1. Examples for the hysteresis of lemon curd samples at 20  $^{\circ}$ C (control sample the sample made of liquid egg yolk frozen for 14 days and thawed fast (2 hours, 35  $^{\circ}$ C)



B. Effect of Freezing and Frozen Storage of Egg Yolk on the Flow Curve of Lemon Curd

Figure 2. Effect of length of frozen storage (-18 °C) and thawing methods (F-2 h, 35 °C, S-24 h, 5 °C) of the egg yolk on the shear stress-shear rate relationship (flow curve) of lemon curd at 20 °C in the first 14 days of the storage experiment

day 1 F

day 7 S

day 1 S

day 14 F

Fig. 2 and Fig. 3 show the phase with decreasing shear rate of the flow curves of the examined lemon curd samples. Fig. 2 shows that there is a large difference in the shape of the flow curves on the first three measurement days compared to the control sample. Lemon curd samples of these days are less viscous, they possess lower yield stress values and a lower shear stress appears by the same shear rate values compared to the control sample. From the first month on (Fig. 3), the flow curves of the prepared lemon curds are much more similar to the control sample.

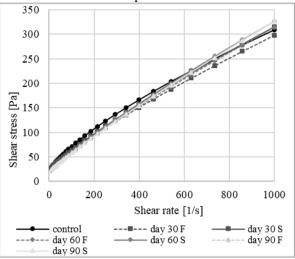


Figure 3. Effect of length of frozen storage (-18  $^{\circ}$ C) and thawing methods (F-2 h, 35  $^{\circ}$ C, S-24 h, 5  $^{\circ}$ C) of the egg yolk on the shear stress-shear rate relationship (flow curve) of lemon curd at 20  $^{\circ}$ C from day 30 to day 90

# C. Effect of Freezing and Frozen Storage of Egg Yolk on the Rheological Properties of Lemon Curd

Fig. 4, Fig. 5 and Fig. 6 show the constants of the Herschel-Bulkley model fitted to the flow curves.

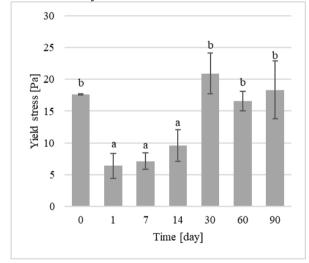


Figure 4. Yield stress values of the lemon curd samples on measurement days calculated by Herschel-Bulkley model (values with different letters are significantly different at P<0.05)

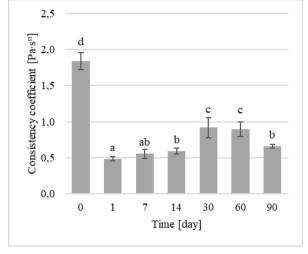
Yield point values varied in the range of 5 to 23 Pa. Scientists in earlier studies determined bigger yield stress values [11], [12], [14]. One of the reasons for the difference may be that they examined lemon curd produced industrially. Besides that, they had completely different values in the range of 77 to 135 Pa.

From the first month, yield point values are similar to the control sample. The reason of this can be the regeneration of LEY during the frozen storage. In contrast, the tendency for the consistency coefficient changed during the experiment and the flow behaviour index was higher than that of the control sample in each

control

day 7 F

day 14 S



case. A big difference in these parameters can be seen in comparison to the above-mentioned studies, as well.

Figure 5. Consistency coefficient values of the lemon curd samples on measurement days calculated by Herschel-Bulkley model (values with different letters are significantly different at P<0.05)

The method of thawing of the LEY samples had no significant effect on the rheological properties of lemon curd samples at p<0.05. The combined effect of the way of thawing and storage time was not significant in neither case, so in the following we examined the effect of the storage time. The length of frozen storage had significant effect on each rheological parameter at p<0.05. Multiple comparison was performed by Games-Howell test in case of the yield stress, Tukey test was used to analyze consistency coefficient and flow behaviour index data. Results of the post hoc test are shown in Fig. 4, Fig. 5 and Fig. 6.

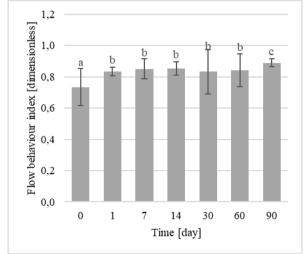


Figure 6. Flow behaviour index values of the lemon curd samples on measurement days calculated by Herschel-Bulkley model (values with different letters are significantly different at P<0.05)

Based on the statistical analysis, yield stress data belong to two groups. Values on days 1, 7 and 14 differed significantly from the yield stress values of the control sample and samples on days 30, 60 and 90. It has been shown above that there is a large difference in the rheological behavior for 1-14 days of the experiment compared to the control sample, and then it is reduced from the 30th day. The biggest consistency coefficient can be seen in case of the control sample. First, values decrease significantly, then they increase under frozen storage, but values do not reach the control sample. For the flow behaviour index, we can see that the control sample is significantly different from the frozen and then thawed samples.

# IV. CONCLUSION

In this study, we examined the rheological properties of homemade lemon curd samples. We applied frozen LEY and we investigated how the freezing and frozen storage affects the rheological characteristics of the samples. Besides that, LEY samples were thawed by two different thawing methods, a slower (2 hours, 35 °C) and a faster (24 hours, 5 °C). It was found that the rheological properties of lemon curd can be described by the Herschel-Bulkley equation. We stated, that the method of thawing does not affect the rheological parameters of the lemon curd samples. However, freezing and frozen storage caused great changes in all three rheological parameters. The change decreased after a month of frozen storage.

In our next experiment, we add sugar as cryoprotectant to LEY before freezing. The protective role of sugar is based on the fact that it prevents the structural transformation and gelling of egg yolk.

#### CONFLICT OF INTEREST

The authors declare no conflict of interest.

#### AUTHOR CONTRIBUTIONS

K.I.H. conducted the research; A.V. and A.B. analyzed the data; K.I.H and I.G. wrote the paper; I.Cs.Ny-Z, L.F. and Cs.N. corrected the conclusions and the paper and all authors had approved the final version.

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