

# Influence of Food Composition on Freezing Time

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## ABSTRACT

Freezing is still the best way to preserve food when is carried out properly and allows production of food without any chemical preservatives. It is important to accurately predict the freezing time of different kinds of food to assess the quality and safety of the foodstuffs, processing requirements, and economical aspects of food freezing. The aim of this research was to investigate the influence of food product composition on freezing time. The research based on hypothesis: the composition of food product has significant effect on freezing time considering that the products have the same shape, mass and relief. In our research we recorded four batches of standard product with three different fillings (chocolate, vanilla and forest fruit) at the same freezing conditions. The products from puff pastry were always made from the same ingredients and also mass, shape, relief, slab and surface were constant. The core temperature after 41 minutes of freezing at air temperature  $-32\text{ °C} \pm 2\text{ °C}$  and constant velocity reached in chocolate product  $-24.4\text{ °C} \pm 0.2\text{ °C}$ , meanwhile in forest fruit  $-17.0\text{ °C} \pm 0.2\text{ °C}$  and vanilla only  $-12.8\text{ °C} \pm 0.2\text{ °C}$ ; thus confirmed our hypothesis. For SMEs (small and medium size food enterprisers) which represent 99 % of all enterprises in the EU, it is essential to establish their own model for predicting freezing time which is more applicable that general theoretical models that require a lot of data which are not common accessible for SMEs.

## KEY WORDS:

Freezing, Food composition, Freezing time, Freezing point, Food quality, Food safety.

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## INTRODUCTION

Like most food preservation methods, the freezing process for food has evolved over a significant period of time. As an extension of cooling, the primary goal of food freezing has been the reduction of microbial growth and/or control of reactions causing spoilage of the food, as well as the corresponding extension of product shelf-life. In addition, the process has extended the availability of many food commodities for consumption at any time during the year. Frozen food can be transported for longer distances and the process has contributed to making foods available on a worldwide basis. For most applications, the food products are held in storage for some period of time before thawed, and prepared for consumption. More recently, the process has evolved to include a variety of products that are consumed in a frozen state [1]. The principal objective of the new general and specific hygiene rules is to ensure a high level of consumer protection with regard to food safety. Food business operators shall ensure that all stages of production, processing and distribution of food under their control, satisfy the relevant hygiene requirements, laid down in Regulation (EC) No. 853/2004. This regulation emphasizes the importance of cold chain throughout HACCP (Hazard Analysis and Critical Control Point) system for all foodstuff that require cooling or freezing regime [2]. Kassianenko and co-workers [3] emphasize the importance of cold chain, which is a vital part of modern global trade as it has impact on all food commodities. In today's modern society, refrigerated storage is one of the most widely practiced methods of preserving perishable food. Improper use of this process increases the potential risk and microbial hazards will advance, thus leading to the food borne illnesses.

The purposes of food freezing is preservation of food, reducing the activity of enzymes and microorganisms, reducing the amount of liquid water for microbial growth and reducing water activity of foods [2,4,5,6]. Freezing is still the best way to preserve food when is carried out properly and allows production of food without any chemical preservatives. Thermo physical properties of frozen food are used to estimate the rate of heat transfer and to calculate the heat load in process such as freezing and thawing [7]. The early calculations and analyses associated with freezing and thawing, primary used constant and uniform thermo physical properties [8]. The calculations and analyses were typically oversimplified and inaccurate. Numerical analyses such as finite difference methods were used widely to analyze thermal food processes [9]. The large number of food products, available today, create a great demand for knowledge on thermo physical properties. Since foodstuff are composite materials, the thermo physical properties are clearly a function of the components [10,11]. The core temperature for frozen food are prescribed by the Regulation upon safety of frozen food, 609/2002 [12] (the Council directive of 21 December 1988 on the approximation of the laws of the Member States relating to quick-frozen foodstuffs for human consumption (89/108/EEC)) and so it is an obligation to release the products on market with proper core temperature which has to be below  $-18\text{ }^{\circ}\text{C}$ . Nevertheless the monitoring of tempera-

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It is important to accurately predict the freezing time of foods to assess the quality and safety, processing requirements, and economical aspects of food freezing.

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ture during the transport is regulated with the Commission Regulation (EC) No. 37/2005 [13] of 12 January 2005 on the monitoring of temperatures in the means of transport; warehousing and storage of quick-frozen foodstuffs intended for human consumption and allowed short time deviation of temperature should not exceed 3 °C.

It is important to accurately predict the freezing time of foods to assess the quality and safety, processing requirements, and economical aspects of food freezing [14]. But freezing process has a problem with moving boundaries [15]. Food, undergoing freezing, release latent heat over a range of temperatures. Freezing does not occur at unique temperature. In addition, food do not have constant thermal properties during freezing. As result, no exact mathematical model exists for predicting the freezing time of foods [16,17].

As proposed by Cleland and co-workers in successive papers [4], one of the ways to determine the freezing time of a multi-dimensional product is to know both the freezing time of a slab under the same operating conditions and a geometric shape factor [18]. Authors initially noted such a factor, such as EHTD (the equivalent heat transfer dimensionality). Therefore, the freezing time of a food of any shape can be calculated by quite a lot of different equations and are quite difficult for use in industry because of the wide range of variables. The experimental or testing method which has a lot of process traps in often used in practice. The most often mistake is overlaying the parameters to other similar products (mass, shape, relief, slab, etc.). The freezing time requirement for each entity is estimated by the simulation model using the Plank's equation. Plank developed an equation, based on the unique phase change model, for estimating freezing time for different geometrical shapes, and allowing for varying film coefficients. The equation, derived from one-dimensional infinite slab geometry and has been analytically extended for infinite cylinders, spheres and for finite parallelepiped geometries. Plank's equation is common worldwide used and can be expressed as follows [19], equation 1. Where the  $t_f$  is time required for freezing (in min),  $\rho$  product density,  $\Delta H$  latent heat of freezing,  $R$  characteristic half thickness of the food object,  $h$ ,  $k_f$  are heat transfer coefficient and thermal conductivity (before freezing) respectively  $\theta_{if}$ ,  $\theta_a$  denote the freezing point and ambient temperatures.

$$t_f = \frac{\rho \cdot \Delta H}{\theta_{if} - \theta_a} \left( \frac{R}{h} + \frac{R^2}{2k_f} \right) \quad (1)$$

$t_f$	time for freezing (min)
$\rho$	product density (kg/m <sup>3</sup> )
$\Delta H$	latent heat of freezing (kJ/kg)
$R$	half thickness of the food object (m)
$h$ , $k_f$	heat transfer coefficient (W/(m <sup>2</sup> ·C))
$\theta_{if}$ , $\theta_a$	thermal conductivity (W/(m·C))

Carefully planned and efficiently provided education of employees is of the great importance. Lectures should be carried out by competent ex-

perts with relevant experience, who will provide specific information to every employee according to their assigned task. Employees should be personally responsible for their work [20]. The consumer is also a source of hazard for itself and that should be incorporated in the risk assessment. The results from research carried out by Ovca and Jevšnik [21] confirmed that the term “cold chain” is not well known among consumers. They also confirmed that Slovenian and European consumers place the responsibility of maintaining a cold chain into other parts of the food chain. One of the reasons for poor knowledge of the cold chain could be due to the unsatisfactory efforts of governmental and non-governmental organizations, which are responsible for educating and informing consumers.

The role of the consumer in maintaining a cold chain is more important than is currently thought, and is greater than the importance attached to other parts of the food chain.

## MATERIALS AND METHODS

Samples were prepared as part of daily production on continuous line at food enterpriser according to its production capacity. The product core temperature and time were measured during shock freezing using RT-DTEMP101; SN: M32551, for the range from -100 °C to +600 °C (MadgeTech, Contocook, New Hampshire, USA), calibrated with measurement error 0.2 °C and evaluated using program MadgeTech Data Recorder Software, version 2.00.63. with the statistical parameters included. We recorded core temperature every 6 seconds and thus we got for each measure 410 thermal points for describing the decrease of temperature in core of product. Water activity was measure using Testo 650 with electrode 0628-0044 (910) at 25 °C, meanwhile the compositions were analyzed in Slovenian National Public Health Institute. The results of analyses are stated in Table 1. The puff pastry was always made from the same ingredients; also mass, shape, relief and surface were constant. Times of freezing, air temperature, air speed in continuous shock freezer were also constant and were not changed during the process. Freezing medium was always cooled air with temperature of  $-32.0\text{ °C} \pm 2.0\text{ °C}$ , constant velocity and time of freezing for 41 minutes. Selected fills were vanilla, chocolate and forest fruits. Each piece of product was weighted on continuous packaging equipment ( $n = 9000$ ;  $100.00\text{ g} \pm 2.8\text{ g}$ ). We measured the temperature of each type of product in for parallel batch at the same conditions: (vanilla,  $n = 4$ ; chocolate,  $n = 4$ ; forest fruits,  $n = 4$ ). Predicted freezing time for mass 100 g was 40 min – 45 min at  $-32.0\text{ °C} \pm 2.0\text{ °C}$ , constant velocity, as stated in producer’s instructions. The time of freezing is managed by belt speed. At one time in freezer were 820 kg of product or 8,200 pieces. The results of core temperature after 41 minutes of freezing were processed by the XLSTAT 2009.6.01 Comparison of samples using nonparametric test, multiple pair wise comparisons using the Nemenyi’s procedure/Two-tailed test with significance level 5 %.

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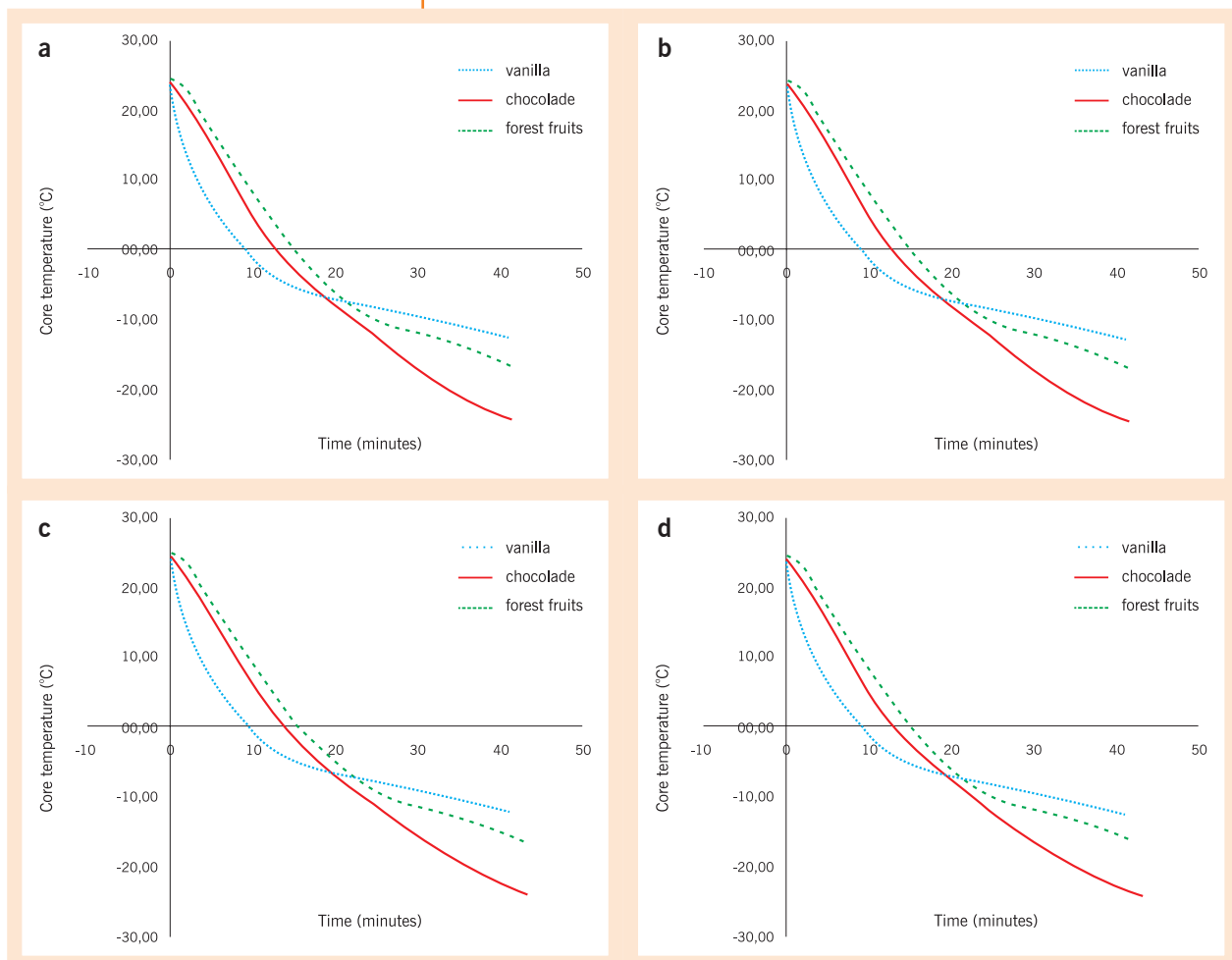
## RESULTS AND DISCUSSION

According to our four batches we recorded core temperature every 6 seconds and thus we got for each measure 410 thermal points for describing the decrease of temperature in core of product. When we compare the data between four parallels within one type of product we ascertain no statistical difference ( $p < 0.001$ ). Results from recorded data for all products are stated on the Figures 1 from a to d. Within one type of product there is no difference within type of product or according of the used fill. As Figure 1 shows that the time of freezing was constant 41 minutes and at this time only one product (chocolate filling) reached the temperature below  $-18\text{ °C}$  and the core temperature after 41 minutes of freezing reached  $-24.4\text{ °C} \pm 0.2\text{ °C}$ . The core temperature for product with forest fruit filling almost reached the required temperature ( $-18\text{ °C}$ ) but nevertheless it was still with its  $-17.0\text{ °C} \pm 0.2\text{ °C}$  more than  $1.0\text{ °C}$  to high. The highest core temperature we measured in product with vanilla filling and it was  $-12.8\text{ °C} \pm 0.2\text{ °C}$  after 41 minutes of freezing. Summary data of four investigated batches are shown in the Table 2. Reason for such differences between similar products is in the specific heat and enthalpy. In general we can conclude that the food with higher content of water have higher specific heat and higher enthalpy (at  $0\text{ °C}$ ) thus need longer freezing time compare to similar prod-

**Figure 1.**

Core temperature in correlation to time of freezing for four batches.

**a.** the first batch; **b.** the second batch;  
**c.** the third batch; **d.** the fourth batch.



**Table 1:**

Composition of food product with three different types of filling.

	C filling	V filling	FF filling	C	V	FF
Proteins	8.15	1.12	1.79	6.17	5.73	5.11
Carbohydrates	56.21	30.86	51.86	45.02	32.89	44.28
Fat	33.52	1.68	1.74	27.24	12.96	18.19
Water	1.08	65.32	43.56	20.5	47.45	31.4
Ash	1.04	1.02	1.05	1.07	0.97	1.02
$a_w$ (25 °C)	0.394	0.972	0.946	0.879	0.952	0.917

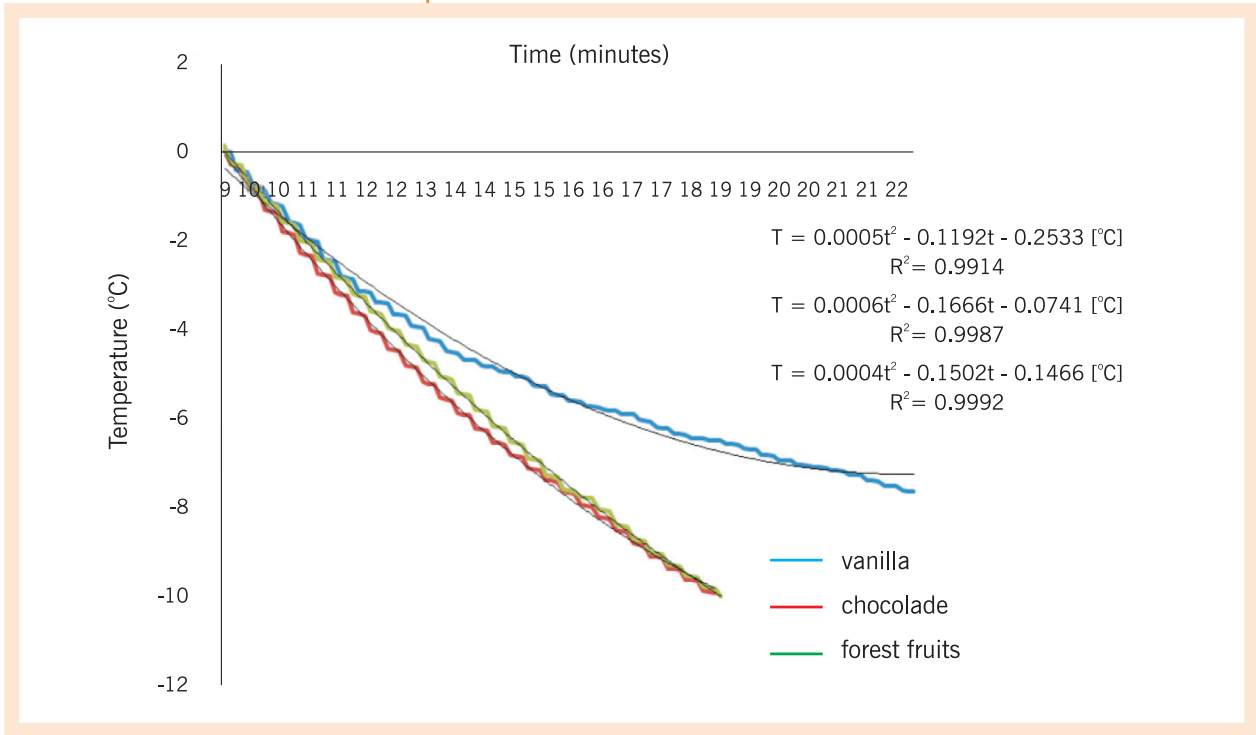
C, V, FF with postscript "filling" are data for pure fill, V: vanilla product, C: chocolate product, FF: forest fruit product,  $a_w$  water activity.

**Table 2:**

Summary of recorded core temperatures in food product with three different types of filling.

t (min.) product		0	5	10	15	20	25	30	35	40
1 <sup>st</sup>	V	23.4	15.9	-1.2	-5.1	-7.1	-8.4	-9.5	-11.2	-12.8
	C	23.9	15.9	4.8	-3.2	-8.3	-12.6	-16.5	-21.1	-24.3
	FF	24.4	15.9	7.5	-0.6	-6.2	-10.4	-11.8	-13.7	-16.9
2 <sup>nd</sup>	V	23.9	15.9	-1.3	-5.3	-7.1	-8.5	-9.5	-11.2	-12.7
	C	23.9	15.9	4.7	-3.4	-8.4	-12.5	-16.6	-21.2	-24.3
	FF	24.4	15.9	7.4	-0.5	-6.3	-10.4	-11.8	-13.9	-16.9
3 <sup>th</sup>	V	23.4	15.9	-1.4	-5.4	-7.0	-8.4	-9.6	-11.2	-12.8
	C	24.1	15.9	4.5	-3.7	-8.3	-12.6	-16.7	-21.3	-24.3
	FF	24.4	15.9	7.3	-0.5	-6.4	-10.4	-11.8	-13.8	-16.9
4 <sup>th</sup>	V	24.1	15.9	-1.4	-5.5	-7.1	-8.5	-9.6	-11.2	-12.8
	C	24.0	15.9	4.4	-3.5	-8.4	-12.7	-16.7	-21.3	-24.4
	FF	24.4	15.9	7.2	-0.7	-6.5	-10.5	-11.9	-13.9	-17.0
$\bar{x}$	V	24.1 <sup>a</sup>	15.9 <sup>a</sup>	-1.4 <sup>c</sup>	-5.5 <sup>c</sup>	-7.1 <sup>b</sup>	-8.5 <sup>a</sup>	-9.6 <sup>a</sup>	-11.2 <sup>a</sup>	-12.8 <sup>a</sup>
	C	24.0 <sup>a</sup>	15.9 <sup>a</sup>	4.4 <sup>b</sup>	-3.5 <sup>b</sup>	-8.4 <sup>c</sup>	-12.7 <sup>c</sup>	-16.7 <sup>c</sup>	-21.3 <sup>c</sup>	-24.4 <sup>c</sup>
	FF	24.4 <sup>a</sup>	15.9 <sup>a</sup>	7.2 <sup>a</sup>	-0.7 <sup>a</sup>	-6.5 <sup>a</sup>	-10.5 <sup>b</sup>	-11.9 <sup>b</sup>	-13.9 <sup>b</sup>	-17.0 <sup>b</sup>
$p$ -value	$p > 0.05$	$p > 0.05$	$p \leq 0.001$	$p \leq 0.001$	$p \leq 0.001$	$p \leq 0.001$	$p \leq 0.001$	$p \leq 0.001$	$p \leq 0.001$	$p \leq 0.001$

t: time of freezing in minutes, V: vanilla product, C: chocolate product, FF: forest fruit product,  $\bar{x}$ : average values for four batches, Significant at  $p \leq 0.001$ ; significant at  $p \leq 0.01$ ; significant at  $p \leq 0.05$ ;  $p > 0.05$ , not significant, Values followed by a different letter are significantly different along the column for the Duncan (0.05) test.



**Figure 2.**

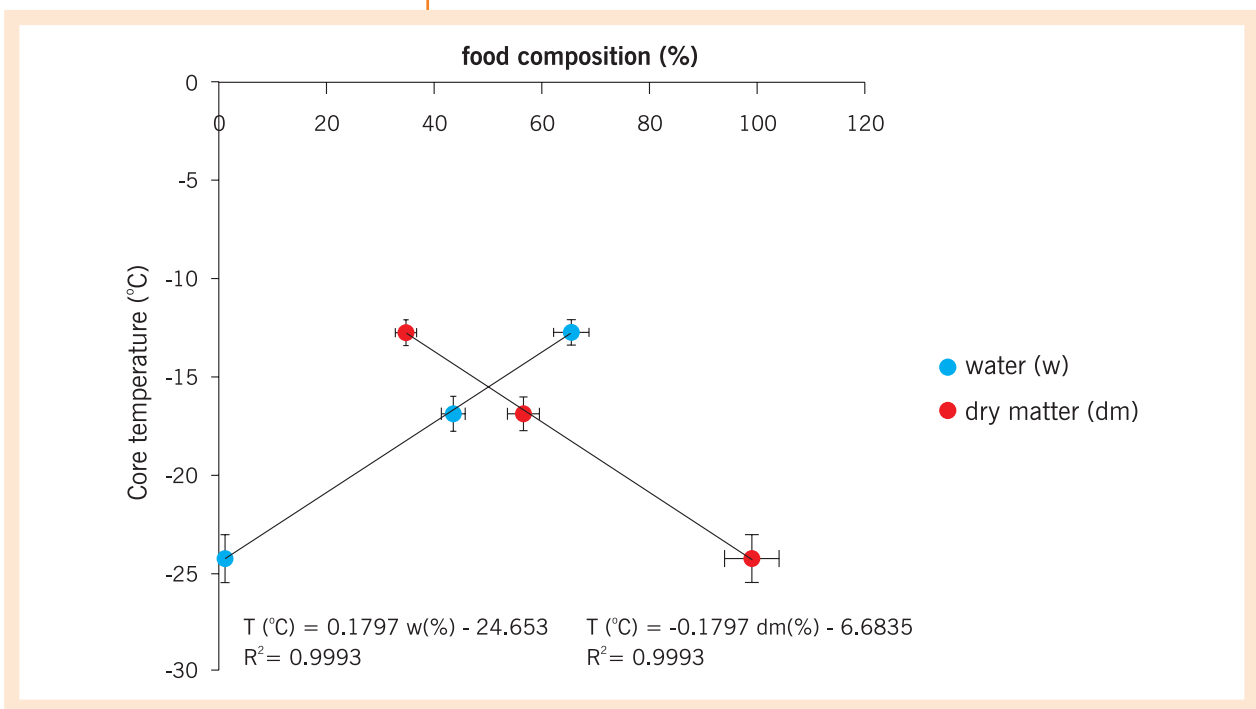
Phase change from liquid to solid (ice).  
 T: predicted temperature (°C);  
 t: time (min) in range from 0 to -12 °C.

ucts with lower water content. Freezing time as correlation to water content as well as dry matter from our research is described with equation on the Figures 2 and 3.

**Figure 3.**

Food composition in correlation to core temperature in sampled products.

There are exceptions like cucumber which has water content 95.4 % and enthalpy 390 kJ/kg (at 0 °C), but low specific heat capacity (1.02 kJ/(kg·K)) thus its water in 100 % freeze at -5 °C, meanwhile tomato pulp with water content 92.9 % and enthalpy 382 kJ/kg (at 0 °C), but higher specific heat capacity (4.02 kJ/(kg·K)) at -5 °C have still 16 % of unfrozen water [11]. The time of cooling when the 0 °C was



achieved, was also different – 9 minutes for vanilla, 13 minutes for chocolate and 15 minutes for forest fruit. So the filling with the highest content of water cool down to 0 °C rapidly compare to other two. The cooling gradient from 24 °C to 0 °C for vanilla was 2.7 °C/min, 1.8 °C/min for chocolate and 1.6 °C/min for forest fruit filling. This temperature cooling gradient is also shown on the Figures 1 to 3. Although any solid food is not solution, the aqueous component in the frozen solid food can be considered as a mixture of ice and a solution of nonaqueous components in liquid water. As water in the food freezes into pure ice during the freezing, the remaining solution becomes more and more concentrated. Thus, depression of the freezing point of the food occurs and continues as concentration increases. The net effects of dynamic freezing point depression are that the initial freezing point of the food is below 0 °C. The freezing process occurs over a range of temperatures, which is different from the freezing process of pure water at unique temperature. The phase changes from liquid water to solid (ice) are shown in the Figure 2.

When the 0 °C was reached after different time in each product, the temperature gradient in all products changed rapidly because of the changing of physical state from liquid to solid (ice). At this point the phenomena discussed above appear, so the products with higher content of water need more energy to convert all water to ice. The product with chocolate filling which has the highest content of fat continues to cool down at the quite the same temperature gradient. It can be assumed that the fat in the chocolate consist majority from saturated fatty acids which are solid at room temperature, thus there is less water than in other two cases that has to be converted from liquid to solid.

## CONCLUSIONS

As expected, the product containing the highest level of water (vanilla filling) has not reached the target temperature and it was with the core temperature extremely above the -18 °C and thus can represent a food safety hazard as well as risk for consumers. There are data confirming microbial growth of some Gram-negative bacteria at -4 °C and growth of bacilli, that is, Gram-positive bacteria at -7 °C [4]. The problem of higher core temperature than -18 °C in frozen food products, which is also obligatory by the law, is not concerning only the primary production process or enterprisers but concerns all actors in food supply chain [2].

The data of food composition as well as shape, density, surface are necessary to predict freezing conditions. On one hand it is possible to reach target temperature by pre-cooling of raw material (where possible), while on the other hand we can prolong the time of freezing and lower the temperature of freezing media (air) and increase its velocity for better heat transfer (not always applicative). In general we need longer time of freezing for foodstuff with high water in the water soluble components. Because of the effect of lowering freezing point as result of concentrating the solution in food, as well as knowing the mean of the importance of specific heat. There are also some other methods to

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In practice every part of food supply chain has the manage system which pushes the action to limits like just in time management.

decrease freezing time such as use of cryogenic freezing, where extreme low temperatures are used by liquid gasses ( $N_2$ , He,  $CO_2$ ) or increasing the dry matter by adding hydrocolloids, proteins and sugars.

Global food safety will be achieved, when every single link in the food chain will entirely (in its indoor and outdoor environment) become master of its particular area and will trust in activity of both previous and following link in the food safety circle "from farm to table", not ignoring consumer [22]. Usually in practice every part of food supply chain has the manage system which pushes the action to limits like just in time management. So there is possibility that frozen food with inadequate temperature from enterpriser over the logistics, reach the retailer shelves thus even consumer, who can storage such a frozen food according to expire date. For SMEs is essential to establish their own model for predicting freezing time which is more applicable than general theoretical models that require a lot of data which are not common accessible for SMEs.

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