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CHANGES IN QUALITY ATTRIBUTES OF GREY MULLET FISH (*Mugil cephalus*) ROE DURING COLD STORAGE AT (4± 1°C)

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Abstract

The fishing industry is expected to develop products from edible parts of the fish whenever possible. When fish is caught during spawning season, roe can comprise a considerable percentage of the female bodyweight. The roe is an excellent raw material for the production of diverse delicacies that can be sold at good prices in many markets. So, in this study chemical composition, qualitative, microbial and sensory changes in fresh roe (control), salted and grilled roe of grey mullet fish (*Mugil cephalus*) during refrigerated storage at 4±1°C were investigated at days 0, 3, 6, 9 and 12 of storage. Moisture, protein, fat and ash of fresh and processed roe were determined. The results indicated that moisture, protein and fat contents were 60.41, 22.3 and 12.6% respectively for fresh roe "FR", while sharply decrease in moisture content was noticed for other treatments after processing directly. Protein content was slightly significant increased ($P<0.5$) during storage period and reached to 23.5, 28.8, 32.0 and 32.9% for fresh roe "FR", salted roe "SR", grilled roe outside fish body "GRout", and grilled roe inside fish body "GRin" samples respectively at the end of storage period. Fat content was increased to 14.1, 13.3, and 14.6% for SR, GRout and GRin at zero time respectively, but was significantly decreased ($P<0.5$) with progress of storage time for all treatments.

In the quality analysis findings TMA-N, TVB-N and TBA values of SR and GRout samples were stayed within the acceptability limit values till the day 9 of storage period.

Microbiological analyses revealed the processed roe had lowest total bacterial count (TBC) and psychrophilic bacteria (PsBC) compared with raw roe. According to sensory results, it was observed that SR and GRout samples were evaluated with higher points in terms of texture, colour and flavour than the fresh roe and grilled roe "GRin" samples during storage period. Thus, the salting and grilling process with cold storage can be good methods to extension shelf life with maintaining high quality in the mullet fish roe samples.

***Keywords*:** Mullet fish (*Mugil cephalus*) roe, salting and grilling process, proximate composition, quality, microbiology and sensory properties, cold storage.

INTRODUCTION

Grey mullet (*Mugil cephalus* L.) is one of the mullet species which is a coastal migratory fish and important for food and roe. In Egypt, the 2015 production of mullets was recorded by the General Authority for Fish Resources Development (GAFRD) at 188,552 tons (GAFRD, 2015). Mullet roe is a source of high quality proteins, ω3 lipids, selenium, iron and calcium. It contains full of vitamin A, vitamin B complex, vitamin C and vitamin E (Altug and Bayrak, 2003 and Bledsoe *et al*., 2003). Also, fish roe is a valuable source of nutritive lipids, especially phospholipids and long chain unsaturated fatty acids which important for human health (Intarasirisawat *et al*., 2011). As fresh fish roe is highly perishable, it's commonly processed to be offered for sale (Krizek *et al*., 2011).

Byproducts such as scales, heads, fat, visceral, and roe are generated increasingly and discarded as waste, without any attempt to recover the essential nutrients (Chalamaiah *et al*., 2010). Among byproducts, roes are highly nutritious material rich in essential fatty acids and amino acids (Heu *et al*. 2006; NarsingRao *et al*., 2012b). Nevertheless, as other fish products, it may become a vehicle and spoiled by many bacterial pathogens. These microorganisms originate from the fish flora and are usually transmitted to the roes during processing in case of limited hygiene conditions (Gram and Huss 1996). Additionally, its microbial status is strictly depending on the microbiological quality of water (Bezirtzoglou *et al*., 1994).

Salting is a traditional preservative method of fish in many countries to increase the shelf life of food product. Length of salting period as well as salt concentration depends on the expected final product (Bellagha *et al.,* 2007 and Chaijan, 2011). The preservative effect of salt is mainly due to the decrease in water activity which has suppressive effects on growth of many spoilage organisms leading to an increase of the shelf-life time (Aubourg and Ugliano,

2002 and Frangos *et al*., 2010). Salted-dried fish roes are one of the most popular forms of roe products in many countries. Among these, the best-known is the salted-dried mullet (*Mugil* spp.) roe, called ‘‘Karasumi’’ in Japan, ‘‘Avgotaracho’’ in Greece, and ‘‘Bottarga’’ in Italy (Rosa *et al.,* 2009). Dried mullet roe is considered a stable natural source of health beneficial ω3 fatty acids (Scano *et al.,* 2009). In addition, processing by heat is hypothesized to increase food digestibility due to breakdown of complex proteins and carbohydrates. Despite this, however, vitamins, minerals, some essential amino acids, and other beneficial nutrients are lost (Mirnezami *et al.,* 2002).

The microflora of the caviar is composed of microorganisms multiplying at 35°C, such as cocci, coli-like bacteria, yeasts and moulds. These micro-organisms arise from the flora of the fish and can be transmitted to fish roes in the course of processing and it can affect the product in a negative way due to the lack of hygiene and sanitation in the course of caviar production (Brunner *et al*., 1995).

Storage time and temperature are the major factors affecting the rate of loss of quality and shelf life of fish (Whittle, 1997). So, mullet roe is either sold chilled or frozen as raw material for being further processed into dried-salted roe. However, one must remember that the composition of Bottarga may change according to both manufacturing and storage conditions, due to hydrolysis and oxidation processes affecting mainly the lipid components (Rosa *et al.,* 2009; Scano *et al*., 2009 and Rosa *et al.,* 2012). Hence, the objective of this study was to investigate the qualitative changes in fresh and processed roe of grey mullet fish (*Mugil cephalus*) during refrigerated storage at (4±1°C).

Materials and Methods

Samples:

The research material, female grey mullet (*Mugil cephalus*) samples were purchased from fishmonger at Sharkia governorate, Egypt during fish hatchery season.

1. Female mullet selection:

Press and soften the abdomen with the finger to distinguish the mullet is female or male. From the liquid flow out, milky liquid indicates the mullet fish is male while golden yellow liquid indicates female. All selected female fish were transported to the laboratory after 30 min with using ice box.

2. Roe gathering and processing:

For all sampling 16 individual female fish were selected with the average weight of 10Kg. All fish were divided into four groups each group contain four fishes. For the first three groups roe were removed as followed, hold the pectoral fin fish with the left hand and deep cut with a small knife in the right hand until the anus then carefully the roe pull up by hand. The roe must be fresh, of good colour, and the skin of roe sac must not be broken. The roe must not be over-ripe nor should it be too green or underdeveloped. First roe group was used fresh roe (FR) as control, each pair of mullet roe were placed in aseptic bags and cold storage at (4±1°C).

Roe from the second group (SR) were used to salting process by "dry-salting" method. Blood and other wastes on the roe were removed with cold water (special care has been given to avoid harvesting roe until after approximately 15 minutes of cooling the fish). Then the surface moisture of the roe was removed with a clean cloth. Roe samples were put in polystyrene boxes with one layer of salt and one layer of roe for 4 hours. After that, roes were left standing in a dry cool place (approximately 20°C) to be dried and they were then put on the wooden board, and slightly dries the mullet roe in the open air and then cover a layer of clean cloth and another wooden board on which the brick or iron block is placed to be shaped into the traditional Bottarga. After shaped for nearly four hours, the mullet roe is taken out to wipe away oil stain and salt water and exposed to the sunlight. Repeat pressing and exposure for several times until the mullet roe gets dry and hard.

Fish in the fourth group were grilled as it is in local oven at medium temperature for 30 min, after that roe were removed, thus roe was grilled inside fish body (GRin). While, each pair of mullet roe from third group were smeared with bread crumbs then roe was grilled outside fish body (GRout) by the same previously oven for 15 min. Slightly dry the mullet roe in the open air and packed in aseptic bags and cold storage at (4±1°C). All roe samples were stored in refrigerator for measuring the chemical, quality, microbial and organoleptic parameters during 0, 3, 6, 9 and 12 day.

Analytical procedures:

Proximate composition:

Roe samples were analyzed in laboratory for moisture, protein, fat and ash by using (AOAC, 2005). Moisture was determined by oven drying at 105 ºC to constant weight as mentioned in the (AOAC, 2005). Crude protein content was measured by determining nitrogen content (× 6.25) using micro-Kjeldahl unit (LABCONCO 60011) and distilling unit (KJE-LEC system 1002). Fat was determined by petroleum ether extraction using(Tecator soxtec system HT 1043 extraction unit).Ash was determined by combustion to a constant weight in a muffle at 550°C (Ney Vulcan 3-550 Furnace).Total carbohydrates were calculated by difference according to (Egan *et al*., 1981). Total carbohydrates % = 100 - (% moisture + % crude protein + % total lipids + % ash).The energy value was calculated according to Atwater method, that was formulated as Energy (kcal/100 g) = (Lipid × 9) + (Protein × 4) + (Carbohydrate × 4) according to (Falch *et al*., 2010).

Quality examination:

Total volatile basic nitrogen (TVB-N), was measured by Pearson's method (Egan *et al.,* 1997) and (TMA-N) was determined according to (AMC 1979). Thiobarbituric acid reactive substances (TBA; mg malondialdehyde (MD) kg-1 sample) was measured according to (Inanli and Coban, 2010).

Microbiological analysis:

Total bacterial count (TBC) was performed according to (AOAC, 2005). Psychrophilic bacteria count (PsBC) was detected according to (Swanson *et al.*, 1992).

Organoleptic evaluation:

The mullet roe samples were sensory evaluation at certain days of storage. The samples have been characterized by 5 panelists and were rated between 1 and 5 points as follow colour, texture, flavour and overall acceptability. 1= Very poor, 2= Poor, 3= Normal, 4= Good, 5= Very good (Kurtcan and Gonul, 1987).

Statistical analysis:

Three replicates of each trial were performed for analysis. Chemical composition, qualitative values and sensory data during storage period were statistically analyzed using tow-way ANOVA. A probability at level of 0.05 or less was considered significant. Standard errors were also estimated. All statistics were run on the computer, using the SAS program (SAS, 2000).

RESULTS AND DISCUSSIONS

Proximate Composition:

The results of chemical composition of mullet fish roe showed that fresh roe (control) contains 60.41, 22.3, 12.6, 2.02 and 2.67% for moisture, protein, fat, ash and carbohydrates respectively. While, after processing directly (zero time), the moisture content were reduced for all treatments (salted and grilled roe) and ranged from 38.70 to 46.30%. It decreased resulted may be from vaporization that was occurred during the grilling processes, also it thought that has occurred due to moisture content was cleared off from roe samples as a result of the dry salting and drying procedure**s** (Hünkar *et al*., 2008 ; Kaba *et al*., 2013 and Pourashouri *et al*., 2015). The mean protein contents for processing roes were 27.8, 30.1 and 32.3 % as wet weight basis for salted (SR), grilled roe out (GRout) and grilled roe in (GRin) respectively, which was in

good agreement with (Mahmoud *et al*., 2008). The percentage values of crude fat were increased after roe processing and reached to 14.1, 13.3, and 14.6% for SR, GRout and GRin respectively, also ash and carbohydrates were increased after processing for all treatments these results are agreement with (Caprino *et al*., 2008 and Çelik *et al*., 2012).

Changes in chemical composition of fresh and processed mullet roe during cold storage are shown in Table 1. From these data, it could be observed that moisture content of fresh roe (60.41%) was significantly higher ($P<0.5$) than that of other treatments, while the moisture contents were decreased for all treatments and it was calculated as 58.1, 45.0, 37.2 and 39.3% for fresh, salted, grilled out and grilled in roe respectively at the end of cold storage period. these results are agreement with (Hünkar *et al*., 2008 and Hojat *et al*., 2015). Crude protein was significantly increased ($P<0.5$) in all treatments compared to fresh roe (control) during cold storage. At the end of storage period, the mean protein contents of salted roe (SR), grilled roe out (GRout) and grilled roe in (GRin) were 28.8, 32.0 and 32.9 % respectively, which was in good agreement with (Mahmoud *et al*., 2008).

For processed roe, maximum fat content was recorded for (GRin) samples as ranged 13.6-14.6%, while minimum fat content was 11.5-13.3% for (GRout) samples. Fat content was slightly decrease by increasing storage time for all treatments. The present outcomes may be explained that presence of salt in mullet roe processing prevented from diminishing fat and fortifying it during the cold storage (Hojat *et al*., 2015). The reason for the latter is that adding low salt into the roe, blocks enzymatic activities that are responsible for hydrolyzing lipid (Yasemen *et al*., 2005).

The results of ash in mullet roe showed in Table 1. After processing, the amount of ash in fish roe was increased. Ash content was 2.02% for control (FR), while was 4.68, 5.21 and 4.98 for (SR), (GRout) and (GRin) respectively at the zero time. Ash content was changed significantly ($P<0.5$) during storage among different roe samples an reached to 1.88, 4.16, 5.10 and 4.84% for (FR), (SR), (GRout) and (GRin) respectively at the end of storage period. The present finding is similar to (Sengor *et al*., 2000 and Gessner *et al*., 2010).

Table 1. Chemical analysis values in the fresh, salted and grilled roe during the cold storage at 4±1°C.

Treatments	Storage Period (days)				
	0	**3**	**6**	**9**	**12**
	Moisture %				
FR	60.41 ± 3.55^{aA}	60.0 ± 4.51^{aA}	59.6 ± 4.11^{aA}	58.6 ± 4.01^{bA}	58.1± 4.05^{bA}
SR	46.3 ± 2.37^{aB}	46.1 ± 3.06^{aB}	45.8 ± 3.71^{bB}	45.3 ± 3.72^{bB}	45.0 ± 3.33^{bB}
GRout	38.7 ± 2.17^{aC}	38.4 ± 2.33^{aC}	38.1 ± 2.91^{bC}	37.7 ± 1.17^{bC}	37.2 ± 2.19^{cC}
GRin	40.41 ± 2.51^{aC}	40.2 ± 2.88^{aB}	40.0 ± 2.59^{bC}	39.7 ± 2.55^{bC}	39.3 ± 2.65^{cC}
	Protein %				
FR	22.3 ± 1.22^{cD}	22.5 ± 1.21^{bD}	22.8 ± 1.11^{bC}	23.1 ± 1.26^{aC}	23.5 ± 1.23^{aC}
SR	27.8 ± 1.54^{cC}	28.0 ± 1.17^{bC}	28.1 ± 1.32^{bB}	28.4 ± 1.32^{bB}	28.8 ± 1.33^{aB}
GRout	30.1 ± 1.71^{dD}	30.5 ± 2.11^{cD}	30.9 ± 1.73^{cA}	31.4 ± 1.56^{bA}	32.0 ± 2.11^{aA}
GRin	32.3 ± 1.87^{bA}	32.7 ± 1.75^{bA}	32.0 ± 1.86^{cA}	32.4 ± 2.04^{bA}	32.9 ± 2.06^{aA}
	Fat %				
FR	12.6 ± 0.37^{aC}	12.3 ± 0.21^{aB}	12.1 ± 0.13^{bC}	11.8 ± 0.07^{cD}	11.4 ± 0.09^{cC}
SR	14.1 ± 0.15^{aA}	13.9 ± 0.13^{aA}	13.6 ± 0.11^{bB}	13.2 ± 0.08^{cB}	12.8 ± 0.13^{cB}
GRout	13.3 ± 0.12^{aB}	13.0 ± 0.17^{aB}	12.6 ± 0.14^{bC}	12.1 ± 0.11^{cC}	11.5 ± 0.12^{dC}
GRin	14.6 ± 0.14^{aA}	14.4 ± 0.11^{aA}	14.1 ± 1.15^{bA}	13.9 ± 0.17^{bA}	13.6 ± 0.16^{cA}
	Ash %				
FR	2.02 ± 0.01^{aD}	2.00 ± 0.01^{aD}	1.97 ± 0.01^{bD}	1.93 ± 0.01^{cD}	1.88 ± 0.01^{dD}
SR	4.68 ± 0.04^{aC}	4.59 ± 0.04^{bC}	4.49 ± 0.03^{cC}	4.34 ± 0.03^{dC}	4.16 ± 0.05^{eC}
GRout	5.21 ± 0.07^{aA}	5.20 ± 0.04^{aA}	5.17 ± 0.05^{aA}	5.13 ± 0.05^{bA}	5.10 ± 0.08^{bA}
GRin	4.98 ± 0.06^{aB}	4.96 ± 0.06^{aB}	4.93 ± 0.07^{bB}	4.89 ± 0.07^{cB}	4.84 ± 0.05^{cB}

FR=Fresh roe SR= Salted roe GRout = Grilled roe outside fish body GRin = Grilled roe inside fish body.

$^{A-D}$ Superscripts in a column are significantly different ($P < 0.05$).

$^{a-e}$ Superscripts in a raw are significantly different ($P < 0.05$).

Table 2. Changes in carbohydrates and energy for fresh, salted and grilled roe during the cold storage at 4±1°C.

Treatments	Storage Period (days)				
	0	**3**	**6**	**9**	**12**
			*Carbohydrates %		
FR	2.67± 0.01^{dC}	3.20 ± 0.01^{cC}	3.53 ± 0.01^{cC}	4.57± 0.02^{bC}	5.12± 0.06^{aC}
SR	7.12 ± 0..22^{cB}	7.41 ± 0.12^{cB}	8.01 ± 0.13^{bB}	8.76 ± 0.11^{bB}	9.24 ± 0.21^{aB}
GR out	12.69 ± 0.36^{cA}	12.9 ± 0.41^{bA}	13.23 ± 0.37^{bA}	13.67 ± 0.33^{bA}	14.20 ± 0.36^{aA}
GRin	7.71 ± 0.13^{cB}	7.74 ± 0.14^{cB}	8.97 ± 0.11^{bB}	9.11 ± 0.12^{aB}	9.36 ± 0.14^{aB}
			Energy (kcal/100 g)		
FR	213.28 ± 11.66^{aC}	213.50 ± 10.52^{aC}	214.22 ± 11.44^{aC}	216.88 ± 16.12^{aC}	217.08 ± 14.06^{aC}
SR	266.58 ± 20.20^{aB}	266.74 ± 13.55^{aB}	266.84 ± 14.64^{aB}	267.44 ± 10.66^{aB}	267.36 ± 18.05^{aB}
GRout	290.86 ± 16.66^{aA}	290.60 ± 12.90^{aA}	289.92 ± 16.12^{aA}	289.18 ± 16.76^{aA}	288.30 ± 11.26^{aA}
GRin	291.44 ± 15.92^{aA}	291.36 ± 17.12^{aA}	290.78 ± 12.24^{aA}	291.14 ± 14.18^{aA}	291.44 ± 14.15^{aA}

FR=Fresh roe SR= Salted roe GRout = Grilled roe outside fish body GRin = Grilled roe inside fish body

$^{A-C}$ Superscripts in a column are significantly different ($P < 0.05$).

$^{a-d}$ Superscripts in a raw are significantly different ($P < 0.05$).

*calculated by difference.

On the other hand, results from Table 2 showed the carbohydrates content (2.67%) for fresh roe (FR) and increased to 7.12%, 12.69% and 7.71% for (SR), (GRout) and (GRin) samples respectively at zero tome, while reached to 5.12%, 9.24%, 14.20% and 9.36% for (FR), (SR), (GRout) and (GRin) respectively at the end of storage time. It was seen that addition of bread crumbs into the roe samples (GRout) lead to increase the carbohydrates value. Also, carbohydrates values were significantly increased ($P<0.5$) in all processed roe during cold storage, these results are agreement with (Kaba *et al*., 2013). In addition, it was observed that the calorie values were affected from this increase and reached to 217.08, 267.36, 2.88.30 and 291.44 kcal at the end of cold storage for fresh roe, salted roe, grilled roe out and grilled roe in samples respectively. Changes in chemical composition of roe may be due to several factors which affect on these parameters. Thus, Inanli *et al*., (2010) has

recorded that the chemical composition of the resulting caviar depends on fish species and processing techniques. Also the difference in chemical composition of various fish roe is mainly attributed to biological factors, including species, maturity stages, diet, season, harvest area and processing conditions (Mahmoud *et al*., 2008).

Quality control criteria:

Quality control values of fresh, salted and grilled roe during the cold storage at (4±1°C) illustrated in Table 3. In fresh fish, TMA-N values should be close to 1 mg N/100g and in spoiled samples it is more than 8 mg N/100g (FAO, 1986). Changes in TMA-N levels of mullet roe during 12 days of refrigerated storage are shown in Table 3. Initially (0 time), the TMA-N content of all mullet roe samples was lower than 6 mg N/100 g indicating the good quality of these products. While, TMA-N values of roe samples were significantly increase ($p<0.05$) during storage period and reached to 14.4; 7.9; 9.2 and 12.6 N/100 g for (FR), (SR), (GRout) and (GRin) respectively. On the 9th day of storage, fresh roe, grilled out and grilled roe in samples were spoiled based on TMA-N analysis but the salted roe samples were not spoiled and retained quality to the end of storage duration. While, TMA-N is not found suitable for measuring quality changes of salted and grilled roe products since bacterial activity could be decreased depending on low water content. Also at low temperatures, protein denaturation was reduced by inhibition of some enzymes and microorganisms which are normally present in caviar. Such findings are in agreement with (Çelik *et al.,* 2012).

TVB–N content was increased in the fresh roe from 12.20 to 39.70 mg N/100g sample, in the salted roe from 15.70 to 28.61 mg N/100g of sample, in the grilled roe out from 16.51 to 30.80 mg N/100g of sample and in the grilled roe in from 17.20 to 35.77 mg N/100g of sample during the cold storage. TVB-N used indicator for all the volatile N containing compounds during decomposition. So, this parameter is accepted as a spoilage index for fish and seafood (FAO, 1986), which has indicated that samples with TVB-N value less

than 25 mg N/100g are ‘perfect quality’, samples with up to 30 mg N/100g are ‘good quality’, samples with up to 35 mg N/100g are ‘marketable quality’ and the samples with TVB-N value more than 35 mg N/100g are indicated as spoiled (Özyurt *et al.,* 2009). According to TVB-N values for fresh roe (control) was spoiled on day 9 followed by grilled in roe (GRin) treatment was marketable quality. Based on the TVB-N content, salted roe (SR) and grilled roe out (GRout) samples consider high quality on day 9. Also, salted roe was high quality till the end of cold storage, indicating that the addition of salt could apparently have some inhibitory effects on spoilage of salted roe samples due to the decrease in water activity which has suppressive effects on growth of many spoilage organisms, it was in agreement with (Horner, 1997; Kung *et al.,* 2008 and Frangos *et al.,* 2010).

The TBA index is a widely used indicator for the assessment of the degree of lipid oxidation (Nishimoto *et al*., 1985). The amount of TBA less than 3 mg malonaldehyde in roe shows very good quality, 3 to 5 mg malonaldehyde, indicates good quality and the authorized consumption level is 7 to 8 mg malonaldehyde into 1000 g of roe these measurements according to (Inanli and Coban, 2010).

Data in Table 3 illustrated that TBA value was gradually increased from 0.66 to 1.80 mg malonaldehyde/kg for (FR) samples during storage period. TBA value in salted roe was changed from 3.45 to 3.12 mg malonaldehyde/kg; in the grilled roe out from 3.55 to 3.90 mg malonaldehyde/kg and in the grilled roe in from 2.89 to 4.40 mg malonaldehyde/kg during storage period. TBA values were diminished in the 3 day of cold storage for salted roe and grilled out roe, after that slightly increase were observed for all roe samples. Significant differences ($P<0.5$) were observed in TBA values between fresh roe and processed roe samples by increasing storage duration. The reason of this increase in TBA value is the effect of salting and grilling process which may be caused increase lipid oxidation. However, TBA findings of this study it is seen that all processed roe had a good quality until the 12 day of storage. Such

findings are in agreement with those of reported by (Hünkar *et al*., 2008 and Rosa *et al*., 2009).

Table 3. Quality control values of fresh, salted and grilled roe during the cold storage.

Treatments	Storage Period (days)				
	0	**3**	**6**	**9**	**12**
	TMA-N (mg/100g)				
FR	5.6 ± 0.13^{cA}	6.2 ± 0.04^{dA}	7.6 ± 0.06^{cA}	10.3 ± 0.12^{bA}	14.4 ± 0.19^{aA}
SR	4.1 ± 0.13^{dD}	5.7 ± 0.04^{cB}	6.2 ± 0.05^{cC}	7.1 ± 0.06^{bD}	7.9 ± 0.07^{aD}
GRout	4.6 ± 0.17^{eC}	5.9 ± 0.03^{dB}	6.8 ± 0.04^{cB}	8.4 ± 0.06^{bC}	9.2 ± 0.11^{aC}
GRin	5.1 ± 0.09^{eB}	6.1 ± 0.17^{dA}	7.3 ± 0.17^{cA}	9.5 ± 0.05^{bB}	12.6 ± 0.18^{aB}
	TVB-N (mg/100g)				
FR	12.20 ± 0.13^{dD}	14.45 ± 0.96^{dD}	25.50 ± 1.66^{cA}	35.30 ± 2.37^{bA}	39.70 ± 2.11^{aA}
SR	15.70 ± 1.06^{dC}	18.15 ± 1.13^{cC}	20.90 ± 1.37b^{cC}	23.18 ± 1.65^{bD}	28.61 ± 1.79^{aD}
GRout	16.51 ± 1.02^{eB}	19.20 ± 1.22^{dB}	22.10 ± 1.29^{cB}	26.01 ± 1.24^{bC}	30.80 ± 1.89^{aC}
GRin	17.20 ± 1.11^{eA}	21.90 ± 1.23^{dA}	25.10 ± 1.52^{cA}	31.50 ± 2.51^{bB}	35.77 ± 2.33^{aB}
	TBA (mg malondialdehyde kg-1)				
FR	0.66 ± 0.002^{dC}	1.32 ± 0.01^{cD}	1.40 ± 0.01^{cD}	1.54 ± 0.02^{bD}	1.80 ± 0.02^{aD}
SR	3.45 ± 0.03^{aA}	2.15 ± 0.02^{dC}	2.30 ± 0.01^{dC}	2.80 ± 0.02^{cC}	3.12 ± 0.03^{bC}
GRout	3.55 ± 0.02^{bA}	2.82 ± 0.02^{cB}	2.93 ± 0.01^{cB}	3.44 ± 0.03^{bB}	3.90 ± 0.04^{aB}
GRin	2.89 ± 0.01^{dB}	3.20 ± 0.04^{cA}	3.80 ± 0.02^{bA}	4.00 ± 0.03^{bA}	4.40 ± 0.04^{aA}

FR=Fresh roe SR= Salted roe GRout= Grilled roe outside fish body GRin= Grilled roe inside fish body

$^{A-D}$ Superscripts in a column are significantly different ($P < 0.05$).

$^{a-e}$ Superscripts in a raw are significantly different ($P < 0.05$).

Microbiological analysis:

Microbiological analysis results of the fresh, salted and grilled roe during the cold storage at (4±1°C) are given in Table 4 and Figure 1. It was observed in the microbiological analysis results that, microbial load of the fresh roe samples were decreased with salting and grilling process. Thus processing

techniques were caused decreased total bacterial count (TBC) in all treatments and were 2.30, 2.61 and 3.20 Log10 CFU/g for (SR), (GRout) and (GRin) at zero time, respectively, compared with fresh roe (3.36 Log10 CFU/g). While throughout a gradual significantly increase ($p<0.05$) in TBC and Psychrophilic bacteria count (PsBC) were observed during storage duration. FR and GRin treatments showed the highest TBC content and were 7.71and 7.61 Log10 CFU/g respectively at the end of storage period. At the day 12 of cold storage PsBC contents were 6.84 and 6.54 Log10 CFU/g for FR and GRin treatments respectively. The salted roe showed the lowest TBC and PsBC content as compared with the other processed roe. The salty treatment caused decreasing microorganism growth and increasing shelf life of the roe due to inhibitory specifications of salt by decreasing humidity degree and pH (Altug and Bayrak, 2003). Thus, the salting can be a good way to increase shelf life with maintaining high quality in the roe. The results of this research are in accordance to Safari and Yosefian (2006) and Inanli *et al.* (2011).

Table 4. Changes in Total bacterial count and Psychrophilic (Log10 CFU/g) of fresh, salted and grilled roe during the cold storage at 4±1°C.

Treatments	Storage time(days)				
	0	**3**	**6**	**9**	**12**
	Total bacterial count				
FR	3.36 ± 0.15^{cA}	3.45 ± 0.17^{cA}	5.71 ± 0.14^{bcA}	6.0 ± 0.16^{bA}	7.71 ± 0.21^{aA}
SR	2.30 ± 0.07^{cB}	2.82 ± 0.05^{bcAB}	3.10 ± 0.08^{bB}	3.78 ± 0.08^{abC}	4.46 ± 0.10^{aC}
GRout	2.61 ± 0.05^{AB}	2.97 ± 0.07^{bcAB}	3.70 ± 0.06^{bB}	4.65 ± 0.05^{abB}	5.98 ± 0.08^{aB}
GRin	3.20 ± 0.11^{cA}	3.40 ± 0.11^{cA}	5.54 ± 0.12^{bA}	5.97 ± 0.13^{bA}	7.61 ± 0.13^{aA}
	Psychrophilic bacteria				
FR	1.30 ± 0.05^{eA}	2.20 ± 0.07^{AB}	4.45 ± 0.13^{cA}	5.72 ± 0.15^{bA}	6.84 ± 0.15^{aA}
SR	0.98 ± 0.03^{deA}	1.65 ± 0.05^{dC}	2.40 ± 0.07^{cC}	3.21 ± 0.12^{bBC}	4.88 ± 0.11^{aBC}
GRout	1.12 ± 0.07^{dA}	2.60 ± 0.12^{cA}	3.20 ± 0.14^{bcB}	3.97 ± 0.12^{bB}	5.12 ± 0.15^{aB}
GRin	1.18 ± 0.04^{eA}	2.00 ± 0.08^{dB}	3.35 ± 0.15^{cB}	5.51 ± 0.17^{bA}	6.54 ± 0.19^{aA}

FR=Fresh roe SR= Salted roe GRout= Grilled roe outside fish body GRin= Grilled roe inside fish body

$^{A-C}$ Superscripts in a column are significantly different ($P < 0.05$).

$^{a-e}$ Superscripts in a raw are significantly different ($P < 0.05$) .

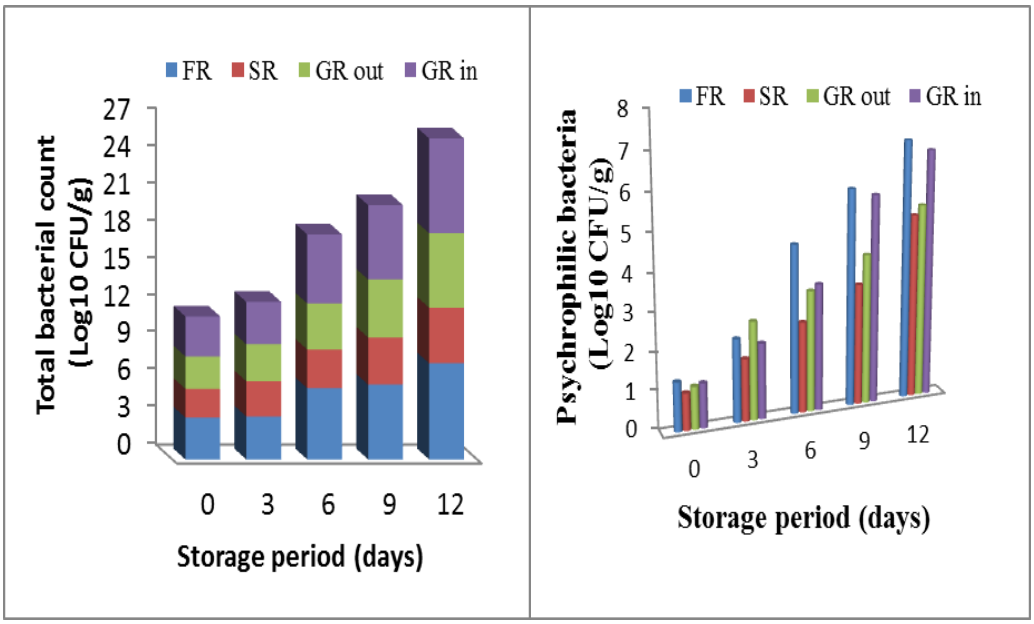

Figure 1. Changes in Total bacterial count and Psychrophilic (Log10 CFU/g) of fresh, salted and grilled roe during the cold storage at 4±1°C.

Organoleptic evaluation:

The results of organoleptic properties of mullet roe were depicted in Figure 2. Colour measurement is one of the important parameters in processed fish products because of consumers associated with a natural and characteristic caviar colour (Bekhit *et al.*, 2009 and Çelik *et al.*, 2012). According the result of the sensory evaluation grilled roe out (GRout) samples which were prepared by adding bread crumbs, it had light brown colour and attractive flavour and good texture until the day 9 of storage period, while salted roe had a good texture, colour and flavour to the end of cold storage period. The colour of fish roe is different according to fish species, diet, and age and carotenoids pigments dissolved in lipids such as lutein, astaxanthin, cantaxanthin, zeaxanthin, betacorothin. These compounds are very sensitive to processing conditions such as heat and oxidation. The colour and flavour of roe indicated its quality from sanitary and health perspective. The present finding is the similar to (Bledsoe *et al.*, 2003, Inalli and Coban 2010, and Inali *et al.*, 2011).On the other hand, sensorial scores of (FR) and (GRin) samples were

crumbling after 6 days of storage period. In addition, overall acceptability of roe was affected by its colour. In general, texture; colour; flavour and overall acceptability in the fresh, salted and grilled roe samples were decreased during the cold storage at (4±1°C).

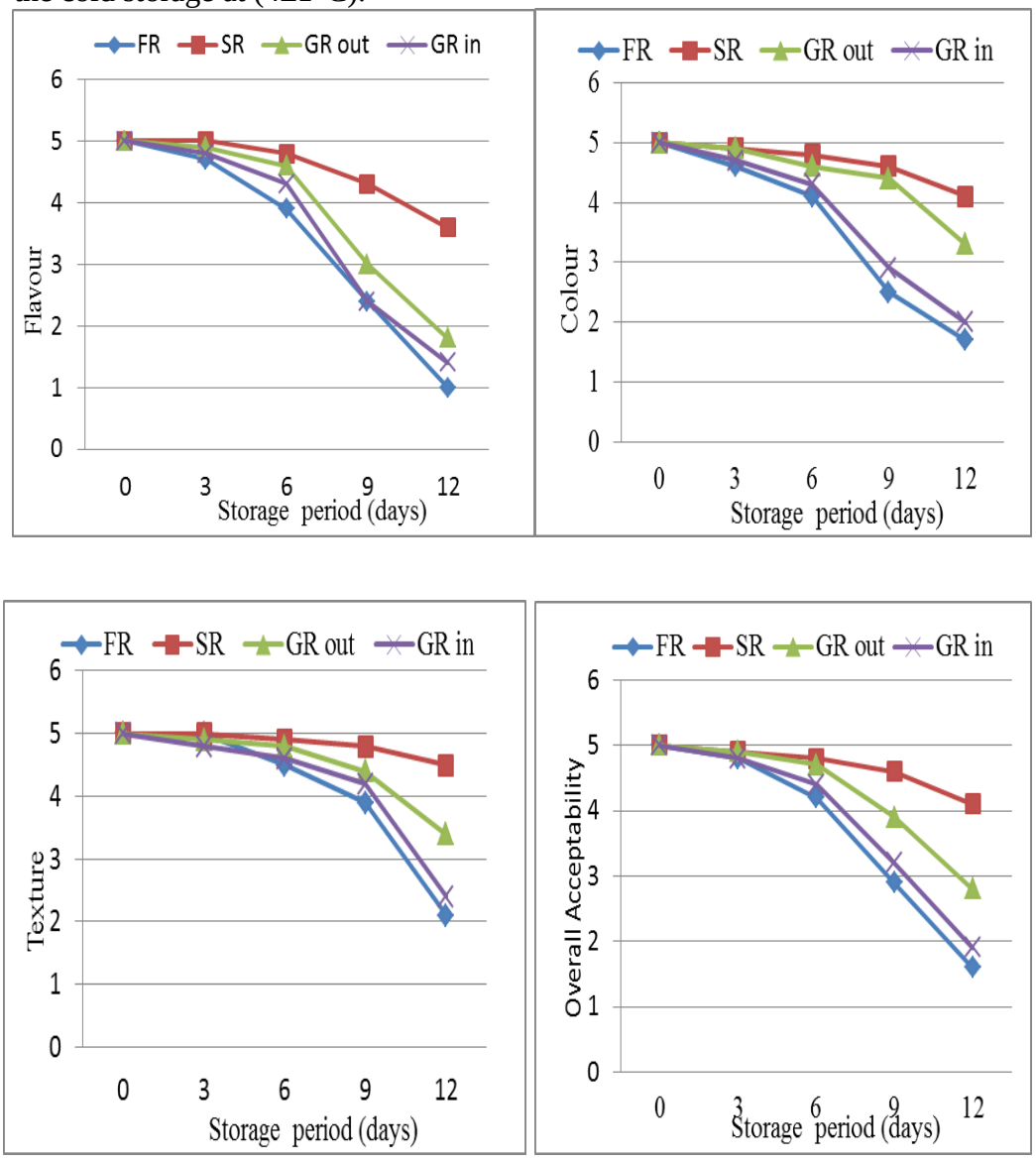

Figure 2. Organoleptic values of fresh, salted and grilled roe during the cold storage at 4±1°C.

CONCLUSIONS

Chemical, quality, microbial and sensory characteristics of experimentally prepared four treatments of grey mullet fish roe during refrigerated storage at 4±1°C were investigated. As the results of this study, it was found that roe of mullet fish can be processed to marketable products like that salted and grilled roe with a good quality. Due to processing techniques were caused reduced microorganisms, so salting and grilling process had good quality and positive effect on increasing shelf life. Conversely, quality characteristics of fresh roe were declined during cold storage.

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التغيرات فى خواص الجودة لبطارخ سمك البورى المخزنة بالتبريد على درجة حرارة ٤± ١ °م

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الملخص العربى

تتوقع صناعة الأسماك حدوث تطور فى منتجات الأجزاء المأكولة من الأسماك ، فعندما يتم صيد الأسماك خلال موسم التبويض فان بطارخ الأسماك تشكل نسبة كبيرة بالنسبة لوزن الأسماك الاناث. كما ان بطارخ الأسماك تعتبر مادة خام ممتازة لتصنيع منتجات طيبة ومتنوعة يمكن بيعها باسعار جيدة فى العديد من الاسواق، لذلك اجريت هذة الدراسة لتقدير التغيرات فى التركيب الكيميائى وخواص الجودة والخواص الميكروبية والحسية للبطارخ الطازجة لسمك البورى (الكنترول) والبطارخ المملحة والبطارخ المشوية خارج وداخل جسم السمكة خلال فترات التخزين (٠، ٣، ٦، ٩، ١٢ يوم) بالتبريد على درجة ٤±١° م.

اشارت النتائج الى ان محتويات الرطوبة والبروتين والدهن كانت ٦٠.٤١، ٢٢.٣، ١٢.٦ % للبطارخ الطازجة على التوالى. لوحظ انخفاض حاد فى نسبة الرطوبة لكل المعاملات بعد التصنيع مباشرة. ارتفاع معنوى ($P<0.5$) فى محتوى البروتين اثناء فترة التخزين حيث وصل الى ٢٣.٥ ، ٢٨.٨، ٣٢.٠، ٣٢.٩ % فى نهاية فترة التخزين لعينات البطارخ الطازجة، البطارخ المملحة ، البطارخ المشوية خارج جسم السمكة والبطارخ المشوية داخل جسم السمكة على التوالى. ايضا ارتفع محتوى الدهن الى ١٤.١، ١٣.٣، ١٤.٦% للبطارخ المملحة ،البطارخ المشوية خارجيا، البطارخ المشوية داخليا على التوالى فى بداية التخزين، ولكن لوحظ انخفاض معنوى ($P<0.5$) فى نسبة الدهن مع تقدم فترة التخزين لكل المعاملات. اوضحت نتائج تحليل خواص الجودة ثبات قيم الـ TMA-N, TVB-N, TBA لعينات البطارخ المملحة والبطارخ المشوية خارجيا عند الحدود المقبولة حتى اليوم التاسع من التخزين. انخفاض العدد الكلى للبكتريا والبكتريا المحبة للبرودة فى البطارخ المصنعة مقارنة بالبطارخ الخام. وطبقا لنتائج التقييم الحسى لوحظ ارتفاع نقاط قيم القوام واللون والنكهة لعينات البطارخ المملحة والبطارخ المشوية خارجيا مقارنة بعينات البطارخ المشوية داخليا والبطارخ الطازجة خلال فترة التخزين. وهكذا تكون عملية التمليح وعملية الشى مع التخزين بالتبريد طرق جيدة لاطالة العمر التخزينى والحفاظ على جودة عينات بطارخ اسماك البورى.