

**Physical and Quality Attributes of Salted cod (*Gadus morhua* L.) as
Affected by the State of *Rigor* and Freezing Prior to Salting**

Kristin Lauritzsen ^{a,*}, Leif Akse ^a, Arvid Johansen ^b Sjurður Joensen ^a,
Nils Kristian Sørensen ^a, Ragnar L. Olsen ^c

^a Norwegian Institute of Fisheries and Aquaculture Research, Tromsø, Norway

^b The Norwegian Fishermen's Sales Organisation, (Norges Råfisklag), Tromsø, Norway

^c Norwegian College of Fishery Science, University of Tromsø, Norway

* Corresponding author:

Postal Address: Muninbakken 9-13, P.O. BOX 6122, 9291 Tromsø, Norway

Tel: +47 77 62 90 00

Fax: +47 77 62 91 00

E-mail address: kristin.lauritzsen@fiskeriforskning.no

Abstract

The effects of the *rigor* state and freezing of cod prior to salting on the mass transfer during production and the quality of heavily cured cod have been investigated. *Pre-rigor* salting lead to a larger reduction in weight, a higher water loss and a lower uptake of NaCl than in fish salted *post-rigor* and in fish salted after frozen storage. The cause of this is believed to be the simultaneous influx of NaCl and *rigor* contractions in the fish muscle. In order to reduce the loss of proteins from the raw material and to increase the instrumental lightness values (L^*) of the salt ripened product, the fish should be salted in *pre-rigor* state. However, if the primary focus is on the weight yield, the fish should be salted *post-rigor* after chilling or freezing *pre-* or *post-rigor*. Freezing and thawing of cod prior to salting *post-rigor* increased the firmness of the salt cured product compared to chilled fish salted *post-rigor*.

Keywords: Cod, salting, *rigor* state, frozen, yield, mass transfer, quality.

1. Introduction

Salt-cured cod (*Gadus morhua* L.) is a highly appreciated and traditional product due to its excellent storage stability, characteristic taste, and nutritional value. The cod is usually stored for 3 to 4 days in ice after slaughtering to resolve *rigor mortis* before processing to salt-cured products. Filleting *pre-rigor* instead of *post rigor* reduces the amount of fillet “gaping” during chilled storage. Fillet gaping is the phenomenon by which the tubular junctions between the ends of the muscle cells and the *myocommata* are broken. In cod fillets, an average shrinkage of 24-25% has been reported after *pre-rigor* processing (Sørensen, Arason & Nielsen, 1995). Filleting of salmon *pre-rigor* is reported to give a permanent length reduction of 10-14% (Andersen, Strømsnes, Steinholt & Thomassen, 1994; Sørensen, Brataas, Nyvold & Lauritzsen, 1997; Skjervold, Fjæra, Østby, & Einen, 2001a; Einen, Guerin, Fjæra & Skjervold, 2002). To increase the supply of fresh fish, farming of cod has received much attention (Jobling, 1988; Tilseth, 1990; Rustad, 1992; Lauritzsen, 1993; Olsen, 1997; Puvanendran & Brown, 1999; Morais, Bell, Roberston, Roy & Morris, 2001; Hemre, Karlsen, Mangor-Jensen & Rosenlund, 2003). Currently, this industry is growing in Norway, making *pre-rigor* fish more available for processing.

Much work has been carried out on the effects of freezing of cod in different stages of *rigor mortis*. Peters et al. (1968) found that freezing *pre-rigor* was preferable to freezing *post-rigor* with respect to the sensory properties while Love (1988; 1992) showed that the degree of gaping was least in thawed marine species when the fish had been frozen *pre-rigor*. However, when fish is frozen *pre-rigor*, the energy state of the muscle at thawing is very important since the fish may develop a strong *rigor mortis*. This phenomenon is called “thaw *rigor*” and can lead to excessive loss of water and solutes producing a tough and dry texture (Marsch & Thompson, 1957; Love & Haraldsson, 1958; Jones, 1969). Thaw *rigor* is assumed to occur because ice crystals disable the sarcoplasmic *reticulum* and/or mitochondria in *pre-*

rigor frozen muscle to maintain low levels of calcium in the sarcoplasm, causing irreversible acto-myosin contractions of the myofibrils during thawing (Ma & Yamanka, 1991; Fennema, 1996). However, it has been reported that thaw *rigor* was avoided in *pre-rigor* frozen codling fillets stored at -7 to -22°C for about 2 months prior to thawing (McDonald & Jones, 1976). The maximum breakdown of high energy phosphorous compounds in frozen cod muscle has been found to occur, around -3/-2°C (Nowlan & Dyer, 1974; Cappeln, Nielsen & Jessen, 1999). Prolonged thawing around -3/-2°C is, therefore, favourable to avoid thaw-*rigor* in cod. It has been reported recently that the ATP content of cod muscle decreased during frozen storage and that glycogen may not necessarily decrease at the same rate (Cappeln & Jessen, 2001).

Very little work has been done on how the state of *rigor* and freezing/thawing affect salt curing of cod. In our previous work, the effects of *rigor* on properties of lightly salted cod, was investigated. Such *pre-rigor* salting resulted in a lower weight yield of the product and unfavourable changes in texture and appearance compared to salting of *post-rigor* fillets (Sørensen et al., 1997).

The aim of the present work was to investigate the effects of state of *rigor* and freezing prior to salting on the mass transfer during production and the quality of heavily cured cod. Instrumentally measured colour and shear force values, water holding capacity, and pH of the cured muscle were used as quality indicators.

2. Materials and methods

2.1 Raw materials

Cod (*Gadus morhua* L.) was caught by net or danish seine in January, February and March. The fish was either killed immediately with a blow to the head, bled for approximately 30 minutes in iced sea-water (0°C) and then gutted and headed before storage in ice, or kept alive in seawater net-pens. The fish kept in seawater net-pens were transported alive in tanks of 400 litres with oxygenated seawater for 1-2 hours and then slaughtered as described above before processing. The fish were either processed to split fish or fillets. The weights of the headed and gutted fish individuals were in the range of 870-5030 g. Coarse refined salt (The Norwegian Salt Company Ltd., Bergen, Norway) with purity 99.4% NaCl, 643 ppm Ca, 795 ppm Mg, 0.014 ppm Cu, 3.1 ppm Fe and 0.026% insolubles, was used both for picklesalting and kench curing.

2.2 Rigor state and freezing of the cod prior to salting

Heavy salt curing of cod using chilled or frozen fish in different states of *rigor* as raw material was carried out in 8 different experiments as described in Figures 1(experiments 1-5) and 2(experiments 6-8) respectively. In the first two experiments, danish seine caught cod were slaughtered, gutted, headed and were either split (n=30, experiment 1) or filleted (n= 30, experiment 2) within the first 2 hours post mortem and then immediately salt cured. In experiment 3, net-caught cod (n=65) were slaughtered, gutted, headed and stored for 3 days iced in polystyrene boxes before splitting and salting. In experiment 4, danish seine caught cod (n=30) were slaughtered, gutted, headed and stored for 4 days iced in polystyrene boxes before splitting and salting. In experiment 5, danish seine caught cod (n=30) were slaughtered, gutted, headed and stored for 6 days iced in polystyrene boxes before manually filleting and salting. In experiment 6, danish seine caught cod (n=30) were slaughtered,

gutted, headed and frozen pre-rigor at -40°C within 2 hours post mortem. The fish were then stored frozen at -35°C for 59 days, thawed in running tap water with a temperature of $6-7^{\circ}\text{C}$ for 24 hours and manually filleted prior to salting. In experiment 7, danish seine caught cod ($n=30$) were slaughtered, gutted, headed, stored for 6 days iced in polystyrene boxes and frozen at -40°C . The fish were then stored frozen at -35°C for 59 days, thawed in running fresh tap water holding a temperature of $6-7^{\circ}\text{C}$ for 24 hours and manually filleted prior to salting. In experiment 8, net-caught cod ($N=65$) were slaughtered, gutted, headed, stored for 3 days iced in polystyrene boxes and frozen at -30°C . The fish were then kept for 11 days at -30°C , thawed in running tap water with a temperature of $6-7^{\circ}\text{C}$ for 24 hours before splitting and curing.

Fish were split using BAADER 440 splitting machines (Baader Ltd., Köhln, Hamburg, Germany) at the Tromvik Fishing Industry Ltd or at the Norwegian Institute of Fisheries and Aquaculture Research. To remove blood from the backbone of the split fish and from the neck region after splitting, the fish were washed in running tap water ($6-7^{\circ}\text{C}$) and additionally cleaned with a rotating plastic brush. Both split cod and fillets were individually tagged immediately before salting by penetrating plastic number tags through the skin using a Marking pistol (Floy Tag & MFG, INC, Seattle, WA, USA).

2.3 Salting

The split cod and fillets were picklesalted by stacking fish and solid salt (weight ratio 1:1) in layers in plastic tubs of 700 litres. After picklesalting for 4 or 7 days, the split cod were removed from the brine, cleaned of used salt and restacked with fresh dry salt. The fillets were restacked after 1 day of picklesalting. Kench curing of split fish lasted for 21 days while kench curing of fillets lasted for 28 days. After 11 days of kench curing, the split fish was restacked with fresh dry salt while the fillets were restacked after 7, 14 and 21 days of

kench curing. The weights of the individual fish were recorded as fresh fish, after picklesalting, during kench curing and when the fish was regarded salt ripened, i.e. after 25 or 28 days of salting of split fish and after 29 days of salting of fillets.

2.4 Mass transfer

To estimate the accumulated uptake of NaCl, water, and protein loss in cured cod muscle, the change in weight of the cured fish at the time of sampling was used. The change in weight (ΔM_t^0) was calculated by the formula as follows and given in %:

$$\Delta M_t^0 = [(M_t^0 - M_0^0) / M_0^0] \cdot 100\%$$

M_0^0 = initial wet weight of sample

M_t^0 = wet weight of sample at time t

The accumulated change (ΔM_t^y) in the NaCl, water and protein content from raw material to cured sample was calculated by the formula and given in % (Barat, Rodriguez-Barona, Andres & Fito, 2003):

$$\Delta M_t^y = [(M_t^0 \cdot X_t^y - M_0^0 \cdot X_0^y) / M_0^0] \cdot 100\%$$

X_0^y = the Y (NaCl, water or protein) wet weight fraction of raw material at time 0

X_t^y = the Y (NaCl, water or protein) wet weight fraction of the cured muscle at time t

2.5 Colour measurements

The instrumental colour of the muscle surface was determined by using a Minolta Chromameter, CR-200 (Minolta Camera Co. Ltd., Osaka, Japan). The detector was placed at the dorsal and the ventral side of the central line in the muscle surface of the fillet and the L* a* b* modus was recorded, obtaining a mean value and standard deviations. Prior to the

measurements, excessive salt was carefully removed from the muscle surface. The colour was determined on 15-20 split fish/fillets (n=15-20) from each group.

2.6 Texture measurements

The instrumental shear force value was determined by using a KGS Systems texture analyser (KGS Systems, Tromsø, Norway) with a one blade Kramer shear force cell. A loin part (6 cm width x 20 cm length) of the skin and boneless split fish/fillets was manually excised by a knife and used in the texture measurements. The steel cutting blade (8.3 cm wide and 1 mm thick) of the Kramer cell had an inclined edge and a flat centre. A 100 kg measuring load cell (type U1, FNr B71949, Hottinger Baldwin Messtechnik, Darmstadt, Germany) was used to cut each piece of fillet transversally 6 times. The texture analyser was run at a speed of 0.94 mm s^{-1} , and the shear force values are presented as maximum peak height, giving units of Newton (N). The shear force was determined on 5 individuals (n=5) from each group.

2.7 Chemical analysis

Water holding capacity (WHC), water, protein, NaCl, Ca, and Mg determinations were performed on pooled samples. The pooled samples were prepared by coarsely homogenising skin and boneless split fish/fillets (n=5) including the loin part used in the texture measurements, in a tap water cooled Stephan mixer, UM12 (Hameln, West Germany) for 3 x 5 seconds. Samples for WHC determinations were taken from this homogenate. The mince was further homogenised in a Dito Sama (K55) Food Processor (Abusson, France) for approximately 1 minute providing samples for determination of water, protein, NaCl, Ca, and Mg content. The samples were either analysed immediately (WHC) or kept in sealed plastic bags and stored for 1 to 4 weeks at -80°C before analysis.

The water content in the muscle samples was determined by drying to constant weight at 105°C (AOAC, 950.46, 1990) and the protein content of muscle samples was determined as Kjeldahl protein (AOAC, 981.10, 1990). The NaCl content of muscle samples was determined by standard procedure (AOAC, 937.09, 1990). There were used 3-6 replicates of each sample for determination of the water, protein and NaCl content.

The concentrations of Ca and Mg in the minced muscle samples were determined as described (Simpson & Blay, 1966) using a Perkin Elmer 3110 atomic absorption spectrometer (Perkin Elmer Co. Ltd., Norwalk, CT, USA). The method was modified by including 1% (w/w) La₂O₃ p.a. in the extract prior to the analysis as recommended for samples with chemical interference (Beaty & Kerber, 1993). Four or five extracts from each sample were made and average and standard deviation calculated from the values obtained.

The WHC was measured on coarsely homogenised muscle tissue at 4-5°C by determining the weight of the liquid lost after a low speed centrifugation as described earlier (Ofstad, Kidman, Myklebust & Hermansson, 1993). The WHC was expressed as weight of water retained (g 100g⁻¹). Mean values were calculated from 4-6 replicates for each sample.

The pH of fresh muscle tissue was measured in a 1 : 1 mixture of muscle homogenate and 0.15 M KCl p.a., while the pH of cured muscle was recorded in a 1 : 5 weight ratio of muscle homogenate and distilled water. A PHM 80 Radiometer (Copenhagen, Denmark) with a glass electrode was used and the 3-6 replicates of each sample were made to obtain a mean value.

2.8 Statistical analyses

Statistical analysis of the analytical data from muscle samples was performed on the software program SAS version 6.12 (SAS Institute, Cary, NC). Data were subjected to one-way analysis of variance (ANOVA) by using the general linear model procedure. Where

statistical differences were noted for a measurement, differences among sample means were determined using the Tukey's Multiple comparison test. The level of significance was set at $p = 0.05$ for all tests.

3. Results and discussion

3.1 Mass transfer

It is well known that heavy curing causes a substantial reduction in the weight of the fish due to large osmotic forces from the salt on the moisture of the muscle cells (Dyer, 1949; Beatty & Fougere, 1957; Tülsner, 1978). One of the aims of our work was to investigate the effects of *rigor* and freezing of cod prior to salting on the mass transfer and yields during heavy salting. In Figure 3, the reduction in weight by curing of split cod is shown. A large and significant ($p < 0.05$) difference in the weight reduction was found between the fish salted in *pre-* and *post-rigor* state. The chilled fish that was salted *pre-rigor*, had a weight reduction of 33% after 25 days of salting and the fish salted *post-rigor*, independent of chilled or frozen storage prior to splitting and salting, had a weight reduction around 28%. Similar difference in the weight reduction was also found between the fillets salted in *pre-* or *post-rigor* state (Figure 4). In addition, it was found that salting of fillets from fish frozen *pre-rigor* and stored frozen for 2 months, resulted in approximately the same weight reduction as when salting *post-rigor* fillets after chilled or chilled and frozen storage of the fish.

We believe that the main cause of the large reduction in weight of chilled fish salted *pre-rigor* is that the *rigor* contractions are induced earlier and stronger during salting than during ordinary chilled storage of the fish muscle. It has been reported that 2-4% NaCl added to ground *pre-rigor* beef muscle produces a faster break-down of adenosine triphosphate (ATP) and adenosine diphosphate (ADP) and increase in inorganic phosphate (van Hoof & Hamm, 1973) and inhibition of lactate formation (Honikel & Hamm, 1978). In the present

work, the NaCl concentration of the muscle increased rapidly to saturated level (Figure 6). Salting of *pre-rigor* fish may have increased the rate of hydrolysis of ATP and given a more rapid decline in the muscle-pH. It is known that the muscle shrinkage during *rigor* is dependant on the ability of the muscle to contract freely, i.e. not being connected to the backbone (Sørensen et al., 1997; Einen et al., 2002). When the muscle tissue is exposed to large osmotic forces during such contractions, one may assume that synergetic effects occur with regard to squeezing moisture out of the muscle tissue. Although, split cod has a large part of the backbone present, the muscle contracts more freely than in gutted fish. Due to the short icing time, chilled fish salted *pre-rigor* probably had a slightly higher body temperature at start of the salting process than fish salted *post-rigor*. This may also have contributed to strong *rigor* contractions (Iwamoto, Yamanka, Watabe & Hashimoto, 1987; Skjervold et al., 2001a).

After death, the creatine phosphate of the muscle cells is rapidly used up and anaerobic glycolysis continues to regenerate some ATP with the end product, lactate, accumulating (Fennema, 1996). In frozen fish, the enzymatic reactions responsible for anaerobic glycolysis and ATP breakdown act independently of each other (Cappeln & Jessen, 2001). The moderate reduction in weight registered during heavy salting of fillets frozen *pre-rigor* makes it reasonable that *rigor* contractions did not occur in these fillets. The reservoirs of ATP and glycogen have probably been depleted during the frozen storage and/or the thawing prior to salting. Energy may have been dissipated as heat rather than used as mechanical energy, and hence unavailable for contractions at thawing (Cappeln et al., 1999; Cappeln & Jessen, 2001) and salting. Recent investigations have shown that fresh *pre-rigor* salmon fillets contracted 14% in length during *rigor*, whereas fillets frozen *pre-rigor* did not develop thaw *rigor* muscle contraction (Einen et al., 2002). The changes in weight found in the present work, were in general much higher than what found in our earlier study on lightly

salted cod fillets (Sørensen et al., 1997). This is due to the larger osmotic forces in the muscle tissue during heavy curing. Both light and heavy curing of cod in the *pre-rigor* state gave a reduced yield favouring *post-rigor* salting. Cod frozen *pre-rigor* and stored and thawed under controlled conditions, resulted in a similar weight reduction as for *post-rigor* salted fish.

The mass transfer in cod muscle tissue during heavy salting was investigated to obtain a quantitative picture of the changes. The accumulated changes in water, protein, and NaCl content were determined by using the relative weight fraction (%) of the respective parameters and the weight of the sample. The water loss by heavy curing of cod fillets is presented in Figure 5 and of split cod in Table 1. As expected from the weight reductions, extensive losses of water, i.e. 40-50% of the water in the raw material, occurred during the salt curing processes. The cod salted both as split fish and fillets *pre-rigor* had a larger loss of water than the other experimental groups. In general, the pH, the *rigor* state, the ionic strength and the osmotic pressure of the muscle tissue *post mortem* are decisive for the water holding properties of the muscle proteins (Hamm, 1960; Asghar, Sameijma & Yasui, 1985; Love, 1988; Honikel, 1989; Ofstad, Kidman & Hermansson, 1996). Cod fillets had a muscle-pH of 6.5 when starting the *pre-rigor* salting 2 hours *post mortem*, and pH 6.4 after 24 hours. The fillets frozen *pre-rigor* and stored for 2 months had a muscle-pH of 6.3 after thawing. The muscle-pH fell to 6.2 during the first 24 hours of salting (results not shown). Split cod had a muscle-pH of 6.7 *post rigor*. After 28 days of salt curing, the pH had declined to 6.2 (Table 2). These pH values indicate that the large difference in water loss between the fish salted *pre-rigor* and the fish salted *post-rigor*, was not only related to the pH at salting or the lowering of the pH during the salting process. Additional factors such as high ionic strength, osmotic pressure and state of *rigor* at salting may have synergistically created reductions in the interfilamental space within the myofibrils, in the space between the myofibrils and in the extracellular space of the *pre-rigor* fish muscle tissue. Preliminary findings at our institute,

suggest that the larger water loss found during salting of cod *pre-rigor* is not regained during rehydration.

The uptake of NaCl into the muscle tissue by heavy curing of fillets is presented in Figure 6 and of split cod in Table 1. A significant ($p < 0.05$) lower uptake of NaCl was found in fish salted *pre-rigor* compared to the other experimental groups. Different uptake of NaCl in the muscle tissue may have been influenced by the amount of ATP present in the muscle at salting. The ATP-driven ionic pumps may have been able to maintain concentration gradients across the membranes in pre-rigor muscle, leading to higher resistance to NaCl distribution in this fish muscle at start of the curing (Wang, Tang & Correia, 2000). A lower uptake of NaCl during salting of *pre-rigor* fish was also found in our previous work on light salting of cod (Sørensen et al., 1997). In addition to causing a reduced ATP level in the muscle (Cappeln & Jessen, 2001), freezing and thawing may also lead to muscle fibre shrinkage, increase in the extracellular space (Sigurgisladottir, Ingvarsdottir, Torrissen, Cardinal & Hafsteinsson, 2000), and partly disintegration of membrane structures (Ma & Yamanaka, 1992). The highest uptake of NaCl was found in cod frozen *pre-* or *post-rigor* prior to salting. Destruction of the cell membranes and other cell damage may have accelerated the NaCl diffusion into the muscle tissue. However, at the end of the curing process, the uptake of NaCl in chilled fillets salted *post-rigor* was just slightly lower than the uptake of NaCl in *post-rigor* frozen fillets. Therefore, we believe that the *rigor* state at salting was the main cause to the large differences in the uptake of NaCl in the muscle.

The protein fraction explains most of the nutritional, water holding and sensory properties of the salt ripened products and is, therefore, the most valuable to the consumer. The accumulated protein loss from raw cod fillets during heavy curing is presented in Figure 7. Significantly ($p < 0.05$) less protein was lost by salting fillets *pre-rigor* compared to the other experimental groups. The highest protein loss during salting was from fillets that

previously had been frozen *post-rigor*. At the end of the curing, the protein loss from these fillets was approximately the same as from chilled fillets salted *post-rigor*. The muscle membranes were probably more intact in chilled fish *pre-rigor* than in *post-rigor* fish (Montero & Mackie, 1992) and may have retarded the leakage of proteins out of the muscle. Formation of extracellular drip channels has been related to *post mortem* changes in cytoskeleton and weakening of the integrity of the cod muscle structure and is a time dependant process (Morrison, Bremner & Purslow, 2000). In addition, the strong contraction suggested in the fish salted *pre-rigor* may have reduced passage through these channels. The salt solubility of the myofibrillar proteins may also have been affected by the state of *rigor*. Such proteins in an *in-rigor* state are highly overlapped and cross-linked and less salt soluble than myofibrillar proteins in *pre-* and *post-rigor* states (Hamm, 1960; Hamm, 1986; Honikel, 1989).

3.2 The quality of cured split cod

Usually, lightness and firmness together with a low yellow colour of the fish muscle surface are highly regarded sensory properties of the final products. The second aim of this work was to investigate the effects of *rigor* state and freezing of the raw materials prior to salting on the quality of the salt ripened split cod. The effects of *rigor* and freezing on the instrumental colour and shear force values, water holding capacity and muscle-pH of the cured split cod were determined and used as quality indicators (Tables 1 and 2). Table 1 shows that both the lightness (L^*) and yellow colour (b^*) values were significantly ($p < 0.05$) higher in cod salted *pre-rigor* than in chilled and/or frozen cod salted *post-rigor*. Instrumental colour values are based on the reflectance of light at specific wavelengths from the fish muscle surface. The elevated L^* and b^* values of the *pre-rigor* salted fish, are explained by the light scattering properties of the muscle surface. As discussed earlier, the differences in

the accumulated water loss and uptake of NaCl in both split fish and fillets could probably be explained by the unrestrained *rigor* contractions occurring during salting. Such contractions may have reduced the light transparency through highly overlapped actin and myosin filaments. In addition, increased level of insoluble proteins may have been formed by the more rapid drop in muscle-pH post mortem of *pre-rigor* salted fish (Warriss & Brown, 1987; Robb, Kestin & Warriss, 2000). Recent investigations on salmon fillets have shown that fresh fillets produced *pre-rigor* had a higher colour score (more intensive colour) during 5 days of cold storage compared to fillets going through *rigor mortis* attached to the backbone (Skjervold, Rørå, Fjæra, Vegusdal, Vorre & Einen, 2001b; Einen et al., 2002).

Instrumental shear force values were measured to detect the firmness of the final cured product. A significant ($p < 0.05$) higher firmness was found in salted fish previously frozen and thawed compared to fish salted after cold storage (Table 2). It has been reported from studies on salmon fillets that freezing and thawing reduce the initial firmness and cause fibre shrinkage of the fillets (Einen et al., 2002). Others have reported increased toughness of the cod flesh after frozen storage and explained it by reduced protein solubility and denaturation of muscle proteins (Shenouda, 1980; Ragnarsson, 1987; Mackie, 1993; Sikorski & Kolakowska, 1994). The progressive increase in the hardness of chilled salted catfish (Yashoda & Suryanarayana Rao, 1998), mackerel meat (Shimomura & Matsumoto, 1985) and salmon fillets (Sigurgisladottir et al., 2000) have been reported to be due to widening of the interstitial space, clumping of cells, decomposition of the muscle proteins and fibre shrinkage. The increased firmness found in salted cod that previously had been frozen is probably caused by protein denaturation occurring both during the freezing and salting processes. When muscle proteins are exposed to high NaCl concentration and traces of Ca and Mg ions, increased cross-linking of the polypeptide chains, shrinkage of the muscle and dehydration may occur (Hamm, 1960; Borgstrom, 1968; Offer & Trinick, 1983; Hamm, 1986; Wilding,

Akahane, Lanier & Hamann, 1986; Ragnarsson, 1987; Morrissey, Mulvihill & O'Neill, 1987). In Table 2, it is shown that the calcium content in the fish, that had been frozen, tended to be higher after 7 days of salting than in the fish chilled before salting. In salt ripened fish, the calcium concentrations were equal while the magnesium concentration was highest in the fish that had been frozen. No significant differences ($p < 0.05$) in muscle-pH were determined between the two groups of salted fish during the salting process (Table 2). The significant ($p < 0.05$) lower water holding capacity (WHC) of the salted muscle that had been frozen, is probably due to the suggested increased protein denaturation and increased cross-linking of the polypeptide chains by the divalent cations; Ca and Mg (Hamm, 1960; Asghar et al., 1985; Shenouda, 1980). It has been reported earlier that Ca ions tighten up the protein structure and shrink the myofibrils of silver carp muscle (Shomer, Weinberg, & Vasiliver, 1987). Work on pork meat has shown that extra calcium added to salt mixtures increased the hardness of bologna sausages (Numata, Kawaguchi, Nakamura & Arakawa, 1992).

4. Conclusions

Rigor contractions simultaneously with a salt influx are probably causing the larger reduction in weight, the higher water loss and the lower uptake of NaCl seen in the muscle salted *pre-rigor*. The absence of such *rigor* contractions probably explains the development in weight, water loss, and uptake of NaCl during salting of chilled and/or frozen fish *post-rigor* and during salting of fish frozen *pre-rigor*. To reduce the waste of proteins from the raw material and to increase the instrumental lightness values (L^*) of the salt ripened product, the fish should be salted in *pre-rigor* state. However, if focus is on the weight yield, the fish should be salted *post-rigor* after chilling or freezing *pre-* or *post-rigor*. Freezing and thawing of cod prior to salting *post-rigor*, increased the firmness of the salt cured product compared to chilled fish salted *post-rigor*. This increased firmness and the lower water holding capacity

are probably caused by protein denaturation occurring both during the freezing and salting processes.

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Figure captions

Figure 1. Schematic illustration of the handling procedure of chilled cod prior to salting and during the heavy salt curing. Five different experiments were carried out. Symbol description: d = time in days, \perp splitting or filleting, \rightarrow chilling, \longrightarrow salting. *Pre-rigor* means within 2 hours *post mortem*.

Figure 2. Schematic illustration of the handling procedure of frozen cod prior to salting and during the heavy salt curing. Three different experiments were carried out. Symbol description: d = time in days, \perp splitting or filleting, \rightarrow chilling, ---- \rightarrow freezing and frozen storage, -- \rightarrow thawing, \longrightarrow salting. *Pre-rigor* means within 2 hours *post mortem*.

Figure 3. The effects of *rigor* state and freezing of split cod prior to salting on the change in weight, (ΔM_t^0 , %) during heavy curing. Symbol description: ■ Chilled and salted *pre-rigor*¹, ▲ Chilled and salted *post-rigor*³, ● Chilled and salted *post-rigor*⁴, Δ Frozen *post-rigor*, stored and salted⁸. Code number relates to experiment number shown in Figures 1 and 2.

Figure 4. The effects of *rigor* state and freezing of cod fillets prior to salting on the change in weight (ΔM_t^0 , %) during heavy curing. Symbol description: ■ Chilled and salted *pre-rigor*², ▲ Chilled and salted *post-rigor*⁵, □ Frozen *pre-rigor*, stored and salted⁶, Δ Frozen *post-rigor*, stored and salted⁷. Code number relates to experiment number shown in Figures 1 and 2.

Figure 5. Estimated accumulated water loss ($\Delta M_t^{\text{water}}$, %) from raw cod fillets during heavy salting as affected by *rigor* state and freezing prior to salting. ■ Chilled and salted *pre-rigor*², ▲ Chilled and salted *post-rigor*⁵, □ Frozen *pre-rigor*, stored and salted⁶, △ Frozen *post-rigor*, stored and salted⁷. Code number relates to experiment number shown in Figures 1 and 2.

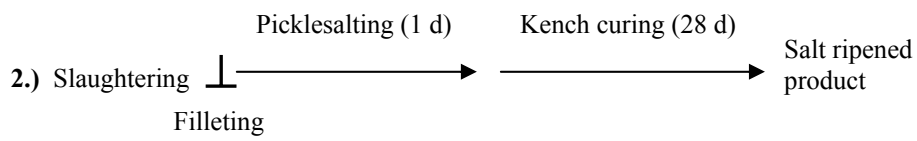
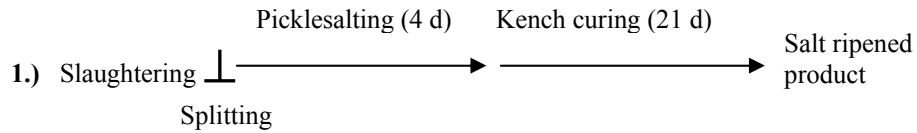
Figure 6. Estimated accumulated uptake of NaCl (ΔM_t^{NaCl} , %) into the raw cod fillets during heavy curing as affected by *rigor* state and freezing prior to salting. Symbol description: ■ Chilled and salted *pre-rigor*², ▲ Chilled and salted *post-rigor*⁵, □ Frozen *pre-rigor*, stored and salted⁶, △ Frozen *post-rigor*, stored and salted⁷. Code number relates to experiment number shown in Figures 1 and 2.

Figure 7. Estimated accumulated protein loss ($\Delta M_t^{\text{protein}}$, %) from raw cod fillets during heavy salting as affected by *rigor* state and freezing prior to salting. Symbol description: ■ Chilled and salted *pre-rigor*², ▲ Chilled and salted *post-rigor*⁵, □ Frozen *pre-rigor*, stored and salted⁶, △ Frozen *post-rigor*, stored and salted⁷. Code number relates to experiment number shown in Figures 1 and 2.

Figure 1

CHILLED FISH

Pre-rigor



Post-rigor

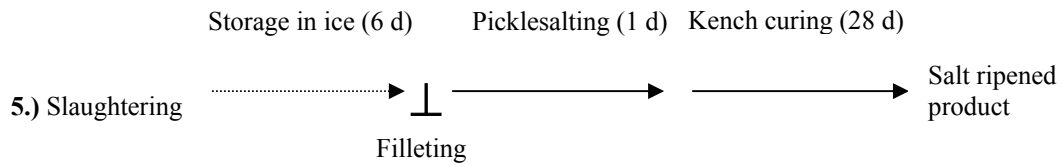
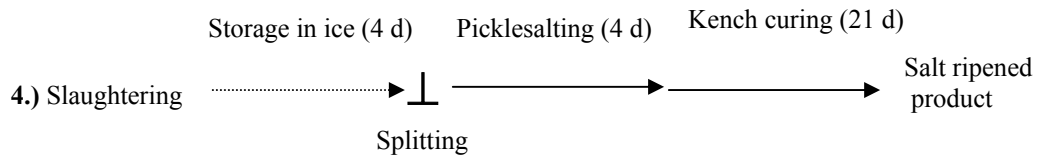
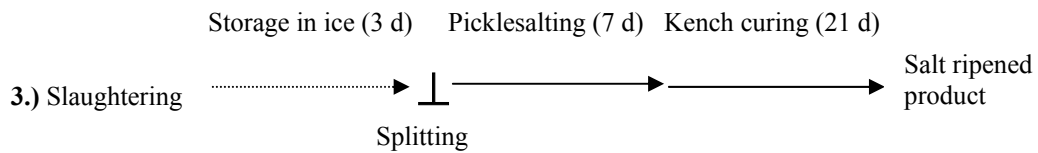
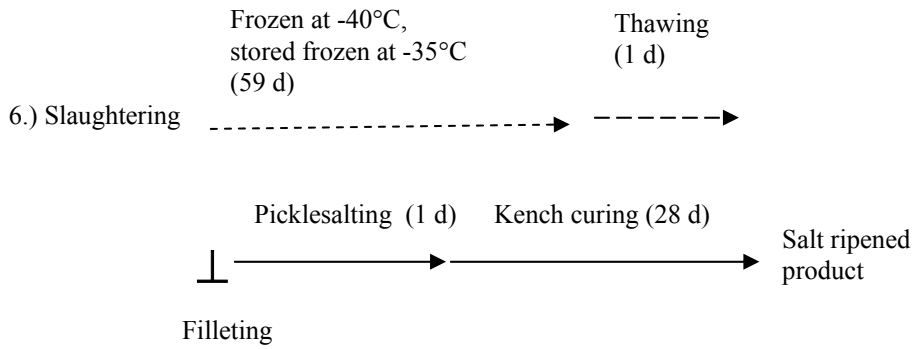


Figure 2

FROZEN FISH

Pre-rigor



Post-rigor

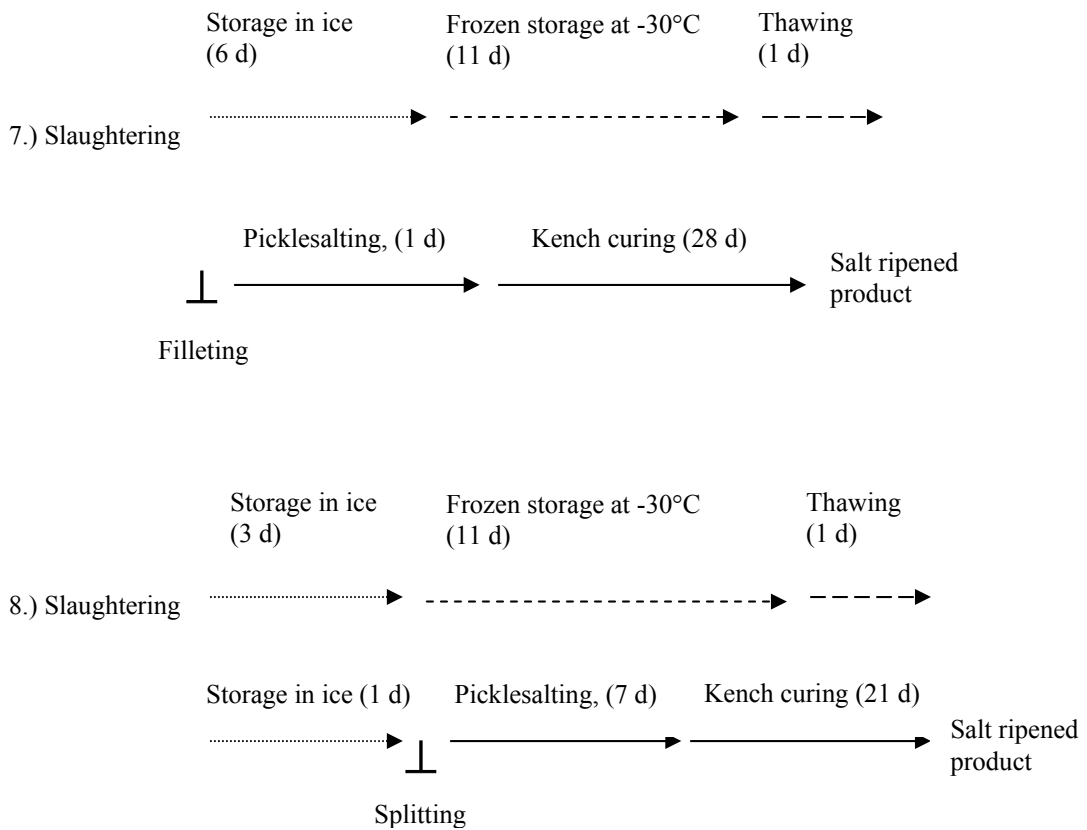


Figure 3

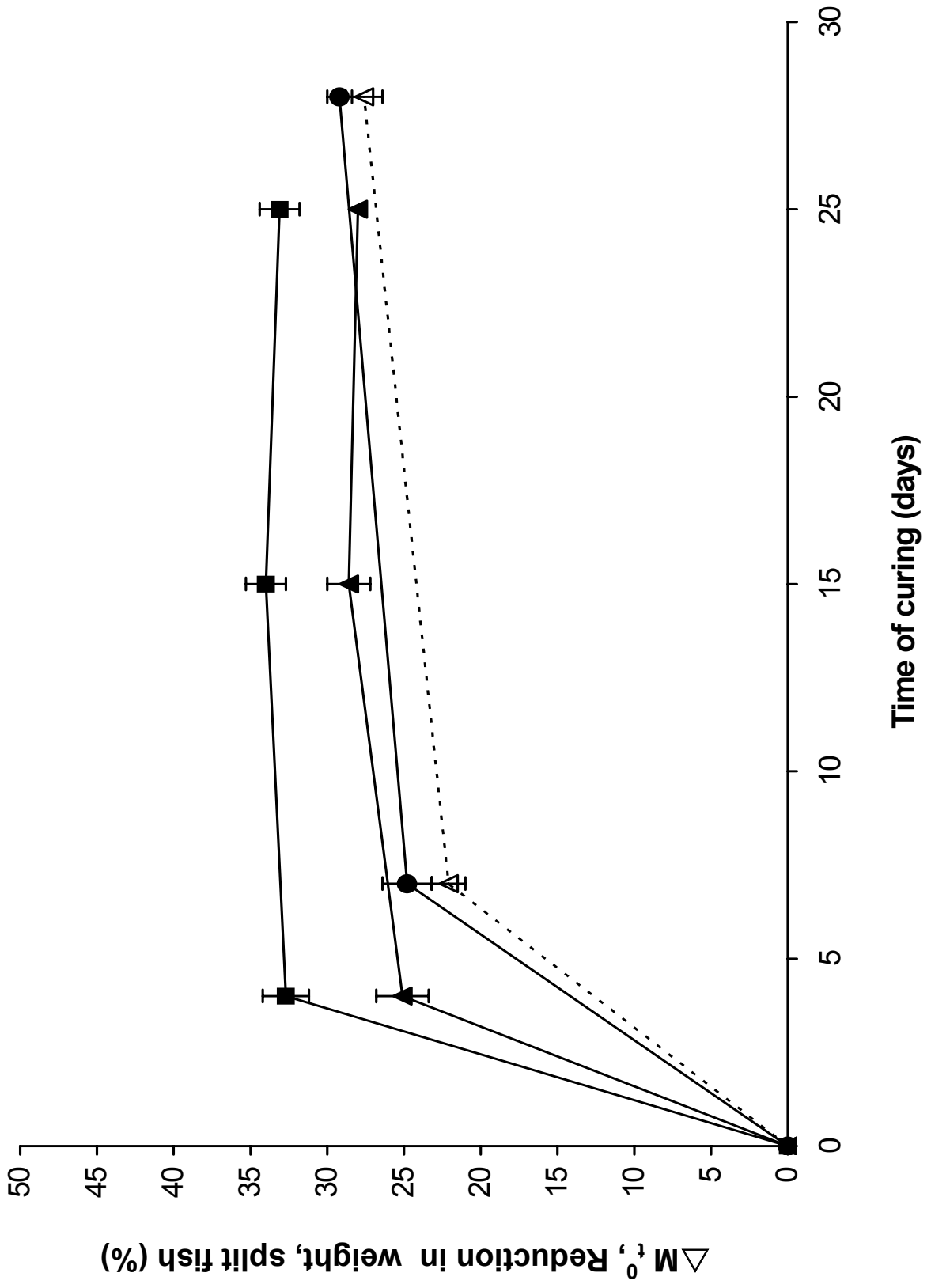


Figure 4

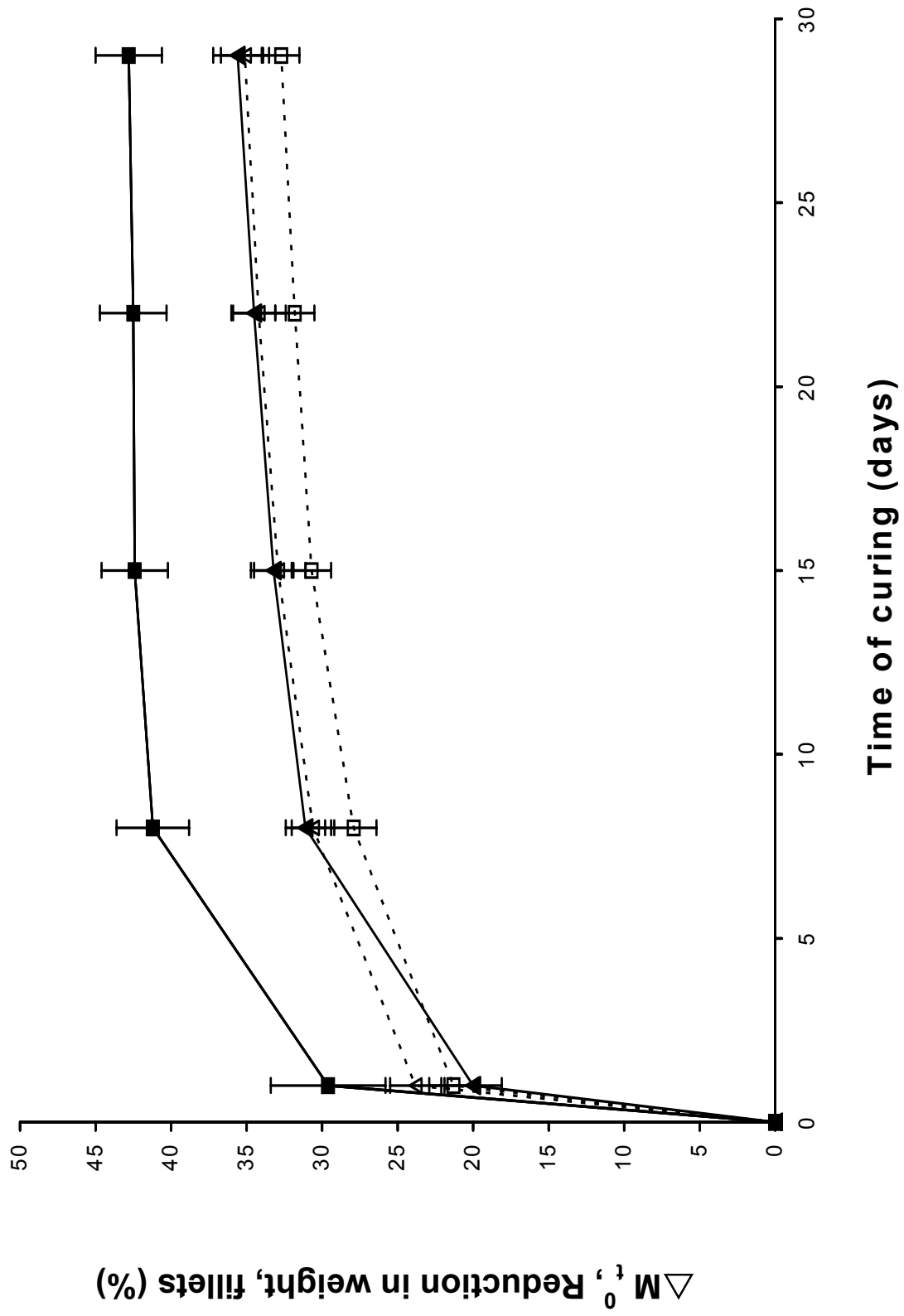


Figure 5

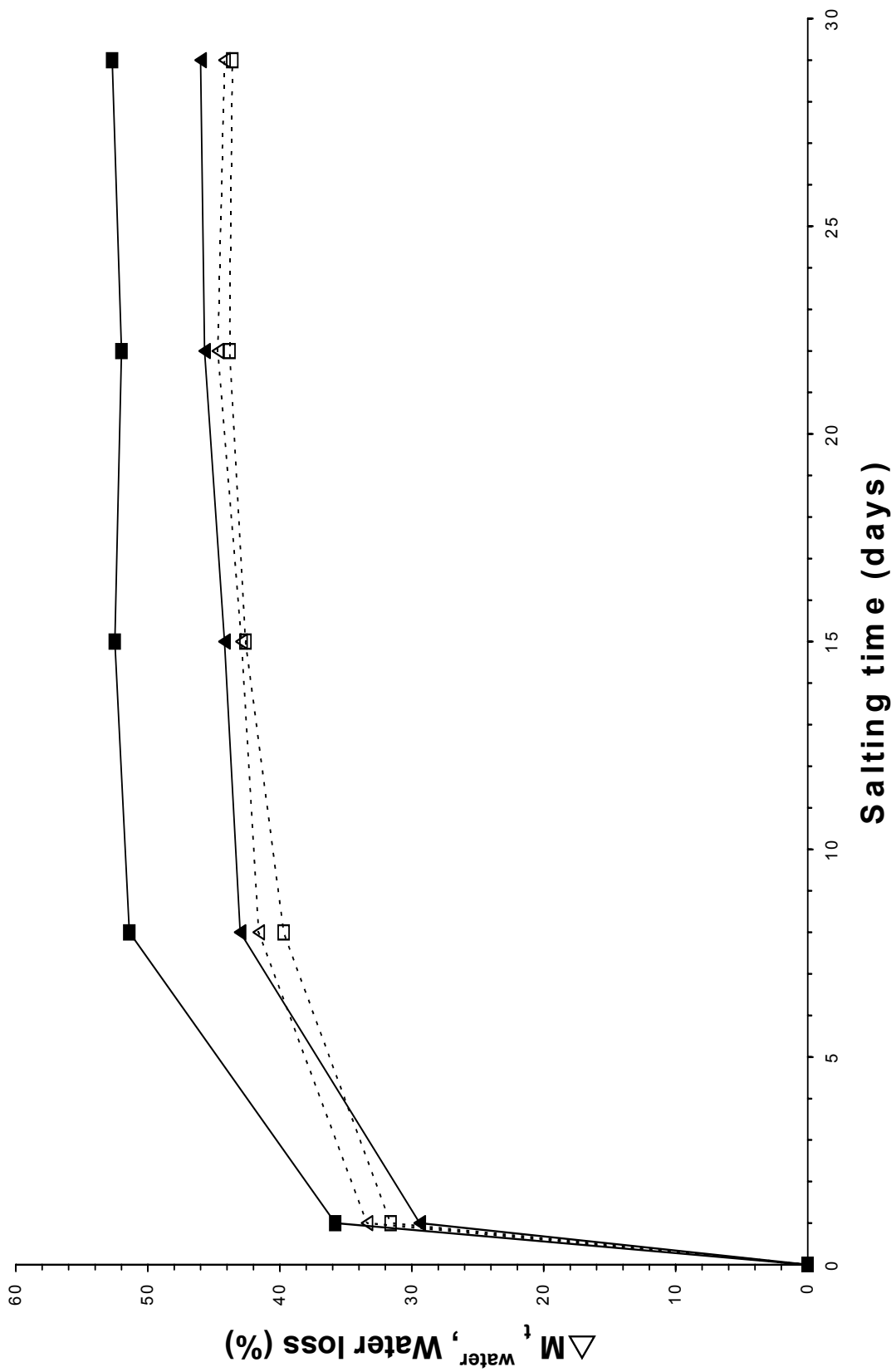


Figure 6

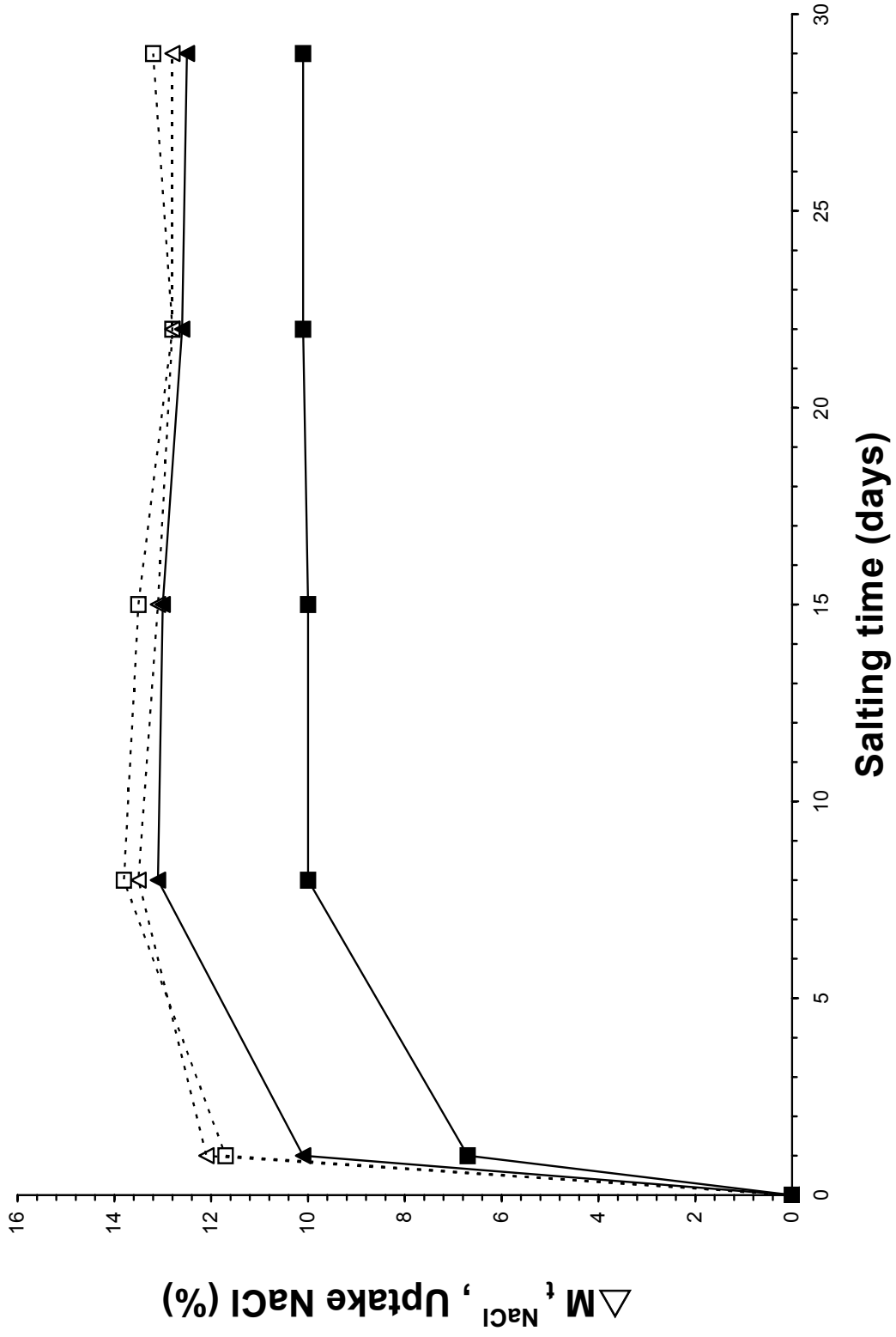


Figure 7

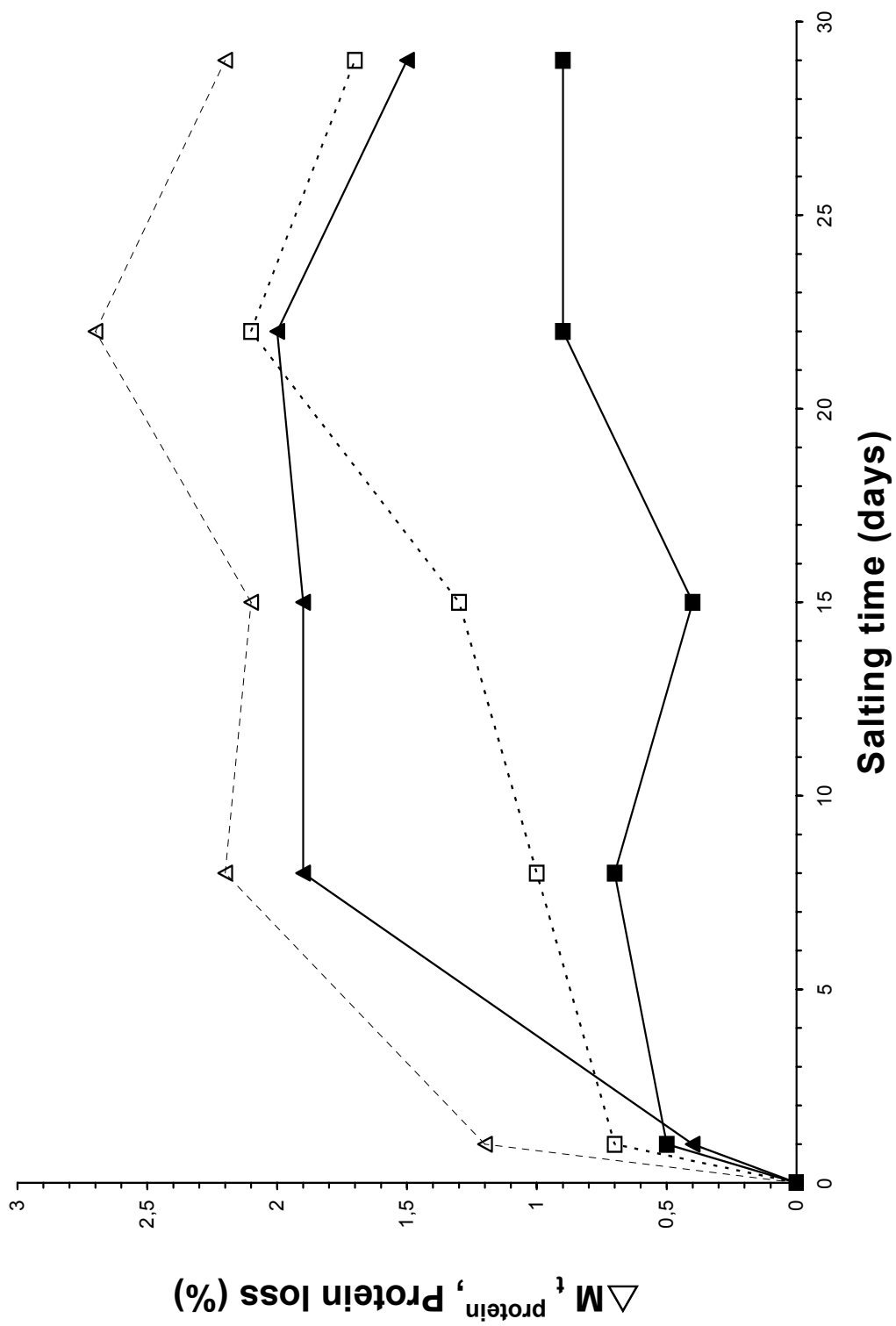


TABLE 1.

EFFECTS OF STATE OF RIGOR AND FREEZING PRIOR TO SALTING ON THE INSTRUMENTAL COLOUR VALUES, WATER AND NaCl CONTENT OF SALT RIPENED, SPLIT COD
(25-28 days of curing)

	Pre-rigor			Post-rigor:		
	Chilled ¹ (n=30)	Chilled ³ (n=30)	Chilled ⁴ (n=65)	Chilled ⁴ (n=65)	Frozen ⁸ (n=65)	Frozen ⁸ (n=65)
Instrumental colour values^A						
L*	62.5 ±2.6	54.0 ±3.3	50.7 ±2.8	50.7 ±2.8	52.2 ±4.6	52.2 ±4.6
b*	0.9 ±1.5	-2.4 ±1.4	-0.1 ±2.8	-0.1 ±2.8	-1.5 ±1.6	-1.5 ±1.6
Water ^B (%)	52.8 ±0.4	55.7 ±0.1	57.3 ±0.2	57.3 ±0.2	57.9 ±0.2	57.9 ±0.2
NaCl ^B (%)	18.4 ±0.3	20.0 ±0.4	19.7 ±0.5	19.7 ±0.5	19.8 ±0.2	19.8 ±0.2
$\Delta W_t^{\text{water}}$ (%)	- 47.1	- 42.3	- 41.8	- 41.8	- 40.5	- 40.5
ΔW_t^{NaCl} (%)	12.1	14.2	13.7	13.7	14.1	14.1

¹ Danish sein caught fish, gutted, split and salted 2 hours after death (pre-rigor)

³ Net-caught fish, gutted and stored in ice for 3 days prior to splitting and salting (post-rigor)

⁴ Danish sein caught fish, gutted and stored in ice for 4 days prior to splitting and salting (post-rigor)

⁸ Net-caught fish, gutted and stored in ice for 3 days, stored frozen for 11 days at -30°C, thawed in running fresh water for 1 day, stored in ice for 1 day and then split and salted (post-rigor)

Numbers 1, 3, 4 and 8 relate to the experiments described in Fig 1 and 2.

^A Instrumental colour determinations; 2 registrations on each of 15-20 split fish individuals (n=15-20) from each group.

^B Water and NaCl determinations; on pooled muscle samples, made by mincing 5 split fish individuals where the skin and bones had been removed (n=5).

The accumulated change in water and NaCl content from raw material to cured sample were estimated as described in Materials and Methods and given as: $\Delta W_t^{\text{water}}$ (%) and ΔW_t^{NaCl} (%).

TABLE 2.

EFFECTS OF FREEZING COD (SPLIT FISH) POST-RIGOR PRIOR TO SALTING
ON THE INSTRUMENTAL SHEAR FORCE VALUE, WATER HOLDING CAPACITY, pH, CALCIUM AND MAGNESIUM CONTENT
OF THE MUSCLE BY HEAVY SALTING

	Salting time:		
	0 day *	7 days	28 days
	Raw materials ^{3,8}	Chilled ³	Frozen ⁸
Instrumental shear force value (N)	13.2 ±1.8	45.0 ±11.7	51.2 ±3.5
WHC (%)	81.7 ±3.4	79.6 ±7.0	64.6 ±1.9
Ca (mg/100 g)	7.0 ±0.8	20.0 ±2.7	25.0 ±1.3
Mg (mg/100 g)	22.0 ±0.8	34.0 ±1.9	35.0 ±0.8
Muscle-pH	6.70	n.d.	6.33
		Chilled ³	Frozen ⁸
		60.6 ±6.0	71.1 ±6.0
		76.7 ±1.0	66.7 ±0.8
		29.0 ±6.9	30.0 ±3.1
		34.0 ±1.2	43.0 ±1.2
		6.23	6.21

n.d. = not determined

Sample size = pooled samples were made by mincing 5 individuals of split fish where skin and bones were removed, n=5.

Numbers 3 and 8 relate to the experiments described in Fig. 1 and 2.

³ net-caught fish, gutted and stored in ice for 3 days (post-rigor) prior to splitting and salting

⁸ net-caught fish, gutted and stored in ice for 3 days (post-rigor), stored frozen for 11 days at -30°C, thawed in running fresh water for 1 day, stored in ice for 1 day and then split and salted.

* values measured immediately before salting

WHC (%) = water holding capacity is retained water in g/100 g of weighted in water of pooled sample after centrifugation at low speed (210 g x 15 minutes)