THE QUALITY AND STORAGE STABILITY OF SALTED REDFISH PRODUCTS AS AFFECTED BY DIFFERENT SALTING METHODS AND STORAGE TEMPERATURES

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ABSTRACT

The aim of the study was to determine how quality and storage stability of the final products are affected by different salting procedures. Four different salting treatments were applied to redfish fillets. Group 1 were only dry salted, group 2 and 3 were treated with 18% brine then dry salted and stored for 41 days at 5°C and 20°C in wax carton boxes. The only difference between the two groups was that group three was treated with polyphosphate. Group 4 was treated with 5% brine and frozen in plastic bags in boxes at -18°C. Quality of final desalted products was determined using physiochemical analysis. A significant increase in yield was obtained during brining. A significant decrease in pH, water activity and moisture were observed in the dry salted product after dry salting. The L*(whiteness) of fillets decreased after brining and increased after dry salting; significant increase in lightness was observed after desalting. The Lipid analysis parameter (FFA, TBARS and lipid content was lower during storage at 5°C than 20°C). A physical quality analysis of the final product after 41 days in storage was conducted. The result showed lipid oxidation in the samples. Fillets stored at room temperatures were high in yellowness. The light salted group stored at -18°C had better physical characteristics, no yellowness in the muscle was observed, and there was no spoilage in taste after cooking.
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<th>Abbreviation</th>
<th>Description</th>
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<tr>
<td>$a_w$</td>
<td>Water Activity</td>
</tr>
<tr>
<td>FAO</td>
<td>Food and Agriculture Organization</td>
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<td>FFA</td>
<td>Free fatty acids</td>
</tr>
<tr>
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<td>Peroxide value (primary oxidation product)</td>
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<tr>
<td>WHC</td>
<td>Water Holding Capacity</td>
</tr>
<tr>
<td>TBARS</td>
<td>Thiobarbituric acid reactive substance (secondary oxidation product)</td>
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<td>STATIN</td>
<td>Statistical Institute of Jamaica</td>
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1 INTRODUCTION

Salting is an old method used in preserving food and involves the diffusion of salt throughout the muscle tissues. Salting leads to a self-stable product, able to be stored for several months. (Andres et al., 2001). Fishery products, including salted fish, play a major role as protein source for Jamaicans (Figure 1). Fish is the second most important contributor to animal-origin protein intake in Jamaica, after poultry meat with annual per capita consumption of fish at 15.73 kg/person, which is one of the highest in the Caribbean region. This high demand for fish shows the potential for expansion of the local Aquaculture sector (FAO, 2014). The consumption of salted cod is firmly rooted in Jamaican culture and has been a part of the Caribbean cuisine from colonial days, being the main constituent of Jamaica's national dish (ackee and saltfish). Even though there is no local production of salted fish in Jamaica, there is a high consumption of salted fish products in Jamaica including salted cod, herring and mackerel. Jamaica imported around 600 tonnes of dried and salted cod from Canada and Norway in 2014 to the value of $8.3 million USD (STATIN, 2014).

Fish products are the third largest food type imported to Jamaica, about 12% of total food imports in 2010. As much as 41 000 tons of fishery products were consumed in 2010. In contrast, the local fishery production has amounted to 19 000 tons in recent years, where 68% originated from wild catches and 32% from aquaculture (Wurmann, 2011).

Figure 1: Jamaica’s fish consumption, fish product import and local fish production from 2000 to 2013 (STATIN, 2014)

Salted fish accounts for most of the imported fish products to Jamaica. The knowledge regarding salting of fish to produce market standard value-added product are currently lacking in Jamaica.

There are vast and untapped opportunities in research and optimization of salting technologies through the aquaculture industry as local salted fish is non-existent on the market and has not been introduced in Jamaica. It has the potential of having a significant socio-economic value due to the consumption pattern of Jamaicans. This technology would present an opportunity for employment as well as diversify the marketing of fishery products of locally produced fish. It has the potential of having a significant socio-economic value due to the consumption pattern of Jamaicans. This technology would present an opportunity for employment as well as
diversify the marketing of fishery products of locally produced fish (Tilapia) in the future. Value addition in in the aquaculture industry is currently low resulting in poor prices. The local fish are sold mainly live, gutted, scaled or to a lesser extent frozen. The cost of production is high leading to low profits and therefore low investment in the sector. There are opportunities to make the sector more marketable and profitable through value addition, such as salting of our locally produced aquaculture fish species.

The Fisheries Division aims to diversify the aquaculture industry, expand the market of Jamaica’s fish product, and increasing the development of value added fish products are of utmost importance. In the recent past, the Fisheries Division has conducted some experiments of salting farmed tilapia. It was apparent that the expertise within this area was minimal hence the end product was not of the desired quality.

The skills and technology acquired for salting of tilapia are desirable and would meet the Department’s objective of developing value-added fish products. Such development would promote the value chain, opening opportunities to potential investors, create employment and aid in the revitalization of the aquaculture industry.

Light salting is a salting procedure which involves bringing fillets at a lesser saturated salt concentration than heavy salted fish. This product is packed and stored frozen. It targets people who like ready to cook food. It does not require desalting, it can just be thawed and cooked as desired. Such a product has its place in every culture including Jamaica, especially among young adults and busy working moms. Hence this research will also explore this salting technology.

**Overall objectives**

The main objectives of the present study are to increase the knowledge of fish salting, compare the effects of different salting methods, and estimate the storage stability of the final products. The knowledge and expertise gained through the project will be transferable to Jamaica’s situation as the intention is to introduce the methods in my place of work on our locally grown species tilapia and catfish (*Pangasius hypophthalmus*). 

The specific objectives of the research are to:

- compare the effects of different salting procedures on the physical and chemical properties of the final products
- determine how quality and storage stability of the final products are influenced by different salting procedures
- find how different salting procedures affect yield and compare the effects of different storage temperatures on the shelf life of heavily salted fish.
- compare the effects of different storage temperatures on the shelf life of heavily salted fish products.
2 LITERATURE REVIEW

2.1 Salting

Salting is a very old method used in preserving food which involves the diffusion of salt throughout the muscle tissues. In addition to preserving fish salting is also used as a preliminary method to some other processing such as smoking, drying and marinating (Andres et al., 2001). Products that are salted are stable and can to be stored for several months but require desalting and cooking before consumption. Salting process implies important changes in composition and structure of the tissue, and the extension of these changes may affect the behaviour during further drying and desalting operations, in terms of mass transfer kinetics and sensorial properties of the final product (Andres et al., 2004). Salting procedures have been altered to improve yield and stabilise quality of the salted products during storage. The production has changed from being a single-step process salting to a multistep procedure, starting with presalting methods like injection and/or brining (Nguyen, 2011).

Salting is usually performed by dry-salting, brine-salting, injection-salting or a combination of these methods. The most commonly used methods by the industry are dry salting (pile) and brine salting (Birkeland, 2005). The main features of salting are the removal of some of the water from the fish flesh and its partial replacement by salt (Turan, 2007). When brine or dry salt are used as salting agents, two main simultaneous flows are usually generated; water loss and salt uptake. According to Thorarinsdottir (2010) “The changes in salting procedures and curing conditions have altered the characteristics of the products, increased weight yields and improved commercial quality.”

Salting methods and salt concentrations are thought to influence the degree of protein denaturation/aggregation, resulting in differences in water retention of fish muscle. Thorarinsdottir et al. (2011) discovered that water holding capacity increases with increasing salt concentration in the muscle in liquid phase (zNaCl), mainly due to muscle swelling. However, a drop in WHC is observed near the zNaCl value of 15% due to protein aggregation. Salting is used to obtain special products as it causes favourable changes in taste and texture of fish muscle. Salted products are used in special and traditional dishes in many countries, e.g. Bacalao in Southern Europe and Latin America and in Jamaica.

2.2 Dry salting

Dry salting is the original salt-curing technique used when salting cod in many countries (Gallart-Jornet, 2007). In the old way of dry salting, butterfly (split) fish or fillets were stacked with alternating layers of dry, coarse salt and kept for weeks. Presently, the fish is piled with alternating layers of salt in plastic tubs with a hole in the bottom to drain away the liquid extracted from the fish. The salt uptake is initiated by extraction of liquid from the fish muscle and solubilisation of the salt. During the salting process, salt diffuses into the fish muscle, while liquid diffuses out. The driving forces for water and salt diffusion are mainly concentration gradient between the fish muscle and the surrounding media and within the fish muscle, pressure gradient and water activity gradient. As a consequence of increased salt concentration in the fish muscle, protein denaturation and aggregation occur, affecting further penetration of the salt into the fish muscle. The water content of the cod muscle is usually reduced from approximately 82% to about 54% during the salt curing process. The salt content of the final product is about 20-22%. The temperature during the process is 2 ± 2 °C and relative humidity (RH) approximately 78 ± 4% (Nguyen, 2011).
2.3 Brining

Brine salting has become popular in recent years for processing salted cod as a pre-salting step, followed by dry salting (Thorarinsdottir et al., 2004). Brine salting is conducted by immersing butterfly (split) fish or fillets into ready-made brine prepared from coarse salt and tap water. There can be a variation of the brine concentration used and other condition used between producers. The brining process is usually carried out for 2-4 days, thereafter pickling and/or dry salting is applied for 14-21 days. During the brining process, the brine concentration and salting rate can be controlled either by increasing the brine to fish ratio or by supplementing salt into the brine to compensate for the salt uptake into the fish muscle. The salting rate also depends on initial brine concentration used. Therefore, brine salting has several advantages over dry salting and pickling including shorter processing time due to higher salt uptake and higher weight yields due to a better control over the rate of salt uptake and water loss in the muscle (Andres et al., 2001; Thorarinsdottir, 2010). The influences of salt concentration on proteins are also believed to influence the rate of salt uptake and water loss. Brining can also improve colour and appearance of salted cod products when applied at levels < 20% (Thorarinsdottir et al., 2004). Brining aids in the increase of salt uptake and it also provides a barrier to contacts of the fish with oxygen, which can initiate rancidity. The strength of the brine also affects the changes in water holding capacity and yield of the final product due to influences on conformational changes and accumulation of proteins in fish muscle (Thorarinsdottir et al., 2004). The pH of the brine is also important to control leaching of salt soluble proteins. More actin and myosin heavy chains are released in brine of pH 6.5 than pH 8.5 (Martinez-Alvarez & Gomez - Guillen, 2005).

2.4 Storage of salted fish

After salting the salted cod is normally packed with excess salt in 25 kg waxed carton boxes and stored at 0-2 ºC and relative humidity of 78%. A heavily salted cod product may experience problems of yellow/brownish discoloration of the flesh which affects the commercial quality rating of the product (Thorarinsdottir, 2010; Nguyen, 2011). A red/pink discoloration of salted cod is thought to be due to the growth of red halophilic bacteria, which produce red carotenoid pigments (Vilhelmsson, 1997). The growth of bacteria and the development of lipid oxidation may be delayed by storing at lower temperatures. These biochemical and chemical changes that occur in the muscle during salting and storage are termed ripening. Ripening results in changes in texture and flavour of the products. Oxidation of highly unsaturated lipids is among the chemical changes that occurs. It which is accelerated by salting and proteins aggregation. (Harris & Tall, 1994). The myosin heavy chain is most susceptible to denaturation by heavy salting (Thorarinsdottir K. A., Arason, Geirsdottir, Bogason, & Kristbergsson, 2002). The salting procedures applied influence the structural changes of proteins in the fish muscle (Barat et al., 2002). Therefore, weight changes during salting and rehydration differ depending on the method used. Injection of salt into fish fillets before brining and dry salting has been shown to increase the water retention in the final product. Injecting salt into fillets will allow for more even distribution of salt already in the beginning of salting, as compared to brining (Thorarinsdottir et al., 2011).
2.5 Rehydration methods

Rehydration is a need before cooking of salted fish. This process is carried out by soaking the salted cod in fresh water to reduce salt content to suitable concentration to be consumed by humans. It removes the salt from the fish muscle to the surrounding water (Barat et al., 2004). In earlier times, it was tradition for rehydration to be carried out in the home. Currently, consumers tend to spend less time on food preparation and prefer more convenient products and ready-to-eat products (Shiu, 2004; Nguyen, 2011). There are many factors that affect the rehydration process. They include the ratio of water to fish, water change frequency during the rehydration process, quality of water, temperature, pressure, how the fish was salted as well as the size and whether the products have skin or are skinless (Andres et al., 2001). The common way to carry out the rehydration process is to soak the salted fish in tap water for at least 24 hrs at refrigerated or ambient temperature. During the rehydration process, water is usually changed one or two times to increase the rehydration rate (Barat et al., 2004).

Desalting of heavily salted fish fillets is an important step in the salt fish processing industry as it is used in the preparation of many traditional dishes. The end user at home mainly carries out desalting by soaking the product in water for 24 hours in a refrigerator or at ambient temperature, and this process can be improved by frequent exchange of water. However, a study done by Barat et al. (2004) showed that when cod fillets were desalted without exchange of water and the desalting time extended, the waste was reduced, and the yield increased. There are two main changes expected during the desalting process, reduction of NaCl concentration to an acceptable level for consumption, and rehydration to obtain high sensory quality and high processing yield.

2.6 Lipid oxidation

Lipid oxidation is a major cause of quality deterioration in muscle food. It results in off-flavour, colour deterioration, loss of nutritional value and generation of potentially toxic compounds, which have negative effects on human health (Erikson et al., 2004). Lipid oxidation is influenced by many factors, including, fatty acid composition, temperature, pressure, water activity, pH, light, salt concentration and processing methods. Sodium chloride has been shown to have a prooxidant effect on lipid oxidation in variety of foods including chicken, pork and fish. Gheisari et al., (2010) studied the effects of different salt concentrations of 1% and 6% on oxidative stability of minced chicken and beef during storage for 20 days. They demonstrated that samples with higher salt concentrations have higher PV and TBARS values. Lipid oxidation increases progressively with increased salt concentration, at relatively low salt contents (0.5-2.5% in refrigerated fish) (Nguyen, 2011). Aubourg & Ugiano (2002) observed a higher peroxide formation during storage pre-treated mackerel with NaCl solutions (5%, 10% and 20%) than that of untreated mackerel.

Colour of fish fillets is a very important sensory attribute in marketing. Colour of fish fillets mainly depends on pH and factors like water content, protein content, lipids content, muscle pigments and their interactions. Lightness (L* value) changes with the content of moisture in the fish muscle. Lipid oxidation increases the yellowness (b* value) of fish fillets while non-enzymatic browning reactions and oxidation of haemoglobin at low pH increase the redness (a* value) of fish muscle.

Lipids are important biochemical compound of fish and play a role as a structural component of the muscle membranes, as storage droplets of triacylglycerol between muscle fibres and as
adipose tissue. Fish are classified into three different groups depending on the lipid content: lean fish - lipid content is less than 2%, semi-fatty fish - lipid content is in the range of 2 to 10%, and fatty fish - lipid content is more than 10%. Redfish is classified as a semi-fatty fish with lipid content of 3.2-8.1% (Murray & Burt, 1969).

2.7 Description of Redfish

The redfish (Sebastes marinus) (Figure 2) is a species commonly found in Icelandic waters, but the main fishing grounds are at the edge of the continental shelf at 200 to 400 m depth south and west of Iceland, (Icelandic Ministry of Fisheries and Agriculture, 2016). Redfish is a moderately fatty fish with fat content 3.2-8.1%, protein content 16.8-19.7 (%), Moisture content 73-79% and energy value of 460-670 calories/lbs (Table 1). This fish has been marketed frozen, whole or as fillet, smoked (cold or hot) (Belitz et al., 2004).

Figure 2: Golden redfish (Sebastes marinus) (Adapted from www.fisheries.is)

Redfish grows slowly and attains an average total length of 35-40 cm though centennial individuals grow up to 100 cm and 15 kg in weight. Fishing of redfish takes place all year with peaks of catches in the late winter. Bottom trawling is the exclusive method used in this fishery (Jonsson & Palsson, 2006). Initially the golden redfish had no commercial value for Iceland and was always discarded in the cod catches but with time fish meal and oil production developed and finally redfish gained recognition as a good food fish. About 50% of the redfish catch is processed and frozen at sea and the rest is iced and exported fresh in containers or by air. The main market for Icelandic redfish is Germany although a considerable percentage is also exported to other western European countries. The market for redfish in eastern Asia is expanding and Japan is now the second largest importer of the golden redfish, (Jonsson & Palsson, 2006).

<table>
<thead>
<tr>
<th>Chemical Composition</th>
<th>Content</th>
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<tr>
<td>Protein</td>
<td>16.8-19.7 (%)</td>
</tr>
<tr>
<td>Lipid</td>
<td>3.2-8.1 (%)</td>
</tr>
<tr>
<td>Moisture</td>
<td>7.3-7.9 (%)</td>
</tr>
<tr>
<td>Energy value</td>
<td>460-670 (Cal/lb)</td>
</tr>
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</table>
3 MATERIALS AND METHODS

3.1 Materials

Fresh redfish fillets (Sebastes marinus) were purchased from HB Grandi Ltd. (Reykjavik, Iceland) and were used for the study. Food grade salt, polyphosphate (carnal) and waxed carton boxes were purchased from Saltkaup Ltd. (Hafnarfjordur, Iceland). Other consumables were collected from the local market, Reykjavik, Iceland.

Chemicals used during analysis of samples were of analytical grade, and purchased from Fluka (Buchs, Switzerland) or Sigma-Aldrich (Steinheim, Germany / St. Louis, MO, USA).

3.2 Experimental design

The study was carried out according to the flowchart depicted in Figure 8. A total of 450 fresh redfish fillets (post rigor mortis) were transported in cold conditions (0 °C) to the laboratory and directly applied to 4 different salting procedures; 1) dry salting only, 2) brining followed by dry salting, 3) brining with polyphosphates (PP) followed by dry salting, and 4) light salting followed by freezing.

The dry salting (group 1) was carried out by placing 90 kg of fillets in a plastic box with alternate layers of salt at a ratio of 1 kg of fish to 0.5 kg of salt. Each fillet was covered in salt for 22 days (Figure 3). The plastic boxes have holes at the bottom to allow excess liquid to drain away. This method is commonly referred to as salting out, getting the water out of the fish.

Figure 3: Experimental group 1

In experimental group 2, brine was applied to the fillets prior to dry salting. The fillets were placed in 18% brine solution at a fish to brine ratio of 1:1 for 48 hours. The fillets were then dry salted as previously described (Figure 4).

Figure 4: Experimental group 2

The salting procedure of the third experimental groups was identical to group 2; except 3% of polyphosphate (carnal) was added to the brine (Figure 5).
The last experimental group (group 4) contained light salted redfish fillets. The fillets were brined in 5% salt solution for 48 hours. These fillets were frozen directly after processing and stored at -18 °C for up to 41 days.

After the salting processes, experimental groups 1, 2 and 3 were stored in waxed carton boxes at 5 °C and 20 °C for up to 35 days (Figure 6).

After the salting processes, experimental groups 1, 2, and 3 were stored in waxed carton boxes at 5°C and 20°C for up to 41 days (Figure 7).
Figure 8: Experimental design for evaluation of different salting procedures of fresh redfish fillets and the storage stability of the final products.

3.3 Sampling

Sampling was performed of the raw material and after each processing step and throughout the storage period of each final product (Table 2). The heavily salted products were desalted prior to analysis on each sampling point. However, the colour of the final product was evaluated both before and after desalting. Analysis performed at each stage are summarized in Table 2. The light salted product was evaluated after freezing, and after 41 days storage at -18 °C.

A total of 25 sampling was performed. On each sampling point, 5 fillets from each experimental group was pooled as one sample, in duplicate, resulting in 10 fillets per group. Additional 5 fillets from each group was used for evaluation of cooking yield. The changes in physical (cooking yield, colour, water holding capacity, water activity and chemical properties (proximate content, salt content, total volatile basic nitrogen, thiobarbituric acid reactive substances, pH, free fatty acids, were evaluated as described in Table 2.
Table 2: Processing activities, sampling and analysis that was performed in present study.

<table>
<thead>
<tr>
<th>Names</th>
<th>Processing Stages</th>
<th>Sampling Dates</th>
<th>Physical and Chemical Analysis</th>
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<tbody>
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<td>14.12.17</td>
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</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>√</td>
</tr>
<tr>
<td>Dry Salting</td>
<td>3</td>
<td>16.12.17-06.01.17</td>
<td>√</td>
</tr>
<tr>
<td>Storage</td>
<td>4</td>
<td>Every 10 days after processing: 0 days 6.01.17</td>
<td>√</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10 days 16.01.17</td>
<td>√</td>
</tr>
<tr>
<td></td>
<td></td>
<td>20 days 26.01.17</td>
<td>√</td>
</tr>
<tr>
<td></td>
<td></td>
<td>30 days 6.02.17</td>
<td>√</td>
</tr>
<tr>
<td></td>
<td></td>
<td>35 days 11.02.17</td>
<td>√</td>
</tr>
</tbody>
</table>

3.4 Physicochemical analysis

Water content was determined according to ISO 6496:1999. About 5g of sample was weighed accurately (±1 mg) and placed in an aluminium foil dish which was prepared with a thin layer of sea-sand and a glass rod. The samples were mixed thoroughly with the sand. The glass rod was kept on the dish and then left to dry for 4 ± 0.1 h in the oven at 103 °C. The dish was removed from the oven and allowed to cool to ambient temperature in a desiccator for about 15 minutes. The water content was calculated using the formula as follows:

\[
W = \frac{m_1 - (m_2 - m_3)}{m_1} \times 100[\%]
\]

Where:
- \(m_1\) was the mass of the test portion (g)
- \(m_2\) was the mass of the dish, test portion sand and glass rod (g)
- \(m_3\) was the mass of the dish, dried test portion, sand and glass rod (g)

Salt content (NaCl) was determined according to a standard method, Volhard titration (Horwitz, 2000)

Water activity was measured using a water activity meter (Aqua Lab). Approximately 2g of homogenised sample was placed onto the sample cup (no more than half full) prior to taking readings.

Colour was analysed using Minolta Chroma meter CR-400 (Minolta Co., Ltd; Osaka, Japan). Colour measurements included; the L value, lightness on the scale of 0 to 100 from black too white; a value, (+) red or (-) green; b value, (+) yellow or (-) blue. The colour was measured at three points laterally from the head to the tail of the fish fillet.
Cooking yield was expressed as percentage of retaining weight compared to the weight of sample before cooking. Three pieces from the middle part of each fillet (n=3) were cooked using a steaming oven (Convotherm OGS 6.10 Combi Convection Steam Oven, Elektrogeräte GmbH, Egolfing, Germany) at 90 °C for 10 min. The cooking yield was calculated as follows:

\[
\text{Cooking yield (\%)} = \frac{W_{\text{cooked}}}{W_{\text{uncooked}}} \times 100
\]

Where:
- \(W_{\text{cooked}}\) is the weight of cooked sample (g).
- \(W_{\text{uncooked}}\) is the weight of uncooked sample (g).

The pH of samples was measured using a digital pH meter (Knick-Portamess 913 pH, Berlin, Germany). The pH values were measured using blended samples of fish fillets. The pH meter was calibrated for pH-4, pH-7 and pH-10 before starting measurements. The average value of two readings were taken for each measurement.

Lipid content was analysed according to the method of Bligh & Dyer (1959). Total lipids (TL) were extracted from 25 g samples (80±1% water) with methanol/chloroform/0.88 % KCl(aq). The lipid content was determined gravimetrically, and results expressed as g lipid / 100 g of the sample.

The free fatty acid content (FFA) was determined according to the method of Lowry & Tinsley (1976)) with modification from Bernardez et al. (2005). About 3 mL of the lower phase resulting from lipid extraction (Bligh and Dyer 1959) was added in a screw cap culture tube. Any solvent present was removed at 55 °C using a nitrogen jet. After cooling down, 3 mL of cyclohexane were accurately added by 1 mL of cupric acetate–pyridine reagent and vortex for ~40 seconds. After centrifugation at 2000 rpm for 10 min at 4 °C, the upper layer was read at 710 nm in spectrophotometer. The FFA concentration in the sample was calculated as µmol oleic acid based on a standard curve spanning a 2-14 µmol range.

3.4.1 Thiobarbituric acid reactive substances (TBARS)

The secondary oxidation product was evaluated according to the method of Lemon (1975) with some modifications used. A sample (5.0 g) was homogenized with 10.0 mL of trichloroacetic acid (TCA) extraction solution (7.5% TCA, 0.1% propyl gallate and 0.1% EDTA mixture prepared in ultrapure water) using a homogenizer at maximum speed for 10 seconds (Ultra-Turrax T-25 basic, IKA, Germany). The homogenized samples were then centrifuged at 5100 rpm for 20 min (TJ-25 Centrifuge, Beckmann Coulter, USA). Supernatant (0.1 mL) was collected and mixed with the 0.9 mL thiobarbituric acid (0.02 M) and heated in a water bath at 95°C for 40 min. The samples were cooled down on ice and immediately loaded into 96-well microplates (NUNC A/S Thermo Fisher Scientific, Roskilde, Denmark) for reading at 530 nm (Tecan Sunrise, Austria). A standard curve was prepared using tetraethoxypropane. The results were expressed as µmol of malonaldehyde diethylacetal per kg of wet muscle.

3.5 Statistical analysis

The results were analysed using Microsoft Excel 2007 to calculate the means and standard deviations for all multiple measurements and to generate graphs. One-way ANOVA were used to compare data sets with Duncan’s multiple range test. The significance level was set at \(p \leq 0.05\).
4 RESULTS

4.1 Chemical composition of redfish (Sebastes marinus)

The chemical composition of the raw fillets was measured. The water content was 79.0±4%, the salt content was 0.3±3%, the water holding capacity was 86.4%, and the lipid content was 4.2 %. Additionally, total volatile basic nitrogen (TVB-N) was determined to be 12.7±9 mg N/100g.

4.2 Yield

4.2.1 Curing Yield

All 3 groups of redfish fillets increased in weight during brining. Fillets that were brined prior to dry salting maintained a higher yield throughout dry salting than the group that was only dry salted (Figure 9). This group showed greater water absorption, which was demonstrated by the higher weight values in fillets after brine salting. The yield of both dry salted only and pre-brined fish fillets gradually decreased during dry salting.

![Figure 8: Changes in curing yield of redfish fillets during brining and dry salting.](image)

4.2.2 Soaking Yield

Soaking yield was determined during storage by calculating the difference in yield before and after desalting. Soaking yield ranged from 124-141% (Figure 10). The group that was only dry salted maintained a higher yield throughout storage at both temperatures (20°C, 5°C) than the two groups that were brined pre-dry salted. There was an increase in yield during cold storage for the fillets that were pre-brined, whereas the group that was dry salted only decrease in yield.
Figure 9: Changes in soaking yield of redfish fillet during storage at 5°C and 20°C

4.2.3 Cooking yield

The cooking yield of raw material was 88%. The cooking yield increased during brining for all 3 groups which ranged from (88%-92%). Fillets that were light salted had a significantly higher cooking yield (p<0.05). However, there was no significant difference between the fillets that were treated with 18% brine and 18% brine +pp. Cooking yield decreased during dry salting for all groups, however there was no significant difference among the groups (Figure 11).

Figure 10: Cooking yield of red fish fillets during curing for all treatment groups.

During storage fillets that were light salted gave a significantly higher yield than the other groups (p<0.05). All three groups decreased in yield during storage at 5°C. The group that was dry salted only maintained a higher yield, though not significant. During storage at room temperature the group that was dry salted only maintained a higher yield than the groups that were pre-brined. After 20 days the fillets that were dry salted only and those treated with 18% brine increased significantly in yield, however fillets that were treated with 18+pp decreased significantly (Figure 12).
Figure 11: Cooking yield of redfish fillet during processing, and storage at stored at 5\(^\circ\) C, 20\(^\circ\) C and -18\(^\circ\) C

4.3 pH

The pH of the raw material of redfish was 6.65 which decreased during brining for 2 groups (18\% brining + dry salting and 18\% brining + pp) however, the pH increased significantly with the group that was light salted (p<0.05) (Figure 13). There was a sharp increase in pH after desalting for all 3 groups that were dry salted. The groups that were brined prior to dry salting had a higher pH than the group that was only dry salted, (0.994 ± 0.86\%, 0.995 ± 0.60\% and 0.993 ± 0.87) though not significant (p<0.05).

Figure 12: Changes in pH value of salted redfish of raw material and during processing.

4.4 Water activity

Water activity decreased from 0.990 in raw fillets to 0.940 and 0.930 during brining for the groups 18\% Brine and 18\% brine +PP respectively, there was however an increase in water
activity during light salting though not significant (Figure 14). Water activity of all groups increased during desalting, however there was no significant difference among the groups.

![Figure 13: Changes in water activity of redfish fillet raw material and after brining and desalting for all groups.](image)

### 4.5 Water content

Water content for red fish fillet was initially 79%±4% (Figure 14). After brining the fillets that were light salted increased in moisture 81%, however the fillets that were treated with 18% brine and 18%+pp decreased in moisture (75% and 71%). The moisture content for all groups decreased significantly after dry salting (p<0.05). Fillets that were dry salted only had a lower moisture content (Figure 15).

![Figure 14: Water content of redfish fillets during curing for all treatment groups](image)
dry salted only decreased significantly in water content in comparison to the other groups (p<0.05) (Figure 16).

4.6 Water holding capacity

The water holding capacity of redfish raw material was 86.4% (Figure 17). Water holding capacity increased significantly during brining for all groups with no significant difference among the groups. It then decreased among all groups after desalting.

Figure 17: Water holding capacity of redfish fillet raw material and after brining and desalting for all groups
4.7 Salt content

The salt content in the raw fillets was 0.3%±3% (Figure 18). There was a significant increase in salt content during brining for all groups (p<0.05). Fillets that were treated with 18% brine and 18%brine+pp had a significantly higher increase during brining than the group that was light salted. All 3 groups had a significant increase in salt content during dry salting. Fillets that were dry salted only had a significantly lower increase in salt content during dry salting than the groups that were brined pre-dry salting.

![Figure 17: Salt content of redfish fillet during curing](image)

During storage there was no significant difference among groups at the same storage temperatures. At storage temperature of 5°C, fillets that were brined pre-dry salting increased in salt content, however fillets that were dry salted only decreased in salt content (Figure 19). During storage at 20°C the group that was treated with 18%brine + polyphosphate and the group that was dry salted only increased in salt content. However, the group that was treated with 18% brine decreased in salt content significantly after day 13. Fillets that were light salted and stored at -18°C maintained a higher salt content throughout storage.
The lightness of redfish fillets decreased during brining for all groups (Figure 20). The group that was treated with polyphosphate had a higher brightness than the other brined groups. Salt decreases the lightness of red fish fillet. After dry salting the lightness of the fillets in all groups was reduced though it was not significant. After desalting the lightness of the group increased significantly (p<0.05) to the lightness in the raw material.

**Figure 18**: Salt content of redfish fillet during storage at different temperatures (5°C, 20°C and -18°C)

### 4.8 Colour

#### 4.8.1 L-value

The lightness of redfish fillets decreased during brining for all groups (Figure 20). The group that was treated with polyphosphate had a higher brightness than the other brined groups. Salt decreases the lightness of red fish fillet. After dry salting the lightness of the fillets in all groups was reduced though it was not significant. After desalting the lightness of the group increased significantly (p<0.05) to the lightness in the raw material.
4.8.2 L-value salted fillets

Fillets treated with 18% brine + pp increased in lightness throughout storage at both storage temperatures (Figure 21). This group was significantly lighter than the group that was light salted and dry salted only. There was not significant difference between fillets that were treated with polyphosphates and fillets treated with 18% brine throughout both storage temperatures.

At storage temperature of 5°C the group that was only dry salted had a significantly lower brightness than the other groups when stored at the same temperatures, however the brightness increased with storage days.
4.8.3  *L*-value desalted fillets

The desalted fillets increased in lightness throughout for all groups at both storage temperatures (Figure 22). The fillets that were treated with polyphosphate were significantly lighter than fillets that were treated with 18% brine and fillets that were dry salted only. The fillets that were dry salted only were darker than the fillets that were brined prior to dry salted throughout storage at both storage temperatures.

![Figure 21: Lightness (L-value) of desalted redfish fillet during storage at different temperature (5°C and 20°C) for all treatments.](image)

4.8.4  *a*-value

The *a*–value (redness) describes the intensity of green colour (negative) and in red colour (positive) of redfish fillet. Significant difference in redness was observed between salted and desalted red fish fillet (*p*<0.05). The fillets decreased in redness during brining for all groups with a further significant decrease during dry salting. The redness significantly decreased in all groups after desalting (*p*<0.05) (Figure 23).

![Figure 22: The redness a-value of redfish fillets during curing.](image)
All groups that were stored at room temperature had a negative value throughout storage. The red intensity decreased (Figure 24). Significant difference was observed between the groups that were brined prior to dry salting and the group that was dry salted only at both storage temperatures. The group that was dry salted only had a significantly higher intensity in redness throughout both storage temperatures in comparison to the group that was treated with 18% brine and the group that was treated with polyphosphate. Significant difference was observed in the fillets that were light salted and stored at -18°C. These fillets had a positive a-value throughout storage.

![Figure 23: The redness a-value of salted redfish fillets during storage at different temperatures.](image)

In the desalted fillets, the a-values of all groups were negative at both storage temperatures (20°C and 5°C) (Figure 25). There was no significant difference in redness among groups at both storage temperatures. The fillets that were dry salted only had a higher intensity of redness at both storage temperatures. All treatment groups decreased in redness throughout storage at both temperatures.

![Figure 24: The redness a-value desalted redfish fillets during storage at different temperatures.](image)
4.8.5  \textit{b-value}

The \textit{b} value (yellowness) describes intensity of blue (negative) and yellow (positive) colour of the redfish fillet. The \textit{b} value was negative for all groups during brining and dry salting (Figure 26). Significant difference was observed between the yellowness of the fillets after brining for all groups and after dry salting of all groups. After desalting the yellowness increased significantly for all groups.

![Figure 25: The yellowness \textit{b}-value of redfish fillets raw material and during brining, dry salted and desalted for all groups.](image)

The yellowness in all groups decreased at all the storage temperatures throughout storage (Figure 27). Significant difference was observed among groups at the different storage temperatures. Group 1 had a significantly higher yellowness at both storage temperatures in comparison to the other groups. There was no significant difference between groups 2 and 3 within the different storage temperatures. At 34 days in storage (day 56) the \textit{b} value of each group at 20°C equals the \textit{b} value of the same group stored at 5°C.

![Figure 26: The yellowness (\textit{b}-value of) salted redfish fillets raw material and processing for all groups.](image)
The $b$ value of the desalted fish fillets of all groups decreased throughout storage at both storage temperatures (Figure 28). Significant difference was observed in Group 1 (dry salted only) at $20^\circ$C. The yellowness was higher than the two groups that were brined prior to dry salting. There was no significant difference among the groups stored at cold storage.

![Graph showing the change in $b$ value over time for different groups.](image)

**Figure 27:** The yellowness ($b$-value) of salted redfish fillets raw material and processing and storage for all groups at different storage temperatures.

### 4.9 Lipid Quality

TBARS, Lipid content, and free fatty acids content (FFA) are shown in the figures below.

#### 4.9.1 TBARS

The TBARS of raw material was 1.54 $\mu$mol/kg (Figure 29). After brining TBARS increased in all groups to 15.5 $\mu$mol (18% brine), 9.6 $\mu$mol/kg (18% +pp) and 3.5 $\mu$mol/kg (light salted).

![Graph showing TBARS values for different conditions.](image)

**Figure 28:** TBARS ($\mu$mol/kg) of redfish fillets during curing and storage.
During storage TBARS for all treatments gradually increased up to day 20 then it gradually decreased throughout the end of storage decreased throughout for both temperatures (Figure 30). TBARS value is more stable in the group that was treated with polyphosphate than the other groups. Significant difference was observed with the light salted group (p<0.05). The TBARS value was significantly higher throughout storage.

![Graph showing TBARS (µmol/kg) of redfish fillets during curing and storage.]

**Figure 29:** TBARS (µmol/kg) of redfish fillets during curing and storage.

### 4.9.2 FFA

The FFA value for raw material was 2.6gFFA/g lipid. FFA decreased significantly during brining for all groups (Figure 31).

![Graph showing FFA values of redfish fillets of raw material during curing and storage of all groups at different storage temperatures.]

**Figure 30:** FFA values of redfish fillets of raw material during curing and storage of all groups at different storage temperatures.
FFA increases gradually throughout storage at 20°C and 5 °C storage except for group treated with polyphosphate stored at 5°C. The FFA value for light salted group stored at -18°C had a lower FFA value than all the other groups (Figure 32).

Figure 31: FFA values of redfish fillets of raw material during storage of all groups at different storage temperatures.

The lipid content of raw material was 4.2 % (Figure 33). During brining lipid content decreased in the group that was light salted and increased in the groups that were treated with 18% brine and 18%bine +polyphosphate. The group that was treated with polyphosphate had a significantly higher lipid content (will be treated as an outlier).

Figure 32: Lipid content of red fish fillet during brining.

During storage lipid content of group 3 and group 4 increased. At both storage temperatures of 5°C and 20°C the lipid content in all groups decreased (Figure 34).
At the end of storage (41 days in storage) a quality test was done on all groups at the three different storage temperatures by assessing the sensory characterises of the fillets. The fillets were desalted and thawed in the case of light salted fillets. They were observed for colour and then they were steamed, and a taste testing was done. All fillets were exhibiting signs of rancidity except for the light salted group that was stored at -18°C. The fillets that were stored at room temperature were most yellow and were gaping. Fillets that were dry salted only had a higher intensity of yellowness except for the light salted group that was stored at -18°C. The fillets stored at 5°C also exhibit yellowness but not as significant as those at room temperatures. The fillets that were treated with 18% brine + pp was lighter than the other two group of fillets. However, the fillet stored at freezing temperature looked fresh, there was no signs of rancidity and when cooked the muscle was white (Figure 35 & 36).

After tasting the fillet, it was concluded the light salted fillet were of the best quality, followed by the fillets that were treated with polyphosphate. It was also noted that the dark muscle in the light salted fillets had an off-flavour taste. The white muscle on the other hand was quite tasty.
Figure 34: Samples of cooked desalted and thawed redfish fillet that were stored at different temperatures after a storage period of 41 days.

Figure 35: Samples of cooked desalted and thawed redfish fillet that were stored at different temperatures after a storage period of 41 days.
5 DISCUSSION

The shelf life of food is the period for which it remains safe and suitable for consumption. Shelf life is also correlated to quality loss in processed and perishable food items. In this research the shelf life study of salted redfish was done by assessing their physiochemical characteristics (lipid content, TVB-N, FFA, TBARS, moisture content, water holding capacity, colour) at different storage temperatures using differ salting procedures.

Yield is a very import aspect from an economical point of view. The increase in yield during brining is attributed to greater water absorption during this process. Water and salt diffusions during the brining process are mainly due to the differences in concentration between the fish muscle and the surrounding solution (brine) and within the fish muscle. This is because brining controls the uptake of salt and reduces the aggregation and denaturation of proteins. This observation agrees with research done by Medina-Vivanco et al. (2002). This study showed that the group with the lowest salt concentration (light salted group) had the higher yield. According to Barat et al., (2002) brines with small concentrations of salt also promote the yield and water holding capacity more than saturated brines of more than 25%. Brining at low salt concentration (6%-15%) increases the water holding capacity due to salting-in effect in the beginning, but higher concentration of salt decreases the water holding capacity due to aggregation of protein (Nguyen et al., 2010). The results show the group that was dry salted only, had a higher soaking yield in comparison to the other groups. this is because of protein denaturation, resulting in low bonding capacity in the fish muscle. In agreement with (Gallart-Jornet, 2007) the degree of protein denaturation/aggregation also affected water holding capacity affects the WHC. The salting out effect (brining) affects the water holding capacity. In this study light salted group had a higher water holding capacity than the groups that were heavy salted.

In agreement with Nguyen (2011) it was observed that the salt content in the fillets group that was only dry salted were significantly higher than in pre-brined fish fillets after dry salting. This is due to the covering of the fillets with salt during dry salting a creates a huge difference in salt concentration between the outside and inside of fish fillets causing higher dissemination rate of salt. The salt can penetrate the flesh effectively.

During storage the fillets that were dry salted only had a higher cooking yield than the fillets that were treated with polyphosphates. This indicates that added phosphates does not affect yield in salted redfish fillet. During the storage time, the cooking yield of salted redfish fillets in all groups stored at 5 °C was lower compared to other fillets stored at different temperatures (20°C and -18°C). That can mainly be caused by degradation of organic substances in the muscle by enzymes. The cooking yield of the light salted fillets stored at -18 °C was higher that all the other groups. It was due to lower temperature resulting in a lower microbiological activity, enzyme activity and protein denaturation.

Moisture content and salt percentage play an important role in the keeping quality of salted fish products. During salting, the mass transfer occurs basically between salt and water: the fish muscle takes up salt and loses water.

Water activity in redfish fillets decreased during brining and the fillets had a further significant decreased (p<0.05) after dry salting. The water activity values of the fillets that were brined pre-dry salting were higher than in dry salted only fillets This may be due to the increase of salt content and decrease of water content of dry salted only fillets.
The pH values of raw material and after brining ranges from a value of 6.5 to 6.9 in all groups and the water activity was also high which resulted in these groups having a high WHC. However, after dry salting the pH, water activity and WHC decreased significantly in all groups. This agrees with Hamm (1986) whose studies state that if the pH value is higher than the isoelectric point of protein in fish muscle (pH 5). If pH is higher than this, water holding capacity and water absorption of the muscle can improve.

Colour is an important quality measure in salted fish. It’s a sensory attribute that can signifies rancidity and spoilage. Colour is also very important in marketing salted fish. The lightness in the muscle is more favourable and demanded by consumers. In this study there was no significant difference in the lightness of fish muscle after brining. However, the fillets increased in lightness among group during storage at all 3 temperatures. The desalted fillets were higher in lightness than the desalted fillets which is agreeable to an extent with research by (Brass & Costa, 2010) stated that whiteness of the fish fillets increased during the dry salting period and after desalting. It was observed that fillets that were treated with polyphosphate and stored at both 5°C and 20°C were lighter that the fillets in the other groups. This is because polyphosphate acts as an antioxidant therefore it slows down lipid oxidation in the fillets. Results showed that the yellowness in fillets increased in dry salting and desalting. During storage at temperatures of 5°C and 20°C the fillets that were dry salted only had a higher yellowness than those treated with 18% brine and 18% brine and polyphosphate. This indicates that lipid oxidation was higher in those fillets and this is being obvious with the quality test down which showed that samples that were dry salted only were the most rancid and of the poorest quality in respect to physical appearance, taste and texture.

The lipid content for fillets in all treatment groups was low throughout storage at both low at both storage temperatures. The reduction or loss of lipids could be explained by its degradation. It was however observed that fillets that were treated with polyphosphate had a high lipid content (8.2%) after brining. This can be treated as an outlier as it is not possible for lipid content to be so high within two days. This could be because of the sample may not have been homogenized properly.

Generally, the increase in TBA indicated the formation of secondary oxidation products such as aldehydes and other volatile compounds responsible for rancid flavour and off odours as well as colour and texture deterioration. TBA values of less than 3 mg malanaldehyde/kg of sample are considered to indicate good quality fishery products. High level of free fatty acid is an indication of microbial spoilage activity. Most fat acidity begins to be noticeable to the palate when the free fatty acid value of oleic acid is about 0.5 - 1.5% in the fish lipid, but the acceptable limit of FFA was 2 - 5%. It was observed that FFA content and TBARS content for fillets that were dry salted only and stored at 20°C were the highest throughout storage. Also, the fillets stored at room temperature had higher FFA and TBARS values that fillets stored at cold storage. The increased FFA in filets treated with 18% brine and fillets that were dry salted only and stored at 20° may also be due to the degradation of protein which leads to lipid oxidation.

The effects of adding phosphates to the brine on the retardation of lipid oxidation of salted redfish fillet during salt curing and processing were observed. The results obtained indicate that added phosphates significantly retarded lipid hydrolysis and lipid oxidation progress, resulting in lower FFA content and TBARS content.
At the end of storage, a quality test was done by tasting and assessing the colour of the fillets at different temperatures. The results showed that storage temperature affects the quality of the fillets as well as polyphosphate helps to lower oxidation. The samples stored at room temperature were very rancid and the yellow colour was very visible. The fillets stored at -18°C were of the best quality with white muscle. From these observations, it can be concluded that the phosphates blend used during brining strongly retarded lipid oxidation of redfish fillets and improved the appearance of the final product.

6 RECOMMENDATION

One of the aims of this research was for this project to be able to adapt salting technology to the local situation in Jamaica. The different salting methods applied in the research produced products with different shelf life. It is recommended that similar research or a pilot project be carried out in Jamaica on tilapia with a sensory component to get the local view of the product. In addition, it is advisable that if the local fish has a lot of dark muscle, it should be deep skinned before salted to eliminate the off-flavour taste.

It is also recommended that the light salted technology can be made adaptable to the Jamaican culture. It is an easy method to carry out, it doesn’t require a lot of time since it does not need to be desalted and it’s a ready to cook product. Fillets that were light salted also increased in yield.

The heavy salted method recommended would be 18% brine followed by dry salting, but it must be stored at cold temperatures such as 0-2°C.

7 CONCLUSION

Salting procedures affected the total weight changes of fillet. The weight changes of fillets increased with decreasing brine concentration. Brining before dry salting increases the weight in fish fillets compared to dry salting only. The use of lower brine concentration are known to increase WHC, due to lower degree of protein denaturation resulting in a total higher yield of the salting process. The physicochemical changes of salted redfish fillets muscle during salting depended on the salting method and storage temperatures. The lipid oxidation as measured by free fatty acid and thiobarbituric acid reactive substance, was highest in fillets that was dry salted only and at room temperatures. Lower storage temperature reduces lipid oxidation hence, better quality salted product and longer shelf life. In this study, quality deterioration in redfish fillets stored at room temperature fish can been attributed to both changes in lipids and loss. The light salted fillets stored at freezing temperature were of better quality. The shelf life of dry salted fish at room temperature can lose its quality with 41 days in storage.
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LIST OF REFERENCES


