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Acute Toxicity of a Glass Manufacturing Effluent in Midwestern Nigeria on the African Catfish (*Clarias gariepinus*)

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ABSTRACT: Untreated and improperly treated industrial effluents endanger the aquatic fauna of the receiving water bodies, making the appropriate monitoring of the effluent discharges from industrial facilities of utmost importance. The potential ecological impact of the effluent from a glass manufacturing facility was investigated using the African catfish (*Clarias gariepinus*). Renewable static bioassay with continuous aeration was used to evaluate the acute toxicity of the different dilution percentiles of the wastewater for 96 h under laboratory conditions. The physicochemical and metal assays of the industrial effluent were carried out using standard methods. Significant concentration-dependent increase ($p < 0.05$) in mortality rates was established. Respiratory disturbance, erratic swimming, loss of equilibrium, lethargies and sudden death were observed in the exposed fish and these varied greatly with increase in concentration of the industrial effluent. The mortality rate of 46.25% was obtained with the 96-h LC_{50} of 13.80 mg/L using the Probit analysis statistical tool. Several physicochemical parameters (pH, dissolved oxygen, turbidity, electrical conductivity, total suspended solids, nitrates, and phosphates) were non-compliant with the standards set by the National Environmental Standards and Regulations Enforcement Agency (NESREA). The assayed metals were within the set limits, except for Mg, Cu, Fe, and Zn. The non-conformity of the industrial effluent discharge to established standards may pose threat to aquatic faunas present in the receiving water bodies.

Keywords: Industrial effluent; Glass manufacturing; Acute toxicity; African catfish; Midwestern Nigeria

Introduction

The global economy is driven by industrialisation which entails the harnessing of available resources for the comfort and better living experiences of humans. Industrialisation comes with its attendant challenges, and a huge load of waste (air, water, solid) generated by the industries is a major one (Menbere and Menbere, 2019; Patnaik, 2018). Most industrial processes utilise a high volume of water during stages of manufacturing, and these processes produce effluents of varying degrees of contamination. The effluents are discharged into surface water systems where they affect the ecosystem of the receiving water bodies if not treated or properly treated (Preisner *et al*., 2020; Akpor and Muchie, 2011). The various pollutants of different natures and types present in industrial effluents endanger the aquatic flora and fauna of the receiving water bodies. Hence, appropriate monitoring of the effluent discharges from industrial facilities is of utmost importance.
The glass manufacturing process uses high water volumes in the different production stages- to wash, cool, grind and separate the cullet (Gholipour *et al*., 2020). In modern times, numerous glassware and objects are

manufactured using raw materials such as alumina, silica, feldspar, nepheline, limestone, dolomite, sodium carbonate, potassium carbonate, potassium nitrate, sodium nitrate, barium sulphate, boron, arsenic trioxide, antimony trioxide, fluorite, cryolite, sodium fluosilicate, cerium oxide, erbium oxide, neodymium oxide, iron oxide, titanium dioxide, and many more (Moretti and Hreglich, 2013). Additionally, secondary raw materials such as glass cullet, chunks, and ingots are employed for glass making. These raw materials generate a complex mix of contaminants in the wastewater from the production processes. The wastewater from glass manufacturing processes contains high concentrations of silicon dioxide particles in the nanometre to millimetre size ranges, which combine with other glass splinters to increase the effluent turbidity; the effluent equally contains dissolved salts, heavy metals, and oil and greases employed in the cutting processes (Gholipour *et al*., 2020).
Conventionally, most effluent assessment regimes rely on the use of physical, chemical, and microbiological parameters. In Nigeria, this set of parameters is the only monitoring criteria established by the environmental regulatory body - the National Environmental Standards and Regulations Enforcement Agency, NESREA (NESREA, 2011). The numerous chemicals utilised in the manufacturing of glass may generate a complex mixture of pollutants in the effluent, and the overall effect of the effluent in aquatic ecosystems may not be fully captured by the use of physical, chemical, and microbiological parameters only. Toxicological assays are now being recommended as complementary tools to evaluate the effluent quality (Akharame *et al*., 2022; Pereao *et al*., 2021). The use of the African catfish (*Clarias gariepinus*) for acute toxicity assessment is adjudged to be extremely sensitive (Olorunfemi *et al*., 2019); hence, it can be utilised as an effective biomarker for contaminants in an aquatic ecosystem. More so, the fish inhabits streams, swamps, rivers, and lakes in Nigeria and is also largely consumed (Olaniran *et al*., 2019). Consequently, this study is aimed at utilising the African catfish (*C. gariepinus*) as a biomarker to assess the possible impact of the effluent obtained from a glass manufacturing facility in Delta State, Nigeria on the aquatic ecosystem (especially fishes) of the receiving water body.

Materials and methods

Sampling sites and collection of effluents: The effluent samples were obtained from a glass manufacturing facility situated at Ughelli (GPS coordinates 5° 30' 0.6732" N and 5° 59' 37.8024" E) in Delta State, Nigeria. The samples were collected from the discharge point of the effluent using 10 L plastic containers. Before the sampling, thorough washing of the containers and rinsing with distilled water was effected, with further rinsing (three times) using the effluents before filling up at the discharge point. The samples were kept in an ice box during transportation to the laboratory and stored at 4 °C in a refrigerator before usage for the analyses.
Water quality parameters measurements: The water quality parameters measurements were done in triplicates following standard procedures recommended by APHA (1998). A field pH meter (HANNA pH/Temperature Tester) was used to carry out the *in situ* pH measurements, while an Adwa AD8000 multi-parameter meter from Adwa instruments was utilised for the electrical conductivity (EC). Total hardness was evaluated using the EDTA titrimetric method, and the dissolved oxygen (DO) measurements were carried out using the Azide modification of the Winkler method. The total suspended solids (TSS) level was measured following the method established by APHA (1998). The ammonia (NH_4^+), nitrates (NO_3^+), phosphates (PO_4^{3-}), sulphates (SO_4^{2-}), and chlorides (Cl^-) levels were determined using a HACH DR 2000 spectrophotometer. The metal levels were determined using the Unican 929 AA spectrometer for calcium (Ca), magnesium (Mg), potassium (K), sodium (Na), cadmium (Cd), chromium (Cr), copper (Cu), iron (Fe), lead (Fe), manganese (Mn), nickel (Ni), silver (Ag), and zinc (Zn).
Acute toxicity assessment: The procedure recommended by UNEP (1989) was followed in carrying out the toxicity assessments. Hundred (100) juveniles of the African catfish (*C. gariepinus*) were sourced from a fish farm situated in Benin City, Nigeria. The juveniles were kept to acclimatise to the laboratory conditions in a glass tank (20×15×30 cm) filled with de-chlorinated water and at room temperature (26.0 - 28.0 °C). The acclimatisation was carried out for 2 weeks with the de-chlorinated water changed regularly every 2 days to prevent metabolic waste build-up. The fish were fed twice a day (morning and evening) with a commercial fish feed at 3% body weight. The feeding was stopped 24 h before the commencement of the 96 h exposure of the fish to effluent samples. The experimental set-up comprised eight (8) fishes in each tank with the different dilution percentile (effluent in de-chlorinated water: 0, 1, 10, 20, 30%) of the effluents. Behavioural responses and mortality were observed for the fishes at the time intervals of 12, 24, 48, 76, and 96 h, respectively. The immobility of a fish with no opercula and tail movements was used to determine their mortality.

Data analysis: The Probit analysis data interpretation was done using the MedCalc® Statistical Software version 20.113. The regression analysis provided the percentage mortality and the 96 h LC_{50} toxicity (Randhawa, 2009).

Results and Discussion

Physicochemical and elemental (metals) assessment of the effluent quality: The measured physicochemical data from the analysis of the effluent obtained from the glass facility is presented in Table 1. The pH value of 6.20±0.040 falls into the slightly acidic pH range, and below the 6.5-8.5 stipulated by the National Environmental Standards and Regulations Enforcement Agency, NESREA (NESREA, 2011). The EC and turbidity values were 830±2.410 µS/cm and 31.7±1.050 NTU representing the effluent's dissolved solids (salts) and suspended particles, respectively. There was no stipulated limit for both parameters in the NESREA (2011) guidelines; however, the EC level recorded was high, while the turbidity is above the 5 NTU limit for potable water (WHO, 1996). Typically, effluents from glass manufacturing facilities contain dissolved salts and high concentrations of silicon dioxide particles in the nanometer to millimeter size ranges which may account for the high EC and turbidity values (Gholipour *et al*., 2020). A TSS value of 0.94±0.010 mg/L is recorded for the effluent, which is higher than the recommended limit of 0.75 mg/L and corroborates with the turbidity value. Turbidity and TSS have been adjudged to have a strong linear relationship in their levels in effluents (Hannouche *et al*., 2011). The measured DO concentration of 3.1±0.845 mg/L is lower than the minimum permissible limit of 4.0 mg/L required for effluent discharge and the survival of aquatic life (fisheries) as stipulated by NESREA (2011). The low DO level correlates with the nitrates (64±0.950 mg/L) and phosphates (91.5±1.730 mg/L) levels recorded for the effluent; the values obtained for both parameters were higher than the permissible limits and are indicative of a high organic matter load present in the effluent (Olaniran *et al*., 2019). The levels of ammonia (0.03±0.005 mg/L), chloride (142±1.850 mg/L), and sulphates (71.5±0.100 mg/L) were lower than the set limits for effluent discharge and fisheries survival (NESREA, 2011).

Table 1: The physicochemical and metals levels of effluent from the glass facility

		NESREA Standards (2011)	
Parameter	**Glass facility effluent**	**Effluent discharge, irrigation and reuse**	**Fisheries and recreational quality**
pH	6.20±0.040	6.5 - 8.5	6.5 - 8.5
EC	830±2.410	NS	NS
DO	3.1±0.845	4.0	4.0 – 6.0
TSS	0.94±0.010	0.75	0.25
Turbidity	31.7±1.050	NS	NS
Ammonia	0.03±0.005	2.0	0.05
Nitrates	64±0.950	40	9.1
Phosphates	91.5±1.730	3.5	3.5
Sulphates	71.5±0.100	500	100
Chloride	142±1.850	350	300
Calcium	70.5±1.115	180	180
Magnesium	50.5±0.765	40	40
Potassium	30.5±0.555	50	50
Sodium	51.4±0.066	120	120
Cadmium	ND	0.01	0.005
Chromium	0.1±0.001	0.5	0.001
Copper	0.1±0.001	0.01	0.001
Iron	1.5±0.030	0.5	0.05
Lead	0.1±0.005	0.1	0.01
Manganese	0.3±0.015	NS	NS
Nickel	ND	0.1	0.01
Zinc	4.1±0.521	0.2	0.01

All values are expressed in mg/L except temperature (°C), turbidity (NTU), conductivity (µS/cm), and pH (no unit). DO = Dissolved Oxygen, TSS = Total Suspended Solids, NS = Not Stated, NESREA = National Environmental Standards and Regulations Enforcement Agency (2009).

Metals or heavy metals are characteristically present in glass manufacturing effluents (Gholipour *et al*., 2020). The recorded levels of alkali metals (sodium and potassium) and alkali earth metals (calcium and magnesium) were mostly below the recommended limits, except for the magnesium (50.5±0.765 mg/L) which was above the maximum permissible limit of 40.0 mg/L. For the heavy metals, the measured values for copper (0.1±0.001 mg/L), iron (1.5±0.030 mg/L) and zinc (4.1±0.521 mg/L) were above the set limits of 0.01 mg/L, 0.5 mg/L, and 0.2 mg/L, respectively (NESREA, 2011). The levels for cadmium, chromium, lead, and nickel were well below the recommended limits or below the detection limits of the AAS instrument used for the measurements. However, the chromium level of 0.1±0.001 mg/L is much higher than the 0.001 mg/L recommended for fisheries; and may adversely affect the aquatic organisms present in the ecosystem of the receiving water body.

The effluent from the glass manufacturing facility was non-compliant with the standard set by NESREA for most of the parameters assayed. The trend in the non-conformity of industrial effluent discharges have been reported by several investigators; and the general assertion of possible adverse environmental implications was noted by the reports of other researchers (Gbarakoro *et al*., 2020; Adegbite *et al.*, 2018; Akharame *et al*., 2017; Ologbosere *et al*., 2016).

Acute toxicity responses of the African catfish to the industrial effluent: The mortality data for the African catfish subjected to the different dilution percentile (effluent in water ratio: 1, 10, 20 and 30%) of the glass facility effluent is presented in Table 2. The fish exhibited behavioural responses which include restlessness, erratic movements, and gasping for breath.

Table 2: Mortality rate of African catfish (*C. gariepinus)* juveniles exposed to varied concentrations of effluents

Effluent type	**Dilution percentile of effluent in water (%)**	**Mortality (h)**					
		12	**24**	**48**	**72**	**96**	**Mortality ratio**
Duplicate 1	0 (control)	0	0	0	0	0	0/8
	1	0	0	1	1	1	3/8
	10	0	0	1	1	1	3/8
	20	0	0	2	1	2	5/8
	30	0	0	2	2	3	7/8
Duplicate 2	0 (control)	0	0	0	0	0	0/8
	1	0	0	2	1	1	4/8
	10	0	0	1	2	1	4/8
	20	0	0	3	1	1	5/8
	30	0	0	2	1	3	6/8

Number of fish in effluent (n) = 8

The behavioural responses became more intense with increase in the concentration of the effluent and eventually led to sluggishness and death of some of the fish. The observed responses were consistent with reports of several investigations for different industrial effluents using African catfish for acute toxicity studies (Ariyomo *et al*., 2021; Ibrahim and Imam, 2019; Otong *et al*., 2018; Adeogun *et al*., 2013; Dahunsi and Oranusi, 2012).

The Probit result (Figure 1) revealed that the mortality rate of the fish was 46.25%, while the 96-h LC_{50} was 13.80 mg/L (95% confidence interval of 0.57-1.97). Increases in the concentrations of the effluent increased the mortality of the fish with a linear relationship established. A key contributor to the observed mortality may be the low DO concentration in the effluent which was below the minimum permissible limit of 4.0 mg/L stipulated by NESREA (2011) for the sustenance of fisheries.

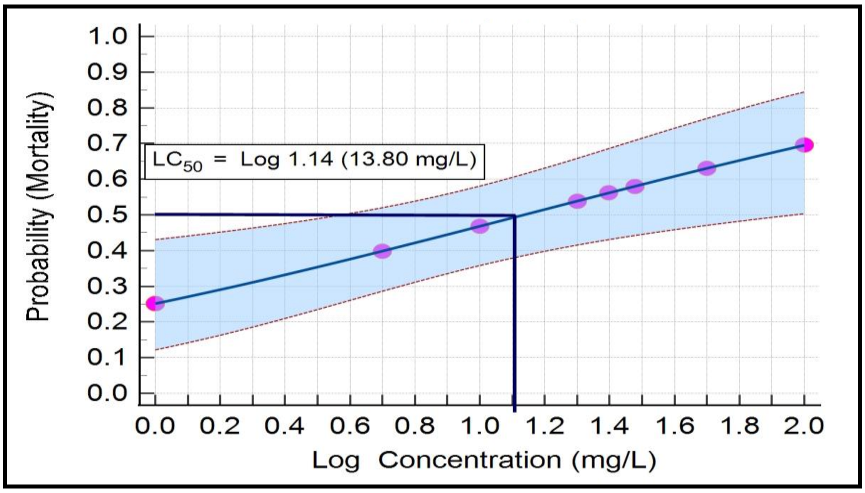

Figure 1: Probit mortality and exposure concentration of *C. gariepinus* juveniles exposed to effluent from the glass industry for 96 h

Essentially, fishes are gravely affected by low levels of DO in aquatic systems leading to difficulty in breathing, tensed behavioural activities, and suffocation (Knight *et al*., 2013). Additionally, most of the physicochemical parameters were above the recommended thresholds and could contribute individually or in combination with others to exert the observed mortalities.

Conclusion

African catfish juveniles (*C. gariepinus)* were exposed to effluent from a glass manufacturing facility in Delta State, Nigeria to assess the potential ecological impact of the effluent discharges to the receiving water body. The mortality rate of 46.25 % was recorded with the 96-h LC_{50} of 13.80 mg/L. A concentration-dependent increase in mortality rates was established. A key parameter such as DO was below the NESREA's stipulated threshold for the sustenance of fisheries, while the other parameters such as pH, turbidity, EC, TSS, nitrates, and phosphates were mostly non-compliant. The assayed metals were within the set limit, except for Mg, Cu, Fe, and Zn. The non-conformity of industrial effluent discharges to established standards may pose threat to aquatic faunas present in the receiving water bodies. Hence, NESREA needs to expedite necessary actions in its monitoring and enforcement functions to ensure industrial effluent discharges meet set standards.

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