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## Glazing of frozen fish: Analytical and economic challenges

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#### ABSTRACT

Adequate glazing (6–10%) of fish fillets prior to frozen storage protects the final product from dehydration, oxidation and quality loss. Excessive glazing (>12%) on the other hand may significantly affect the economic value and end user satisfaction of frozen fish fillets. This paper describes the optimization, validation and application of a gravimetric procedure for the quantification of the ice-glaze content of frozen fish fillets (accredited under ISO 17025). This procedure has been utilized to determine the glazing percentage of multiple batches (n=50) of 11 different fish species sampled from 2005 until 2009. Average glazing percentages were  $8.7 \pm 2.0\%$  for the pooled samples (n=712), and ranged between 6.6  $\pm 2.2\%$ (salmon/cod) and 10.6  $\pm 1.6\%$  (plaice). The lower threshold value of 6% glazing for sufficient protection was violated in only one batch, whereas none of the batches exceeded the 12% excessive glazing threshold. The annual market place value of one %-point glazing is estimated at 1 million Euro in a low to moderate fish consumption market like Belgium. The large variability of glazing, combined with this technology's possible implications with respect to end-product-quality and economic value urges for technology improvement, monitoring and more controlled application of the glazing process in the frozen fish industry.

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#### 1. Introduction

Consumer demand for seafood has steadily grown during the last decades [1]. This evolution was accompanied by a growing importance of frozen as opposed to fresh fish [1]. In 2006, 54% of the 110 million tons of worldwide fish produced for human consumption underwent some form of processing. The share of frozen fish in the total quantity of fish being processed before consumption amounted up to 42% in 2006 [1]. The success of this processing method may be explained by its efficacy with respect to the preservation of an otherwise highly perishable product. A progressive loss of intrinsic and sensory seafood quality has indeed been reported throughout chilled and frozen storage of fish [2,3]. One particular established technology generally applied during freezing and frozen storage of seafood is the application of a layer of ice to the surface of a frozen product by spraying or brushing on water or by immersing the product in a water bath [4], referred to as glazing.

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During frozen or cold storage, seafood products may develop surface drying and dehydration, which may lead to freezer burn, and may suffer from quality loss owing to oxidation or rancidity. Glazing of seafood products typically prevents the incidence of these processes during frozen storage [5]. The ice layer excludes air from the surface of the product and as such reduces the rate of oxidation [4]. Glaze is typically applied from 4% to 10% depending on the product, though ranges from 2% to 20% have been reported as well [6]. In extreme cases up to 25–40% glaze has been observed for some seafood products [4,7], although it should be noticed that seafood products such as shrimps and squid rings as a result of their high surface to volume ratios can have unavoidable water-ice glaze up to 25%.

Determination of the ice-glaze content of fish fillets is relevant for multiple purposes. Firstly, the degree of glazing affects the quality of the product offered; in particular a too low degree of glazing (<6%) may lead to a hampered protective function. Secondly, glazing is relevant from a market and economics perspective. Excessive glazing (>12%) might imply additional direct profits for sellers at the expense of buyers, which may lead to trade conflicts, and misleading of consumers. In any case, the risk of yielding customer dissatisfaction, either from inferior quality frozen fish caused by a too low degree of glazing, or from the perception of being ripped off since buying water for fish, is substantial. Moreover, a too high degree of glazing may contribute to the ecological foot-

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#### Table 1

Fish species or products for which the glazing percentages were determined; period 2005-2009.

English name	Dutch name	Scientific name	Number of pieces per bag	Average price (Euro/kg)
Anglerfish	Staartvis	Lophius piscatorius	8–10	28.70
Witch flounder	Hondstong	Glyptocephalus cynoglossus	16–23	7.80
Haddock	Schelvis	Melanogrammus aeglefinus	10-13	13.50
Cod	Kabeljauw	Gadus morhua	9–13	14.60
Rose fish	Roodbaars	Sebastes marinus	14–16	10.20
Ray	Rog	Raja clavata	8-9	7.80
Pollack	Koolvis	Pollachius pollachius	9–12	6.30
Wolf fish	Zeewolf	Anarhichas lupus	9	10.50
Plaice	Pladijs	Pleuronectes platessa	10-11	14.60
Salmon/witch flounder <sup>a</sup>	Zalm/hondstong	S.salar/Glyptocephalus cynoglossus	8-9	17.50
Salmon/cod <sup>a</sup>	Zalm/kabeljauw	S.salar/Gadus morhua	23–26	10.50

<sup>a</sup> Combinations of 2 fish species.

print of seafood since unnecessary amounts of ice (water) are being cooled, stored, shipped and transported. The latter argument for a better control of the glazing process is increasingly important, owing both to the increase in frozen fish trade and to the public debate concerning sustainability that is gaining momentum [8].

Although glazing is a widely applied technology for fish products whose market shares are increasing, surprisingly little research has been published in this domain, apart from a few notable studies focusing either on microbiological safety [9] or quality preservation, in particular lipid oxidation [10] effects from glazing. A number of methods exist for the determination of the net contents and glaze contents, notably for the case of frozen shrimps. The CODEX ALIMENTARIUS procedures developed by the FAO/WHO and applications thereof are among these [11,12]. Some more recent publications deal with the development and application of an enthalpy technique for measuring the glazing percentage of frozen shrimps [13-15]. But to the best of our knowledge, the application and/or accreditation of an analytical procedure for the determination of glazed fish fillets have not been reported yet. Therefore, the goal of this study was to develop, optimize and validate a procedure for the quantification of the glazing percentage of different types of frozen fish species. The accreditation under ISO 17025 of this validated procedure is reported and the results obtained with this technique over a period of five years as a quality control measure for the glazed fish products marketed by a major retailer on the Belgian market is presented. Finally, the economic importance of glazing is evaluated and discussed.

#### 2. Materials and methods

#### 2.1. Origin and preparation of commercial material

The different fish species used for commercial purposes in Belgium (Table 1) were captured in the North East Atlantic Region (FAO Statistical Area 27), except for the salmon, which was farmed in Chile. Upon catching, the fish was stored in ice up to three days before landing. Whole fishes were washed, cleaned and fish fillets were prepared whenever required. Upon cleaning the whole fish or fish fillets were deep frozen and merged in a 0 °C water bath for 30 s to apply an even layer of glaze. Glaze was subsequently allowed to set by storage at -18 °C, the whole fish or fish fillets packed in bags and transferred to the cold store.

# 2.2. Gravimetric procedure for determining the glazing percentage

The percentage of glazing was determined according to the following procedure. First, the frozen fish sample (at  $-18 \degree C$  or below) was removed from the freezer and its gross-weight (=*W*1) determined on a PB 3003-5 scale (Mettler Toledo S.A., Greifensee, Switzerland). Subsequently, the frozen sample was immersed into

a water bath (GFL mbH, Burgweidel, Germany) and gently agitated for about 30 s until all visible ice-glaze was removed. This was checked by carefully feeling the fish surface with the finger tips. When the smooth surface of the glaze disappeared and the rough surface of the fish itself could be felt, the deglazing procedure was stopped. Ideally, the water bath contains an amount of fresh water equal to about 10 times the declared weight of the product; the temperature should be adjusted to about  $20 \pm 2$  °C. Finally, the sample was carefully dipped dry (without pressure) with a cotton rag and the non-glazed or net-weight (= W2) determined. The percentage of glazing or glaze-weight relative to gross-weight was then calculated as follows:

$$\% \text{ Glazing} = \frac{(W1 - W2) \times 100}{W1}$$

The procedure for the determination of the percentage of glazing is described in the CODEX ALIMENTARIUS Committee for Fish and Fishery Products: CODEX STAN 190-1995 [11].

#### 2.3. Validation study

Validation studies were performed by using a batch of eight fish samples, consecutively re-glazed for every test phase by immersing the fish samples in a water bath at 0 °C and allowing the glaze to settle overnight. Precision of the gravimetric procedure, determined as relative standard deviation, was obtained from the repeated determination (n = 5) of the glaze content of the aforementioned batch by one analyst on three consecutive days (repeatability, n = 24) and by three different analysts on randomly selected days (reproducibility, n = 40). Recovery of the method was evaluated by comparing the repeated gravimetric results (n = 24) under reproducibility conditions with the initial glaze percentages, as obtained from the fish processing company.

#### 2.4. Data analysis procedures

The dataset used for analysis contained data from 712 individual pieces from 50 different batches (see Table 1 for information about the species). Data analysis procedures include descriptive statistical analyses with determination of mean weights, glazing percentages for individual pieces and batches, either pooled per species or time period. Standard deviations of glazing percentages are provided in tables or graphs as a measure of dispersion around the mean. The relationship between glazing percentage as dependent and net-weight, species and season as independent variables was analyzed through regression analysis using SPSS 15.0. Market data from FAO (2009) [1] and GfK (2009) [16] are used for the evaluation and discussion of economic implications.

### 42 **Table 2**

Descriptive statistics of the samples subjected to glazing determination (n = 712).

	Min.	Max.	Mean	SD
Gross-weight W1 (g)	29.00	349.00	124.72	69.76
Net-weight W2 (g)	26.00	326.00	113.67	63.34
Glazing percentage (%)	2.90	16.00	8.73	2.04

#### 3. Results and discussion

#### 3.1. Validation and accreditation of the fish glaze quantification

An optimized version of the CODEX procedure for the quantification of the percentage of glazing, as described above, has been validated at our laboratory. Since the procedure is gravimetric, an essential parameter for validation was the calibration of the balance used. In our laboratory, balances were calibrated daily with weights of 1, 5, 10, 50 and 100 g, respectively. The temperature of the water bath was also monitored and limits were set between  $18 \,^\circ$ C and  $22 \,^\circ$ C. Moreover, it was observed that the manner of dipping the sample prior to determining the net-weight (*W*2) was a crucial parameter. Initially, paper towels were utilized but pieces of paper tended to stick to the frozen fish causing deviations on *W*2 and thus underestimating the glazing percentage. Therefore we switched to cotton rags, which eliminated this problem.

Since there is no reference material available the determination of precision as described in the guidelines for single laboratory validation of methods [17] was performed by consecutively re-glazing the same pieces of fish within one batch under identical conditions, herewith minimizing the inherent variation in glaze application. Applying this design, satisfactory performance in terms of repeatability, reproducibility and recovery was shown. The RSD values obtained from repeatability and reproducibility studies ranged from 1.5% to 13.5%, with an average of 8.9% and from 7.8% to 19.1%, with an average of 13.6%, respectively. Recovery values of 87.4–121.4%, with an average of 99.9% and SD of 17.7% were obtained for the repeated glaze measurements.

Upon validation, the procedure was successfully accredited (ISO 17025) at our laboratory and has been employed for routine measurements for almost five years since 2005.

#### 3.2. Screening of glaze percentage of different fish species

The mean ( $\pm$ SD) glazing percentage across all samples was 8.73% ( $\pm$ 2.04) (Table 2). From the total of 712 individual samples analyzed, 8.3% had a glazing percentage below the 6% threshold for adequate protection, whereas 5.6% of the samples had excessive glazing beyond 12%. The glazing percentage of 86.1% of the individual samples fell within the 6–12% range (see also histogram in Fig. 1). It should be noted though that one fifth (20.5%) of the individual samples fell in the 10–12% glaze range.

As our data show large variations between the glazing percentages of different pieces in one bag (Figs. 1 and 2), average statistically relevant glazing percentages need to be determined to allow extrapolation from a single bag to an entire batch and an appropriate number of individual samples is requisite. Therefore, it was agreed with the retailer that only sampling of at least one whole bag (approximately 1.5–2.0 kg) of the same species or product per batch could lead to a representative result of the glazing percentage produced by the supplying seafood company. The initial data based on the analysis of merely two pieces of fish per batch were as a consequence omitted from our final datasheet as presented in Figs. 2–4, resulting in a final dataset of 712 values.

Glazing percentages of the different pieces from one bag may indeed easily differ 4–5%-points, as can be deduced from the standard deviations in Fig. 2. In addition, large variations in glazing



Fig. 1. Histogram of glazing percentage of individual samples (n = 712).

percentages between the different batches (bags at different time points) from one species were observed (Fig. 2). Indeed, the application of glaze has been reported to be difficult to control [12,13], and therefore the amount of glaze is generally not constant and the thickness not uniform.

A relevant finding in our data is the sinusoid trend in the glazing percentages over time, suggesting temporal variations in the glazing process (Fig. 3). This trend may be explained by seasonal variations, as the highest glazing percentages are visible in the early months of the year (i.e. winter period in Belgium). The potential of seafood products to freeze surface water is closely related to the product temperature before and after glazing [5]. Effectively, mean glazing percentages differed significantly between summer (June, July, August; mean glazing=8.16%; SD=2.13), mid season (other six months; mean glazing=8.61%; SD=1.95) and winter (Decem-



**Fig. 2.** Mean glazing percentages  $(\pm SD)$  of the different species of fish measured during this study for their respective batches.



Fig. 3. Evolution of the mean glazing percentages  $(\pm SD)$  of the different batches and species of fish over time.

ber, January, February; mean glazing = 9.55%; SD = 1.94) (ANOVA F = 20.59; p < 0.001). Differences in product temperature throughout the year may therefore explain the temporal variability in the glazing percentage observed.

Significant differences in glazing percentages over time or between different batches may have significant end-productquality and/or economic implications. As contractually agreed between the fish processing company (supplier) and the retailer (buyer), acceptable glaze percentages range between 6% and 10%. If the glazing percentages are below 6% or above 12% the batch could be rejected by the retailer, while for glazing percentages between 10% and 12% a financial retribution for the batch can be requested from the fish processing company. As can be seen in Fig. 2 only in one case at batch level (a salmon/cod sample) the mean value of the relative glazing weight is below 6%, while in 14 batch samples (out of the total of 50, i.e. more than one quarter) the mean value is ranging between 10% and 12%. The value of 12% glazing was however not exceeded in this series of analyses.

Excessive glazing has been has been reported for example for shrimps, with ice coatings as thick as 25–45% [18]. As a result, the Food Advisory Committee (FAC) addressed the problem and decided that standardized procedures for the determination of ice-glazing of shrimps needed to be introduced, i.e. the CODEX procedures. A more recent report from the import and export division

of Singapore however mentioned that the bulk of frozen sushi fillets had excessive glazing with 40% or more [7]. These data are indeed excessive in comparison to our data. It should be noted however that smaller pieces, i.e. fillets with a lower netweight such as is the case for sushi fillets [7] or other seafood products such as shrimps [18], generally yield higher glazing percentages due to their relative high surface area. The ice-uptake on a product is indeed determined by its surface area-to-volume ratio [5]. This high surface area-to-volume ratio of sushi fillets and shrimps, which is a direct consequence of their lower net-weight, was also the case for the salmon/cod fish batches that were tested during our study, but did not result in high or excessive glazing percentages (Fig. 4).

Multiple regression analysis with glazing percentage as dependent variable and net-weight, species and season as independent variables (with species and season dummy variables entered in block) yielded a model with  $R^2 = 0.298$ . An initial model with only net-weight as a predictor yielded a  $R^2$  of only 0.008. The  $R^2$  increments from adding season and species were however significant at p < 0.001 and amounted to 0.05 and 0.24, respectively. The regression coefficient of net-weight in the final model was -0.006(t = -3.26; p = 0.001) indicating a negative but only weak relationship between net-weight and glazing percentage. These findings confirm that weight in general is a relatively poor predictor of glazing percentage, whereas seasonal and between-species dif-



Fig. 4. Mean (±SD) percentage of glazing for the different species and batches of fish as a function of their mean (±SD) non-glazed net-weight (=W2).

ferences account for a much larger proportion in the variance of glazing.

#### 3.3. Economic importance of glazing determinations

As indicated in the introduction, adequate glazing and determination of glazing can have important economic repercussions. First, a too low as well as an excessively high degree of glazing as compared to contractual agreements may lead to substantial rates of rejection of consignments and related direct and indirect (e.g. transaction and opportunity) costs. Considering the analyzed samples at batch level, only in one single case such costs have been incurred during the considered time period 2005-2009. Note that if the determination of glazing had been performed on individual sample instead of batch level, about 13% of the batches would have been rejected based on unjustified grounds. This provides an additional economic rationale for determining glazing percentages on the level of a whole bag, i.e. a complete consumer sales unit. Nevertheless, for 28% of the batches (14 out of 50) a financial retribution could contractually be charged by the buyer at the expense of the seller. Hence, from the seller's or fish processing company's point of view, the negative financial impact of a difficult or inadequately controlled technological process can be quite substantive. Since these batches did not exceed the 12% glazing threshold, they were marketed to consumers as end users with a relatively high glazing percentage, thus relatively much water for fish. In economic terms, this represents a relatively small value of around 0.50 eurocent per consumer sales unit. However, in consumers' perception this excessive amount of water may be perceived more as rip-off than reality would suggest, especially for a product with an a-priori expensive image such as fish fillets [19].

Second, the total market volume of frozen finfish in Belgium of around 12,526 tons (gross-weight) with an average retail price of 8.21 Euro kg<sup>-1</sup> [16] represents a total annual market value of 102.8 million Euro [16]. Under the assumption that most of the frozen finfish underwent glazing, the total Belgium market place value of 1%-point glaze is estimated around 1 million Euro annually. Although speculative, extrapolation to the world seafood market would suggest that 1%-point glazing represents an annual business value of 2–4 billion Euro. Obviously, such economic values underscore the importance of adequate glazing technology, and monitoring and determination of the glazing percentage in particular.

#### 4. Conclusions

The objective of this study was to optimize, validate and apply an analytical procedure for the determination of the glaze percentage of frozen fish fillets. The successful validation (repeatability 8.9%, reproducibility 13.5%, recovery 99.9%) and accreditation (under ISO 17025) of an optimized version of the CODEX ALIMENTARIUS method [11] were reported. Over a period of five years the glazing percentage of 50 bags of fish of 11 different species representing 712 individual samples was determined using this method. In only

one case the lower limit of 6% glazing was not met. The upper limit of 12% was never exceeded at batch level, but in 14 cases a mean glazing percentage between 10% and 12%, leading to a possible retribution of the fish processing company to the retailer, was observed. The economic importance of an adequate glazing technology, its application and determination, have been highlighted. One %-point glazing is estimated to represent a marketplace value of 1 million Euro in a low to moderate fish consumption market like Belgium. The large variability of glazing as well between and within species as over seasons, combined with the substantial implications of glazing towards end-product-quality and economics urge for technology improvement, stringent monitoring and more controlled application of glazing in the frozen fish industry. Last but not least, business-to-business relations, reseller reactions and consumer perceptions towards excessive glazing merit attention in future research.

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#### References

- FAO, The state of world fisheries and aquaculture (SOFIA), Rome, FAO Fisheries and Aquaculture Department, 2009. http://www.fao.org/fishery/sofia/en.
- [2] O. Rodríguez, J. Barros-Velázquez, A. Ojea, C. Piñeiro, J.M. Gallardo, S. Aubourg, Proceedings First Joint Trans-atlantic Fisheries Technology Conference 33rd WEFTA and 48th AFTC Meetings, Reykjavik, Iceland, 2003, pp. 73–74.
- [3] E. Martinsdóttir, K. Sveinsdóttir, D. Green-Petersen, G. Hyldig, R. Schelvis, in: T. Børessen (Ed.), Improving Seafood Products for the Consumer, Woodhead Publishing, Cambridge, 2008, pp. 40–62.
- [4] Research and Development Fact Sheet Glazing, FS2-05-08 May 2008, Grimsby, Seafish.
- [5] S. Jacobsen, K.M. Fossan, J. Food Eng. 48 (2001) 227-233.
- [6] W.A. Johnston, F.J. Nicholson, A. Roger, G.D Stroud, FAO Fisheries Technical Paper. No. 340. Rome, FAO, 1994.
- [7] C. Lay Har, Import and Export Division for Director-General Agri-Food and Veterinary Authority of Singapore, 2007.
- [8] W. Verbeke, F. Vanhonacker, I. Sioen, J. Van Camp, S. De Henauw, Ambio 36 (2007) 580–585.
- [9] Y.C. Su, M.T. Morrissey, J. Food Prot. 66 (2003) 812–818.
- [10] A. Soyer, M.E. Sahin, Turk. J. Vet. Anim. Sci. 23 (1999) 575-584.
- [11] CODEX STAN 190-1995, Standard for quick frozen fish fillets, Codex Alimentarius Committee for fish and fishery products, FAO/WHO, Rome.
- [12] M. Kent, G. Stroud, J. Food Eng. 29 (2009) 313-321.
- [13] S. Jacobsen, K.M. Fossan, J. Food Eng. 40 (1999) 21-26.
- [14] S. Jacobsen, S.W. Pedersen, Food Sci. Technol. -Lebensm. -Wiss. Technol. 30 (1997) 578–584.
- [15] N. Antonacopoulos, Deut. Lebensm. -Rundsch 89 (1993) 81-82.
- [16] GfK, Household consumer panel data: GfK ConsumerScan. GfK Panel Services Benelux. Brussels. 2009.
- [17] M. Thompson, S.L.R. Ellison, R. Wood, Symposium on Harmonization of Quality Assurance Systems for Analytical Laboratories, Budapest, Hungary, 1999, pp. 835–855.
- [18] I.P. Beckman, P. Mattson, Var Föda 32 (1980) 530-532.
- [19] K. Brunsø, W. Verbeke, S.O. Olsen, L. Fruensgard-Jeppesen, Br. Food J. 111 (2009) 669–685.