INTRODUCTION
Due to poor fish holding facilities in markets that result into spoilage of fresh fish, most farmers in Nigeria are exploring the idea of preserving fish in ice because it is locally available. An earlier study on catfish revealed that fresh fish can remain in acceptable condition while kept in ice for no less than two weeks. This notion stems from the background that temperature plays a key role in fresh fish spoilage which directly causes changes in the nutritional composition of fish. In the high ambient temperatures of the tropics, fresh fish will spoil within 12 hours. It is widely reported that chilling or icing of fish immediately after catch reduces its spoilage. On the other hand, physical properties of fresh fish such as firmness and appearance of the skin are greatly influenced by storage time due to cellular flaking of autolytic and microbial changes (Chisomo et al., 2016).

Fresh fish will spoil very quickly. Once the fish has been caught, spoilage progresses rapidly. In the high ambient temperatures of the tropics, fish will spoil within 12 hours. Using good fishing techniques (to ensure the fish is barely damaged) and cooling the fish, with the help on ice on board, can increase the storage life of fresh fish (Brigitte et al., 2004).

Since spoilage causes changes in the freshness and nutritional composition of fresh fish, it is necessary to assess the extent of the changes with respect to time so that consumers have maximum nutritional benefits from fish. This is also important for fish processors to avoid processing fish which has compromised nutritional and fresh quality (Chisomo et al., 2016). The present concern about the environmental resources challenge all over the effect of prolonged storage in ice on nutrient composition and sensory quality of whole fresh pond raised catfish; bacteria are capable of causing spoilage because of two important characteristics. First they are psychrotrophic and thus multiply at refrigeration temperatures. Secondly they attach various substances in the fish tissue to produce compounds associated with off-flavours and off-odours. The objective of the study was to assess the effect of prolonged storage in ice on nutrient composition and sensory quality of whole fresh pond raised Catfish (Clarias gariepinus).

MATERIALS AND METHODS
The Study Area
The study was carried out at the Research and Teaching Farm of the Department of Fisheries, University of Maiduguri (latitude 14° 0’ N and 13° 0’ E )

Fish Sample Collection and Preparation
Live fish samples, with an average weight of 500g were purchased from custom market in Maiduguri Borno state. The live fish samples were transported in plastic bucket containing water to the Department of Fisheries, Research and Teaching Farm, University of Maiduguri where the experiment was carried out. The fish were degutted, washed and iced immediately. Data was analyzed using a statistical package SPSS version 20.0. The result of the study shows that that sensory quality of fresh C. gariepinus declines with time when stored in ice and that storage for not more than 15 days can maintain acceptable quality. The nutritional quality of cat fish deteriorated with time when stored in ice. Crude protein decreased from 30% at day1 to 21.57% at day9. Similarly, NFE decrease from 17.78 at day1 to 2.30% at day 15. However, Carcass fat increased with storage time, from 25.25 at day1 to 36.9% at day12. Dry matter content decreased with increasing time of storage indicating increased moisture content of the product. The result shows that that like any other fish C. gariepinus has a relatively longer shelf life when kept in ice. It is concluded that there is a significant change in the proximate composition of the fish flesh with an increase in storage time. Based on the fining of the study it is recommended that C. gariepinus fish stored in ice should be kept for a limited number of days of not more than 15 days.

Keywords: Storage, Ice, Proximate composition, Sensory quality

ABSTRACT
Fish storage on ice has been one of commonly used methods to preserve fresh fish for subsequent processing and consumption. This study was carried out to assess the effect of prolonged storage in ice on nutrient composition and sensory quality of whole fresh pond raised Catfish (Clarias gariepinus). Live fish samples, with an average weight of 500g were purchased from custom market in Maiduguri Borno state and transported in plastic bucket containing water to the Department of Fisheries, Research and Teaching Farm, University of Maiduguri where the experiment was carried out. The fish were degutted, washed and iced immediately. Data was analyzed using a statistical package SPSS version 20.0. The result of the study shows that that sensory quality of fresh C. gariepinus declines with time when stored in ice and that storage for not more than 15 days can maintain acceptable quality. The nutritional quality of cat fish deteriorated with time when stored in ice. Crude protein decreased from 30% at day1 to 21.57% at day9. Similarly, NFE decrease from 17.78 at day1 to 2.30% at day 15. However, Carcass fat increased with storage time, from 25.25 at day1 to 36.9% at day12. Dry matter content decreased with increasing time of storage indicating increased moisture content of the product. The result shows that that like any other fish C. gariepinus has a relatively longer shelf life when kept in ice. It is concluded that there is a significant change in the proximate composition of the fish flesh with an increase in storage time. Based on the fining of the study it is recommended that C. gariepinus fish stored in ice should be kept for a limited number of days of not more than 15 days.

Keywords: Storage, Ice, Proximate composition, Sensory quality
finally at end of experiment. The samples analyzed for dry matter, crude protein, crude fibre, ether extract or fat, ash and carbohydrate according to AOAC method (1990).

**Dry matter**
The dry matter content of the samples were determined by weighting 10g of samples were into petri dish while placed in hot oven at 105°C for 24 hours. And then removed and placed in dessicator to cool, after cooling reweighed.
The dry matter content was calculated using the formula:

\[ \text{W}_1 - \text{W}_2 \times 100 \]

where \( \text{W}_2 \): weight of petri dish with sample in grammes before oven dried, \( \text{W}_3 \): weight of petri dish with sample in grammes after oven dried, \( \text{W}_1 \): weight in grammes of empty petri dish.

**Crude Protein**
Crude protein contents were analyzed using 2g of samples weighed into a digestion tube and 2 keljedal tablets was added. 20ml of concentrated sulphuric acid was added onto the tube and digested at 420°C for 3 to 5 hrs. After cooling, 90mls of distilled water was added into digested solution. About 50mls of 40% caustic soda (NaOH) was added on to 50mls of digested and diluted solution and placed on heating section of the distillation chamber, 30mls of 4% boric acid, plus bromocresol green and methyl red as an indictor was put onto conical flask and placed underneath the distribution chamber for collection of ammonia, the solution of hydrochloric acid (HCL) was weighed into burrette. The conical flask containing the solution was titrated until the colour changed from green to pink. The burette reading was taken and the crude protein was calculated using the formulae:

\[ \% \text{CP} = \frac{\text{(A-B)} \times \text{N} \times \text{F} \times 6.25 \times 100}{\text{Mg of samples}} \]

A: mls of acid used for titrating the samples, B: ml of acid used for titrating blank samples (0), N: normality of acid used for titration, F; factor is 14.007, 6.25: is a constant, 100: conversion to percentage

**Crude Fibre**
Crude fibre was determined by weighting 2g of samples was placed in a round bottom flask and 50mls of tri-chloroacetic acid reagent (TCA) was added. The mixture was boiling and refluxed for 40 minutes. Filter paper was removed and cooled to room temperature. Filter paper was used to filter the residue. The residue obtained was washed 4 times with hot water and once with petroleum ether then the filter paper plus the sample were folded together and dried at 30°C -60°C in an oven for 24 hours. Reweighted and then at 650°C and then reweighed.

\[ \% \text{CF} = \frac{\text{Differences in weight x 100}}{\text{Weight of sample on DM basis}} \]

**Ether Extract (FAT)**
The ether extract was determined by using soxhlet apparatur, 2g of the fish sample was weighed into a thimble and 200 Mls of petroleum ether was measured to measuring cylinder, the solution was put into round bottom flask and was at 45°C for 1 hour interval for 2hours. The collecting flask was removed and cooled into dessicator for 15 minutes and percentage fat samples are determined by using the formulae:

\[ \% \text{fat} = \frac{\text{weight of fat x 100}}{\text{Weight of the sample}} \]

**Ash**
To determine the ash content 2 g of sample was weighted into crucible and dried at 105°C for 24 hours, then cooled in the dessicator for 15 minutes and reweighed, it was then charred at 600°C in muffle furnace for 2-3 hours. Then cooled for 15 minutes and reweighed.

\[ \% \text{Ash} = \frac{\text{loss in weight x100}}{\text{Initial weight}} \]

**Carbohydrate**
A percentage carbohydrate was determined by computing indirectly by difference using formulae:

\[ \% \text{carbohydrate} = 100 - (\% \text{MC} + \% \text{ash} + \% \text{CP} + \% \text{CF}) \]

**Sensory Analysis**
Sensory analysis was carried out by a pre-trained sensory evaluation panel consisting of 6 members. A developed quality index scheme (QIM) was used to assess the quality changes of the fresh fish at a 3 day interval involving the following parameters: skin appearance, slime, fresh firmness, odour, colour of the eyes and colour of the gills. The sensory demerit scores were compared using one way analysis of variance (ANOVA). Means and standard errors (±SE) were reported at 5% level of significance. Duncan’s Multiple Range Test (DMRT) was used to separate significantly different means.

**Statistical Analysis**
Data was analyzed using a statistical package SPSS version 2.0. Changes in mean proximate composition, and sensory scores were compared using one way analysis of variance (ANOVA). Means and standard errors (±SE) were reported at 5% level of significance. Duncan’s Multiple Range Test (DMRT) was used to separate significantly different means.

Table 1: Score card for Fresh C. gariepinus

<table>
<thead>
<tr>
<th>Score</th>
<th>Eyes</th>
<th>Gills</th>
<th>Skin</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>9 (Excellent)</td>
<td>Flat, transparent or slightly grey pupil</td>
<td>Red or rose coloured. Transparent mucus</td>
<td>Bright, very dark and clear colour</td>
<td>Flesh is firm and springs immediately released</td>
</tr>
<tr>
<td>8 (Excellent)</td>
<td>Very slightly sunken</td>
<td>Dark-red, mucus</td>
<td>Bright, dark colours</td>
<td>Reasonably firm, thumb indentation slowly fills out</td>
</tr>
<tr>
<td>7 (Good)</td>
<td>Slightly sunken, cloudy</td>
<td>Red-brown, mucus slightly opaque, gill-like colour</td>
<td>Some loss of brightness</td>
<td>Moderately firm</td>
</tr>
<tr>
<td>6 (Poor)</td>
<td>Sunken, very cloudy</td>
<td>Brown, abundant harel-colour mucus</td>
<td>Dull colours</td>
<td>Very slightly soft flesh skin coming up</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION

Proximate Composition

The proximate composition of *Clarias gariepinus* preserved in ice over different time intervals is presented in Table 1. There were significant differences (P<0.05) in moisture, protein, fat and ash contents with a decreasing trend against ambient storage time. The lowest moisture, fat and ash were observed in samples at 15 hours of storage. Significant changes (P<0.05) in the nutrients were observed at 6 hours of storage time also agreeing with results from sensory and organoleptic tests while protein contents were higher in 16 days of the experiment. All nutrients highly negatively correlated with storage time, -.972, -.926, -.916 and -.966 for moisture, ash, fat and protein respectively (P<0.05) denoting loss of nutrients with time in storage.

The result of the study showed that fish at first day of experiment displayed all the characteristics of freshly caught fish with a score of 10.0. Proximate analysis of *Clarias gariepinus* exhibited a decrease in the protein content from 1st to 2nd day and increase on the third day and decreased from 6 to 12 days while 15 days has the highest protein contents of the fresh fish iced (Table 1).

The crude protein content in *Clarias gariepinus* in ice was observed to decrease from 36.57±0.02 % to 21.37±0.13 % after nine days of storage. The protein increased from 21.37±0.13 % to 41.00±0.00 % after 15 days in the ice (Table 1). Proximate composition is a reliable objective indicator for determining nutritional value and quality of fish (Ravichandran et al., 2011). The moisture content in fish samples of *Clarias gariepinus* stored in ice was increased from 7.37±0.03% to 8.83±0.02%, and was decreased from 8.83±0.02% to 6.90±0.00% after the 15th day (Table 1). Ash content of *Clarias gariepinus* stored in ice decreased from 19.42±0.07% to 17.85+0.18%, at day 6 while increased from 17.85±0.18% to 17.67±0.03 at 15 days (Table 1).

**Table 1: Proximate composition of *Clarias gariepinus* preserved in ice over different time intervals**

<table>
<thead>
<tr>
<th>Days</th>
<th>Moisture</th>
<th>Ash</th>
<th>Protein</th>
<th>Fat</th>
<th>CHO</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>7.37±0.03d</td>
<td>19.42±0.07a</td>
<td>30.22±0.11d</td>
<td>25.25±0.43d</td>
<td>17.75±0.45b</td>
</tr>
<tr>
<td>3.00</td>
<td>7.76±0.02c</td>
<td>19.51±0.01a</td>
<td>31.57±0.32c</td>
<td>36.15±0.22a</td>
<td>5.01±0.57d</td>
</tr>
<tr>
<td>6.00</td>
<td>8.32±0.03b</td>
<td>17.85±0.18cd</td>
<td>36.57±0.12b</td>
<td>28.11±0.36c</td>
<td>9.22±0.11c</td>
</tr>
<tr>
<td>9.00</td>
<td>8.88±0.1a</td>
<td>18.09±0.01bc</td>
<td>21.37±0.13e</td>
<td>27.54±0.44c</td>
<td>24.12±0.22a</td>
</tr>
<tr>
<td>12.00</td>
<td>8.83±0.02a</td>
<td>18.5±0.04b</td>
<td>25.90±0.0f</td>
<td>36.90±0.4a</td>
<td>10.0±0.45c</td>
</tr>
<tr>
<td>15.00</td>
<td>6.90±0.00e</td>
<td>17.67±0.03d</td>
<td>41.00±0.00a</td>
<td>2.13±0.13b</td>
<td>2.30±0.16e</td>
</tr>
</tbody>
</table>

Mean Sensory Scores for Whole Fresh *C. carpio* Stored In Ice

The sensory limit for acceptability of *Clarias gariepinus* by the sensory panelists was observed between 12 and 14 days while the highest total demerit score of 6.0 was observed on day 15. this is close to the value reported by Kapute et al. (2013) where shelf life of Lake Malawi Tilapia was estimated between 15 and 16 days with the highest demerit score observed on the 16th day.

The natural/normal fresh colour of the fresh fish became discoloured during storage. Texture became harder at the beginning due to stiffening of the muscle during rigor mortis process. The concave appearance of the eyes and scales becoming loose could be as a result of prolonged storage in ice earlier reported by Uchoi et al. (2011). All sensory attributes of oven dried samples of the samples which include flavor, odour and texture gave significant relationship with storage time (p<0.05).

**Table 2: Panelist Mean Sensory Scores for Whole Fresh *C. carpio* Stored In Ice**

<table>
<thead>
<tr>
<th>Fresh pond-raised <em>Clarias</em></th>
<th>Days</th>
<th>Eyes</th>
<th>Gill</th>
<th>Texture</th>
<th>General appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>8.7</td>
<td>8.8</td>
<td>8.5</td>
<td>8.0</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>7.6</td>
<td>8.5</td>
<td>7.0</td>
<td>7.8</td>
</tr>
<tr>
<td></td>
<td>12</td>
<td>7.0</td>
<td>7.5</td>
<td>7.0</td>
<td>7.5</td>
</tr>
<tr>
<td></td>
<td>15</td>
<td>6.5</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
</tr>
<tr>
<td></td>
<td>6.0</td>
<td>6.5</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
</tr>
<tr>
<td></td>
<td>5.0</td>
<td>5.0</td>
<td>5.5</td>
<td>5.5</td>
<td>6.0</td>
</tr>
</tbody>
</table>

The panelist evaluation of the Fresh pond raised *C. gariepinus* is presented in Table 3. Sense of smell indicates that the fish maintained an excellent odour from day 1 to day 6 but started deterioration at day 9 and 12 maintaining fairly good quality rated as very good and good respectively. However, complete loss of quality was observed at day 15. Ayodeji et al (2020)
reported similar reports. However, Flavor followed the same trend with the smell. Fish appearance did not show significant variation from day 1 to day 9 and the quality did deteriorate even at day 15 respectively.

Table 3: Sensory Evaluation of Fresh pond Raised C. gariepinus stored in Ice over time intervals

<table>
<thead>
<tr>
<th>Odour</th>
<th>Flavor</th>
<th>Texture</th>
<th>Appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Days</td>
<td>Scores</td>
<td>Days</td>
<td>Scores</td>
</tr>
<tr>
<td>1</td>
<td>Excellent</td>
<td>1</td>
<td>Excellent</td>
</tr>
<tr>
<td>3</td>
<td>Excellent</td>
<td>3</td>
<td>Excellent</td>
</tr>
<tr>
<td>6</td>
<td>Excellent</td>
<td>6</td>
<td>Excellent</td>
</tr>
<tr>
<td>9</td>
<td>V. Good</td>
<td>9</td>
<td>V. Good</td>
</tr>
<tr>
<td>12</td>
<td>Good</td>
<td>12</td>
<td>Good</td>
</tr>
<tr>
<td>15</td>
<td>Poor</td>
<td>15</td>
<td>Poor</td>
</tr>
</tbody>
</table>

Analysis of the constituents of proximate composition: crude protein, moisture and ash revealed significant differences (P<0.05) in the different compositions within the same storage temperatures and between the two temperatures in Clarias gariepinus. The decrease in crude protein of the fish during the storage could be attributed to the gradual denaturation of the crude protein to more volatile products as total volatile bases (TVB), trimethyl amine (TMA) hydrogen sulphide and ammonia. These findings were in line with the results reported by Reza et al. (2015).

The changes in protein and lipid content may be associated with the leaching out to ice of some of the lipid fractions (Odolli et al., 2019). The reduction in crude protein content of the fish may also have been due to a decrease in salt soluble protein and water soluble protein (Mahboob et al., 2015) or due to autolytic deterioration associated with the actions of endogenous enzymes and bacteria (Tavares et al., 2021). The decrease in ash content may be linked with the oxidation of polyunsaturated fatty acids found in fish tissues to other products such as aldehydes, free fatty acids, ketones, and peroxides (Ayala et al., 2014). Ash content decreased during the experimental period. In a related experiment, Ahmed et al. (2022) reported ash content ranged from 3.24% to 5.74% with the highest value seen in case of Clarias gariepinus. Ash content decreased during the experiment.

It can be concluded that sensory quality of fresh C. gariepinus declines with time when stored in ice and that storage for not more than 15 days could maintain acceptable quality. It can also be concluded that the nutritional quality of cat fish deteriorated with time when stored in ice. The general conclusion is that C. gariepinus has a relatively longer shelf life when kept in ice.

**CONCLUSION**

It can be concluded that sensory quality of fresh C. gariepinus declines with time when stored in ice and that storage for not more than 15 days could maintain acceptable quality. It can also be concluded that the nutritional quality of cat fish deteriorated with time when stored in ice. The general conclusion is that C. gariepinus has a relatively longer shelf life when kept in ice.

**RECOMMENDATIONS**

Based on the findings of the study it is recommended that C. gariepinus fish stored in ice should be kept for 15 days at most.

**ACKNOWLEDGEMENT**

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**REFERENCES**


EFFECT OF PROLONGED STORAGE IN ICE

Hassan et al.,

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1454. doi: 10.1002/fsn3.980. PMID: 31024718; PMCID: PMC6475805.


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