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## 1 INTRODUCTION

A series of experiments on superchilling of saithe fillets from Lerøy was performed at the SINTEF Energy Research laboratory in Trondheim in September/October 2009. The experiments are part of the project Competitive Food Processing in Norway and “Temperaturstyring fra fangst til marked”.

The main intention of the study was to check if different freezing regimes (high or low temperature) would result in different physical quality of the product after storage in both superchilled (-1,7 °C) and normal (+2 °C) temperatures.

Earlier experiments performed in the laboratory at SINTEF Energy Research have indicated that low temperature during superchilling could lead to higher drip loss. This could be due to higher degree of recrystallization inside the product during storage at superchilled temperatures. The new crystals that are formed during storage will be larger, due to the slow process, and could therefore demolish the internal structure of the product.

## 2 MATERIALS AND METHODS

The saithe fillets arrived in sizes of approximately 1 kg. The fish was caught and slaughtered 3 days before arrival. From each fillet, samples of 250 ± 20g were cut out. With exception of very large fillets, only one sample was made from each, to keep the shape of the samples as similar as possible. The samples were weighed, vacuum-packed and stored for 1 day, until superchilling. Reference samples for chilled (+2 °C), iced and frozen storage were also prepared.

The superchilling was performed with a FMC FoodTech impingement (IMP) freezer. To test the effect of freezing temperature, three different temperatures were chosen: -45 °C (series 1), -27 °C (series 2) and -10 °C (series 3). In addition, all the temperature series had three different storage

scenarios. The samples were moved from superchilled storage to chilled storage after 1, 3 and 6 days for group A, B and C respectively. This resulted in a total of 9 superchilled test courses.

To keep the ice fraction in all the samples at the same level, the freezing time ( $\tau$ ) was calculated for each of the temperatures with the following simplified formula:

$$\tau_1 = \frac{T_i - T_0}{T_i - T_1} \tau_0$$

$\tau_1$  is the time needed to reach a ice fraction with temperature  $T_1$  equal to that reached with temperature  $T_0$  after the time  $\tau_0$ .  $T_i$  is the freezing temperature (approximately 0 °C). The goal was to reach an ice fraction of about 15 %. Due to the uncertainties in the calculation, a pre-experiment was carried out. The following superchilling periods were chosen based on the calculations and the pre experiment: 57, 105 and 260 s for series 1, 2 and 3 respectively.

For both the pre-experiment and the experiment, ice fractions were measured by a calorimetry method developed by SINTEF ER. 5 samples from each freezing temperature were used for ice fraction measurement.

1, 3 and 6 days after being moved from the superchilled storage, 6 samples from the different superchilled series were wiped off with paper and weighed. The drip loss was then calculated by the percentage loss in weight from pre-packing.

## 3 RESULTS

### 3.1 Ice fraction

The average ice fractions measured by the calorimetry method is shown in Table 1, together with the standard deviations.

**Table 1: Ice fractions**

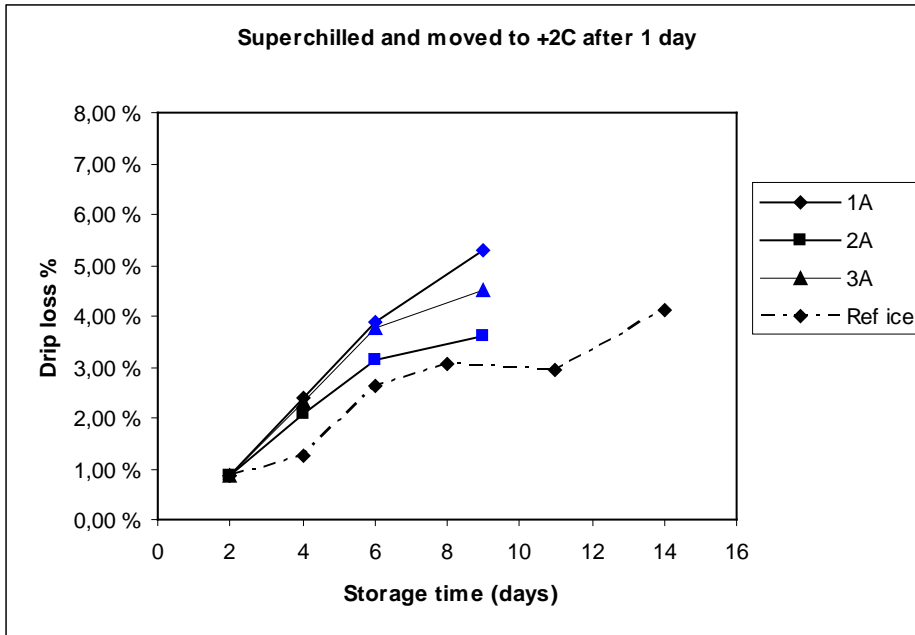
Series	Temperature	Ice fraction	STD
1	-45 °C	16,4 %	1,7 %
2	-27 °C	16,3 %	2,3 %
3	-10 °C	11,9 %	1,5 %

### 3.2 Drip loss

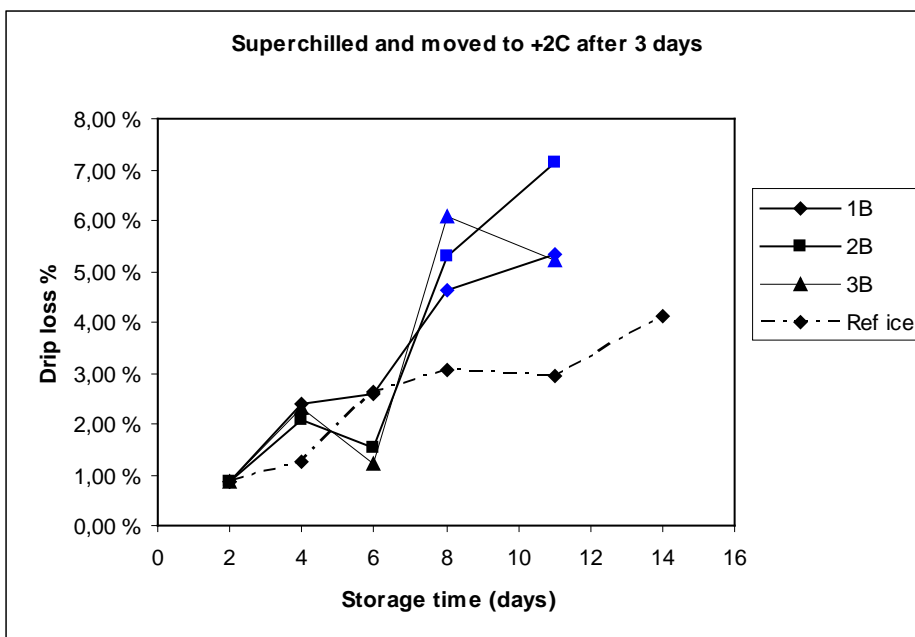
Figure 1, Figure 2 and Figure 3 shows the development in drip loss during storage for series A, B and C respectively. All curves start with a common point, based on drip loss measurement done on the reference at the day of superchilling (day 2).

In addition, the plot for the series that were stored at superchilling temperature for more than one day (B and C) also include the first measurement of the previous superchilled samples (i.e. plot of 1C includes the measurement of 1A and 1B after 1 day at chilled (2 °C) storage. This is done to illustrate the effect of superchilled storage on the drip loss.

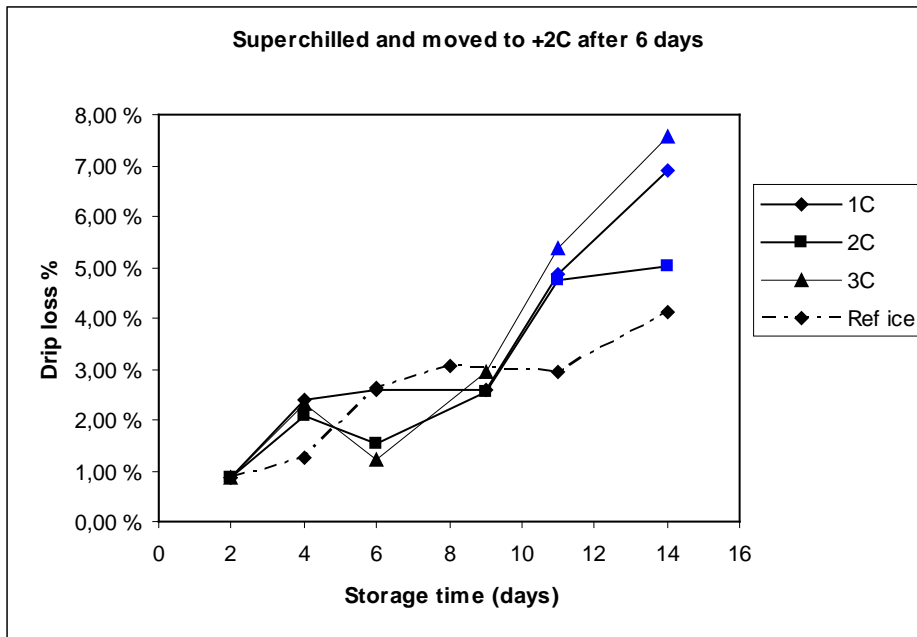
All measurements done after more than one day of storage at 2 °C is marked with blue points.



**Figure 1: Superchilled saithe moved from superchilled storage after 1 day**

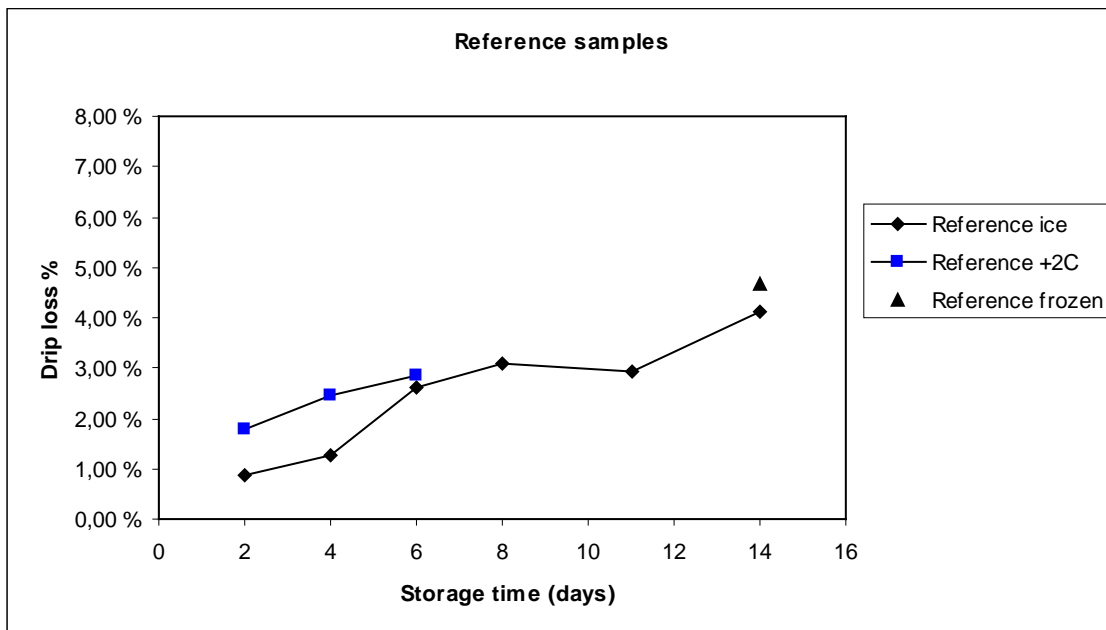


**Figure 2: Superchilled saithe moved from superchilled storage after 3 days**



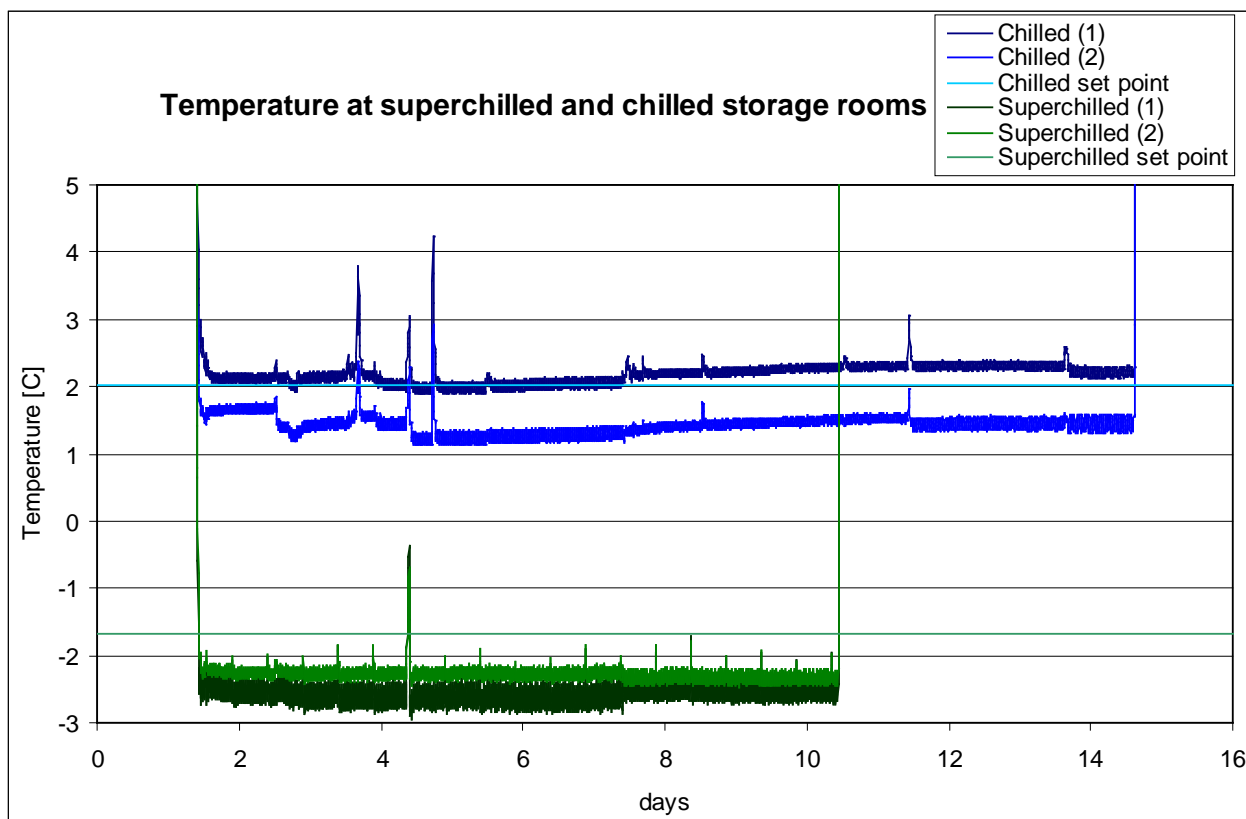
**Figure 3: Superchilled saithe moved from superchilled storage after 6 days**

Figure 4 show the measurements of all the reference samples.



**Figure 4: Reference samples**

Figure 5 shows the temperatures that were logged inside the superchilled and chilled storage.



**Figure 5: Temperatures at superchilled and chilled storage rooms.**

## 4 DISCUSSION AND CONCLUSION

### 4.1 Ice fractions

Table 1 shows that the ice fraction in series 1 and 2 match very well with the goal of 15 %. The ice fraction in series 3 is a bit lower than the goal. Earlier experiments have shown that the ice fraction is not very critical as long as it stays below 30 %.

The standard deviation is also relatively low compared to earlier tests. It is normal that there are large variations in the ice fraction for heterogeneous products such as foods. The saithe fillets will have some variations in thickness and surface areas, and the heat transferred to each sample will therefore vary largely.

### 4.2 Drip loss after superchilling

The drip loss measurements performed 1 day (24 hours) after removal from the superchilled storage was meant to show the drip loss due storage at superchilled temperature. It was therefore important not have the samples stored too long in the chilled storage room, as this could influence the drip loss. However, if the thawing period is too short, ice would still be present in the sample and reduce the drip loss.

Figure 1 to Figure 3 shows that the drip loss after thawing is quite low compared to the drip loss after further storage at chilled temperatures. In addition, the drip loss is not increasing during storage at superchilled temperatures (rather decreasing). This indicates that there was still ice in the samples after 24 hours of thawing. This was confirmed when weighing the samples for drip loss calculations (one could see and feel that there were still ice in the samples). Therefore one should not put too much focus on these results.

It was also observed that there was more ice in the samples from group B and C, which had been stored at superchilled temperatures for a longer period. The reason for this is indicated in Figure 4. The set point for the superchilled storage room was set to  $-1,7\text{ }^{\circ}\text{C}$ . However, the measured temperature is much lower (between  $-2,7\text{ }^{\circ}\text{C}$  and  $-2,2\text{ }^{\circ}\text{C}$ ). This temperature corresponds to higher ice fraction than the initial 15 %. This means that the amount of ice in the samples would increase during storage. This would lead to the need for a longer thawing period before the first measurement, but could also lead to higher degradation of the physical properties of the product.

#### 4.3 Difference between the freezing temperatures

Figure 1 indicates a difference in drip loss depending on temperature used during superchilling. The chart shows that a temperature of  $-27\text{ }^{\circ}\text{C}$  gives the lowest drip loss in this experiment, when the samples are stored at superchilling temperature for only 1 day.

However, Figure 2 shows very different results (almost complete opposite). Here series 2 gives the worst results after 6 days chilled storage. One should notice that in contrast to group A, where the different series had steady development, the drip loss in group B seems more variable. The reason for this is difficult to predict.

The tendency in Figure 3 is more similar to that of Figure 1, with both a more steady development, and an indication that  $-27\text{ }^{\circ}\text{C}$  freezing temperature gives the lowest drip loss.

Based on the results, it is difficult to conclude on the hypothesis for the experiment. However, there is a small indication that neither too low nor too high temperature is ideal, but one should be somewhere in the middle.

#### 4.4 Comparison with the reference samples

The data collected from the reference sample concur well with previous results.

Since the superchilled samples have been partially frozen, it is natural that the drip loss is somewhat higher than the reference kept on ice. This difference is further increased due to the low temperature in the superchilled storage room. The drip loss of the samples stored at  $2\text{ }^{\circ}\text{C}$  is somewhat higher than that stored on ice. It would therefore be interesting to compare this drip loss with that of the superchilled storage over a longer period. This would be a fair comparison since the superchilled samples are stored at  $2\text{ }^{\circ}\text{C}$  after superchilled storage.

#### 4.5 Storage room temperatures

As discussed above, the temperature in the superchilled storage room was measured to be much lower than the set point. There are main possibilities for why this has happened. One could be that the temperature element in the set point controller is badly calibrated.

The other main reason could be due to differences in the temperature depending on location in the room. Figure 4 shows that there are considerable differences in the temperature measurement of the two elements. It is known that one of the elements was placed much closer to the evaporator than the other. However, the difference between the mean temperature measured by the two elements is only  $0,2\text{ }^{\circ}\text{C}$ , while they are more than  $0,6\text{ }^{\circ}\text{C}$  from the set point.

To investigate this further, it has been decided to deploy several temperature elements at different locations in the room, to measure the local temperature differences.

In the chilled storage room, the difference between the temperature measurements is even greater (almost  $0,8\text{ }^{\circ}\text{C}$ ). It is difficult to predict how this influences the drip loss after storage, but it would

certainly influence the thawing period of the superchilled samples. This could be reason for the very low drip loss in two of the series in group B.

## **5 FURTHER WORK**

Before further experiments are performed in the laboratory, it is important to determine why the measured temperature in the superchilled storage room is much lower than the set point.

Since the experiment is inconclusive on the hypothesis. It will be necessary to investigate the issue further. One possibility could be to perform a similar experiment, to see if the results are repeatable. There are large uncertainties when working with heterogeneous products such as fish and the repeatability of experiments can be very low. It would then also be desirable to store samples at chilled temperatures during the whole experiment.

There has also been started an initiative to measure the ice crystal size of superchilled products. This could answer some questions concerning differences in recrystallization, due to different freezing temperature.