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FHF Project 901638

Studies on moisture levels in clipfish (cod and saithe) and methodologies applied by customs authorities (CLIPTURE)

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1. Summary

NORWEGIAN

Implementeringen av den brasilianske forskriften som omhandler saltet og saltet og tørket fisk innebærer et skille for saltfisk- og klippfiskvarer basert på grad av fuktighetsinnhold. Klassifiseringen innebærer et skille for saltfisk der fuktighetsinnholdet er høyere eller lik 53 g/100 g, og klippfisk med fuktighetsinnhold som er lavere enn 53 %-terskelen. Klassifiseringen medfører ikke bare ulike tollsatser, men gir også kommersielle konsekvenser ved POS. Usikkerhet om hvilken målemetode som anvendes for kontroll av vanninnhold forårsaker handelsproblemer for norsk klippfiskindustri.

For å vurdere avvik i målemetodene som anvendes ble analyse–av representative prøver fra tre størrelseskategorier for klippfisk av torsk (8/10, 7/9, 10/12) og klippfisk sei (7/9, 10/12, 16/20) fra seks norske eksportører gjennomført. Resultatene fra bruken av metoden og prøvepreparatet, som er nedfelt i Codex Stan 167/1989, avdekket at middelverdiene for fuktighet for kategoriene er under 53 %, men nærmer seg den kritiske grensen, spesielt for torsk 8/10. Det ble registret en forurolingende grad av manglende overholdelse av den brasilianske forskriften for noen størrelsesklasser, samt betydelig heterogenitet mellom produsenter.

Enkelte land har i likhet med de brasilianske myndighetene iverksatt forskjellige og spesifikke betingelser for fuktighetsanalyse av klippfisk (gjøres ved prøveuttak). Imidlertid fører bruken av den brasilianske metoden til et gjennomsnitt som er 1,6 g/100 g høyere enn bruken av referansen nedfelt i CODEX-metoden. Derfor vil grensene for manglende overholdelse være betydelig høyere for alle størrelseskategorier.

Tverrsnittsmetoden i Codex Stan 167-1989 ble definert for å håndtere den langsgående variansen i fuktighetsinnholdet i klippfisk av torsk, slik det er dokumentert i denne studien. Imidlertid er metoden vanskelig å gjennomføre, og i tillegg kostbar, upresis og vanskelig å ta i bruk for standardlaboratorier. Derfor foreslås endringer i standardprosedyren.

ENGLISH

The implementation of the new Brazilian regulation makes a distinction in customs categories for *peixe salgado* (salted fish) with moisture contents higher or equal to 53 g/100g, or *peixe salgado seco* (dried salted fish) with moisture contents below this threshold. The appropriate allocation of fish materials to one or other customs item not only entails different tariff duties but also has commercial and labelling implications, causing some trade issues to the Norwegian clipfish industry.

In order to assess the problem, the analysis of representative samples of three size categories for dried salted cod (8/10, 7/9, 10/12) and saithe (7/9, 10/12, 16/20) from six Norwegian exporters has been carried out. Results from the application of the method and sample preparation laid down in Codex Stan 167/1989 reveal that mean moisture levels for the categories are below 53% but approach the critical limit especially for the cod 8/10 size class. Disturbing non-compliance rates to the Brazilian regulation have been registered for some size classes as well as considerable heterogeneity between producers.

Certain countries, like the Brazilian Authorities, have implemented different and specific conditions for the analysis of moisture content in salted fish. However, the application of the Brazilian method leads to a mean 1.6 g/100g higher result than the use of the reference Codex Stan 167/1989 method, and therefore the border non-compliance rates would be significantly higher for all size categories.

Codex Stan 167-1989 cross-section method was designed to deal with the longitudinal variance in the moisture content in dried salted cod, as it has been documented in this study. However, it is hard to put into practice, costly, unprecise and difficult to be adopted by standard laboratories, so changes to standard procedure are suggested.

2. Introduction

Brazil is one of the most important markets for the Norwegian clipfish industry. The Brazilian Government made effective the new technical regulation for salted fish and clipfish by publishing the *Instruçao Normativa nº1, do 15 de janeiro de 2019*. Among others, this regulation listed denominations for different salted fish cuttings, unacceptable defects, and sampling and microbiological criteria to be applied by custom authorities. It also laid down the differentiation between dried-salted fish (*peixe salgado seco*), which can contain up to 52,9% moisture content, and salted fish (*peixe salgado*) which may vary between 53% and 58%. The categorization of goods in one or other category leads to a different tariff duty.

Companies exporting saltfish or clipfish to Brazil shall therefore have controlled drying processes and uniform product lots to prevent any custom issues. FHF project no. 901307 updated the analytical data of four species of dried salted fish exported by Norwegian companies. Among other results the average moisture contents of dried-salted fish revealed levels bordering the new Brazilian criteria, as well as differences in the degree of drying between different producers for the same product category. The moisture method used and the sample preparation used in this previous project was based on the cross-sectional method of the Portuguese "*Decreto Lei 25/2005*"[1] but with some modifications.

Some companies informed of certain discrepancy on the analytical methodologies to measure moisture content put into force by different Brazilian customs offices, and this dissimilar criterion may have been causing uncertainty and customs issues to the Norwegian industry.

The Brazilian Agriculture Ministry (*Agricultura, Pecuária e Abastecimento*) includes in its *Official Method Manual for the analysis of Food products from animals*[2] detail of the procedure that should be implemented by official Brazilian laboratories (LFDAs & accredited). This document acknowledges either *ISO1442:1997* or *AOAC 2008.06* as valid general methods to determine the moisture content in general food samples; but, considers the *AOAC 950.46B (Moisture in meat)* method as the one to be applied to salted and dried salted fish products.

Moreover, and even though it lays down that the cross-section method described in the CODEX-STAN 167-1989 can be implemented, it also includes some additional methodological aspects (see *Table 1)* that vary from this reference and can eventually lead to misunderstanding by laboratory operators. It should also be mentioned that in addition to imported clipfish, the salted fish category in Brazil includes different species ranging in size and final presentation to consumers, so to define a common sample preparation to all products becomes complicated.

The moisture content methods based in oven drying are probably one of the simplest methods in food analysis, but when applied to clipfish some discrepancies between methods may arise, especially in the laboratory sample preparation. Some of them are summarized in table below.

[1] *Decreto Lei nº 25/2005 de 28 de janeiro do Ministério da Agricultura, Pescas e Florestas. Diário da República – I Série-A. Nº20 .*

[2] *http://www.agricultura.gov.br/assuntos/laboratorios/legislacoes-e-metodos/arquivos-metodos-da-area-poa-iqa/ManualdeMtodosOficiaisparaAnlisedeAlimentosdeOrigemAnimal2ed.pdf*

Table 1: Comparison of moisture content method references applied to salted and dried-salted fish.

	Decreto Lei nº 25/2005 (Portugal)	***Codex Stan 167 / 1989***	***Brazil (Salted & dried-salted fish) (Manual de Análise de Alimentos de Origem Animal - 2º edition - MAPA Brazil-2019 + AOAC 959.46B).***	***ISO 1442:1997. Meat and meat products - Determination of moisture content.***
Cross Section	Withdraw the surface salt excess	Brush the surface salt	Withdraw the surface salt excess.	Sampling is not defined in this reference Std. Suggested sampling method ISO 3100-1. Al least 200 g for a representative sample.
	From pectoral to anal fins	20 mm from earbones to tail body end (caudal fin excluded)	- 3 transversal sections (25 mm): Next to Dorsal fin, between dorsal fin and annus, after annus. - Use the cross-sectional method from Codex STAN 167/1999.	
	20 mm sections separated by 40 mm	2 mm sections separated by 40 mm	25 mm sections or Codex STAN 167/1999.	
Sample selection	Skin & Bone included	Skin & Bone included	Skin & Bone **excluded**	Not defined
Cutting	Mechanically grinded	Cut with scissors.	Mechanically grinded	Use of mechanical grinder is allowed
Sample Weigh	10 g	Unspecified (Use of all material from sections)	Supposedly 2 g (*AOAC 950.46B*)	5-8 g
Use of treated sand	20-30 g	No sand used	No sand used	15-32 g. 3-4 times sample weight.
Replicates / sample	2 replicates /sample	2 replicates /sample	-	2 replicates /sample

During the preparation of this proposal, e-mail and telephone contact to some Brazilian official laboratories (*Laboratórios Federáis de Defesa Agropecuária (LFDA/SDA*)) has been carried out. From the information gathered, it seems that since 2019, and as a consequence of a claim from Norwegian Authorities, the reference moisture content method for "bacalhau" changed from ISO 1442:1997 to AOAC 950.46B with the sample preparation also shifting from AOAC 937.07 to Annex B of the Codex Stan 167-1989. These changes were, therefore included in sections 5.25.1, 5.1.1 and 5.1.3 of the last version of the *Official Method Manual for the analysis of Food products from animals* [2], but in a rather unclear way, and this is probably causing discrepancy across Brazilian laboratories regarding sample preparation of "bacalhau" materials for moisture content analysis. It has also been appointed that the Brazilian Authorities are presently studying whether the analytical results from the application of different procedures differ, which is one of the objectives of this project.

Project organization

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3. Objectives

Appropriate labelling and trade is within the priorities of the "clipfish and saltfish" sector in 2020. As a tool to assess potential trade issues and promote fairness, effectivity and homogeneity in the inspection of imported clipfish by custom authorities in third countries, the objectives of this proposal become necessary.

1. To provide, with a scientific approach, the difference between moisture methodologies and consequent deviation acceptance used as a basis for correct labeling of clipfish or saltfish exported to Brazil.
2. To document nonconformities/variation in test results due to size and species.
3. To compare differences in moisture evaluation derived from the application of the Annex B of CODEX STAN 167/1989 vs. present Brazilian Methodology.
4. To calculate the longitudinal variation in moisture content in a split clipfish piece.
5. To define a new sampling procedure that could be easier, avoids misunderstandings and show same results as the cross-sectional method from CODEX STAN 167/1989 –Annex B.
6. To write a scientific publication to be used by the industry when meeting Food Authorities in Norway and in Brazil.

4. Project execution

The project has been structured in two separated but interconnected tasks. The first one intends to define the moisture content levels and variation in clipfish (cod and saithe) produced by the Norwegian industry based on the method laid down in Codex Stan 167-1989. The second task investigates the effects of different sample preparation, current methodology applied by Brazilian Authorities and the aforementioned Codex method, on final moisture contents. In addition the change in moisture content along fish length is also tested.

Sampling

Six Norwegian producers supplied the project samples (350 kg.) corresponding to dried salted cod and saithe. Three size categories for cod (8/10, 7/9, 10/12) and saithe (7/9, 10/12, 16/20) were included. Each one of the companies selected for each species and size category collected four salted fish samples from different production lots. Therefore 60 analyses for each of the two species were carried out as part of work package one.

Table 2: Sampling scheme of task 1.

COD				
8/10	5 companies	4 samples /company	20 samples	60 cod fishes
7/9	5 companies	4 samples /company	20 samples	
10/12	5 companies	4 samples /company	20 samples	

SAITHE				
7/9	5 companies	4 samples /company	20 samples	60 saithe fishes
10/12	5 companies	4 samples /company	20 samples	
16/20	5 companies	4 samples /company	20 samples	

In addition, the twenty fish samples of the 8/10 category of cod were additionally included in task 2.

Table 3: Sampling scheme of task 2.

		Codex Stan 169 - 1989					Analysis Brasil
COD	Size class	Sampling	Analysis CODEX (x2)	Anterior	Media	Posterior	Mix (x2)
Company 1	8/10	4	4 x 2	4	4	4	4 x 2
Company 2	8/10	4	4 x 2	4	4	4	4 x 2
Company 3	8/10	4	4 x 2	4	4	4	4 x 2
Company 4	8/10	4	4 x 2	4	4	4	4 x 2
Company 5	8/10	4	4 x 2	4	4	4	4 x 2
		20	40	20	20	20	40

Task 2 totals 140 analyses. The average results of duplicates of the implementation of the Codex method in 8/10 cod samples are complementary included in task 1.

The collection of the samples and shipping of the materials from Norwegian companies was coordinated by FHF. Materials were received at ANFACO-CECOPESCA facilities and stored in a cool chamber.

The storage conditions were registered by means of a temperature and relative humidity sensor. The temperature was maintained between 2,2 and 3,4 °C, but the average relative humidity inside the cool chamber (RH= 60,5%) what was below the desired level (75-78%). In order to prevent moisture loss, boxes were sealed and covered with plastic bags. The analysis of the full sample set was executed in three months from arrival.

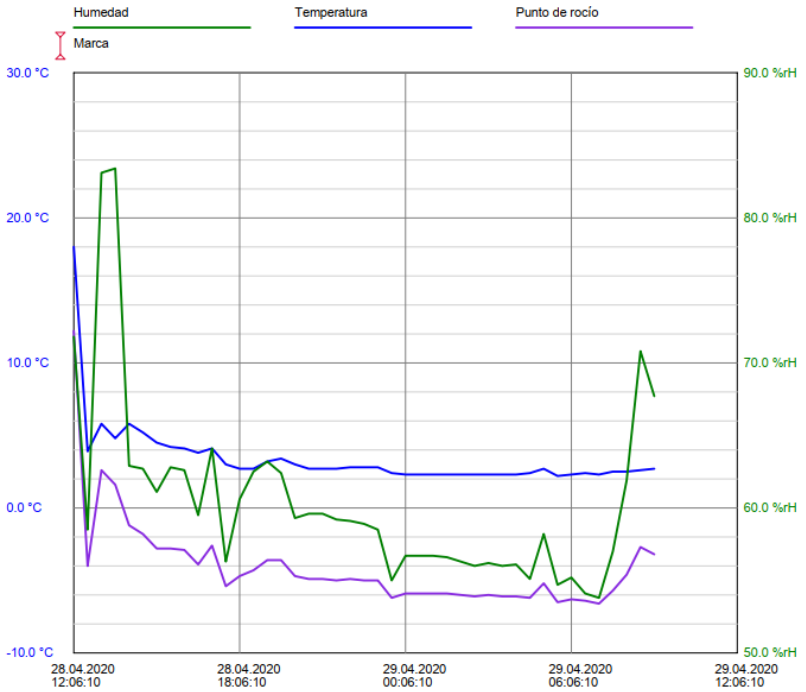

Figure 1: Register of storage conditions of clipfish samples inside the cool chamber.

Sample preparation for Codex analysis

In order to get the laboratory samples for Task 1, the procedure detailed in Codex Stan 167-1989 was implemented.

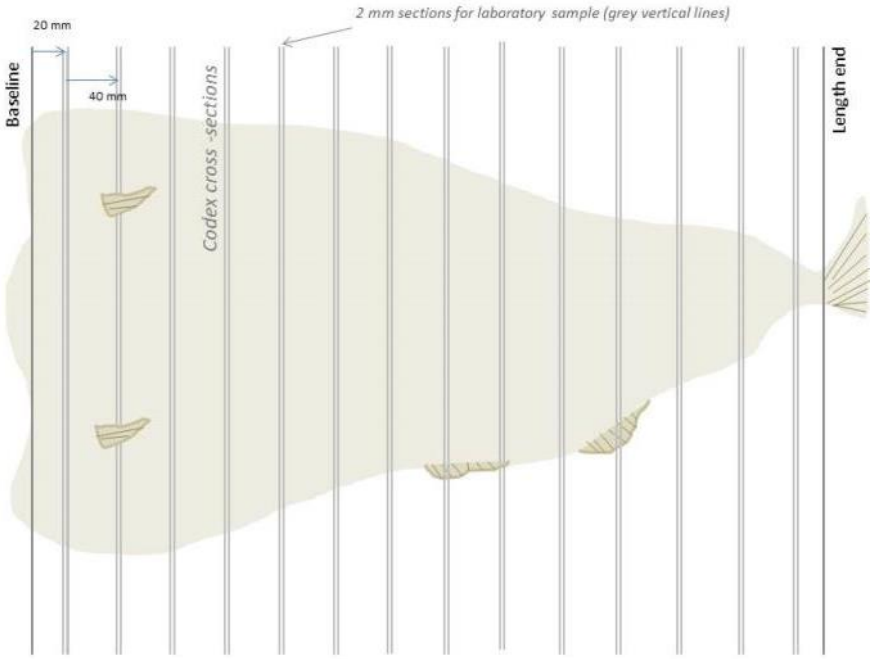

Figure 2: Codex Stan 167/1989 cross-sectional method.

Samples were selected and internally codified, and then surface salt was gently brushed away. Fish weigh and the length from the cleft in the tail and a line drawn between the tips of the earbones were registered.

Figure 3: Sample codification, salt brushing, weighing and length measurement.

The cross section method was applied using a mechanical band saw. As it would be more in depth detailed in the discussion section, the cross sections selected to create the laboratory sample were as

thin as possible but thickness was not very precise since it was difficult and dangerous to perform 2 mm cuts.

Figure 4: Laboratory sample preparation according to Codex Stan 167-1989.

All cross sections, including bone and skin, were cut in small pieces using scissors, then were collected inside a sealed plastic bag and immediately processed in the laboratory.

Sample preparation for method comparison

Twenty dried salted cod fillets corresponding to 8/10 size class were sectioned and different parts were selected. Cross-section method following Codex Stan 167-1989 detailed above was also carried out but in this case, two separate samples were made from the same homogenate in order to get a duplicate analysis.

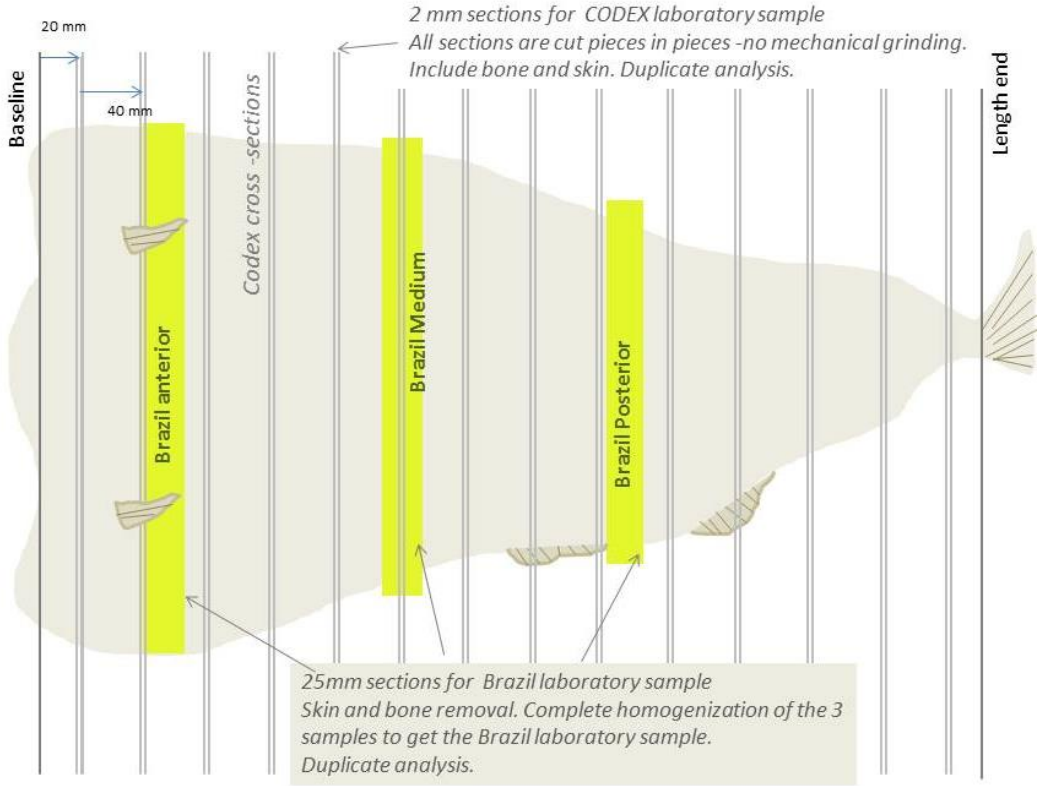

Figure 5: Sample preparation for method comparison.

The **Brazilian methodology** was based in sections 5.25.1, 5.1.1 and 5.1.3 of the last version of the *Official Method Manual for the analysis of Food products from animals* [2], as well as detailed information

obtained from direct contact to Brazilian technicians. Three 25 mm wide sections were selected to create an aggregate sample; one just after the pectoral fin, another one at the anus, and the remaining at the middle position between the other two sections. Bones and skin were removed and a mechanical grinder was used for the homogenization of the three sections (full speed – 20 s). Two samples were also made of from the same homogenate in order to get a duplicate analysis.

Sample preparation for the study of longitudinal variance

On the same fish that was used for method comparison, three sections of 20 mm were selected to compare the variation in moisture content along the fish length. The **anterior** one at a 15% length of the total length measured as detailed in Codex Stan 167-1989, **medium** at 50% and **posterior** at 80% of the total length. Samples corresponding to sections were processed as laid down in Codex Stan 167-1989; including bone and skin and avoiding mechanical grinding.

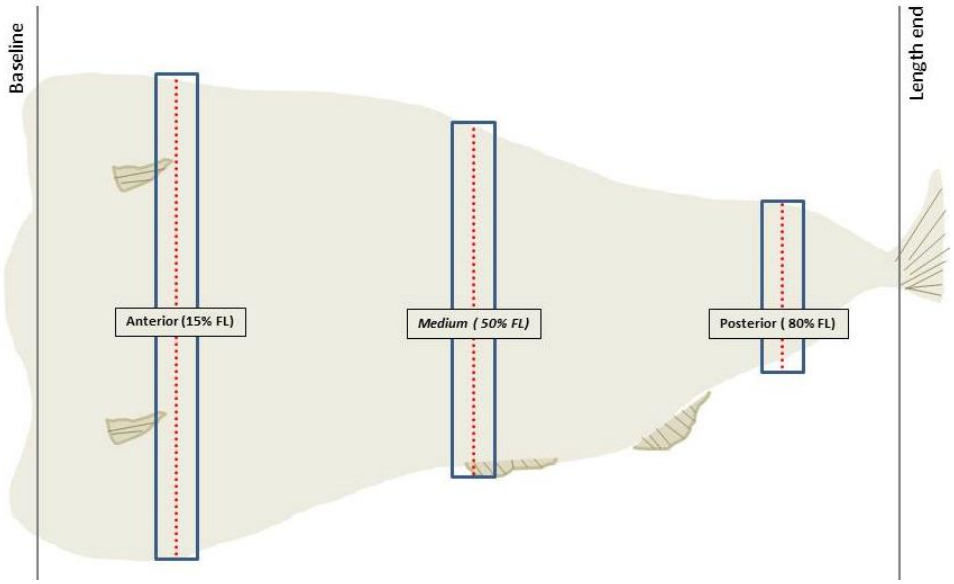

Figure 6: Defined sections for the study of the moisture content longitudinal variance in clipfish.

Seven samples from each fish were obtained; two replicates of the CODEX sample cross-section method, two replicates from the homogenization of the three sections of the Brazilian protocol, and three samples for the longitudinal variance study.

Figure 7: Seven samples obtained from each of the 8/10 class cod individuals.

The moisture method applied is under accreditation by the Spanish National Accreditation body – ENAC. It basically consists in weighing around 20 g (no additional laboratory homogenization is required) of sample in pre-tared and pre-dried basin. The sample is dried in an oven at a controlled temperature of 103ºc overnight until constant weight is registered in two consecutive measurements.

5. Results and discussion

Length, weight and moisture content results.

In addition to the moisture content, the application of the Codex Stan 167/1989 method involves recording of fish weight and length. The mean results of the twenty analyses carried out for each class is displayed in Table 4 and figures below.

Table 4: Mean length and weight for cod & saithe in each size class.

		N	Mean Lenght (cm)	Mean Weight (g)
8/10	Cod	20	71,9	5193
7/9	Cod	20	60,9	2938
	Saithe	20	57,6	3208
10/12	Cod	20	56,0	2302
	Saithe	20	50,8	2208
16/20	Saithe	20	44,1	1454

The obtained Pearson correlation coefficient for length and weight data gets to 0,905 demonstrating that, as expected, these are strong positively correlated variables. Besides, the internal variance increases as the size class is bigger, so 8/10 class includes more different fish (based on weight and length) than 7/9, 10/12 or 16/20, and this occurs either for cod and saithe.

Based on the length data, considering the same size class (7/9 and 10/12) cod is significantly longer than saithe (*t-student test* ($p<0.05$)) but this does not occur for weight data.

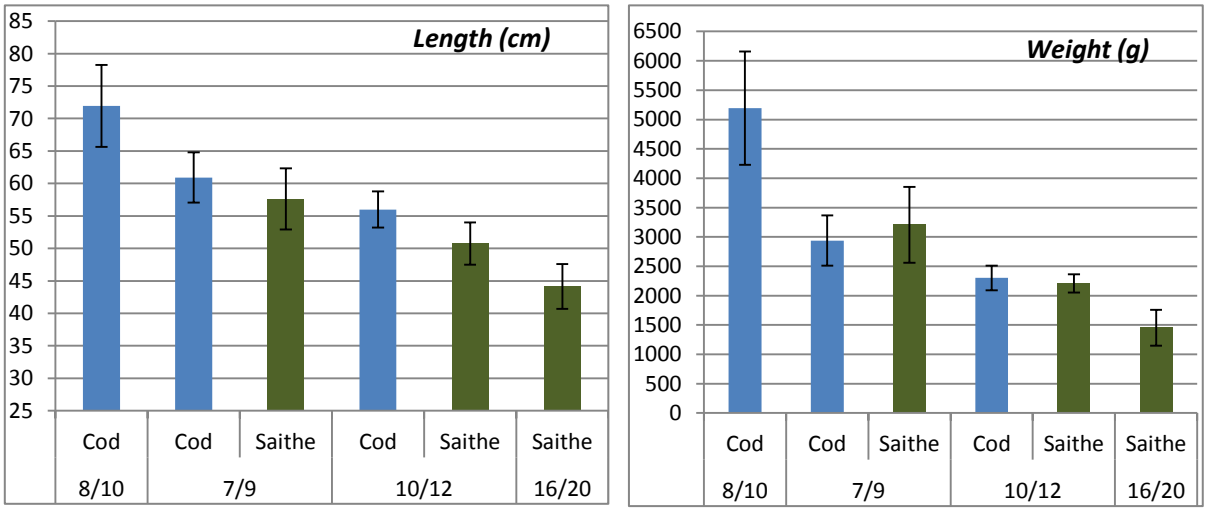

Figure 8: Length and weight of cod and saithe for each size classes .

From the application of the method laid down in Codex Stan 167-1989, the mean moisture content values are below the 53% threshold defined in the Brazilian legislation for heavy salted fish. However mean values considerably approach to this critical value especially for the 8/10 size class for cod, causing that 7 of 20 sample results were higher than 53 g/100g, which means that a 35% of the production would not be appropriately classified. Based on the statistical distribution of the results in each class we can estimate the percentage of samples that would not comply with the dry-salted category by the Brazilian legislation.

Table 5: Mean and SD for moisture levels in dried salted cod and saithe size classes. Observed and estimated moisture content compliance rates with the Brazilian regulation.

Size class		N	Moisture Mean (g/100g)	Moisture SD (g/100g)	Number of samples not complaying with the Brazilian regulation	% of samples beyond the 53% limit (estimated statistically)	Statistical method.
8/10	Cod	20	**52,5**	1,3	7 (35%)	**35%**	*T-test*
7/9	Cod	20	**51,0***	1,1	0 (0%)	**4,2%**	*T-test*
10/12	Cod	20	**50,7***	1,3	2 (10 %)	**4,8%**	*T-test*
7/9	Saithe	20	**50,5***	1,6	1 (5%)	**7,2%**	*T-test*
10/12	Saithe	20	**49,5****	1,6	0 (0%)	**2,0%**	*T-test*
16/20	Saithe	20	**49,8****	1,8	1 (5%)	**4,0%**	*T-test*

**, ** results not statistically different.*

The compliance rates show remarkably high percentage of non-compliance (35%) for the 8/10 class, and around 2-5% for the rest of the groups. It should be discussed if this risk of non-compliance is assumable by the Norwegian industry or to the contrary drying processes should be extended or modified.

Based in the t-student distribution the 8/10 class mean values are significantly different *($p<0.05$)* to the rest of the groups but, no significant differences were found between the moisture content levels in the 7/9 size class for cod and saithe, and with the 10/12 size class for cod *($p>0.05$)*. The contrary occurred in the 10/12 category where saithe and cod mean values significantly differed *($p<0.05$)*. Finally, saithe mean moisture content seems also not to differ between 10/12 and 16/20 size classes *($p>0.05$)*.

Internal variability and between companies.

The data obtained was also used to determine and compare the variability in moisture content from production lots from the same company. To do this the standard deviation (SD) of the four samples for each size class corresponding to different production lots of the same company was calculated and averaged. The assigned value for each company is presented in *Fig. 9* below.

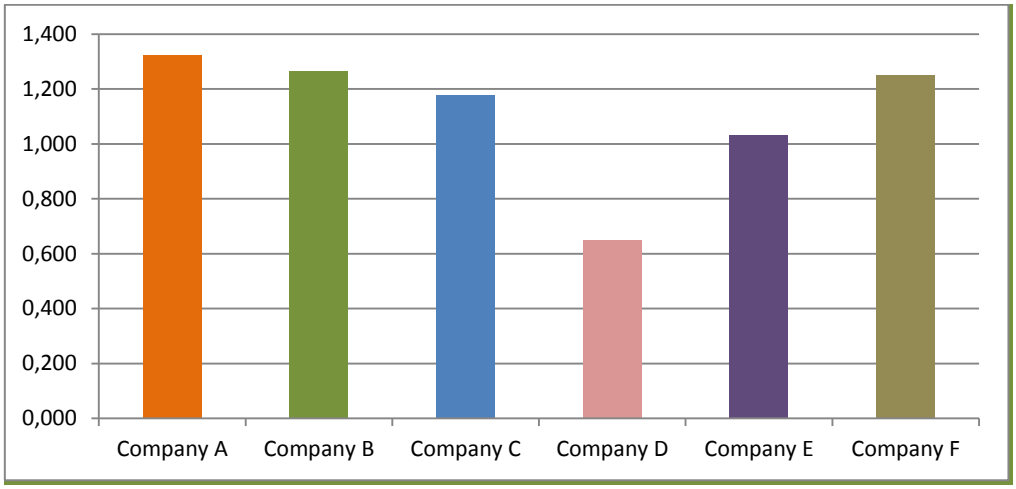

Figure 9: Internal variability (SD) in production lots for each of the companies.

With the exception of Company D, whose precision in production nearly doubles the rest of the companies, the variation approaches a mean value of 1.12 g/100g, which is reasonably low considering the method uncertainty and the nature of the materials and production process.

It is also interesting to examine the differences between companies. To do this a BIAS was calculated for every sample as a subtraction of the moisture content to the mean moisture content of the whole size class (all companies). The biases corresponding to the same company are then averaged and mean values are presented in figure below.

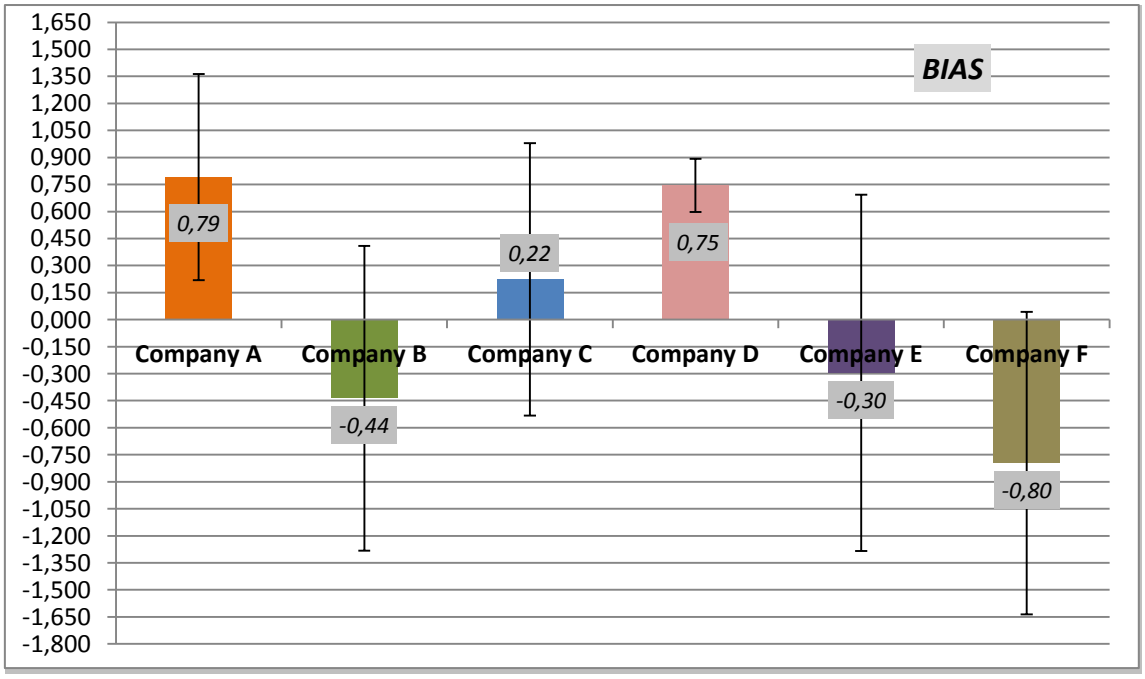

Figure 10: Variation of moisture content values between participant producers.

Companies A and D show higher moisture content results in dried-salted products than the rest of the companies (0,79 and 0,75 g/100g higher respectively) . To the contrary, company F seems to have a more intense drying process attending to the mean moisture contents (–0,80 g/100g below average) of its samples. As it can be seen on *Figure 10*, attending to their mean results and internal variance of their data, the rest of the companies seem to be closer to mean data for the size class.

Moisture content variation in the fish length.

In order to check the differences in moisture content and the effect of the drying process along the fish length, three sections were collected in each one of the 20 dried cod samples. As it was detailed in section 4.2 , a first **anterior** section at 15% length of the total length measured as detailed in Codex Stan 167-1989, a **medium** at 50% and a **posterior** section at 80% of the total length are taken. These samples were processed and analyzed as detailed in Codex Stan 167-1989.

The mean results of the samples for the anterior, medium and posterior part were 52,4 %, 51,7% and 48,5% respectively. By using a *T-student distribution* (*n=20; p<0.05*) means from groups were compared for equality. Despite it seems there is a decreasing gradient of moisture levels as we move along from anterior to the posterior parts of the fish, statistical significance of the differences were only found for the posterior part to the other two sections. No significant differences were found between the anterior and medium parts.

However, the calculation of a bias as the subtraction of the result of every section to the result obtained from the application of the Codex Stan 167/1989 cross-section method in the same sample is presumed to be more indicative. Therefore, **the mean deviation to the Codex method result was -0.09 for the anterior part, -0.87 for the medium and -4.02 for the posterior part of the fish.** In this case, if we compare the mean bias for the anterior, medium and posterior part (*t-student* (*n=20; p<0.05*) we find that the differences are significant among all groups, but it should be mentioned that the anterior and medium groups show statistical values close to the critical limit of significance (*One-way ANOVA: α=o,o5*). The differences of moisture contents at different lengths are correlated with the thickness of the fish at the point of sampling. In the 8/10 cod size class used for this study, the thickness at medium locations in fish was sometimes thicker than the anterior part.

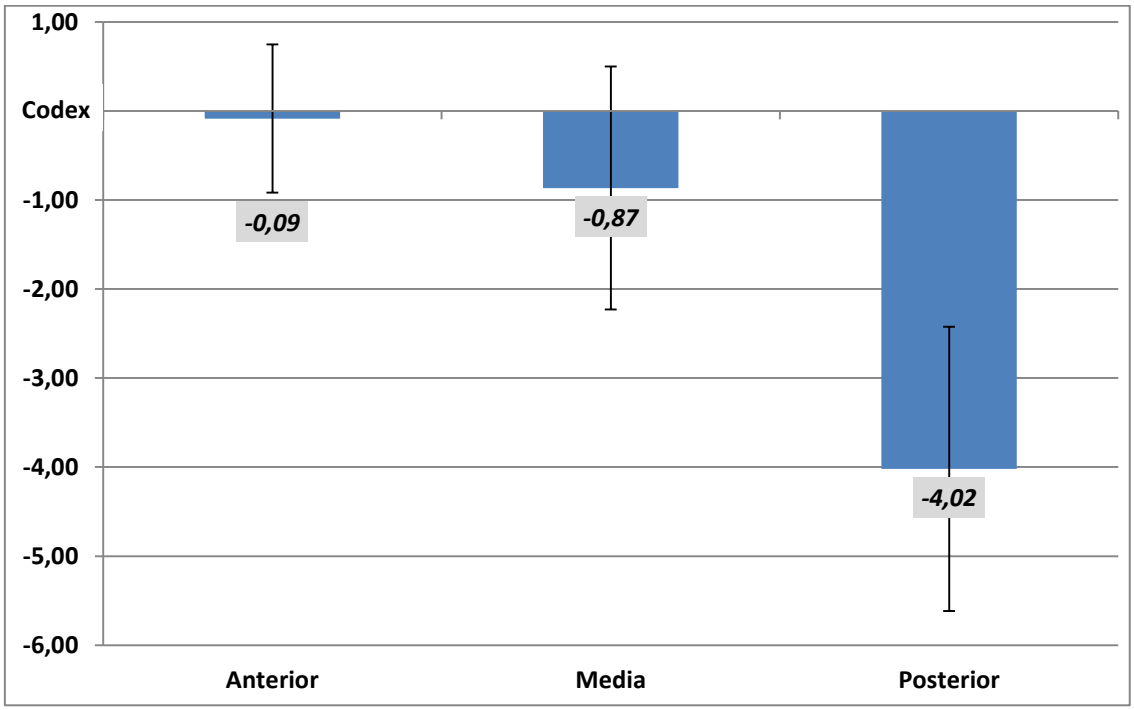

Figure 11: Mean calculated bias from Codex results for anterior, medium and posterior sections.

The anterior section is the area that best resembles the mean moisture content when the cross-section method from the Codex Stan 167-1989 is applied. However, further study should be carried out to confirm this also occurs in other size classes.

Differences between the Brazilian and Codex Stan 167/1989 sampling methodologies.

The main differences between these methods correspond to sample preparation for analysis, as it has been previously detailed in paragraph 4.3. The Brazilian method excludes bone and skin and uses a mechanical grinder obtaining more homogeneous laboratory samples. The samples under the Codex Stan 167/1989 method conditions are made up of bigger and heterogeneous pieces including bones fragments and skin strips (*see Figure 7).* This is the reason behind the great differences found in the precision of the final results (replicates from the same laboratory sample). **The precision error for Codex method (SD=1,10 g/100g) nearly doubles the one obtained for the samples processed under the Brazilian method (SD=0,58 g/100g).**

To compare the method performance a BIAS was calculated as the subtraction of the result from the Codex Stan 167/1989 to the result from the implementation of the Brazilian method on the same fish. Then a mean BIAS was calculated based on the 20 samples results. **The Brazilian method gives a 1,63 ± 0,76 g/100g higher result than the CODEX method**, with all the individual results higher for the Brazilian method than the CODEX method (all bias were positive). The differences in the method performance are statistically confirmed (*One-way ANOVA: α=0.05).*

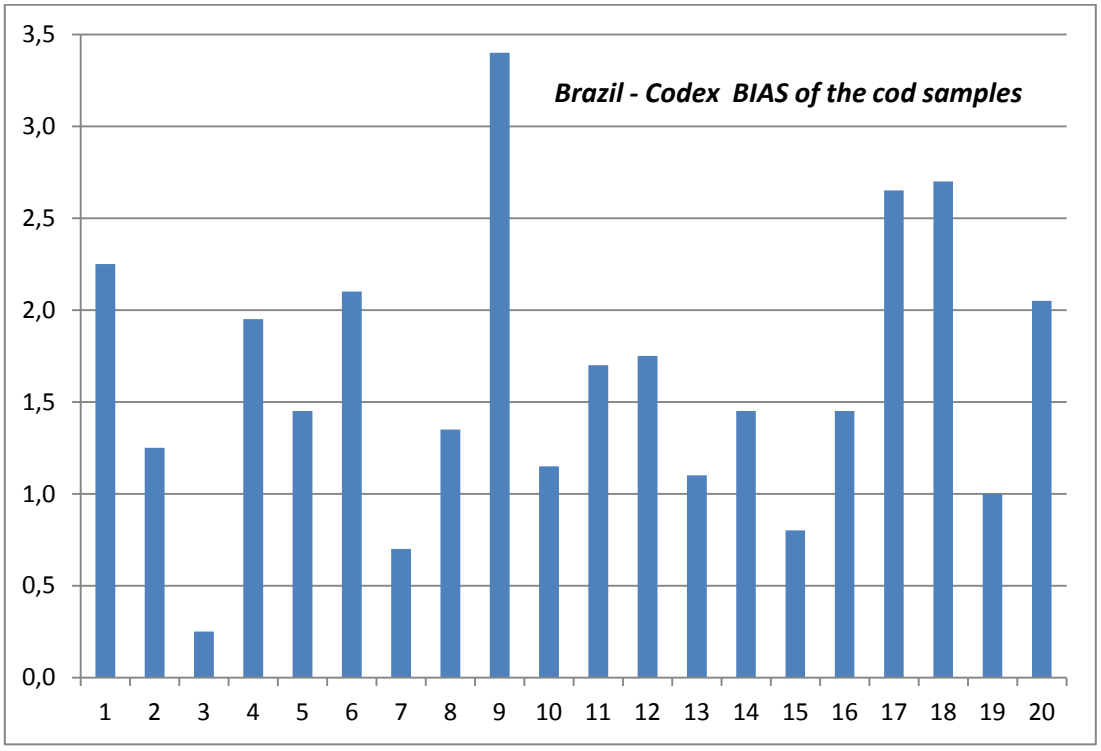

Figure 12: Individual bias (Brazil vs.Codex) of the samples analyzed.

It is convenient at this point to recall some data from the previous FHF project no. 901307[3]. The analysis of different salted and dried salted products had been carried out using the Portuguese method from *Decreto Lei 25/2005*, with the exception of excluding bone and skin. The comparison of the mean moisture contents displayed in *figure 13* of dried salted cod (*type B = 53,2 g/100g*) and saithe (*53,6 g/100g*) with the corresponding samples according to size in the present project (10/12); cod (*50,7 g/100g*) and saithe (*49,5 g/100g*) reflects that the method, and more specifically the sample preparation, critically conditions the final results obtained.

[3] *Updating of analytical data for the nutritional labelling of traditional (klipfish, saltfish, stockfish) fish.* https://www.fhf.no/prosjekter/prosjektbasen/901307/

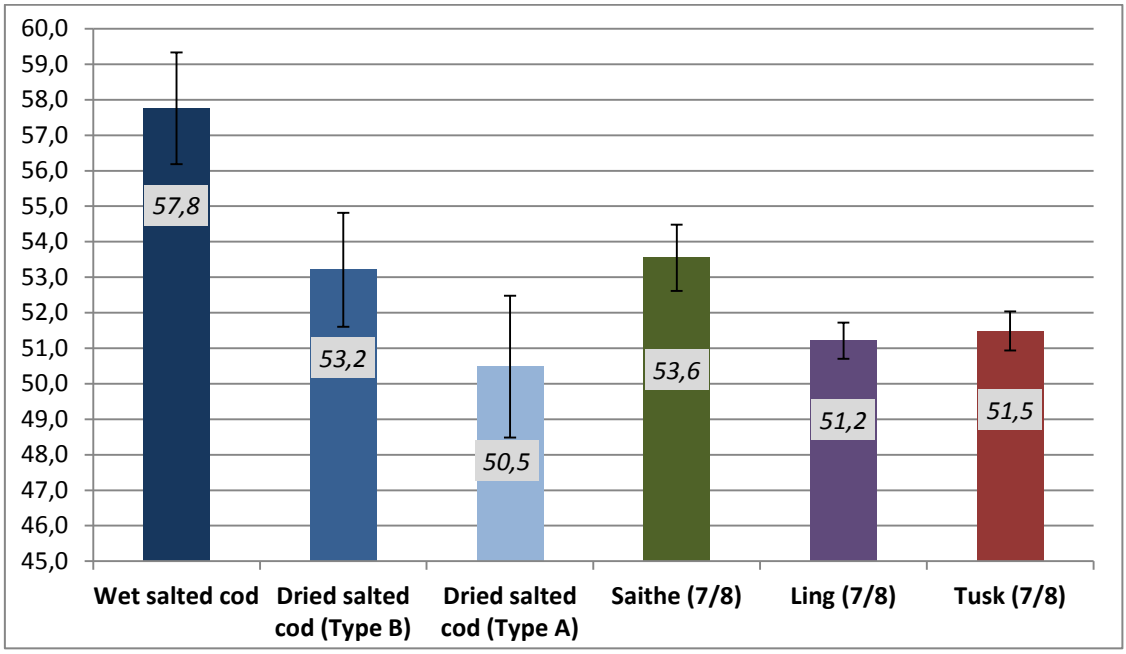

Figure 13: Mean moisture contents (n=10) in saltfish and clipfish from FHF Project no.901307.

Discussion

Anatomical variation of moisture content in dried salted fish.

The moisture content variation along a split dried salted fish has been confirmed. The parts closer to tail (posterior) contain significantly lower moisture contents than sections taken at a closer distance to the front part. Moreover, fish does not only contain longitudinal but also transversal moisture content variability. Fish is much thicker at the loins and close to spine than at the ventral part (*wings*), and this is more evident at the anterior parts of the fish than at posterior, where it is barely perceptible. When carrying out cross-sections, it occurs that some fish, more commonly saithe than cod, are nearly the same thick or even thicker at medium areas (*50% of total length*) than at anterior parts (*15% of total length*). In addition the wing is more present at anterior than medium areas and obviously than posterior areas. All these factors are behind the relative closeness in the results between the anterior and medium sections and the great differences found to tail areas.

The mean moisture content of the anterior sections closely resembles the moisture content from the application of the Codex Stan 167-1989 cross- section method, so the use of single sections could be an easier and more accessible alternative methodology for moisture content analysis. However, the results found should be corroborated in different clipfish size classes since only the big 8/10 class for cod was used for the study.

Practical suitability of the moisture content method from Codex Stan 167-1989.

The moisture content **method laid down at Codex Stan 167-1989** is well designed to get a representative sample of the entire splitted fish, but in practice **becomes really hard to implement**. The numerous and extremely thin (2 mm) sections to be obtained require the use of specific instruments like a mechanical band-saw, which is usually not available at standard laboratories. As it is also stated in Annex B of the Codex Stan 167-1989, it becomes difficult to make precise cuttings of 2 mm all along the entire fish due to presence of wings and fins, bones, and different thicknesses. In addition it is much easier to handle and cut parts that are more dry and rigid than other wetter and more flaccid. In practice, this difficulties lead to the inclusion of sections of a different width, that can definitely affect the representability of the laboratory sample. Moreover, from a direct interpretation of the reference

method it seems that the entire laboratory sample, composed of trimmings of all cross-sections, is weighed in the tared basins. In practice, due to imprecise cuttings and the fish size, the laboratory sample may weigh more than 200 g, and therefore only a representative portion of the laboratory sample can be weighed and analyzed.
Finally, section 4.7 of the procedure states that "*All sections of 2mm, marked II, IV, VI, VIII in the figure, even numbers, are collected to a collected sample",* meanwhile in the figure at the end of the document it is laid down that "*All section labelled by even number, II, IV,VI,VIII **etc.** are collected to constitute one sample". Unless an in depth reading is not made.* Misinterpretations may occur in case a complete and thorough reading is not carried out, leading to only include sections II, IV, VI, and VIII to the laboratory samples.

Comparison of moisture content methods.

Even though the Codex Stan 167-1989 cross-section method is the main reference to be applied by authorities to this food item category at border inspections, there are some national alternatives that usually simplify the sampling procedure. The methodology laid down in the currently in force *Decreto Lei 25-2005* [1], performs a more simple procedure that excludes the tail part of the fish, among some other particularities. Due to some similarities, it is probable that the latter reference was adopted and adapted by Brazilian Authorities to get the method [2] detailed above, that reduces the sections to three. Alike the Portuguese method the Brazilian sample preparation is skewed to the front due to the exclusion of sections from anal fins to tail.

From direct contact to Brazilian laboratory technicians it seems that some parts of the protocol are applied in a rather unclear way. It should be also fair to mention that there are several and very different dried salted fish products in the Brazilian market and the implementation of a single protocol for the category may be difficult, and would necessarily require internal interpretations from the standard protocol. **Our studies demonstrate that there is a bias in the final results when the Brazilian procedure or the Codex cross-section method is applied to classify imported lots of this food commodity.** This should be taken into account not only by Norwegian exporters by means implementing measures to handle the increased risk of non-compliance of materials at border inspection, but also by the Brazilian Authorities promoting the homogenization of criteria between laboratories.

To the contrary of other references the Codex Stan 167/1989 method does not permit the use of mechanical grinding, probably in order to prevent sample heating and moisture loss. The use of handheld scissors to turn sections to pieces, in addition to put more difficulties in the practical implementation, does not allow getting a satisfactory homogenate sample. Bones and skin become also part of the laboratory sample, whereas in the Brazilian standard are withdrawn. These are probably the reasons why **the precision of the Brazilian method is much higher than the precision of the method laid down in Codex Stan 167-1989.**

It would be a matter of discussion the benefits and drawbacks of including bone and skin to the laboratory sample. The inclusion of bones and skin would resemble more precisely the nature of the material especially when dealing with trade issues, but probably the same sample could not be used for other purposes (nutritional, food safety, etc.) where the analysis requires to be performed exclusively on the edible parts. In a different approach, the precision could be improved to some extent by the permission of moderate mechanical grinding for homogenization when bones and skin are included. This would also ease the sample preparation since we can exclude the step of separating edible and non-edible parts.

Moisture content variation in Norwegian production.

Despite the sampling to the moment is still short to set solid conclusions and assuming that samples supplied by companies match the sampling criteria of belonging to different production lots, it seems that **the internal homogeneity of the companies is high as moisture contents did not considerably**

vary. To the contrary, and even though differences are not of a major importance, it has been observed that some companies put into market more dried products than others and therefore can be more confident on not having issues at border inspection by Brazilian Authorities.

From the results obtained from the implementation of the Codex method, **the Norwegian production of dried salted cod and saithe may have some compliance problems with Brazilian standards** especially with the 8/10 size class of cod. The results in smaller fish classes have reduced non-compliance rates but probably still not within the safe margins that companies would desire. **In the case of the implementation of the Brazilian method, problems of compliance of the Norwegian production would be severe**, since in average gives a 1,63 g/100 higher result than the Codex method.

Suggestions

Based on the information gathered by the project, a **new sample preparation procedure is suggested** for discussion and testing by laboratories. The procedure selects three sections of 20 mm at 20%, 50% and 80% of total length. Bones and skin are included and moderate mechanical grinding is allowed to get a homogeneous laboratory sample.

Finally, it is important to remind the potential of modern techniques like infrared spectroscopy (NIR) and hyperspectral imaging in the quantification of moisture contents of food products. The simplicity of use, low cost, fast response and non-destructive nature are their major advantages. At present there are a great variety of instruments that could be used for direct scanning and on-line measurements, or even portable handheld instruments for at-line applications. During the last years the miniaturization of the hardware, the interconnection to common interfaces like PDAs or mobile phones, and the reduction of the prices of the instruments shorten the distance from laboratory to industry applications. However, there are still some unavoidable drawbacks like the specificity and high cost of getting a calibration that suits the needs of the industry.

6. Main findings

- **It has been documented that there is an important longitudinal moisture content variance in a piece of splitted clipfish, with much reduced values at the tail part.**

- **Codex moisture content method is imprecise, high cost and difficult to put into practice in a efficient way by standard laboratories and industries. Suggestions for protocol changes have been laid down.**

- **Internal variability from companies seems to be low meanwhile there are some evident differences in moisture contents of clipfish between different producers.**

- **From the results of the Codex method the 8/10 size class of dried salted cod has an expected non-compliance rate of a 35%. For the rest of the size classes the rates are lower.**

- **Present Brazilian method gets, in average, a 1,6 g/100g higher moisture content result than method laid down in Codex Stan 167-1989. The use of the Brazilian method may lead to unacceptable non-compliance rates at border inspection for the Norwegian industry, so strategies to handle risks of non-compliance at border inspection should be considered by export companies.**

7. Deliverables.

- **Final report of the tasks carried out and results obtained.**

- **Power point file used for the presentation of the results at the meeting with the clipfish sector at 01/09/2020.**

- **Peer reviewed journal publication in progress.**