

TOXICOLOGICAL EVALUATIONS OF TREATED COSMETIC EFFLUENT ON *CLARIAS GARIEPINUS* (THE AFRICAN SHARPTOOTH CATFISH)

*Sogbanmu, T. O, Bawa-Allah, K. A. and Oroboade, D. F.

Ecotoxicology and Conservation Unit, Department of Zoology, Faculty of Science, University of Lagos, Akoka 101017, Lagos, Nigeria.

*corresponding author: tsogbanmu@unilag.edu.ng

ABSTRACT

The preponderance of industrial activities has led to the discharge of often untreated effluents into the aquatic environment. Some industries endeavour to carry out some form of treatment on their effluent before discharge into the environment; however, there is need to determine the efficiency of such treatment by evaluating potential biological effects in aquatic organisms. In this study, we investigated the physico-chemical parameters of a treated cosmetic effluent as well as the biochemical and histological effects of sublethal concentrations of the effluent in *Clarias gariepinus* (The African sharptooth catfish) over a period of 28 days. Some physico-chemical parameters including zinc and lead were higher than the Federal Ministry of Environment set limits while others such as phthalate, triclosan and parabens were not detected. The median lethal concentration of the effluent to *C. gariepinus* was 4.14 mL/L (0.41%). Significant changes ($p \leq 0.05$) were observed in the levels of reduced glutathione and malondialdehyde in the liver of *C. gariepinus* exposed to sublethal concentrations of the treated effluent compared to control after 14 and 28 days of exposure. Histological alterations observed in *C. gariepinus* exposed to sublethal concentrations of the treated effluent were shortening/blunting of secondary lamellae in the gills and hepatic central veins infiltrated with red blood cells in the liver. The results show that long-term exposure to sublethal concentrations of the treated cosmetic effluents may cause deleterious effects to animals inhabiting recipient freshwater ecosystems. Further studies to explore other potential biological effects of treated cosmetic effluents are recommended in order to provide holistic and evidence-based information to relevant stakeholders and environmental regulatory agencies.

Keywords: Biochemical indices, *Clarias gariepinus*, Cosmetic effluent, Freshwater ecosystems, Histological alterations

INTRODUCTION

The global rise in human population and industrialization has led to increased anthropogenic impacts on the environment (Akoteyon and Soladoye, 2010). Despite the economic downturn in the past 20 years, one of the major industries that has been adopted and embraced is the cosmetics industry which has grown by 4.5% yearly due to increasing global demands (Zulaikha *et al.*, 2015). Cosmetics are products applied to the body for the purpose of cleansing, beautifying or improving and enhancing appearance (Singh, 2010). Due to the economic benefits, it is almost impossible for the manufacturer to produce a good quality

product at a low cost and in an environmentally friendly at the same time (Zulaikha *et al.*, 2015). Cosmetics include chemicals like skin softeners, surfactants, dyes, humectants, preservatives, antioxidants, emollients, emulsifiers, ultraviolet absorbers and acrylates (Chude and Ekpo, 2010). These chemical additives are sometimes hazardous and prohibited due to the health risks they pose. Most of these chemicals are added in the form of preservatives and fragrances, to increase the shelf-life of the products, to have a good odour and appearance (Adepoju-Bello *et al.*, 2012).

Some of these chemicals are toxic and prohibited from use as ingredients because they have the potentials to cause cancer, mutation, reproductive toxicity and endocrine disruption (Amasa *et al.*, 2012). Trace elements whose toxicity are well documented (Adepoju-Bello *et al.*, 2012; Popoola *et al.*, 2013; Ramakant *et al.*, 2014; Zulaikha *et al.*, 2015) are also incorporated into beauty products for many purposes. Biomarkers (biological responses elicited in living organisms as a results of exposure to or effects of toxicants) such as antioxidant enzymes activity, lipid peroxidation which indicates oxidative stress, histological alterations in tissues of animals have been used to evaluate the potential effects of effluents on model organisms in the receiving aquatic ecosystems (Adeogun *et al.*, 2012; Adeogun and Chukwuka, 2012; Sogbanmu and Otitoloju, 2014; Sogbanmu *et al.*, 2018).

The African sharptooth catfish (*Clarias gariepinus*) is valuable commercial and ecologically important fish species in Nigeria. It is a freshwater fish that forms the diet of most individuals due to their relative ease of culture and high protein content (Adeogun and Chukwuka, 2012). Ecologically, they are important components of the food web in freshwater ecosystems. The aim of this study was to investigate the acute toxicity as well as biochemical and histological effects of sublethal concentrations of treated cosmetic industry effluent on a model aquatic organism, *Clarias gariepinus* (the African Sharptooth Catfish).

MATERIALS AND METHODS

Collection of Treated Cosmetic Effluent and Physico-Chemical Analysis

Treated cosmetic industry effluent was obtained from a cosmetics producing company in Ogba, Lagos, Nigeria. The effluent was collected in a ten (10) L plastic keg, transported to the laboratory and refrigerated (at 4°C) until use. The effluent was analysed at the Chemistry Department, University of Lagos for the following physico-chemical parameters; pH, total dissolved solids (TDS), total suspended solids (TSS), electrical conductivity, turbidity, dissolved oxygen (DO),

biochemical oxygen demand (BOD), chemical oxygen demand (COD), total hardness, oil and grease, potassium, sodium, calcium, chlorides, sulphates, magnesium, total chromium, copper, zinc, cadmium, nickel, cobalt, iron, lead, according to (Mombeshora *et al.*, 2001; Ajuzie and Osaghae, 2011).

Evaluation of Phthalates, Triclosan and Parabens in the Treated Cosmetic Effluent

Determination of Phthalates in Treated Cosmetic Effluent

Phthalates in the cosmetic effluent was evaluated using a GC-MS (Gas Chromatography - Mass Spectrometry) with ultraviolet (UV) detection (Celeiro *et al.*, 2019). The GC (CP-3800; Varian, Palo Alto, CA) was equipped with a 1079 universal capillary injector and a fused capillary SPB-5 column coated with a film of 5% phenyl–95% dimethylpolysiloxane (30 m × 0.25 mm, 0.25 µm film thickness). The injector temperature was 260 °C and injections were in splitless mode. The oven temperature was programmed from 60 °C (held 1 min) to 280 °C at 20 °C/min (held 5 min). Helium (purity of 99.99%) was used as the carrier gas at a flow rate of 0.8 mL/min. The quadrupole MS (Saturn-2000, Varian) was equipped with a computer data analysis system and a mass spectral library (National Institute of Standards and Technology (NIST)). The MS parameters were 1 µs/scan, 10 µA filament current and 260 °C transfer line temperature. The MS was operated in full scan electron ionization–total ion chromatogram and selective ion monitoring modes from m/z 50 to 400.

Determination of Triclosan in Treated Cosmetic Effluent

Triclosan in the cosmetic effluent was determined using a Varian 3800 CX GC directly connected to a Saturn 2000 ion-trap mass spectrometer (Walnut Creek, CA, USA) (Cheng *et al.*, 2011). A ChromatoProbe (Varian) and a temperature-programmed injector (liner: 3.4mm i.d) were used to introduce large volume of samples for injection-port derivation (Adriano, 2001). 10 µL of effluent was mixed with MTBSTFA (1 µL) introduced into a micro-vial, the vial was placed into the ChromatoProbe via holder, and then positioned into the GC injection-port. The temperature was held at 100 °C for 2 mins for TBDMS derivation and solvent vaporization, and then the temperature was rapidly increased to 270 °C to allow introduction of TBDMSTCS into the analytical column. A DB5-MS capillary column (30 m × 0.25 µm film thickness; Agilent, Santa Clara, CA, USA) was used. The oven temperature was programmed from 120 °C for 2 mins, a temperature ramps of 25 °C/min u to 300°C, and maintaining this for 3 mins. The temperature of the transfer line was set at 280 °C. Full-scan electron-impact ionization spectra was at an emission current of 20 µL at an electron energy of 70

eV. Solvent delay time was 9 mins with an ion trap temperature of 200 °C at a mass range of 150-450 m/z (Onwusah *et al.*, 2011).

Determination of Parabens in Treated Cosmetic Effluent

0.5 mL of methyl paraben standard solution 100 µg/mL and 0.5 mL of 1 M sodium hydroxide solutions were added to 0.5 mL of orthoaminobenzoic acid and 0.5 mL of 1% sodium nitrite and 0.5 mL of 1 M HCl were mixed together and made up to mark with 5 mL of effluent sample into a 10 mL volumetric flask and stirred (Dhahir and Hussein, 2013). The resulting solution was then cooled in a ice bath for 2 mins until an orange colour developed and the absorbance was measured at a wavelength at 442 nm, against a blank solution prepared by the same method but without methyl paraben (Barlow *et al.*, 2004).

Ethics Statement

This study followed the principles in the Declaration of Helsinki on the humane treatment of animals used in research (<http://www.wma.net/en/30publications/10policies/a18/>).

Collection and Acclimatization of Test Animals

Fingerlings (age: 4 weeks old; weight range: 5 – 9 g; length range: 4.8 – 6.0 cm) and juveniles (weight range: 17 – 25 g and length range: 12.2 – 14.3 cm) of *Clarias gariepinus* (pisces, cypriniformes, clariidae) were procured from a farm at Ikorodu, Lagos and transported to the Zoology laboratory in oxygenated polyethylene bags. Fish were stocked in 50 L plastic tanks that was three-quarter filled with dechlorinated tap water. The fish were allowed to acclimatize to laboratory conditions (temperature: 28 ± 2°C; relative humidity: 78 ± 4%) for a period of 7 days prior to the experiments. The fishes were fed with Coppens fish feed and mortality was assessed daily. A fish was considered dead if it failed to respond to mechanical stimuli (Arun *et al.*, 2011). Dead fish were removed from stock tanks to prevent contamination.

Acute Toxicity Studies with Treated Cosmetic Effluent

A range finding test was conducted to establish the dose range of the effluent for the experiment before the definitive toxicity tests. A static bioassay protocol (in which the toxicant was not renewed over the period of 96 hours) was employed for the acute toxicity test of the effluents against fingerlings of *C. gariepinus* according to OECD (1992) and Sogbanmu and Otitoloju (2014). Acute toxicity tests were carried out in duplicates. Fish were exposed to the following test

concentrations – 2 mL/L, 4 mL/L, 6 mL/L, 8 mL/L, 10 mL/L of the treated cosmetic effluent and control (water alone).

Experimental Design for Sublethal Toxicity Studies

Juveniles of *C. gariepinus* of similar sizes were randomly selected from the stock tank and exposed to sublethal concentrations (1/10th 96 hLC₅₀ (0.4 mL/L) and 1/100th 96 hLC₅₀ (0.04 mL/L)) of the treated cosmetic effluent derived from the acute toxicity test results and control (water only). A semi-static bioassay test protocol was adopted in which the test media was changed once every 72 h to fresh media of the same concentration and untreated control (Arun *et al.*, 2011). The experiment was carried out in triplicates for each concentration, hence 9 plastic tanks of 5 L volume each.

Biochemical Studies with *Clarias gariepinus*

On the 14th and 28th days post-exposure, fishes from the test media including control were randomly selected; euthanized, liver samples were excised and homogenized according to Onwusah *et al.* (2015). The tissue homogenates were subjected to determination of Malondialdehyde (MDA) an index of lipid peroxidation, reduced glutathione (GSH) and antioxidant enzymes (superoxide dismutase (SOD) and catalase (CAT) (King *et al.*, 2012; Sogbanmu and Otitolaju, 2014).

Histology Studies with *Clarias gariepinus*

On the 14th and 28th days post-exposure, fish from the experimental groups as well as control were randomly selected for histological studies. The selected fishes were euthanized, the gills and livers were excised and fixed in 10 % formosaline (Amasa *et al.*, 2012). They were processed routinely, embedded in paraffin wax, sectioned at 4-5 µm thickness, stained with haematoxylin and eosin (H and E) (Sogbanmu *et al.*, 2018). Permanent slides were prepared with tissue sections, examined with the aid of a light microscope (Nikon TE 3000) and photomicrographs taken with a digital camera (Nikon 9000).

Statistical Analysis

The physico-chemical parameters of the samples were analysed using SPSS version 20.0. The 96 hLC₅₀ value of acute toxicity was determined by probit analysis (using SPSS version 20.0.) Data obtained for sub-lethal toxicity tests were subjected to One-way analysis of variance (ANOVA) test to test for the significance between the treatment means. Significant means (at p<0.05) were separated with Least Significant Difference (LSD) using SPSS version 20.

RESULTS

Physico-Chemical Analysis of Treated Cosmetic Effluent

Physico-chemical analysis of the effluent showed that conductivity, total dissolved solids, zinc and lead were higher than Federal Ministry of Environment set limits. Oil and grease, total suspended solids, ammonia, nickel, cadmium and phenol were not detected (Table 1). Phthalate, triclosan and parabens were below detectable limits (Table 1).

Table 1: Physico-Chemical Parameters of Treated Cosmetic Effluent

Parameters	Effluent	FMEnv Limit
Odour	Objectionable	NS
Appearance	Clear	NS
Colour (Lovibond Units)	NS	7
pH	7.74	6-9
Electrical Conductivity EC ($\mu\text{S}/\text{cm}$)	6000	NS
Total Dissolved Solids TDS (mg/L)	310	2000
Total Suspended Solids TSS (mg/L)	ND	30
Salinity (ppt)	0.18	NS
Turbidity (NTU)	30	NS
Nitrate (mg/L)	8.11	20
Phosphate (mg/L)	0.02	5
Oil and grease (THC) (mg/L)	ND	10
BOD ₅ (mg/L)	4.0	50
Dissolved oxygen DO (mg/L)	5.1	NS
Chloride (mg/L)	100	600
Sulphate (mg/L)	8.0	500
Ammonia (mg/L)	ND	5
Phenols (mg/L)	ND	0.2
Heavy Metals		
Iron (mg/L)	0.08	20
Copper (mg/L)	0.04	<1
Nickel (mg/L)	ND	<1
Lead (mg/L)	3.7	<1
Cadmium (mg/L)	ND	<1
Zinc (mg/L)	3.52	<1
Cosmetic Products		
Phthalate (ppt)	BDL	NS
Triclosan (ppt)	BDL	NS
Paraben (ppt)	BDL	NS

KEY: NS- Not Specific, ND- Not Detected, BDL- Below Detected Level. Source: (FMEnv (formerly FEPA), 1991).

Acute Toxicity Studies of Treated Cosmetic Effluent on *Clarias gariepinus*

The acute toxicity studies showed that the mortality of the fishes was dose-dependent, in which more fishes died with increase in concentration of the test effluent. The relative acute toxicity of the treated Cosmetic effluent on *Clarias gariepinus* fingerlings revealed that the 96 hLC₅₀ (median lethal concentration to 50% of the exposed fishes) was 4.14 mL/L (0.4%) (Table 2).

Table 2: Acute toxicity of treated cosmetic effluent against *Clarias gariepinus*

Treatment (mL/L)	Test Animal Life Stage	96 hLC ₅₀ Value	Slope S.E.	±	Probit Line Equation	D.F.
Treated Cosmetic Effluent	Fingerlings	4.14 mL/L (0.4%)	0.25 0.39	±	Y = 0.25±0.50X	2

KEY: S.E: Standard Error, D.F: Degree of Freedom, 96 hLC₅₀: Lethal concentration of the test chemical that would cause 50% mortality in test organisms over a period of 96 hours.

Biochemical Analysis of *Clarias gariepinus* exposed to Treated Cosmetic Effluents

There was a significant ($p < 0.05$) increase in malonaldehyde (MDA) levels in the liver of *Clarias gariepinus* in the two effluent treatments (0.04 and 0.4 mL/L) after 14 days of exposure when compared to the control group. At day 28, there was a significant ($p < 0.05$) increase in MDA level in liver of *C. gariepinus* juveniles in the 0.4 mL/L group but no significant ($p > 0.05$) change was observed in the 0.04 mL/L group compared to the control group (Table 3).

There was a significant ($p < 0.05$) decrease in reduced glutathione (GSH) levels in the liver of *C. gariepinus* in the 0.4 mL/L group but no significant ($p > 0.05$) change was observed in the 0.04 mL/L group after 14 days of exposure to the effluent compared to control. However, there was a significant decrease ($p < 0.05$) in GSH level in liver of *C. gariepinus* in both treatments compared to the control group after 28 days of exposure to the treated cosmetic effluent (Table 3).

After 14 days of exposure to treated cosmetic effluent, there was a significant ($p < 0.05$) decrease in the catalase (CAT) level in the liver of *C. gariepinus* juveniles in the 0.4 mL/L group but no significant ($p > 0.05$) change was observed in the 0.04 mL/L group compared to the control group. No significant ($p > 0.05$) differences was observed in CAT level in the exposed groups after 28 days of exposure compared to control.

There was no significant ($p < 0.05$) difference in superoxide dismutase (SOD) level in the liver of *C. gariepinus* juveniles in both treatments (0.04 and 0.4 mL/L) after 14 and 28 days of exposure to the effluent compared to control.

Table 3: Biochemical effects in the liver of *Clarias gariepinus* exposed to sublethal concentrations of treated cosmetic effluent over a period of 28 days

Treatment (mL/L)	MDA		GSH		CAT		SOD	
	Day 14	Day 28	Day 14	Day 28	Day 14	Day 28	Day 14	Day 28
Control (0.0)	1.91±0.1 1	2.02±0.0 8	27.47±1.6 7	30.30±1.0 0	17.19±1.6 1	22.64±1.13	4.24±0.1 3	4.77±0.0 6
0.04	2.32±0.0 7*	2.72±0.1 9	23.53±1.9 0	23.38±1.4 5*	20.55±0.3 8	23.90±6.89	4.46±0.7 5	4.69±0.2 7
0.4	3.10±0.6 6*	5.20±0.9 0*	10.94±5.9 0*	21.09±0.6 6*	10.20±1.7 0*	21.56±5.87	4.62±0.2 3	4.75±0.4 8

KEY: Each value represents mean ± S.D.; n = 2. GSH – reduced glutathione, MDA – malondialdehyde, CAT – catalase, SOD – superoxide dismutase, *: Significant difference compared to control using one-way analysis of variance (ANOVA) test at $p \leq 0.05$

Histological Alterations in *Clarias gariepinus* exposed to Treated Cosmetic Effluent

The histological alterations observed in the gills of *C. gariepinus* were shortening/blunting of secondary lamellae (SL) but preservation of primary lamellae (PL) after 14 days of exposure to 0.4 mL/L (Figure 1B) and 0.04 mL/L (Figure 1C) of cosmetic effluent compared to control (Figure 1A). Destruction of both primary and secondary lamellae of the gills were also observed after 28 days of exposure to 0.4 mL/L (Figure 1E) and 0.04 mL/L (Figure 1F) of cosmetic effluent compared to control (Figure 1D).

The histological evaluations in the livers of *C. gariepinus* showed radially arranged plates of normal hepatocytes (NH), no cytoplasmic fat vacuoles or abnormalities were observed after 14 days of exposure to the treatments (Figures 2B and 2C) compared to control (Figure 2A). However after 28 days of exposure to 0.4mL/L cosmetic effluent, hepatic central veins were observed to be packed with red cells (RC) (Figure 2E) compared to control (Figure 2D) while radially arranged plates of normal hepatocytes (NH), no cytoplasmic fat vacuoles or abnormalities were observed after 28 days of exposure to 0.04 mL/L (Figure 2F) of cosmetic effluent compared to control (Figure 2D).

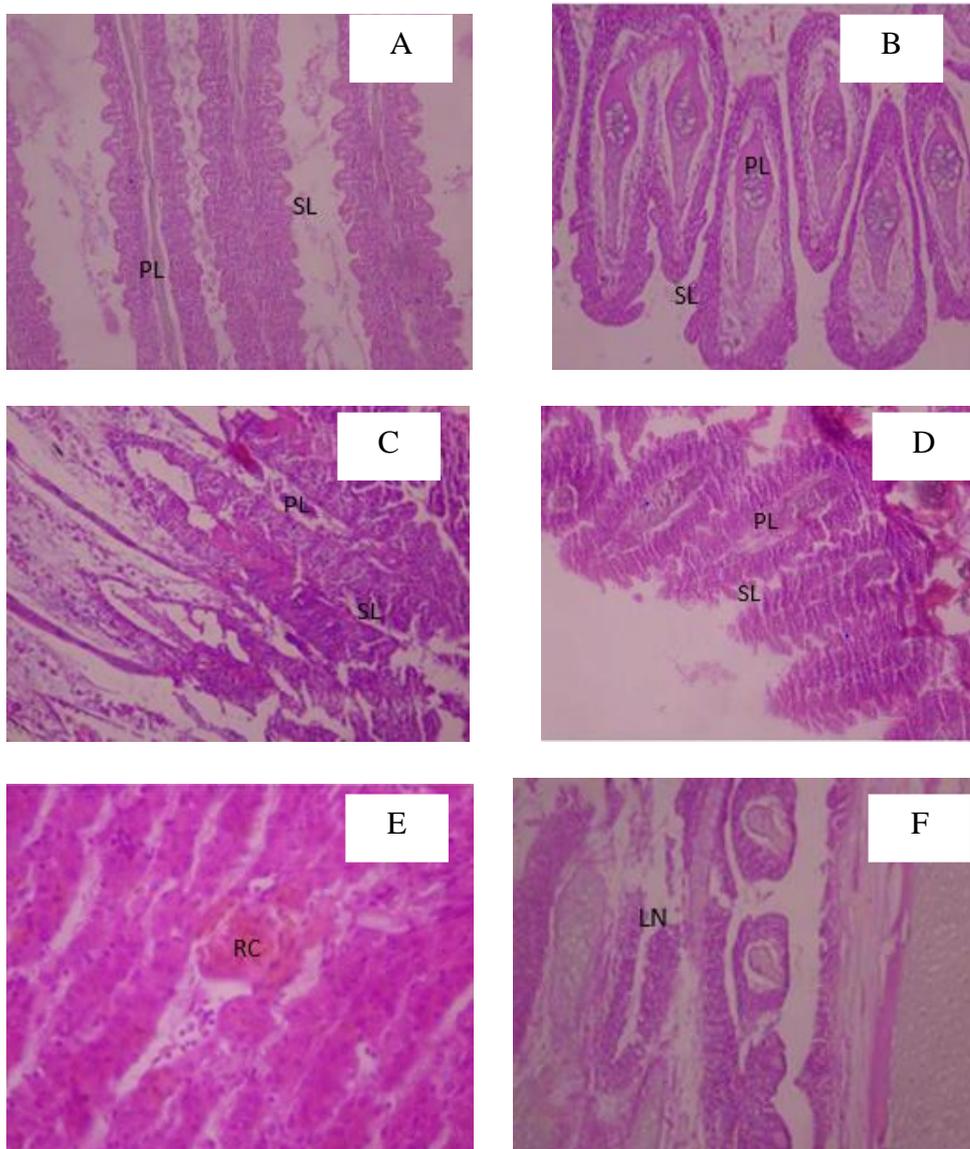


Figure 1: Photomicrographs of histological sections of gill samples of *Clarias gariepinus* juveniles in the control (A, D) and treated groups (0.4 mL/L – B, E; 0.04 mL/L – C, F) after 14 days (A, B, C) and 28 days (D, E, F) (magnification – 100x)

KEY: SL – Secondary Lamellae, PL – Primary Lamellae, LN – Lamellar Necrosis

Notes: Figure 1A – No alterations observed; Figure 1B – Mild lamella necrosis; Figure 1C - Mild lamella necrosis; Figure 1D - No alterations observed; Figure 1E - Severe lamella necrosis Figure 1F - Severe lamella necrosis

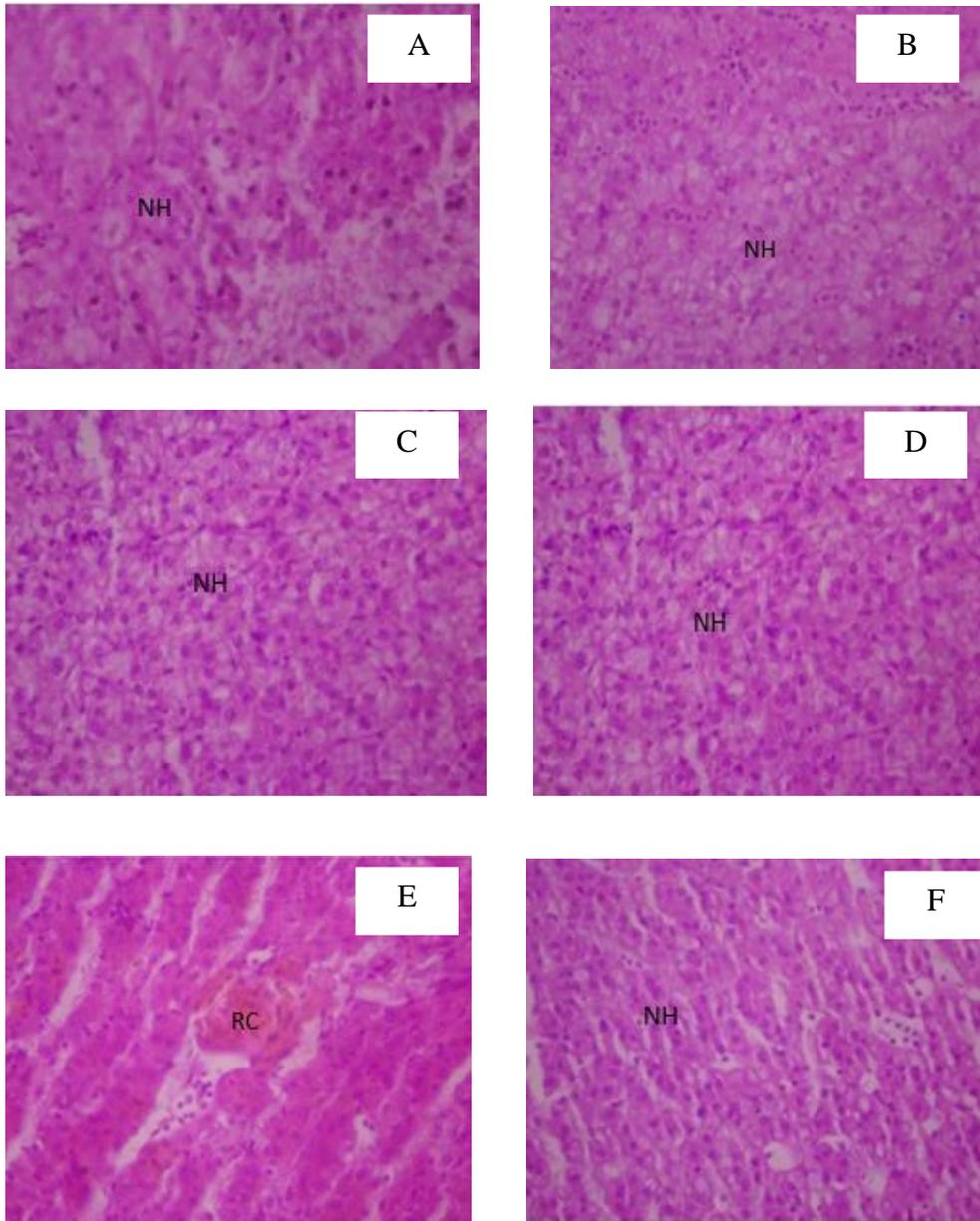


Figure 2: Photomicrographs of histological sections of liver samples of *Clarias gariepinus* juveniles in the control (A, D) and treated groups (0.4 mL/L – B, E; 0.04 mL/L – C, F) after 14 days (A, B, C) and 28 days (D, E, F) (magnification – 100x)

KEY: NH– Normal hepatocytes, RC – Red cells

Notes: Figure 2A – No alterations observed; Figure 2B – Normal hepatocytes; Figure 2C - Normal hepatocytes; Figure 2D - No alterations observed; Figure 2E – Central venous congestion; Figure 2F - Normal hepatocytes

DISCUSSION

Environmental issues in Nigeria top the agenda at both international and national levels due to environmental degradation, deterioration and under-development (Oyedeji *et al.*, 2011).

The physico-chemical parameters of the effluent in this study showed risk of contamination. Since trace elements are non-biodegradable, they are capable of bioaccumulating in aquatic organisms over time causing both acute and chronic effects (Bautista *et al.*, 2007). Zinc is one of the most abundant essential trace elements in biological systems and it is a co-factor of numerous enzymes, however it can be toxic at high concentrations. Zinc has been reported to be bioaccumulated in the liver of fish (Gairdina *et al.*, 2009) and has been shown to cause gill damage and increased mucus secretion (Skidmore, 1970). Lead is a non-essential trace element that has no known function in biological systems. It is highly toxic at low concentrations and has immunotoxicological effects in fish making them susceptible to pathogens (Paul *et al.*, 2014). Other parameters were within acceptable limits for the growth and survival of *C. gariepinus* (Miege *et al.*, 2009; Oyedeji *et al.*, 2011). Phthalates, Triclosan and Parabens were not detected as they were below detectable limits but this does not imply that they are absent (Akoteyon and Soladoye, 2010). The lack of detection could be as a result of the low detection range (part per thousand) of the equipment used for the analyses (Asonye *et al.*, 2007).

The acute toxicity result supports the observation of Akaniwor *et al.* (2007) that in all toxicants, a threshold is reached above which there is no drastic survival of animals. Below the threshold, an animal is in a tolerance zone, above the tolerance zone is the zone of resistance (Nadal *et al.*, 2004). The established 96 hLC₅₀ value of 4.14 mL/L showed that the treated cosmetic effluent was quite toxic to the exposed fish due to the relatively low value (0.4%) of the 96 hLC₅₀ value effluent (Miege *et al.*, 2009). Although *C. gariepinus* has been proven to be relatively resistant to various toxicants when compared to other species (Patkar, 2008; Onwusah *et al.*, 2015).

The increased malondialdehyde concentration in the exposed fish indicates increased lipid peroxidation due to oxidative stress which may have been elicited by trace elements present in the effluent (Oyedeji *et al.*, 2011; Zhang *et al.*, 2013). Lipid peroxidation has been suggested to be one of the primary mechanisms of cell injury by xenobiotics (Bautista *et al.*, 2007). Reduced glutathione (GSH) plays an important role in the metabolism of reactive oxygen species, protecting

cells from lipid peroxidation (Ramakant *et al.*, 2014). The GSH levels in the liver of the African catfish were significantly lower than those of the control group after 28 days. This result agrees with that of Saliu and Bawa-Allah (2012) who found that zinc and lead significantly affected reduced glutathione concentration in the liver of *Oreochromis niloticus* (Nile Tilapia). The apparent decrease in GSH may also demonstrate the inefficiency of the fish liver in neutralizing the impact of peroxides, resulting in increased lipid peroxidation, or may be related to the decreased availability of glutathione needed to reduce the reactive oxygen species (Ramakant *et al.*, 2014). Catalase (CAT) enzyme activity was significantly decreased initially (after 14 days) in the liver of *C. gariepinus* compared to the control group. The initial (after 14 days) decrease of the catalase enzyme activity in the liver of *C. gariepinus* may be due to direct trace element-mediated structural alteration of the enzyme (Miege *et al.*, 2009), depression of catalase synthesis (Patkar, 2008) and inhibition of the enzyme activity by superoxide radicals (Arun *et al.*, 2011). Superoxide dismutase (SOD) enzyme activity was not significantly changed in the liver of *C. gariepinus* exposed to treated cosmetic effluent compared to the control group. The absence of significant change in stimulation of the superoxide dismutase activity may be an adaptive response elicited by the increased production of superoxide radicals in the presence of trace element pollution (Chude and Ekpo, 2010).

The gills were the primary target tissue affected by the treated cosmetic effluent. Gills are generally considered good indicator of water quality (Chude, 2008), being models for studies of environmental impact (Chude and Ekpo, 2010; Onwusah *et al.*, 2015), since they are the primary route of entry of most contaminants. After 28 days of exposure, a total and partial fusion of secondary lamellae, mainly epithelial oedema was observed. The changes in appearance of the secondary lamellae result from the collapse of the pillar cell system and breakdown of vascular integrity with release of large quantities of blood that push the lamellar epithelium outward (Bautista *et al.*, 2007). Necrosis and cell desquamation of the gill epithelium observed may be due to the direct toxic effects of trace metals, while epithelial oedema and partial and total fusion of the lamellae represent a defence response (Ramakant *et al.*, 2014). The liver is the main organ for detoxification (Arun *et al.*, 2011) that suffers serious morphological alterations in fish exposed to toxic trace elements (Saliu and Bawa-Allah, 2012). The liver of the exposed fish had vacuolated cells showing evidence of fatty degeneration. Necrosis of some portions of the liver tissue that were observed resulted from the excessive work required by the fish to get rid of the toxicant from its body during the process of detoxification, (Shalom *et al.* 2013).

The inability of the fish to regenerate new liver cells might also have led to necrosis.

CONCLUSION AND RECOMMENDATIONS

In conclusion, the discharge of inefficiently treated industrial effluent into receiving water bodies invariably leads to accumulation of pollutants in the water and sediment. These pollutants end up depleting the water quality which increases the mortality rate of aquatic organisms. The alterations in the levels of biochemical and histological indices of *C. gariepinus* proved that the treated cosmetic effluent may still contain pollutants that are toxic to fish and could create hazard in water bodies. We recommend that regulatory agencies should identify and monitor indiscriminate discharge of effluents and endeavour to create adequate awareness. Management strategies should be developed, the populace should be informed about the adverse effects of effluents and the role they play in our immediate environment. Further studies to explore other biological potential effects of treated cosmetic effluents are recommended.

ACKNOWLEDGEMENT

The authors acknowledge Mr Adenekan, Department of Biochemistry, University of Lagos for his technical assistance with the biochemical aspects of this study.

REFERENCES

- Adeogun, A. O. and Chukwuka, A. V. (2012). Altered reproduction in *Clarias gariepinus* exposed to industrial effluents. *American Journal of Agricultural and Biological Sciences* **7**(1): 61-70.
- Adeogun, A. O., Ogdan, I. M., Ibor, O. R. and Chukwuka, A. V. (2012). Long-term exposure to industrial effluent induces oxidative stress and affects growth in *Clarias gariepinus*. *Research Journal of Environmental and Earth Sciences* **4**(7): 738-746.
- Adepoju-Bello, A. A., Oguntibeju, O. O., Adebisi, R. A., Okpala, N. and Coker, H. A. B. (2012). Evaluation of the concentration of toxic metals in cosmetic products in Nigeria. *African Journal of Biotechnology* **11**(97): 16360-16364.
- Ajuzie, C. U. and Osaghae, B. A. (2011). The bacterial and physico-chemical properties of hair salon wastewater and contaminated soil in Benin metropolis. *African Journal of Biotechnology* **10**(11): 2066-2069.
- Akaniwor, J. O., Anosike, E.O. and Egwim, O. (2007). Effect of Indomie industrial effluent discharge on microbial properties of new Calabar River. *Scientific Research and Essays* **2**(1): 1-5.

- Akoteyon, I. S. and Soladoye, O. (2010). Statistical study of physicochemical properties of wastewater in Odogunyan, Lagos, Nigeria. *European Journal of Chemistry* **8**(4): 2010-2015.
- Amasa, W., Santiago, D., Mekonen, S. and Ambelu, A., (2012) Are cosmetics used in developing countries safe? Use and dermal irritation of body care products in Jimma Town, Southwestern Ethiopia. *Journal of Toxicology* **30**: 1-8.
- Arun, K., Mishra, A. K., Mishra, A., Ghosh, A.K. and Chattopadhyay, P. (2011) Evaluation of skin irritation of herbal O/W sunscreen cream on rabbit model. *Journal of Pharmaceutics and Cosmetology* **1**: 44-49.
- Asonye, C. C., Okolie, N. P., Okenwa, E. E. and Iwuanyanwu, U. G. (2007). Some physicochemical characteristics and heavy metal profiles of Nigerian rivers, streams and waterways. *African Journal of Biotechnology* **6**(5):617-624.
- Bautista, P., Mohedano, A. F., Gilarranz, M. A., Casas, J. A. and Rodriguez, J. J. (2007). Application of Fenton oxidation to cosmetic wastewaters treatment. *Journal of Hazardous Materials* **143**:128-134.
- Celeiro, M., Lamas, J. P., Vila, M., Garcia-Jares, C., Homem, V., Ratola, N., Dagnac, T. and Llompert, M. (2019). Determination of multiclass personal care products in continental waters by solid-phase microextraction followed by gas chromatography-tandem mass spectrometry. *Journal of Chromatography A* **1607**: 440398.
- Cheng, C.-Y., Wang, Y.C., Chen, H.-C. and Ding, W.H. (2011). Simplified derivatization method for Triclosan determination in personal care products by gas chromatography-mass spectrometry. *Journal of the Chinese Chemical Society* **58**: 49-52.
- Chude, L. A. (2008). Toxicological Study of six locally generated pollutants on fingerlings of *Oreochromis niloticus* and *Clarias gariepinus* in the laboratory. *Ph.D. Thesis*, Michael Okpara University of Agriculture, Umudike, Abia State, Nigeria. 174p.
- Chude, L. and Ekpo, I. E. (2010). Laboratory comparison of hair dressing effluent on fingerlings of *Oreochromis niloticus* and *Clarias gariepinus*. *Nigerian Journal of Agriculture, Food and Environment* **6**(1&2): 104-109.
- Dhahir, S. A. and Hussein, H. J. (2013). Spectrophotometric determination of methyl parabens in pure and pharmaceutical oral solution. *Advances in Natural Science* **6**(4): 69-74.
- Federal Ministry of Environment (FMEnv) (1991). Interim effluent limitation guidelines in Nigeria for all categories or industries. *In: Guidelines and standards for Environmental Pollution Control in Nigeria*. Page 33.

- Giardina, A., Larson, S. F., Wisner, B., Wheeler, J. and Chao, M. (2009). Long-term and acute effects of zinc contamination of a stream on fish mortality and physiology. *Environmental Toxicology and Chemistry* **28**(2): 287-295.
- King, M. A., Sogbanmu, T. O., Osibona, A. O., Doherty, V. F. and Otitolaju, A. A. (2012). Toxicological evaluations and usefulness of lipid peroxidation as biomarker of exposure to crude oil and petroleum products tested against *Clarias gariepinus* and *Clibanarius africanus*. *Nature Environment and Pollution Technology* **11** (1): 1-6.
- Miege C., Choubert J. M., Ribeiro L., Eusebe M., Coquery M. (2009). Fate of pharmaceuticals and personal care products in wastewater treatment plants – conception of a database and first results. *Environmental Pollution* **157**: 1721-1726
- Nadal, M., Schuhmacher, M. and Domingo, D. L. (2004). Metal pollution of soils and vegetation in an area with petrochemical industries. *Science of the Total Environment* **321**(1-3): 59-69.
- Onwusah, B.O., Isoboye, M.A. and Omokaro, O. (2015). Mycoflora of different wastewater from some hair salons in Port Harcourt in Nigeria. *Current Studies in Comparative Education, Science and Technology* **2**(2): 303-311.
- Organization for Economic Cooperation and Development (OECD) (1992). Fish acute toxicity test. *OECD Guidelines for Testing Chemicals, No. 203*, adopted 17.07.1992, pp.1-9.
- Oyedeji, F. O., Hassan, G. O. and Adeleke, B. B. (2011). Hydroquinone and heavy metal levels in cosmetics marketed in Nigeria. *Trends in Applied Sciences Research* **6**: 622-639.
- Patkar, K. B. (2008). Herbal cosmetics in ancient India. *Indian Journal of Plastic Surgery* **41**: S134-S137.
- Paul, N., Chakraborty, S. and Sengupta, M. (2014). Lead toxicity on non-specific immune mechanisms of freshwater fish *Channa punctatus*. *Aquatic Toxicology* **152**: 105-112.
- Popoola, O. E., Bisi-Johnson, M. A., Abiodun, A. and Ibeh, O. S. (2013). Heavy metal content and antimicrobial activities of some naturally occurring facial cosmetics in Nigeria. *Ife Journal of Science* **15**(3): 637-644
- Ramakant, S., Poornima, S., Sapina, J., Mathur, H. B. and Agarwal, H. C. (2014). Heavy metal in cosmetics. *Centre for Science and Environment* **45**: 3–28.
- Saliu, J. K. and Bawa-Allah, K. A. (2012). Toxicological effects of Lead and Zinc on the antioxidant enzyme activities of post juvenile *Clarias gariepinus* fry. *Journal of Fisheries and aquatic Science* **3**: 280-290.
- Shalom, N. C., Dominic, E. A., Victor, C. O., Iweala, E. J., Afolabi, S. I., Uhuegbu, C. C., and Oranusi, S. U. (2013). Skin care product usage:

- implication on Health and wellbeing of Africans. *Journal of Applied Science* **13**(3): 430–436.
- Singh, S. K. (2010). Haematological parameters of tench, *Tinca tinca* after acute and chronic exposure to lethal and sublethal mercury treatments. *Bulletin of Environmental Contamination and Toxicology* **73**:911-918.
- Skidmore, J. F. (1970). Respiration and osmoregulation in rainbow trout with gills damaged by zinc sulphate. *Journal of Experimental Biology* **52**(2):481-494.
- Sogbanmu, T. O and Otitolaju, A. A. (2014). Joint action toxicity and biochemical effects of binary mixtures of Forcados Light Crude Oil and three dispersants against *Clarias gariepinus*. *International Journal of Environmental Research* **8**(2): 395-402.
- Sogbanmu, T.O., Osibona, A.O., Oguntunde, A.O. and Otitolaju, A.A. (2018). Biomarkers of toxicity in *Clarias gariepinus* exposed to sublethal concentrations of polycyclic aromatic hydrocarbons. *African Journal of Aquatic Sciences* **43**(3): 1-12.
- Zhang, X., Zolfaghari, M. and Singh, S. (2013). Fate and Transport of Fragrance Materials in Principal Environmental Sinks. *Chemosphere* **93**(6): 857-869.
- Zulaikha, S. R., Norkhadajah, S. I. and Pravenna, S. M. (2015). Hazardous ingredients in cosmetics and personal care products and health concern: A review. *Public Health Research* **5**(1): 7-15.