

RESEARCH ARTICLE

# Comparative Study of Changes in Muscle Protein of Cultured and Wild *Clarias gariepinus* in Frozen Storage

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## Abstract

Fish is valued for its nutritional qualities as a good source of protein, minerals and vitamins in the amount required for healthy human growth. African catfish, *Clarias gariepinus* is widely consumed in Nigeria but prone to spoilage after harvest. In this study, samples of both cultured and wild *Clarias gariepinus* (5kg/each) were subjected to frozen storage immediately after harvest to arrest spoilage after removing the slimes and blood clots from the skin, labeled as wild whole fish (WWF), and cultured whole fish (CWF) respectively. Proximate, minerals and biochemical analysis of fish samples were determined at fresh state, seven days and fourteen days of freezing respectively and the results obtained were analyzed using pie charts and t-test table mean of values ( $P < 0.05$ ). The crude protein decreased steadily with increase in number of days of freezing in both WWF ( $59.37 \pm 0.78 - 54.19 \pm 2.36$ ) and CWF ( $68.82 \pm 0.09 - 61.92 \pm 0.11$ ) respectively. Conversely, the free fatty acid (FFA %) values increased with the number of days of freezing in WWF ( $0.13 \pm 0.08 - 0.18 \pm 0.08$ ) and CWF ( $0.12 \pm 0.07 - 0.22 \pm 0.07$ ) respectively. Mineral analysis indicated high calcium levels in both fish samples. Assessment of biochemical indices related to lipids damaged in *Clarias gariepinus* after 14 days frozen storage showed Total Volatile Basic Nitrogen (TVBN) value higher in CWF (26.47%) than WWF (7.46%), indicating higher perishability in cultured fish than the wild species.

## 1. Introduction

Fish provides 22% of protein intake and exceeds 50% in some countries where animal protein is expensive and it is an indispensable source of micronutrients such as iron, iodine, zinc, vitamin A and B (Dominguez *et al.*, 2021). *Clarias gariepinus* belongs to the family Clariidae (air-breathing catfishes) and it is considered to be one of the most important catfish species for aquaculture in Nigeria (Fawole *et al.*, 2007). Fish is prone to decomposition, rancidity and microbial spoilage immediately after harvest, therefore, there is need to process and preserve harvested fish in order to extend its shelf life for human consumption (Eyo, 2001). Some processing techniques are based on temperature control with the use of ice, refrigeration or freezing; while others are based on the control of water activity through drying, salting, smoking and freeze-

drying. Freezing is popular because it can effectively reduce the activity of enzymes without heating or adding preservatives (Oyelese, 2006a). However, freezing and frozen storage cause undesirable protein denaturation due to the formation of ice crystals, which seriously affect the quality and acceptability of fish products (Benjakul, *et al.*, 2003).

Fish protein includes essential amino acids which improve its digestibility. Fish has health benefits with its high-protein, low-fat content. In particular, white-fleshed fish is lower in fat than any other source of animal protein, and oily fish are high in omega-3 fatty acids. Since the human body must supply these essential nutrients, fish are an important part of human diets. Fish proteins have several peptides having functional properties such as antioxidant, anti-diabetic, anti-proliferative, antimicrobial, anti-

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coagulant, anti-hypertensive, and calcium-binding activities, thereby, improving human health (Khan *et al.*, 2022). Although frozen storage is an excellent method of preservation, the quality of fish is still decreased and some chemical and physical changes of protein occur such as the changes of muscle texture and flavor loss which is liable to cause consumer rejection (Wang *et al.*, 2010).

In catfish meat, as in all meats, the deterioration process starts as soon as the fish dies, including the degradation of proteins and ATP, altered pH, fat oxidation and production of undesirable compounds such as trimethylamine (TMA-N) and volatile bases of low molecular weight (TVB-N), which are produced by bacterial action and generate changes in texture, color, odor and flavor (Li *et al.*, 2011). These changes can be classified as biochemical, physical and microbiological, and determine the degree of acceptance by consumers along with nutritional valuation, shelf life (Millward *et al.*, 2008).

Due to irregular supply of electricity in Nigeria, the denaturation of the protein is one problem caused by slow freezing and the formation of ice crystals during freezing damages the ultrastructure and concentrates the solutes in the meat, which, in turn, leads to alterations in the biochemical reactions that occur at the cellular level and influence the quality of physical parameters of the meat (Badii and Howell, 2002). In addition, species, initial micro flora, handling stress, storage temperature as well as type of processing and packaging atmosphere are among factors affecting the rate and nature of quality changes in the fish muscle during storage (Olafsdottir *et al.*, 2004). In order to ensure product quality and stability, controlled temperatures throughout the cold chain is necessary, the required temperature must be maintained from production to consumption. The international and national regulation about the storage temperature of frozen foods is set at  $-18^{\circ}\text{C}$  as the highest temperature during storage and distribution (Rediers *et al.*, 2009).

Cultured catfish *Clarias gariepinus* is good for human consumption just like the wild counterpart. It is a good source of essential fatty acids but affected by the changes in the biochemical state of the flesh due to stress, freshness, freezing speed, storage temperature, temperature fluctuations and drying and thawing conditions (Adebayo *et al.*, 2016). The main objective of the study was to determine changes in the muscle protein of *Clarias gariepinus* from both culture and

wild sources under frozen storage by analyzing the proximate, mineral and biochemical changes in the flesh of the fish at both fresh and frozen states.

## 2. Materials and Methods

### 2.1 Fish Samples Collection and Preparation of Fish Samples

Five kilogram each of live fish samples of average weight of one kg/one labeled Wild Whole Fish (WWF) and Cultured Whole Fish (CWF) of *Clarias gariepinus* were sourced from fishermen at Ogbese Reservoir, Ise Ekiti, Ekiti State and Afe Babalola University Farm, Ado Ekiti, Ekiti State, Nigeria respectively. The live fish samples were moved to the Department of Fisheries and Aquaculture Laboratory. Fish were percussively stunned (rendered instantaneously insensible) with a blunt instrument, washed properly with clean water to remove the slime and blood clots from the skin. After washing, the fish samples were immediately frozen at  $-18^{\circ}\text{C}$  whole labeled as Wild Whole Fish (WWF) and Cultured Whole Fish (CWF) respectively.

### 2.2 Chemical Analysis

Proximate analysis was performed at the fresh state of the fish samples, on the 7<sup>th</sup> day and 14<sup>th</sup> day respectively, using AOAC, 2012.

### 2.3 Analysis for Trace Elements, Free Fatty Acid (FFA) and Thiobarbituric Acid (TBA)

Trace elements, free fatty acids and Thiobarbituric acid levels in fish samples were analyzed using standard methods (AOAC, 2012)

### 2.4 Statistical Analysis

Average values per each sample were determined and analyzed using t-test table mean of values ( $P < 0.05$ ) and pie charts

## 3. Results

The results of the proximate composition of the fish samples at fresh and frozen states are presented in Tables 1, 2 and 3 below. The results clearly showed that the moisture content of all the fish samples analyzed were significantly different ( $P < 0.05$ ). While protein values were decreasing from the fresh state of fish samples to 14<sup>th</sup> day of frozen, the lipid content was inversely increasing. The moisture content significantly increased with the increase in storage time.

**Table 1.** The proximate composition of wild and cultured *Clarias gariepinus* in fresh state

Parameters	Wild (%) (WWF)	Cultured (%) (WCF)
Moisture	5.71 ± 1.41b	7.10±0.02a
Ash	9.75 ± 0.18 a	9.47±0.13b

Crude protein	59.37 ± 0.78b	68.82±0.09a
Lipid	17.58 ± 0.42 a	13.78±0.35b

**Table 2.** The proximate composition of wild and cultured *Clarias gariepinus* after 7 days of frozen storage.

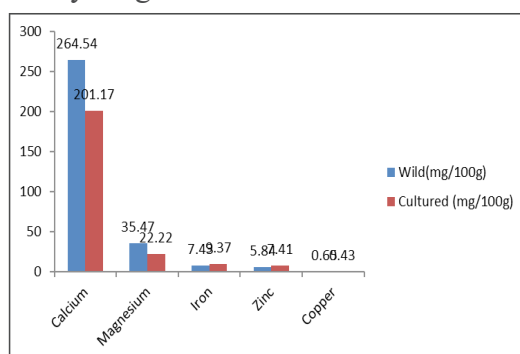
Parameters	Wild (%) (WWF)	Cultured (%) (WCF)
Moisture	6.71 ± 1.41b	8.20±0.00a
Ash	13.76 ± 0.18a	9.51±0.35b
Crude protein	57.35 ± 0.78b	64.73±0.12a
Lipid	19.56 ± 0.42 a	15.48±0.52b

**Table 3.** The proximate composition of wild and cultured *Clarias gariepinus* after 14 days of frozen storage.

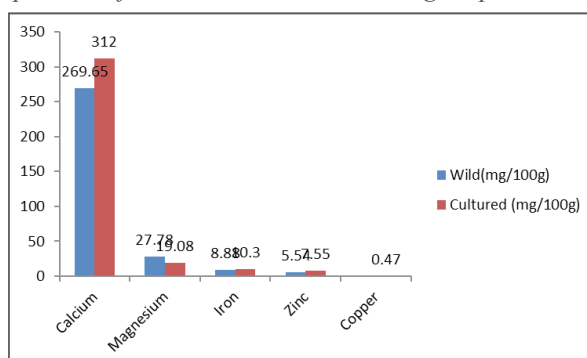
Parameters	Wild (%) (WWF)	Cultured (%) (WCF)
Moisture	6.72 ± 1.25b	8.40±0.70a
Ash	16.76 ± 0.45a	9.81±0.22b
Crude protein	54.19 ± 2.36b	61.92±0.11a
Lipid	21.19 ± 0.50 a	18.37±0.52b

The results of the mineral element concentrations were given in figures 1 and 2. The variations observed in the values obtained showed Calcium to be highest in all the fish samples examined, followed by magnesium.

Iron, Zinc and Copper respectively, though in trace amounts. Calcium increased with storage time with decrease in Magnesium and Iron levels.



**Figure 1.** The Mineral Composition of Wild and Cultured *Clarias gariepinus* in their fresh state before frozen



**Figure 2.** The Mineral Composition of Wild and Cultured *Clarias gariepinus* after 14 days of frozen storage

Tables 4, 5 and 6 show the concentrations of Free Fatty Acid (FFA), Peroxide Value (PV) in milli-equivalents (meq) peroxide/kg of fat extracted from the samples, Thiobarbituric Acid Value (TBA) and Total Volatile Basic Nitrogen (TVBN) in all the samples examined.

The Free Fatty Acid content of all the fish samples were significantly different ( $p < 0.05$ ) with the value ranging from  $0.13 \pm 0.08$ - $0.18 \pm 0.08$  in WWF and  $0.12 \pm 0.07$ - $0.22 \pm 0.07$  in CWF respectively.

**Table 4.** Assessment of biochemical indices related to lipids damaged in fresh state of *Clarias gariepinus*

Parameters	Wild (%) (WWF)	Cultured (%) (WCF)
FFA%	0.13±0.08a	0.12±0.07b
PV *(meq/kg fat)	4.22±1.50a	4.15 ± 1.33b
TBA*(mg malonaldehyde)	0.44±0.04a	0.37±0.04a
TVBN*(mg %)	7.34±0.42a	24.51±0.63a

**Table 5.** Assessment of biochemical indices related to lipids damaged in *Clarias gariepinus* after 7days frozen storage

Parameters	Wild (%) (WWF)	Cultured (%) (WCF)
FFA%	0.15 ± 0.05b	0.16±0.00a
PV *(meq/kg fat)	4.33 ± 0.50a	4.29±1.05b
TBA*(mg malonaldehyde)	0.16 ± 0.04b	0.19±0.04a
TVBN*(mg %)	7.46 ± 0.78b	26.47±0.63a

**Table 6.** Assessment of biochemical indices related to lipids damaged in *Clarias gariepinus* after 14days frozen storage

Parameters	Wild (%) (WWF)	Cultured (%) (WCF)
FFA%	0.18 ± 0.08b	0.22±0.07a
PV *(meq/kg fat)	4.42 ± 1.50a	4.31±1.33b
TBA*(mg malonaldehyde)	0.15 ± 0.04b	0.19±0.04a
TVBN*(mg %)	9.36±0.42b	27.48±0.63a

**Note:** FFA- Free Fatty Acid, PV- Peroxide Value in milli-equivalents (meq) peroxide/kg of fat extracted from the food, TBA- Thiobarbituric Acid Value, VBN- Total Volatile Basic Nitrogen

#### 4. Discussion

In this study, it was observed that the longer the storage time, the higher the moisture content of the stored products. Also, crude protein kept reducing while free fatty acid (FFAs) was increasing with storage time. This was in support of Yu *et al.*, 2018, who observed that low temperature at longer time denatured fish protein. According to the report, the free amino acids and water soluble protein content of tissue serve as an excellent source of autolysis and as a resultant the quantity of protein is decreased. It also helped the bacteria to invade the tissue fast. Findings from the current study on rancidity are in line with the position of Sikorski *et al.*, 2020 who reported that deterioration of fish is aided by lipid oxidation. The Free fatty acid is the resultant products of hydrolytic rancidity of lipids which is expressed as % of oleic acid. The FFA is a result of enzymatic decomposition of lipid during storage leading to lipid hydrolysis (Sriket *et al.*, 2010). The initial FFA (% oleic acid) increased significantly throughout the period of storage. Free fatty acids got generated during storage mostly which may be due to the activity of lipases present both in tissue and also of microbial origin (Rodriguez *et al.*, 2007)

The Total Volatile Basic Nitrogen (TVB-N) content significantly changed during the storage period both in capture and wild fish samples. However, the TVB-N content did not exceed above acceptable limit, i.e. 35 mg N/100 g (FAO, 1986). The increasing in TVB-N value was comparable with the increasing TVBN value as reported by (Stodolnik *et al.*, 2005) in frozen whole mackerel (*Scombus scombus*). This increase could be attributed to the production of ammonia and other volatile amines in the muscle during storage (John and Ohshima, 2010). Present findings are in agreement with the (Lokuruka *et al.*, 2012) that found

increased level of TVBN in the ungutted and gutted experimental tilapia with long storage time in ice.

In the present study, primary oxidation products of the fish sample were measured by the peroxide value (PV) and are expressed as meq O<sub>2</sub>/kg of fat. It was observed that the PV increased significantly in both culture and wild fish samples with the time of storage. The PV value was observed higher in the wild than culture fish; similar results were also reported by Karlsdottir *et al.*, 2014 in chilled steaks of Silver carp where the PV value increased with increased in storage time. Lipid oxidation in fish depends on numerous factors such as species, storage temperature, fat composition, and it produces a number of substances, some of which have unpleasant flavours and odours. Some may also contribute to texture changes, by forming covalent bonds with muscular proteins (Lehmann and Aubourg, 2008).

The Thiobarbituric Acid Value (TBA) in the present study increased significantly in the treatments during storage period of 14 days in the wild samples while the culture sample values remained unchanged. Fish muscle texture depends on a number of intrinsic biological factors that are related to muscle fibre density, and involving both fat and collagen content (Hernandez *et al.*, 2009). The fish death triggers autolytic and microbiological processes that make the muscle softer and less elastic (Olafsdottir *et al.*, 2004). In the same line of the present findings, earlier study by Lehmann and Aubourg, 2008 showed the textural changes in iced *Labeo calbasu*. They found that the early signs of disorganization of the muscle structure were characterized by a loss of compactness of the fibers after seven days. Similar results have been reported for few marine fish species (Sikorski *et al.*, 2020).

## 5. Conclusion

This study showed that cultured *Clarias gariepinus* had comparatively similar storage results with wild species, except that the values were higher in most of the parameters analyzed in cultured fish samples. This may be attributed to formulated source of feeds to the fish in a controlled environment. And it is expected that spoilage and deterioration would be more pronounced in cultured *Clarias gariepinus* due to nutrient accumulation in the body when compare to the wild species. Though, cultured *Clarias gariepinus* showed relatively similar quality and nutritive value as wild *Clarias gariepinus* but its long frozen storage must be discouraged.

### 5.1 Recommendation

Fish, either wild or cultured is better consumed in the fresh state for better nutrient availability. To further preserve the nutrient of fish and to extend its shelf life, fish should be stored in a frozen state at minimal period to maintain its nutritional value and prevent rancidity. It is recommended that wild *Clarias gariepinus* is better stored in frozen storage as it has less deteriorating muscle proteins than the cultured counterpart.

## 6. References

- Adebayo I. A, Fapohunda O .O and Ajibade A. O. (2016) Evaluation of Nutritional Quality of *Clarias gariepinus* from Selected Fish Farms in Nigeria. American Journal of Food Science and Nutrition Research 3(4): 56 -62
- Association of Official Analytical Chemist (2019): Official Methods of Analysis of (AOAC) 19th Edition by George Latimer (Author) Maryland, USA.
- Badii, F., and Howell, N. K. (2002). Changes in the texture and structure of cod and haddock fillets during frozen storage. *Food hydrocolloids*, 16(4): 313-319.
- Benjakul, S., Visessanguan, W., Thongkaew, C., and Tanaka, M. (2003). Comparative study on physicochemical changes of muscle proteins from some tropical fish during frozen storage. *Food Research International*, 36(8): 787-795.
- Domínguez, R., Pateiro, M., Munekata, P. E., Zhang, W., Garcia-Oliveira, P., Carpena, M., ... and Lorenzo, J. M. (2021). Protein oxidation in muscle foods: A comprehensive review. *Antioxidants*, 11(1): 60.
- Eyo A. A. (2001): Fish Processing Technology in the Tropics. National Institute for Freshwater Fisheries Research. University of Ilorin press pp 66-70.
- Fawole O. O. Ogundiran M. A. Ayandiran T. A. and Olagunju O. F (2007): Proximate and Mineral composition in some freshwater fishes in Nigeria. *Internet Journal of food safety*, volume 9 Pp 52-55.
- John, J. H., and Ohshima, T. (2010). Control of lipid oxidation and meat color deterioration in skipjack tuna muscle during ice storage. *Fisheries Science*, 76(4): 703-710.
- Karlsdottir, M. G., Sveinsdottir, K., Kristinsson, H. G., Villot, D., Craft, B. D., and Arason, S. (2014). Effects of temperature during frozen storage on lipid deterioration of saithe (*Pollachius virens*) and hoki (*Macruronus novaezelandiae*) muscles. *Food Chemistry*, 156, 234-242.
- Khan, S., Rehman, A., Shah, H., Aadil, R. M., Ali, A., Shehzad, Qand Xia, W. (2022). Fish Protein and its derivatives: The novel applications, bioactivities, and their functional significance in food products. *Food Reviews International*, 38(8): 1607-1634.
- Lehmann, I., and Aubourg, S. P. (2008). Effect of previous gutting on rancidity development in horse mackerel (*Trachurus trachurus*) during frozen storage at - 20° C. *International Journal of food science & technology*, 43(2): 270-275.
- Li, X., Li, J., Zhu, J., Wang, Y., Fu, L., and Xuan, W. (2011). Postmortem changes in yellow grouper (*Epinephelus awoara*) fillets stored under vacuum packaging at 0 C. *Food Chemistry*, 126(3): 896-901.
- Lokuruka, M. N. I., Muyela, B., Okeyo, G. O., Shitandi, A. and Otieno, M., (2012). Effect of gutting on sensory, some biochemical and microbiological properties of Nile perch (*Lates niloticus*) and Nile tilapia (*Oreochromis niloticus*) stored in ice. *Cont. J. Fish. Aqua. Sci.*, 6 (3):1-13.
- Millward, D. J., Layman, D. K., Tomé, D., and Schaafsma, G. (2008). Protein quality assessment: impact of expanding understanding of protein and amino acid needs for optimal health. *The American journal of clinical nutrition*, 87(5): 1576S-1581S
- Olafsdottir, G., Nesvadba, P., Di Natale, C., Careche, M., Oehlenschläger, J., Tryggvadottir, S. V., ... and Jørgensen, B. M. (2004). Multisensor for fish quality determination. *Trends in food science and technology*, 15(2): 86-93.
- Oyelese O. A. (2006a) Quality Assessment of Cold Smoked, Hot Smoked and Oven Dried *Tilapia niloticus* Under Cold Storage Temperature Conditions *Journal of Fisheries International* Volume: 1 | Issue: 2 | Page No.: 92-97.
- Rediers, H., Claes, M., Peeters, L., and Willems, K. A. (2009). Evaluation of the cold chain of fresh-cut endive from farmer to plate. *Postharvest Biology and Technology*, 51(2): 257-262.
- Rodríguez, A., Losada, V., Larráin, M. A., Quitral, V., Vinagre, J., and Aubourg, S. P. (2007). Development

- of lipid changes related to quality loss during the frozen storage of farmed coho salmon (*Oncorhynchus kisutch*). *Journal of the American Oil Chemists' Society*, 84(8): 727-734.
- 19 Sikorski, Z. E., Kołakowska, A., and Burt, J. R. (2020). Postharvest biochemical and microbial changes. In *Seafood: Resources, nutritional composition, and preservation* (pp. 55-75). CRC Press.
- 20 Stodolnik, L., Stawicka, A., Szczepanik, G., and Aubourg, S. P. (2005). Rancidity inhibition study in frozen whole mackerel (*Scomber scombrus*) following flaxseed (*Linum usitatissimum*) extract treatment.
- 21 Sriket, C., Benjakul, S., and Visessanguan, W. (2010). Post-mortem changes of muscle from fresh water prawn (*Macrobrachium rosenbergii*) as influenced by spawning stages. *LWT-Food Science and Technology*, 43(4): 608-616.
- 22 Wang, P. A., Vang, B., Pedersen, A. M., Martinez, I., and Olsen, R. L. (2011). Post-mortem degradation of myosin heavy chain in intact fish muscle: Effects of pH and enzyme inhibitors. *Food Chemistry*, 124(3): 1090-1095.
- 23 Yu, L., Jiang, Q., Yu, D., Xu, Y., Gao, P., and Xia, W. (2018). Quality of giant freshwater prawn (*Macrobrachium rosenbergii*) during the storage at -18 C as affected by different methods of freezing. *International Journal of Food Properties*, 21(1): 2100-2109.