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Determination of quality and aqua veterinary drugs residues of some cultured fish and their processed products

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ABSTRACT

This study was planned to determine the quality and aqua veterinary drug residues of some cultured fish and their processed products. Fish samples of the Nile tilapia (*Oreochromis niloticus*) and mullet (*Mugil cephalus*) were purchased from a fish farm at El-Fayoum, Egypt during September 2020 and cooked by grilling and frying methods. Quality indices of pH, total volatile nitrogen (TVN), trimethylamine (TMA), thiobarbituric acid (TBA) as well as safety parameters; eight aqua veterinary drugs residues, total plate count (TPC), and spore-forming bacteria (SFB) were determined. Results showed that values of quality parameters were based mainly on fish species and cooking method conditions and they were within the maximum permissible limits (MPLs). With regard to safety, although TPC and SFB were among permitted counts the raw tilapia samples contained 7428ppb tetracycline and 5000ppb aminoglycosides, respectively, however, residues of these compounds were neither detected in grilled or fried tilapia products nor in raw, grilled or fried mullet products as well. In conclusion, although cultured fish samples are of good quality they are contaminated, especially raw tilapia, by high levels with tetracycline and aminoglycosides residues. It is worthy to mention that, cooking methods could reduce those compounds up to 100%. Hence, in this study, the usage of drugs was recommended to never exceed the MPLs and to ensure withdrawal periods before fish marketing.

INTRODUCTION

Fish are susceptibile to many diseases, hence, antibacterial drugs have been largely used as therapeutic application, prophylactic utilization, and growth promoters. However, overusing of those compounds can create some resistant pathogens; therefore, a proper withdrawal period should be ensured before slaughtering and marketing **(Soumik *et al.*, 2014; Heshmati *et al.*, 2015**; **Huicab-Pech *et al.*, 2016; Shaltout *et al.*, 2019a)**. The global efforts are needed to promote more judicious use of prophylactic antibiotics in aquaculture and others (**Manage, 2018**). Besides, cooking methods; boiling, frying and grilling of chicken muscles can reduce concentrations by 84.52%, 93.62% and 96.58% oxytetracycline (OTC) and 81.22%, 90.54% and 94.5% of ampicillin residues, respectively (**Elbagory** *et al.*, 2016) however, microwaving may not reduce the

concentration OTC below the maximum residue limit if the initial concentration is very high (**Vivienne *et al.*, 2018**). In addition, **Shaltout *et al.* (2019**b**)** found that Ciprofloxacin and OTC were reduced by 20.74% and 87.97% when boiled, 38.14% and 86.95% if microwaved, and 12.84% and 73.98% in roasted samples, respectively. Therefore, this work was performed to determine the quality and aqua veterinary drugs residues of some cultured fish and their processed products, using fish samples of *Oreochromis niloticus* and *Mugil cephalus* that were purchased from fish farm at El-Fayoum, Egypt during September 2020.

MATERIALS AND METHODS

Fish samples

The Nile tilapia (*O. niloticus*) and Mullet (*M. cephalus*) were obtained from fish farm localized at El-Fayoum during September, 2020. Fish specimens were transferred in an icebox to Fish Processing Technology Lab., Fisheries Division, National Institute of Oceanography and Fisheries, Egypt. Tilapia and mullet samples recorded lengths of 27±1.58 and 45±2.48cm, and weights of 508±90.55g and1.0003±143.49g, respectively. Fish samples were washed carefully with tap water, gutted, cut as steaks, rewashed, drained, rubbed in wheat bran and grilled at 170ºC for 20 min using electric pre-heated hot plate. While, other samples were rubbed in maize flour and deep oil fried at 170ºC for 5-7min by a Braun electric fryer.

Analytical methods

The pH value and water activity (**AOAC, 2007**), cooking loss (**Roland *et al.*, 1981**), total volatile basic nitrogen (TVN) and trimethylamine (**AMC, 1979**) and thiobarbituric acid value (**Tarladgis *et al.*, 1960**) were determined. Additionally, total plate count (TPC) and spore forming bacteria (SFB) were examined (**APHA, 2001**). Bioassay procedures for detection and quantitation of antibiotics residues in raw and processed fish products were done according to **USDA (2011)** at Institute of Animal Health Research, Ministry of Agriculture and Soil Reclamation, Egypt. The results obtained (n=3) were statistically analyzed using SPSS (Ver.16) and all results were expressed as Mean ± SD.

RESULTS AND DISCUSSION

1- Cultured fish

1.1.Physico-chemical quality indices

Table (1) shows the physico-chemical characteristics of the cultured fresh and processed tilapia and mullet fish samples. Cooking loss in grilled tilapia (13.60%) was more than that in the grilled mullet (11.74%). The same trend was observed in the case of fried products recording 9.79% and 8.40% for fried tilapia and mullet, respectively. Remarkably, those values are reflected on yield %. Initial pH recorded values were 6.51

and 6.02 for the raw tilapia and mullet samples, respectively. Interestingly, a slight increase was observed in all processed fish products. On the other hand, an increase in pH recorded values (6.30) in grilled and fried mullet was similarly monitored. Values of A_w were similar in the raw tilapia and mullet, recording a value of 0.97, while the value of their processed products was 0.96, indicating a similatity too. The TVN content (18.15mg\100g) in the raw tilapia was lower than that in the raw mullet (24.45 mg\100g). Processing techniques increased TVN content in both grilled and fried products. This increment is mainly due to thermal processes used. The same trend was observed in the case of TMA content. Concerning TBA values, they recorded 0.81 and 1.29 mg MDA/kg in the raw tilapia and mullet, respectively. Furthermore, values of the aforementioned factor increased in all processed products, particularly fried products.

Table 1. Physico-chemical characteristics of cultured fresh and processed tilapia and mullet samples during September, 2020.

Index	Cultured fish *spp.;*					
	O. niloticus			***M. cephalus***		
	Raw	**Grilled**	**Fried**	**Raw**	**Grilled**	**Fried**
Cooking loss (%)	-	13.60	9.79	-	11.74	8.40
Yield (%)	-	85.40	90.21	-	88.26	91.60
pH	6.51±0.01	6.81±0.02	6.90±0.01	6.02±0.02	6.30±0.03	6.30±0.02
A_w	0.97	0.96	0.96	0.97	0.96	0.96
*TVN (mg/100g)	18.15±1.23	20.85±0.86	20.16±0.99	24.45±0.94	28.24±1.12	27.50±0.98
**TMA-N (mg/100g)	6.22±1.28	6.69±1.07	6.45±1.13	8.24±1.07	9.50±0.79	9.32±0.88
***TBA (mg MDA/kg)	0.81±0.33	0.88±0.54	0.92±0.53	1.29±0.18	1.72±0.28	1.77±0.55

*TVN: total volatile basic nitrogen, **TMA-N: trimethylamine nitrogen, ***TBA: thiobarbituric acid.

1.2. Microbial load

Microbial load (log_{10} cfu/g) of the cultured raw and processed tilapia and mullet fish samples is shown in Table (2). Initial TPC in the raw tilapia sample was lower (4.20 log_{10} cfu/g) than that in the mullet (4.46 log_{10} cfu/g). Concerning the effect of processing, it was found that TPC reduced noticeably in processed products, especially fried products. On the other side, SPB in raw tilapia (4.03 log_{10} cfu/g) was lower than that in the raw mullet (4.19 log_{10} cfu/g). Values of SPB increased sharply in fried tilapia (4.26 log_{10} cfu/g) and mullet (4.76 log_{10} cfu/g) than that in grilled products; 4.05 and 4.29 log_{10} cfu/g, respectively.

Table 2. Microbial load (log_{10} cfu/g) of cultured fresh and processed tilapia and mullet fish samples during September, 2020.

Index	Cultured fish *spp.;*					
	O. niloticus			***M. cephalus***		
	Raw	**Grilled**	**Fried**	**Raw**	**Grilled**	**Fried**
TPC	4.20±1.14	4.13±0.92	3.90±0.64	4.64±1.33	4.23±0.94	3.61±1.43
SPB	4.03±0.06	4.05±0.97	4.26±1.24	4.19±1.04	4.29±1.86	4.76±0.93

TPC: Total plate count; SPB: Spores forming bacteria.

1.3. Aqua veterinary residues

Table (3) exhibits aqua veterinary drugs residues (pbb) of the cultured raw and processed tilapia and mullet samples. 8 antibiotic compounds were determined, high levels of tetracycline (7428pbb) and aminoglycoside (500ppb) compounds were found only in raw tilapia sample, whereas processed tilapia products showed no residues. It was noticed that, the raw and processed mullet samples contained no residues of the studied compounds. Moreover, a reduction rate of tetracycline and aminoglycoside recorded 100% in grilled and fried tilapia products.

Table 3. Aqua veterinary drugs residues (pbb) of cultured fresh and processed tilapia and mullet flesh during September, 2020.

Compound	Cultured fish *spp.;*						**MPLs
	O. niloticus			*M. cephalus*			
	Raw	Grilled	Fried	Raw	Grilled	Fried	
Tetracycline	**7428**	Nd	Nd	Nd	Nd	Nd	***200
Beta-Lactam	*Nd	Nd	Nd	Nd	Nd	Nd	
Penicillin	Nd	Nd	Nd	Nd	Nd	Nd	
Streptomycin	Nd	Nd	Nd	Nd	Nd	Nd	
Fluroqinolone	Nd	Nd	Nd	Nd	Nd	Nd	
Macrolide	Nd	Nd	Nd	Nd	Nd	Nd	
Erythromycin	Nd	Nd	Nd	Nd	Nd	Nd	
Aminoglycoside	**5000**	Nd	Nd	Nd	Nd	Nd	Not limited

*Nd: not detected.
** MPLs: Maximum permissible limits as set by **EOS (2008)** and **CAC (2018).**
***: only for Oxytetracycline.

In this study, the pH values of raw fish are in agreement with those reported by **Howgate (2009)**, which can be used as a good indicator for fish freshness, quality and deterioration. Moreover, the increase in the pH values of the processed fish coincides with the findings of **Ersoy *et al.* (2008)**, who reported that the increment in pH values of the processed samples might be due to the formation of some basic compounds as a result of amino acid degradation (**Ruiz-Capillas & Moral, 2001)**. Eminently, cooking techniques caused an increase in TBA, a trend which agrees with that reported by **Sallam *et al.* (2007**). The previous authors assessed that the increase in TBA might be due to the formation of secondary products of lipid oxidation. Concerning microbial load in this work, similar results in TPC of the processed samples were found by **El-Sherif *et al.* (2011)**, who reported that a decrease in TPC of the processed samples might be related to the thermal destruction of microorganisms. However, SPB increased as affected by cooking temperatures used. With regard to the residues of aquadrugs in raw and cooked samples, othe current results concur with those of **Heshmati (2015)**, who stated that drug residues in animal based foods might be decreased due to cooking time and temperature as main factors which affect antibiotic residues. Temperature during cooking has the greatest impact on the loss of tetracycline residues (**Hassani *et al.*, 2008; Abou-Raya *et al.*, 2013**). To illustrate, **Shaltout *et al.* (2019a**) concluded that cooking procedure was one of the most important agents that affected tetracycline residues. However, other

studies concluded that cooking processes do not guarantee a full breakdown of those drugs present in condemned animals (**Javadi, *et al.*, 2011)**.

CONCLUSION

Although cultured fish samples have a good quality but they are contaminated at high levels with tetracycline and aminoglycosides residues. Interestingly, cooking methods can reduce those compounds up to 100%. Hence, it is recommended that the usage of drugs must not exceed the MPLs and withdrawal periods should be safeguarded before fish marketing.

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