Determination of the water holding capacity (WHC) and cook loss of fish muscle

ZENTRIFUGEN

The determination of the water holding capacity (WHC) is an established method of investigating the degree of denaturation of proteins in muscle tissue. Although the emphasis to date has been on raw material, the method described here and the corresponding sample cups have been specially developed for cooked samples.

Where convenience meals are prepared from fish products, for example, the quality of the fish must be kept at a high level. Quality controls are therefore essential at various stages in the production process. Heating in particular causes changes to muscle tissue through denaturation of the proteins. The fish muscle should not disintegrate after cooking and remain tender.

A determination of the WHC and cook loss allows conclusions to be drawn about the degree of denaturation of the proteins and therefore the quality of the fish. It is therefore an objective and reproducible method.

Carrier 4780 with lid 4783 and special accessories for determination of WHC.

Hettich products to determine the WHC and cook loss of fish muscle:
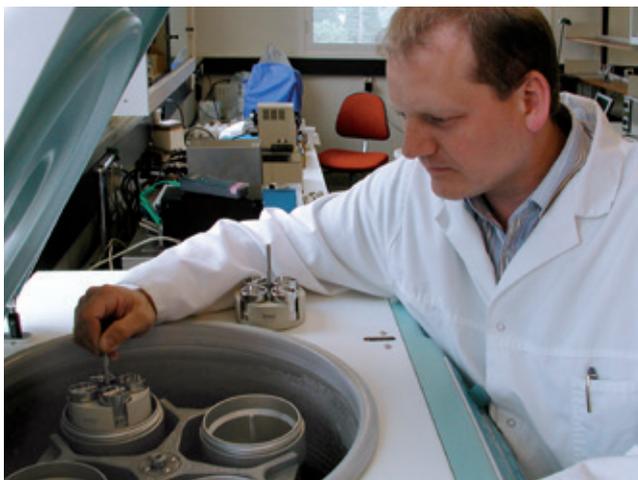
By courtesy of Nofima Norconserv AS, Måltides Hus, Richard Johnsens gt 4, 4021 Stavanger.

Advantages of the method

This method enables the WHC and cook loss of heat treated samples to be determined precisely. The methods used to date have been for investigation of raw samples and are unsuitable for a study of the kinetics of cook loss and the WHC of cooked samples since these require a rapid and uniform heating of the sample.

The special design of the newly-developed sample cups ensures that heat is transferred quickly and uniformly from the heating medium to the sample. The changes in quality as a result of the heating can therefore be determined precisely and the manufacturing processes changed where necessary.

The new formula described by Skipnes et al.[1] factors the cook loss into the calculation of the total WHC (WHC_{TOT}) directly.

[1] Skipnes D., Østby M. L., Hendrickx M. E. 2007: A method for characterising cook loss and water holding capacity in heat treated cod (Gadus morhua) muscle, in: Journal of Food Engineering, Volume 80, Issue 4, P. 1078-1085.

The sample cup

The centrifuge tubes used to date for a determination of the WHC were expensive custom-made products and required transfer of the sample between heating and centrifugation. The sample cups developed specifically for this method offer considerable advantages:

· Simple and exact determination of the water holding capacity.

· Isothermal heating in sample cup without the need to transfer samples between heating and centrifugation.

· Rapid heating in a water bath and direct analysis of the following parameters
    - Water holding capacity
    - Cook loss
    - Texture
    - Colour.

· No liquid loss from the sample cup.

· No absorption of liquid into the sample cup.

· Easy to clean.

· High mechanical durability, allowing higher centrifugal forces.

Bottom lid in base position

Bottom lid in **extended position**

Filter insert

Sample cup

Top lid

Fig. 1: Design of the sample cup

Fig. 2: Bottom lid in base position (left) and in extended position (right)

This flexible solution allows adaptation to different sample quantities and ensures an optimal heat transfer during the heating process.

Preparation

1. Preparation of the fish sample

Cut the raw fish sample into pieces or cut samples so that they have a diameter of 31 mm. Weigh out approx. 5 g of muscle tissue with a total height of approx. 6 mm. The muscle tissue should be homogeneous and free of fat and connective tissue.

2. Preparation of the sample cups

The sample cups should be kept on ice until use. Before filling with the sample, the top lid is screwed onto the sample cup. If the temperature in the sample is being measured then the top lid with the opening for the temperature sensor should be chosen. Weigh the sample cup with the top lid (weight g_1).

3. Filling the cup with the sample

Invert the sample cups (the top lid now forms the bottom), fill it with the prepared sample material and weigh the sample cups again (weight g_2). Screw in the filter until it makes contact with the sample.

Now screw in the bottom lid as far as it will go (until it is in contact with the filter) and weigh the sample cups again (weight g_3).

Fig. 3 shows a longitudinal section through the sample cups, assembled for the heating process. The bottom lid (dark grey) is extended till it meets the filter (blue).

Note: The filter and the sample should be in good contact, but the sample must not be subject to any pressure.
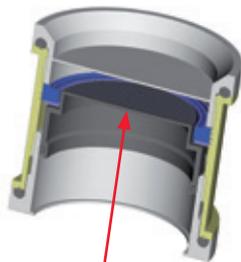

Fig. 3: Longitudinal section through the sample cup assembled for the heating process

4. Heating of the sample

The sample cup is placed for a specific period of time in a water bath preheated to the required temperature (e.g., 10 minutes at 80 °C).

The special design of the sample cup and the lid enable optimum heat exchange.

Note: Ensure that no air bubbles form on the sample cup in the water bath as they would prevent uniform heat exchange.

5. Removal of the exudate

Remove the sample cup from the water bath and allow it to cool in ice water at 0 – 1.8 °C and then dry the outside of the cup well. Unscrew the lower lid and allow the liquid that has been released by the sample during heating to drop out of the cup for 30 seconds. Wipe the remainder of the exudate from the lower lid and inside wall of the sample cup. Ensure that you do not touch the filter during the drying procedure. Weigh the sample in the sample cups together with the dried lid (weight g_4).

6. Centrifugation

Screw the bottom lid back on, this time in its base position and centrifuge for 15 minutes at 1800 min^{-1} and 4 °C.

Fig. 4 shows a longitudinal section through the sample cup, assembled for the centrifugation. The bottom lid (soft and dark grey) is in base position. The liquid lost by centrifugation will be collected in the space between bottom lid and filter (blue).
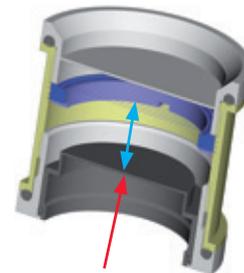

Fig. 4: Longitudinal section through the sample cup assembled for centrifugation

7. Removal of the exudate

After centrifugation, weigh the sealed sample cup. Unscrew the bottom lid in which the liquid that was lost during centrifugation has collected, and allow the sample to stand for 30 sec so that the liquid can drain. Wipe the remainder of the exudate from the bottom lid and the inside wall of the sample cup. When drying the inside of the cup ensure again that the filter is not touched. Weigh the sample in the sample cup together with the dried lid.

Steps 4 and 5 are omitted for the determination of the WHC of raw samples.

Calculations

1. Determination of cook loss

The cook loss is the difference between the g_3 value and the g_4 value. The weight of the sample is given by g_2 minus g_1. The percentage weight loss can therefore be calculated from these values.

2. Determination of WHC of raw samples

The WHC of raw samples is calculated as the ratio of the water remaining after centrifugation to the initial water content of the sample, using the following formula[2]:

$$WHC = \frac{W_0 - \Delta W}{W_0} \times 100 \%$$

$$W_0 = \frac{V_0}{V_0 + D_0} \times 100 \quad \text{and} \quad \Delta W = \frac{\Delta V_0}{V_0 + D_0} \times 100 ;$$

V_0 = Initial water content of the sample
ΔV_0 = Difference in water content of the sample, before and after centrifugation
D_0 = Initial dry mass of the sample. The dry mass can be determined gravimetrically, for instance by drying it for 16 hours at 105 °C.

3. Determination of WHC of heated samples

The fish sample loses liquid when it is cooked. The liquid comprises water, dissolved proteins, ash, salt and fat. The remaining dry material D_1 is therefore somewhat less than the initial dry mass D_0. The sample will lose not just water but also additional dry mass during the centrifugation procedure. As a result, the remaining dry mass D_2 after heating and after centrifugation will be significantly lower than D_0.

This method calculates the water holding capacity on the basis of the water content of the raw sample and takes into account the weight loss on cooking as the total loss WHC_{TOT}:

$$WHC_{TOT} = \frac{W_0 - \Delta W_{TOT}}{W_0} \times 100 \% ;$$

where

$$\Delta W_{TOT} = \frac{\Delta V_1 + C_1}{V_0 + D_0} \times 100 ;$$

V_0 = Initial water content of the sample
D_0 = Initial dry mass of the sample
ΔV_1 = Water loss from the heated sample through centrifugation
C_1 = Weight loss on cooking of the sample

This leads to the following new definition of water holding capacity:

$$WHC_{TOT} = \frac{V_0 - (\Delta V_1 - C_1)}{V_0} \times 100$$

This calculation method describes the change in water holding capacity from raw samples to cooked samples. The percentage of dry mass in the exudate is also taken into consideration in the calculation.

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[2] Skipnes D., Østby M. L., Hendrickx M. E. 2007: A method for characterising cook loss and water holding capacity in heat treated cod (Gadus morhua) muscle, in: Journal of Food Engineering, Volume 80, Issue 4, Issue 4, P. 1080.

Ordering information

Centrifuge and standard accessories	Cat. No.
ROTINA 420 R	4706
4-place rotor	4784-A
Bucket	4780
Lid	4783

Special accessories	Cat. No.
4-places insert	SK 11.07
Sample cup, stainless steel	SK 10.07-2
Lid for temperature sensor	SK 10.07-17
Adapter for texture analyzer	SK 10.07-18

ROTINA 420 R
Benchtop centrifuge, cooled

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100 Cummings Center, Ste 136L

Beverly, MA 02176 U.S.A.

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