Scientific Works. Series C. Veterinary Medicine. Vol. LXIX (1), 2023
ISSN 2065-1295; ISSN 2343-9394 (CD-ROM); ISSN 2067-3663 (Online); ISSN-L 2065-1295

# DETERMINATION OF CHEMICAL-ANALYTICAL QUALITY CRITERIA OF KING SCALLOP (*Pecten maxima*), ATLANTIC SEA SCALLOP (*Placopecten magellanicus)* AND QUEEN SCALLOP (*Aequipecten opercularis*) SAMPLES FROM NORTH ATLANTIC OCEAN, ENGLISH CHANNEL AND FROM THE TRADE MARKET

**Cătălina Nicoleta BOIŢEANU[1], Nicoleta CIOCÎRLIE[1], Florin NEACSU[2], Laurenţiu TUDOR[1]**

[1]University of Agronomic Sciences and Veterinary Medicine of Bucharest, Faculty of Veterinary Medicine, 105 Splaiul Independenţei, District 5, Bucharest, Romania
[2]Baylor University, Department of Chemistry and Biochemistry, Waco, TX 76798, USA

Corresponding author email: catalina-nicoleta.boiteanu@fmvb.usamv.ro

***Abstract***

*King scallops (Pecten maximus), Atlantic sea scallops (Placopecten magellanicus) and Queen scallops (Aequipecten opercularis) were were directly caught from natural environment of North Atlantic Ocean and English Channel Bay of Biscay during four of Walther Herwig's III (WH III) expeditions and compared with an enlarged range of frozen scallop purchased on the German market. Proximate composition, was examined in the muscle to identify changes as a result of freezing and processing. Investigations took place on-board and at the Max Rubner Institute Hamburg. In the investigated purchased bivalve molluscs samples, in two cases (Pecten spp., Placopecten magellanicus) perfect positive correlations (R2 = 1) were observed between the amount of phosphate and the pH values, while in the other 5 samples, the correlations were negative (R2 = −1). A negative but weak correlation was established between protein percentage and TVB-N % (R2 = − 0.26). There were calculated reasonable positive correlations between phosphate and ice glaze (R2 = 0.48).*

***Key words****: phosphates; water content; scallops; proximate composition; additives.*

## INTRODUCTION

Shellfish quality refers to aspects of consumer preference such as freshness, nutrition and the sensory attributes such as color, texture and flavour (Boulter, 1996). Storage and handling practices during *rigor mortis*, resolution of *rigor mortis*, nucleotide degradation (autolysis) and microbial growth affect freshness, flavour and texture. Nucleotide degradation and microbial growth specifically affect the freshness and flavour of fish and shellfish (Tudor, 2013). Tenderness (texture) is an important aspect of scallop quality and can be measured by both instrumental and sensory methods (Ocano-Higuera et al., 2006). Autolysis, microbial and textural changes in scallops under various storage and handling practices have been widely reported (Ocano-Higuera et al., 2006; De Vido & de Mattio et al., 2009; Makri, 2009). Quality can also refer to the physiological condition of an animal and its ability to respond to its environment. A combination of physiological, biochemical and behavioural tests, including condition indices and carbohydrate content was used in order to assess the response of scallops to stress.

Indices mainly measured the physiological activity of an animal at a given time or environmental condition and were computed for specific tissues (adductor muscle) in relation to other tissues after taking morphometric measurements (Beltran-Lugo et al., 2006).

Biochemical composition is an important quality indicator and is closely associated with above mentioned condition indices. Storage of nutrient reserves in the adductor muscle contributes to the nutritional value of the scallops (King et al., 1990; Ocano-Higuera et al., 2006).

## MATERIALS AND METHODS

Established methods were used in the laboratory of Max Rubner Institute (Hamburg, Germnay) to analyse the contents of water, protein (according to Dumas), ash, total phosphate (photometric) and to measure the pH values. The qualitative detection of condensed phosphates was carried out using the photometric reference method, a quantitative analysis.

The percentage of dry matter and water respectively were determined after drying over night at 105ºC aliquots of the homogenized samples. The ash content was measured according to Antonacopoulos (1973) as previously described (Boițeanu & Neacsu, 2022). Samples were mineralised over night into a mufle furnace at 550°C, than cooled in a desiccator and weighed.

In order to measure the total phosphorus content the photometric reference method was carried out. The homogenized samples were dried and calcined, the ash was then hydrolysed with nitric acid. After the addition of ammonium metavanadate and ammonium heptamolybdate to an aliquot part of dilute nitric acid (~20%), a yellow coloration resulted, whose extinction was photometrically measured. Extinction was directly proportional to the phosphorus content.

The percentage of fats from scallop samples was exactly measured by gravimetric method Smedes (1999) as amended by Karl et al., (2012) and as previously described (Boițeanu & Neacsu, 2022). The method is based on the extraction of fat with cyclohexane and 2-propanol followed by the transfer of the fat to the cyclohexane phase by addition of water. Phase separation is made by centrifugation. Finally, the gravimetric determination of fat takes place, after separation and concentration of the cyclohexane phase.

Determination of the percentage of protein substances in fish was based on the principles of the Dumas combustion method (Miller et al., 2007) as previously described (Boițeanu et al., 2014). After well-homogenized samples were obtained, they were heated in a high-temperature furnace where the combustion took place at over 1000°C in the presence of pure oxygen. This heat treatment produced (among others) nitrogen as NyOx (oxides). The gas mixture was passed through a reduction chamber containing copper heated to 650°C in which the nitrogen oxides were converted into elemental nitrogen and the excessive oxygen was collected. Finally, total nitrogen content was measured by a thermal conductivity detector. The entire process took less than 4 minutes. In the end, total protein was measured multiplying the N results by 6.25.

Salt percentage was measured by titration with 0.1N silver nitrate solution using an automatic titrator (Karl et al. 2002) as already described (Boițeanu et al., 2014). The principle of the method is based on the extraction of salt (by means of dissolving into water) from the pre-weighed sample (the sample is suspended in water and acidified with nitric acid). After protein precipitation, the chloride concentration is determined by titration (up to the final potentiometric point), with an aliquot part of the standardized silver nitrate solution (0.1N).

## RESULTS AND DISCUSSIONS

### Water in fresh and frozen scallops

Marketed amount of quick-frozen scallops (*Pectinidae*) has significantly increased in the recent years and has been extended from the king scallop (*Pecten maximus*) to a variety of species. Incorrect labelling of frozen scallops and excessively high water addition are the two associated and hard-to-solve problems.

Due to their tasty large sphincter (adductor muscle), scallops are traded as high-priced seafood compared to many other molluscs. Their taxonomy could be confusing due to the similar appearance and because of assigning names in an unscientific way.

It is not uncommon for other low quality scallops to be incorrectly labelled and to pass as more valuable scallops (*Pecten maximus* or *Pecten jacobaeus*) in order to increase commercial profits (Näumann et al., 2012). Difficulty resides in their differentiation because most of the deep-frozen scallops are available on the european market without the shell, with or without the orange-yellow roe.

Scallops are caught in both the Atlantic and Pacific regions and marketed. Besides wild scallops catches, there is a targeted mussel culture economy that allows for faster and more

reliable harvesting. Processing techniques for commercial purposes vary by species, region and country. In US and Canada trap fisheries targeting sea scallops (*Placopecten magellanicus)*, use the technique consisting in the on-board removal of shells and viscera (to minimize risk of biotoxins formation). The catch is then stored in sacks on ice or in ice and seawater mixture. Catches can be frozen for short fishing trips, if necessary. The shells are also removed after landing.

Different processing techniques are adopted from catching or harvesting to the deep-frozen scallop product and have a significant impact on their quality. The current use of water to separate the innards and cleanse the adductor muscle is a necessary treatment step and inevitably leads to water uptake. Significant amounts of added water could get into the scallops' meat also by prolonged immersion. The use of additives such as polyphosphates increases the water-binding capacity, and leads to high water retention during thawing and preparation (Flick, 2012; Manthey-Karl et al., 2015).

**Criteria for assessing the water content in scallops**

Studies that dealt with the natural proximate composition of scallops and the influence of the various processing technologies on the product quality are to be mentioned. In the '70s and '80s, the US and Canada, both of which have traditionally been important fisheries for scallops, numerous investigations were carried out regarding the biology of these species (Rosseker & McKay, 2010). Untreated fresh scallops of known origin and with exact taxonomy assignation were subjected to studies that underlined the importance of the ripening cycle and seasonal dependence on the composition of the flesh. Interest was given also to the influence of the various processing steps on the end product, which is mainly sold on the market as deep-frozen muscle and the industrial scallops processing methods (Shumway & Parsons, 2006).

There is a considerable range in water content depending on season and food supply. Botta and Cahill (1992) determined between 74.6% and 80.2% in *Placopecten magellanicus*; a Canadian study (DuPaul et al., 1996) comes to similar results with 74.2-80.9%.

Comparable contents were also determined by other authors. Table 1 gives an overview of the composition of the raw materials and the differently treated meat from the literature. No difference was found between wild and cultivated forms of *P. magellanicus* (Naidu, 1978).

The interim storage of the muscle meat, which has already been freed from the shells, in ice/ ice water can cause the water content to rise. In doing so, DuPaul et al. (1996) found an initial natural content in *P. magellanicus* of 73.9-78.9%, whereas 74.2-82.6% was determined for the meat at the time of landing. At the time in North America there was an 80% limit for water content and exceeding this value was considered as added water. Rippen et al. (1998) stated, however, that this limit value is not practicable in connection with the strong fluctuations in the natural water content in the raw material.

In France, scallops (king scallops, *Pecten maximus*) are traditionally traded, most of which come onto the market fresh but also deep-frozen. The global trade in scallops of other origins and species did not stop at the French market either. As early as the late 1980s, research institutes, such as Institut Français de Recherche pour l'Exploitation de la Mer, were trying to find suitable criteria to detect unacceptable additives in foreign water. They examined freshly caught and frozen samples of *Pecten maximus* and *Mizuhopecten yessoensis* (Japanese scallops), which are imported to France in significant quantities from Japan. As a result, France introduced the determination of the water to protein ratio (W/P) as the most appropriate criterion for determining extraneous water. The untreated *Pecten maximus* muscle showed a ratio between 3.9 and 4.7. It was thus comparable to that for *Mizuhopecten yessoensis* (Loreal, 1990). Immersion in water for 15 minutes increased the (W/P) ratio to 5.2. In the case of frozen goods, de-glazing proved to be a critical point and should be carried out according to a standard procedure. Overall, a W/P $< 5$ is derived from the test results, taking into account a 5% increase in weight during

processing such as transport and washing, for *Pecten maximus* and *Mizuhopecten yessoensis*.

**Additives in scallops**

The muscle meat composition and the water to protein ratio (W/P) was determined by Manthey et al. (2015) in samples from the German retail. The results showed that a considerable number of samples had very high moisture contents and W/P ratios > 5. These facts were mainly true for *Placopecten magellanicus* and *Mizuhopecten yessoensis*. There was no conformity with the prescribed declaration of food additives.

**Phosphates**

Scallops are treated with water-binding additives, such as phosphates, in countries where it is permitted. In particular, phosphates (di- and higher condensed phosphates) are used to reduce the so-called "drip loss" that occurs as a result of freezing and thawing. They increase the water-binding ability of the muscles by supporting the dissociation of proteins and thus water retention. In the US, phosphates are classified as GRAS (Generally Recognized As Safe; FDA, 2012) food additives. They can be used in accordance with good manufacturing practice to create the impression of 'natural' succulence in the meat. There is no limit value for phosphorus-based additives in the end product. Significantly higher values are only considered critical in the USA in connection with increased water content (Kennedy, 2011).

According to the EU regulation 1333/2008 (EU, 2008) and the still valid directive 95/2/EG (EU, 1995) fresh molluscs must not be treated with phosphates. They are only allowed for frozen products. However, the added content in the product must not exceed 5 g/kg phosphate (calculated as $P_2O_5$). The Codex Alimentarius draft standard for scallops does not deviate from these restrictions on phosphates. No additives are allowed in the fresh raw product either.

Sidwell et al. (1977, review of the literature) gives an average natural phosphorus value of 0.27 ± 0.038 g P/100 g (≙ 0.62 ± 0.087 g $P_2O_5$/100 g) for scallop species (*Pectinidae* spp.) that are not specified in more detail, with a range of 0.21-0.34 g P /100 g (≙ 0.48-0.78 g $P_2O_5$/ 100 g).

Phosphates get into the product to be processed mainly by dipping. The concentrations of the phosphate baths vary from 2% to 10%, with the necessary exposure times depending on the type of fishery product (Schnee, 2004). Added table salt (NaCl) in concentrations of about 1.0% in a sodium polyphosphate brine can support the water binding capacity of the phosphates (Fisher *et al.*, 1996). Only at higher salt concentrations of over 6%, as is the case in the brines of salt fish, the water is no longer retained in the muscle meat, and dehydration processes occurs (Schröder, 2010). In contrast to the various phosphate compounds, table salt is not an additive, but an ingredient that serves to add flavour and can also increase the water content in the scallop. It must be declared without specifying the exact quantity.

**Investigations in North Sea and North Atlantic**

The sampling of untreated scallops took place on board the FFS "Walther Herwig III" during the voyages to the North Sea and the North Atlantic (232nd, 256th, 304th and 310th , between 2001 and 2007. Objectives of cruises as resumed were investigations on commercially used fish and molluscs species in respect to food safety and food quality.

| Plattform<br>Platform | Reise-Nr.<br>Cruise-No. | Zeitraum<br>Period | Projekt<br>Project | Arbeitsgebiet<br>Working area |
|---|---|---|---|---|
| Walther Herwig III | 232 | 14.09.2001 - 04.10.2001 | | North Atlantic Ocean |

| Fahrtleiter<br>Chief Scientist | Institut<br>Institute | Auslaufhafen<br>Port of departure | Einlaufhafen<br>Port of return | Stationskarte<br>Station map | Schiffsroute<br>Trackchart |
|---|---|---|---|---|---|
| Oehlenschlager Jorg | Institut fur Biochemie u. Technologie der BFA Hamburg | Bremerhaven | Bremerhaven | | |

**Ziel der Reise / Objectives of Cruise:**

Investigation on commercially used fish species in respect of food saftey and food quality.

**Messungen / Measurements**

| Institut<br>Institute | Wissenschaftler<br>Scientist | Anzahl<br>Number | Einheit<br>Unit | Typ der Messungen<br>Type of measurements | Kommentar<br>Comments | Daten im DOD<br>Data in DOD |
|---|---|---|---|---|---|---|
| IBT | Oehlenschlager Jorg | 49 | hauls | B90<br>Other biological / fishery measurements | Inorganic analysis | no |

Figure 1. Objectives and areal of Walther Herwig's III (WH III) 232nd trip

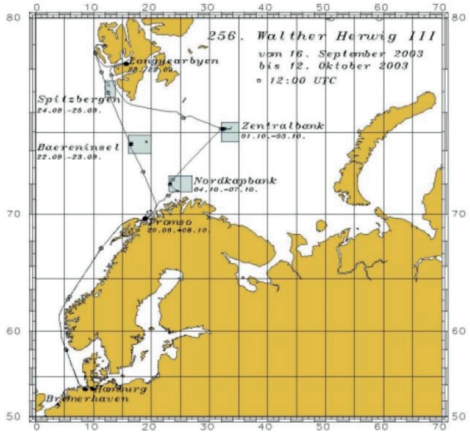


Figure 2. Explored waters
during Walther Herwig's III 256th expedition

## I. On-bord investigations

### Sensory assessment

The sensory assessment of scallops' quality included the on-board assessment of the samples after cooking for 8 minutes in the cooking bag. Inspectors experienced in assessing fishery products, each of whom had to assess all samples, assessed the quality characteristics of smell, appearance, taste and consistency on Torry rating scale of 0 to 5. The better the rating, the higher the number of points awarded.

As expected, the longer the scallops were stored in ice, the less fresh they becomed. The shelf life of scallops in ice (1.5°C) was compared to that under super-chilled conditions (-0.9°C), the latter increasing the shelf life from 9 to 16-17 days. If the pieces were additionally packed in a modified atmosphere, the shelf life in ice increased from 9 to 14 days, while under "super-chilled" conditions a further increase in shelf life to 21 days was observed. However, when the latter combination was applied, texture changes towards "meaty" were noted, possibly caused by increased drip.

At the time of sensory spoilage of ice-stored scallops after 10 days, *Photobacterium phosphoreum* dominated the spoilage flora. Ketones (33%) followed by amines (29%), alcohols (15%), acids (4%), aldehydes (3%) and a small amount of esters (<1 %) were detected as volatile components at the time of spoilage.

In addition to the sensory analysis, the total content of volatile N-bases (TVB-N value) was determined. The preparation of the perchloric acid extract, was made from 20 g of minced sample which were homogenized with 180 ml of 6% (w/v) perchloric acid for 30 s using an UltraTurrax, than the mixture was filtered and the clear perchlorate acid extract was frozen at −28°C until future analysis.

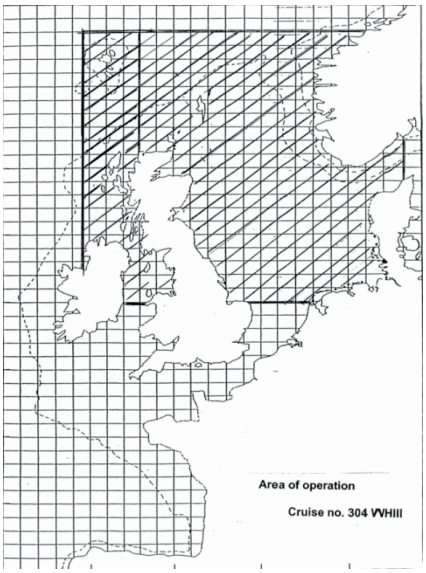


Figure 3. Operational area of Walther Herwig III
during the 304th cruise


Figure 4. Variable stations of fishing and area of
investigation during the 310th WH III cruise

**TVB-N, cooking loss, pH, color and fish tester**

The TVB-N value as an objectively determinable variable, which according to European standards should always be used when sensory findings indicate deviations in quality, showed that during the investigations the total content of volatile basic N-compounds were subjected to only minor changes (figure 7).

The determination of the pH value (using the laboratory pH meter 761, Knick Electronic Measuring Devices, Berlin) was carried out using a puncture electrode at different locations of the molluscs belonging to the test group (n = 4) accordingly.

The pH values are not subject to any significant changes during ice storage, as can be seen from the small standard deviations of the mean values over the entire storage period. However, as expected, the pH value was influenced by the measuring location.

The results of the instrumental color measurements showed only insignificant changes in lightness. The values determined with the Fish Tester Intellectron VI showed a largely linear relationship to the storage time. There was detected a significant decrease along with increasing storage time due to the decrease in the resistance of the samples along with reduced freshness. The values measured on freshly caught molluscs agree well with those previously described.

Modifications of the electrical properties of the muscle tissue were also measured with a torrymeter. Torrymeter values also decreased with decreasing freshness. The correlation of the Torrymeter values with the storage time or with sensory properties was significantly low.


Figure 5. Torrymeter

Measuring the electrical properties of the meat (resistance, conductivity and capacitance) transversely through the adductor muscle with a Fish Tester Intellectron VI is a reliable method with regard to the storage time in melting water ice. The tester value was determined for each scallop (n=5) in a test group measurement, determined at the level of the sphincter.

Figure 6. Wild scallops samples preparation before proximate analysis

**II. Investigations at the Max Rubner Institute**

Results showed large differences in quality of frozen scallop products and the problem of added water in frozen seafood in general and in frozen scallops in particular is remaining practically unchanged during.

The lack of quality and the affordable prices of frozen scallops is a fact due to the large range of products sold under the generic name “Frozen scallops” that are not correctly labelled and identified as species and regarding their added water percent. A too flexible regional

and global legislation led to the apparition of aquaculture assortments such as *Pangasius* spp. usually sold with very high amount of added water and at very cheep prices.
Water and additives contents of seafood were subjects largely discussed in many publications, in the past 20 years, and some laws were adopted by countries (France and USA) to protect consumers from purchasing practically just water instead of seafood they desire.


Figure 7. TVB-N (mg %) in analysed *Pectinidae*: R (refrigerated) and C (congelated) samples

All nutrient-rich seafood products are highly perishable. Large variety of additives and preservatives are used to maintain/ augment aspects regarding the quality, safety, wholesomeness, and consumer acceptance. Analytical methods have been developed to detect and monitor the residues of many additives and preservatives for ensuring consumer health and safety in compliance with regulatory EU standards. Because the current analytical efforts are considered, in their majority, time-consuming more advancement is needed and most important is to ensure rapid and simultaneous detection of multiple ingredients and additives (Surendran Nair et al., 2020).
Water absorption during processing cannot be technologically avoided. Therefore, intakes of 0.5 g/kg are tolerable. Taking into account that raw seafood originally contents 80% water, the final water added product would contain 81%. For a theoretical increase to 85%, percent that in real life is found in many marketed seafood products, is needed > 33% of added water although the labels are showing clean products.
A more serious fact is that phosphates content rarely withstands analytical testing being mostly eliminated before analysis during thawing.

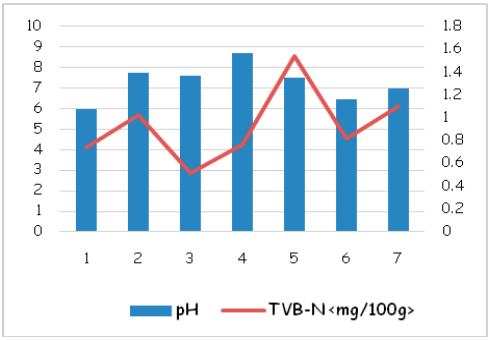


Figure 8. Variation of pH and TVB-N (mg %) in the analyzed *Pectinidae* samples

The consumer is therefore often cheated when buying scallops and/ or other seafood if the composition of the product does not correspond truly to the labelling.

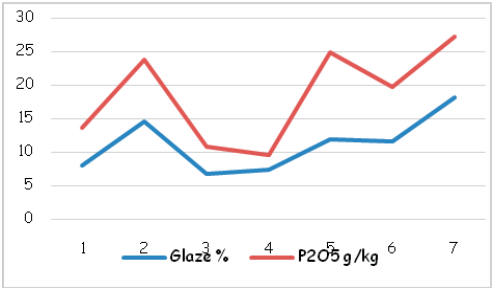


Figure 9. The variation of phosphorus content depending on the proportion of glaze in the analyzed Pectinidae samples

Salutary efforts were made by the CAC in order to develop standards and codes of good practices. Suitable information for the buyers and complete labelling in the food chain, respected both by the producer and the trader is a must for the scallops' and seafood market generally. Question are to be addressed regarding the form in which the manufacturer's information can be checked for traceability reasons (Tudor et al., 2022).
To establish reliable values for litigation purposes, firstly reliable values for the initial proximate composition of the raw material should be settled and published. As an exemple, measuring the water/protein (W/P) ratio offers valuable assessment perspectives.
Summarized in Figure 10 are the most important analysis W/P ratio results of the various samples. The pH values measured were

between 5.98 and 8.71 while fresh scallops have usually pH-values situated around 6.0. Values of pH above 7.0 were previously associated with the use of additives.

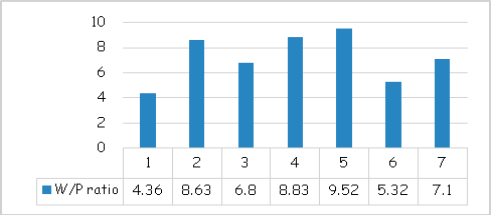


Figure 10. W/P ratio in marketed *Pectinidae*.

All other samples showed a clear relationship between water content and W/P ratio. With a water content of over 80%, the W/P ratio was > 6. As regards W/P ratio calculated, only one product had results in the borderline range with 77.1% water and a W/P of 4.4 (Table 1).

Table 1. Water (W) and protein (P) content of Wild *Pectinidae* in %

| Sample no. | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|---|---|---|---|---|---|---|---|
| W% | 77.12 | 86.53 | 84.49 | 86.7 | 88.03 | 79.21 | 84.35 |
| P% | 17.67 | 10.03 | 12.43 | 9.82 | 9.25 | 14.88 | 11.9 |

Phosphate amount largely varied between the different samples from 2.2 g $P_2O_5$/kg in *Placopecten magellanicus* and 12.9 g $P_2O_5$/kg in *Pecten yessoensis*. This result should also take into account the results obtained using total phosphate determination methods in fishery products which showed very often large natural fluctuations.

Different qualities of *Pecten maximus* were previously found on the european market . None of these marketed scallops declared the additives in the list of ingredients. The calculated W/P ratios were between 4.3 and 8.6. Research on fresh scallops confirmed a water to protein ratio in fresh scallops below 5 (Manthey-Karl et al., 2015).

Table 2. Investigated *Pectinidae* samples from the German market

| Species | Glaze < % > | pH | TVB-N <mg/100g> | DM < g/kg > | Ash < g/kg > | Fat <g/kg> | Protein <g/kg> | $P_2O_5$ < g/kg> | NaCl <g/kg> |
|---|---|---|---|---|---|---|---|---|---|
| *Pecten* spp. | 8 | 5.98 | 0.74 | 228.8 | 12.5 | 7.1 | 176.7 | 5.64 | 3.8 |
| *Pecten* spp. | 14.5 | 7.75 | 1.02 | 134.7 | 20.5 | 4.1 | 100.3 | 9.33 | 3.1 |
| *Placopecten magellanicus* | 6.7 | 7.62 | 0.52 | 155.1 | 17.4 | 4.1 | 124.3 | 4.15 | 5 |
| *Placopecten magellanicus* | 7.3 | 8.71 | 0.77 | 133 | 30.3 | 3.3 | 98.2 | 2.2 | 1.1 |
| *Placopecten magellanicus* | 11.6 | 6.45 | 0.82 | 207.9 | 17.3 | 5.9 | 148.8 | 8.07 | 3.8 |
| *Pecten yessoensis* | 11.9 | 7.52 | 1.54 | 119.7 | 25.2 | 4.5 | 92.5 | 12.94 | 0.9 |
| *Pecten maximus* | 18.1 | 6.99 | 1.1 | 156.5 | 20.6 | 8.3 | 119 | 9.11 | 1.8 |

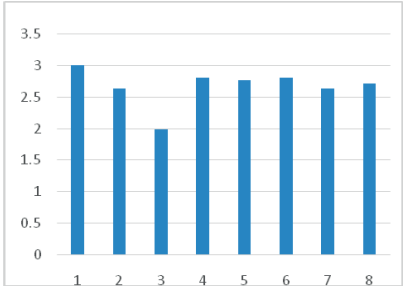


Figure 11. $P_2O_5$ content (oy) as <g/kg> in 8 wild scallops samples (ox)

Investigated purchased from the German market bivalve mollusc samples, showed (cf. Table 3), in two different species (*Pecten* spp., *Placopecten magellanicus*) perfect positive correlations (R2=1) between the amount of phosphate and the pH values, while in the other 5 samples, the correlations were negative (R2 = –1). A negative but weak correlation was established between protein percentage and TVB-N % (R2 = –0.26).

There were reasonable positive correlations between phosphate and ice glaze (R2 = 0.48), a fact that was expected (Figure 12). The highest amount of glaze (18%) overpassing the 10% limit, was found in marketed *P. maximus* percent which was correlated with a high

quantity of phosphates (9 g $P_2O_5$/kg) situated next to the doubled maximal value permitted (5 g $P_2O_5$/kg).

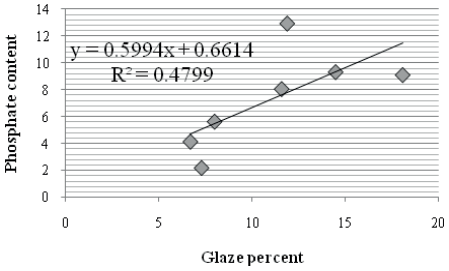


Figure 12. Scatter plot showing a reasonable correlation ($R^2$ = 0.48) between the amount of phosphate (in g $P_2O_5$ /kg) and the proportion of glaze ice (%) in scallops samples from German market

The percentage of water found in *P. magellanicus* was between 79.2 and 86.7%, higher values than those previously reported in the literature (74.2-82.5%) by Du Paul et al. (1996) for untreated scallops. The same parameter, measured in *P.maximus*, was 77.1-86.5%, the values being clearly higher than those reported by Sidwell (1977) in the case of raw scallops. The marketed *P. yessoensis* reached the water percent of 88%, a value clearly higher than those specified for wild scallops by Loreal (1990), namely 77.6-79.2% . The total phosphate content in marketed Pectinidae was in 5 samples perfect negatively correlated with the pH, while in 2 investigated samples the same correlation found was perfectly positive (Table 3).

Table 3. Regression equations for phosphate (y) versus pH in purchased scallops samples

| Species | Regression equations | Pearson correlation coefficient |
|---|---|---|
| *Pecten* spp. | Y= - 4.6471 x + 35.539 | $R^2$=−1 |
| *Pecten* spp. | Y = 10.206 x – 53.411 | $R^2$=1 |
| *Placopecten magellanicus* | Y = 19.147 x - 105.79 | $R^2$=1 |
| *Placopecten magellanicus* | Y = - 15.941 x + 102.85 | $R^2$=−1 |
| *Placopecten magellanicus* | Y = - 4.7647 x + 34.943 | $R^2$=−1 |
| *Pecten yessoensis* | Y= - 6.2353 x + 44.277 | $R^2$=−1 |
| *Pecten maximus* | Y = - 2.12 x + 11.2 | $R^2$=−1 |

Investigated samples caught in the wild during 4 of WH's III cruises showed a small variability of natural phosphates content in the range of 1.98 to 3 g $P_2O_5$/kg (Table 4).

Table 4. Total Phosphates content of Wild *Pectinidae* in g $P_2O_5$/kg

| Species | g $P_2O_5$/kg |
|---|---|
| *Pecten maximus* (Walter Herwigs III 232nd Trip) | *3.01* |
| *Pecten maximus* (Walter Herwigs III 232nd Trip) | *2.64* |
| Pecten spp. (Walter Herwigs III 256th Trip) | *1.98* |
| *Aequipecten opercularis* (Walter Herwigs III 310th Trip) | *2.81* |
| *Aequipecten opercularis* (Walter Herwigs III 310th Trip) | *2.77* |
| *Aequipecten opercularis* (Walter Herwigs III 310th Trip) | *2.80* |
| *Pecten maximus* (Walter Herwigs III 304th Trip) | *2.63* |
| *Pecten maximus* (Walter Herwigs III 304th Trip) | *2.72* |

**Litigation concerns**

Although, there are specific roles for additives in foods (e.g., direct additives such as phosphates added in seafood to retain moisture and protect flavour), the chances of minute amounts of indirect food additives (for instance, packaging substances) finding their way into foods during processing and storage cannot be neglected. Therefore the competent authorities require that all materials coming in contact with food should be certified as safe before they are used in the indicated manner.

Food additives are studied, regulated, and monitored to ensure that consumers feel safe about the foods they eat and are protected from hazards. The entire responsibility rests with the manufacturers which are entitled to ensure that the ingredients are of a food-grade purity and comply with specifications and restrictions.

All international trade risk assessments are conducted by an independent, international expert scientific group, the Joint FAO/WHO Expert Committee on Food Additives (JECFA) (WHO, 2018), ensuring that there are monitoring steps in place at various levels of global food production.

Phosphates are added to seafood to increase water-holding capacity (Sebranek, 2015). Sodium tripolyphosphates are additives of the phosphate family used in the seafood industry with a humectant function, i.e., those substances maintain the moisture of the product, being more used in scallops, shrimp and lobsters processing.

Sodium tripolyphosphates in scallops, added in concentrations from 2.5% to 10% (1 min) until the moisture percent was increased to 82-86%, respectively, in conjunction with 1% of NaCl, was shown efficient to prevent the drip loss during defrosting and after cooking, and inhibited the microbial growth. Generally, a long exposure to tripolyphosphates demonstrated no other notable advantage, while a short exposure of scallops to these additives produced desirable functional effects, generally without exceeding 83% of moisture in the final product (Rippen et al. 1993; Gonçalves & Duarte Ribeiro, 2008).

Health concerns and safety assessment regarding the chronic exposure to food additives and the associated adverse health impacts are growing concerns globally (Maffini et al., 2017). With the evidence unveiled through epidemiological studies linking the consumption of phosphate food additives and increased cardiovascular risk, the European Food Safety Authority is currently pursuing a high-priority re-evaluation of added phosphates in seafood and meat products (Foley et al., 2009; Cancela et al., 2012; EFSA, 2013).
(Surendran Nair et al., 2020)

A maximum water content in scallop meat is not internationally established by food laws. Some authors (Gonçalves & Duarte Ribeiro, 2008) indicate the 5 g $P_2O_5$/kg maximal value adopted in the EU. Codex Alimentarius Committee (CAC) for Fish and Fishery Products, discussed the topic of adding water to frozen scallops, and this subject has been controversial for a long period of time (Schröder & Siegert-Clemens, 2012). At the CAC's 32$^{nd}$ meeting that took place in 2012, the member states (MS) established the labelling options, that were more transparent and consumer-protecting: "Scallop (name…) with added water" or "Product from scallop (name…) with added water" or another labelling announce which fully informes the buyer. Draft standard for frozen scallops was in 2015 at 6/8 stage process, pending acceptance by the CAC and was finally amended in 2016 and revised in 2017. Recommendations regarding the added water were made for "Quick frozen Scallop Meat" and "Quick Frozen Roe-on Scallop Meat" that shall have the mention "added water" as a part of products' name.

## CONCLUSIONS

The very high water content, corroborated with the large amounts of total phosphate (up to 13 g $P_2O_5$/kg) detected in the corresponding samples from the German market, entitle us to say that large amounts of phosphate-based additives (well over the legal limits) were used for traded scallops, intended to increase the water-binding capacity.

On the other hand, samples caught in the wild during 4 of WH's III cruises showed a small variability of natural phosphates content in the range of 1.98 to 3 g $P_2O_5$/kg.

## ACKNOWLEDGEMENTS

This research work was carried out with the support of DAAD, Max Rubner Institut, Professors H. Rehbein, C. Sala and L. Tudor, researchers M. Manthey-Karl, H. Karl and all the staff from MRI Hamburg.

## REFERENCES

Antonacopoulos, N. (1973). Lebensmittelchemisch rechtliche Untersuchung und Beurteilung von Fischen und Fischerzeugnissen. In: *Fische und Fischerzeugnisse* (edited by W. Ludorff & V. Meyer). p. 219. Berlin & Hamburg, Germany: Paul Parey.

Atanasoff A., Dospatliev L., Zapryanova D., & Oner C. (2014). Proximate composition and mineral profile of snail meat (helix lucorum) from trakia valley in bulgaria. *Scientific Works. Series C. Veterinary Medicine,* Vol. LX (1), ISSN 2065-1295, 126-126.

Beltran-Lugo, A. I., A. N. Maeda-Martinez, R. Pacheco-Aguiler, H. G. Nolasco-Soria & V. M. Ocano-Higuera (2005). Physical, textural, and microstructural properties of restructured adductor muscles of 2 scallop species using 2 cold-binding systems. *J. Food Sci.* 70:E78–E84.

Boulter, M. (1996). Scallop quality: the effects of different handling practices and storage times, prior to processing, on scallop quality and investigation of quality assessment methods/indicators including D-lactic acid. Seafish report no. SR469. Grimsby, Scotland: The Sea Fish Industry Authority. 19 pp

Boițeanu C.N.& Neacsu F. (2022). Freshness indicators, phosphorus content and humidity correlations in anglerfish and monkfish samples (*Lophius* spp.) from the North Sea and Atlantic Ocean *Scientific Works. Series C. Veterinary Medicine*. Vol. LXVIII (2), 121-128.

Boițeanu C.N.& Neacsu F. (2021). Phosphorus content, native and added to Pike-perch (*Sander lucioperca*) fillets, sold on the european market and its effects on total products' quality. *Scientific Works. Series C. Veterinary Medicine.* Vol. LXVII (1), 141-147.

Boițeanu C.N., Manthey-Karl M., Karl H., Meyer C. & Savu C. (2014). Proximate Composition, Microbiological Quality and Sensory Attributes of Mahi-mahi (*Coryphaena hippurus*) and Emperor Sea Bream (*Lethrinus* spp.) Fillets Sold on Retail Market. *Bulletin UASVM Food Science and Technology*, 71(2) , 89-95.

Bota, J. R. & Cahill, F. M. (1993). Moisture content of scallop meat: Effect of species, time of season and method of determining "added water". *Proceedings of the 18th Annual Tropical and Subtropical Fisheries Technological Conference of the Americas. Virginia* (USA): p. 43-50.

Brenner K, Oliveira A., Rosenkranz G., Burt R., Spafard M., Bechtel P., Crapo C., & Ralonde R. (2012). Quality of Weathervane Scallops (Patinopecten caurinus) from Eastern and Western Gulf of Alaska. *Journal of Shellfish Research*, 31(4):1123-1132. 2012.

Bundesforschungsanstalt für Ernährung und Lebensmittel, Standort Hamburg, Report of the 304. Cruise of FRV "Walther Herwig III" 19.09. – 2.10.2007. Chief scientist: Dr. Carsten Meyer. Studies on food quality of fishes from the North Sea and substances of content of mussels and crustaceas and collection of reference samples

Cancela, A.L., et al. (2012). Phosphorus is associated with coronary artery disease in patients with preserved renal function. PLoS one 7 (5), e36883.

DuPaul, W. D., R. A. Fisher, & J. E. Kirkely (1996). Natural and exvessel moisture content of sea scallops (*Placopecten magellanicus*). Virginia Marine Resource report no. 96-5. Gloucester Point, VA: Virginia Sea Grant Marine Advisory Program.

FSIS, 2018. Safe and suitable ingredients used in the production of meat, poultry and egg products; http://www.fsis.usda.gov/wps/portal/fsis/topics/regulatory-compliance/labeling.

WHO, 2018. Food additives-Facts sheets; http://www.who.int/news-room/fact-sheets/detail/food additives.

CODEX STAN 315-2014. CAC Standard for fresh and quick frozen raw scallop products. Adopted in 2014. Revised in 2017. Amended in 2016. https://www.fao.org/fao-who-codexalimentarius/sh-proxy/en/

De Vido de Mattio, N. D., M. E. Paredi & M. Crupkin. 2009. Postmortem changes in the adductor muscle of scallop (Chlamys tehuelchus) in chilled and frozen storage. J. Aquat. Food Prod. Technol. 10:49–60.

Elemental Analysis and the Dumas Method https://www.velp.com/en-ww/dumas-method-1.aspx

EFSA, 2013. Assessment of one published review on health risks associated with phosphate additives in food. *EFSA J.* 11 (11), 3444.

Federal Research Centre for Fisheries. Institute for Fishery Technology and Fish Quality Institute for Fishery Ecology. Report of the 256. Cruise of "FFS Walther Herwig III" 16.9.-12.10.2003 Chief Scientists: Hans-Jürgen Kellermann (IFÖ) Ines Lehmann (IFF).

Foley, R.N., Collins A., Herzog C., Ishani A., & Kalra P. (2009). Serum phosphorus levels associate with coronary atherosclerosis in young adults. *J. Am. Soc. Nephrol.* 20 (2), 397 - 404

Gonçalves, A.A & Duarte Ribeiro J.L. (2008). Do phosphates improve the seafood quality? Reality and legislation. Pan-American *Journal of Aquatic Sciences*. Accessed at 12.03.2023 on <https://www.researchgate.net/figure/Use-of-phosphates-in-fish-according-to-Codex-Alimentarius_tbl2_228633771>.

Harlow, J. Sea Scallops: How to avoid getting soaked. Available in: http://www.bpe.com/ food/columns/ harlow/sea-scallops.htm - Access in 10.08.04.

Karl H, Bekaert K., Berge J.-P., Cadun A., Duflos G., Poli B.M., Tejada M., Testi S. and Timm-Heinrich (2013). WEFTA Interlaboratory Comparison on Total Lipid Determination in Fishery Products Using the Smedes Method. *Journal of AOAC International* 95, 1-5.

Karl, H., Äkesson, G., Etienne, M., Huidobro A., Luten, J., Mendes, R., Tejada, M. and Oehlenschläger, J., (2002). WEFTA Interlaboratory Comparison on Salt Determination in Fishery Products. *Journal of Aquatic Food Product Technology* 11, 215-228.

King, I., Childs M. T., Dorsett C., Ostrander J. G., & Monson E. R. (1990). Shellfish: proximate composition, minerals, fatty acids, and sterols. *J. Am. Diet. Assoc*. 90:677–685.

LBFG (Lebensmittel- und Futtermittelgesetzbuch, German Food and Feed Code) (2008). Bestimmung des Gesamtphosphorgehaltes in Fleisch und Fleischerzeugnissen. Photometrisches Verfahren. In: Amtliche Sammlung von Untersuchungsverfahren nach§ 64 LBFG. Belin, Germany: Beuth-Verlag. Available at: http:// www.methodensammlung-bvl.de.

Makri, M. 2009. The biochemical textural and sensory properties of frozen stored (–22C) king scallop (*Pecten maximus*) meats. *Afr. J. Biotechnol*. 8:3893–3903.

Manthey-Karl M., Lehmann I., Ostermeyer U., Rehbein H. & Ute Schröder (2015). Meat Composition and Quality Assessment of King Scallops (*Pecten maximus*) and Frozen Atlantic Sea Scallops (*Placopecten magellanicus*) on a Retail Level.

Manthey-Karl, M., Schröder, U., & Wagler, M. (2014). Zur Qualität tiefgefronerer Kammmuscheln. Rundschau für Fleischhygiene und Lebensmittelüberwachung : RFL, 66(3), 90-93.

Miller E.L., Bombo A.P., Barlow S.M. and Sheridan B (2007). Repeatability and Reproducibility of Determination of the Nitrogen Content of Fishmeal by the Combustion (Dumas) Method and Comparison with the Kjeldahl Method: Interlaboratory Study. *Journal of AOAC International* 90, 6-20.

Naidu, K. S. 1987. Efficiency of meat recovery from Iceland scallops (Chlamys islandica) and sea scallops (Placopecten magellanicus) in the Canadian offshore fishery. *J. Northwest Atl. Fish. Sci*. 7:131–136.

Ocano-Higuera, V. M., A. N. Maeda-Martinez, M. E. Lugo-Sanchez & R. Pacheco-Aguilar. 2006. Postmortem biochemical and textural changes in the adductor muscle of *Catarina scallop* stored at 0C. J. *Food Biochem*. 30:373–389.

Rippen, T.; Sutton, H.; Lacey, P.; Lane, R.; Fisher, R., & Dupaul, W. (1993). Functional, microbiological and sensory changes in sea scallops (*Placopecten megallanicus*) treated with sodium tripolyphosphate during iced storage. *Proceedings of the 18th Annual Tropical and Subtropical Fisheries* Technological Conference of the Americas. Virginia (USA): p. 51-71.

Smedes F. (1999). Determination of Total Lipid Using Non-chlorinated Solvents. *Analyst* 124, 1711-1718.

Surendran Nair, M., Nair, D. V. T., Kollanoor Johny, A., & Venkitanarayanan, K. (2020). Use of food preservatives and additives in meat and their detection techniques. *Meat Quality Analysis*, 187–213. doi:10.1016/b978-0-12-819233-7.00012-4

Tudor L. (2013). *Utilizarea noilor tehnologii de control si expertiza a produselor alimentare*. Controlul calităţii peştelui şi produselor acvatice. Calitatea moluştelor comestibile. Proiectul: perfecţionarea resurselor umane din medicina veterinară. POSDRU/ 81/3.2./S/58833. Online at: https://www.edu-veterinar.ro/ files/download/prezentari/siguranta-alimentelor/Controlul-calitatii-pestelui-si-produselor-acvatice.pdf

Tudor L, Ciocîrlie N, & Boițeanu C.N. (2022). *Medicină Veterinară și Sănătate Publică*. Ed.Universitară Carol Davila București. ISBN978-606-011-243-3. pp 200-204