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Full Length Research Article

Evaluation of Physiochemical Parameters and Some Heavy Metals from Tannery Effluents of Sharada and Challawa Industrial Areas of Kano State, Nigeria

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ABSTRACT
The evaluation of physicochemical parameters and some heavy metals was carried out from six different tannery companies at the industrial areas of Challawa and Sharada, using standard procedures. Temperature, pH, conductivity and total dissolved solids(TDS) were determined using portable digital hand logging meter. The sulphate, phosphate, nitrate, arsenic contents were determined using UV Spectrophotometer, while chloride, dissolved oxygen (DO) and biochemical oxygen demand (BOD) contents were determined using titrimetric method, and the heavy metals by atomic absorption spectrometry. The mean temperature of the water ranged from 36.00 – 39.67^{0}C, pH: 2.63 – 9.33, conductivity: 336.33 – 12351.67µS/cm, TDS: 166.73 – 6017.67mg/L, sulphate: 114.00 – 466.67mg/L, phosphate: 0.29 – 12.80mg/L, chloride: 58.66 – 1577.87mg/L, nitrate: 6.03 - 33.43mg/L, DO: 2.60 – 21.27mg/L and BOD: 0.77 – 12.33mg//L. Water samples from the tannery effluents contained higher levels for both physicochemical parameters and heavy metals in untreated than treated. Cadmium and nickel were within the WHO limit for both treated and untreated while arsenic was within the limit for only treated effluent. Chromium and lead were above the WHO limit for both treated and untreated effluent. The parameters studied showed variations within the tannery companies and this may affect the Challawariver where this effluent is discharged which can have long term health effect on the aquatic animals and humans that depend on the river water for their daily activities.
Keywords: Biochemical Oxygen Demand, Dissolve Oxygen, Heavy Metals and Effluents

INTRODUCTION
Kano Sate is the second largest industrial center in Nigeria and the largest in Northern Nigeria. Due to the large number of privately owned medium and small scale industries in the state producing various items including textile materials, tanned leather, foot wares, cosmetics, plastics, enamel wares, pharmaceuticals, ceramics, steel rods, beverages, animal feeds, furniture, vegetable oil and agricultural implements, the state government established an industrial estates such as Sharada, Challawa and others to meet up with the rapid growing industrialization (Ibrahim, 2003).

Industrial liquid effluents are one of the principal sources of heavy metals responsible for environmental pollution (Solomon*et al.,* 2015). The current scenario of sustainable environment is highly risked since most waterborne waste discharges from both domestic and industrial sources are channeled into natural water bodies (Karthick*et al*., 2014).

In recent years, large scale usage of chemicals in various human activities has grown considerably and pollution has assumed an escalating dimension due to the continual expansion of urbanization, industrial development and agricultural activities (Emmanuel and Adepeju, 2015). Water pollution by industrial effluent has been one of the vital issues of environmental concern today (Uma*et al.,* 2016).
The wastewater from the treatment of hides and skin in industrial effluents contained high levels of metals, toxic chemicals and other undesirable substance. As such, among all the industrial wastes, tannery effluents are ranked among the highest pollutants (Umar *et al.,* 2017).Several chemicals like Cr $(SO_4)_3$, NaCl, Ca $(OH)_2$,

H_2SO_4etc, are extensively used during leather manufacturing processes(Lakshmi and Malliga, 2014), hence, the effluent contain high levels of impurities.Tanneries generate wastewater in the range of 30-35 L/kg skin/hide processed with variable pH and high concentrations of suspended solids, BOD and COD (Ilou*et al.,* 2014).

One of the major factors that affect the beneficial use of surface water such as rivers, streams and ponds for industrial, domestic and agricultural purposes are contamination and pollution due to anthropogenic or human activities (Mustapha*et al.,* 2015).Tannery effluents have been considered the most polluted industrial wastes as in most cases it contains high levels of metals which are very toxic to plants, animals, (Abdulmuminі*et al.,*2015; Nivedita and Raviraj, 2016). Thus, efforts have been made to appraise the quality status of waste water effluent of industrial processes(Bichi and Danazumi, 2010). Such efforts would prove the acceptability of the treatment processes used as well as provide guidance on the use of the treated waste effluent (Hayelon and Adhena, 2014;Varsha*et al.,* 2017).

The aim of the research was to determine the level of some physicochemical parameters and heavy metals in the tannery effluent of Sharada and Challawa industrial areas of Kano State, Nigeria

MATERIAL AND METHODS

Study area

The study areas are Sharada and Challawa Industrial Area (Figure 1) of Kano State, Northern Nigeria.They arelocated on latitude $11^0 58^/$ – $11^0 50^/$N and longitude range of $8^0 31^/$- $8^0 40^/$E at an average elevation of about 430m above the mean sea level (Egwuonwu *et al*., 2011). The study areas,Sharada and Challawa industrial estates are located in Kumbotso and Municipal local Government areas respectively. They are bordered in the South and West by Madobi local Government, in the southwest by RimiGado, in the South by Dala and Gwale and in the East by Dawakin Kudu local government areas.

A gentle slope tending towards the south-west direction characterizes the study site. All the industries located in the area discharge their effluent into Challawa River located at about 2.0 km from Challawa industrial estate and about 20.0km from Sharada industrial estate downstream from the cluster of industries. In Sharada phase I industrial estate, Kano Municipal, liquid effluent was collected from Unique tannery (UQT) and Laquat tannery (LQT) while in Challawaindustrial estate, Kumbotso local Government, samples were collected from GB tannery treated (GBTa), GB tannery untreated (GBTb), Fata tannery treated (FTa), Fata tannery untreated (FTb), Mamuda tannery treated (MDTa), Mamuda tannery untreated (MDTb) and BB tannery (BBT)

Sampling

Liquid effluent samples (treated and untreated) were collected from six tannery companies of Sharada and Challawa industrial estate. The untreated effluent was collected from the effluent storage chamber and the treated effluent was collected directly from the treatment plant. Both treated and untreated effluents were collected in 2L and 4L plastic containers. One cm^3 concentrated HNO_3 was added to sample in 4L container to lower the pH to 2.0 and to maintain the stability of oxidation state of the metals in solution, and it was used for heavy metal analysis while the sample in the 2L container for physicochemical analysis was refrigerated at 4 ºC (APHA, 1992).

Physicochemical Analysis of Tannery Effluent

The pH and temperature were measured at the site of sampling using mercury glass thermometer and pH meter (Jenway 370). Conductivity and total dissolved solid were measured with digital conductivity meter (DR/890) after calibration with 0.01M KCl.

The NO_3^-, SO_4^{2-} and PO_4^{3-}were determined using portable data logging meter (DR/20-10) which was installed with program for the three anions. The program numbers 355, 680,490 were entered to display 500nm, 450nm and 430nm for NO_3^-, SO_4^{2-} and PO_4^{3-}respectively. 10cm^{3}of the water sample was taken into the meter cell, the content of one Nitra Ver 5 nitrate reagent was added and shaken to dissolve. The cell was was allowed to stand in the dark for five minutes and concentration in mg/L nitrate was read on the meter. The same process was repeated for sulphate and phosphate using Sulfa Ver 4 and phosphate reagents respectively (APHA, 1992). Chloride, DO and BOD were determined by titrimetric methods (Ademorati, 1996).

Digestion of Sample

For sample digestion, 1000cm^3 of the pretreated effluent water sample was measured and 750cm^3 was transferred into a 1000cm^3 pyrex glass beaker and evaporated on a hot plate. When the volume had reduced, the remaining 250cm^3 was added and heating continued until the volume was about 50 cm^3. The solution was allowed to cool and 20cm^3 of 0.1M nitric acid was added to the beaker and boiled on a hot plate at 85ºC until a clear solution was obtained. The sample was allowed to cool and transferred into a 100cm^3 standard flask. The resulting solution was then made up to 100 cm^3 mark with deionized water. This was filtered into a 100cm^3 volumetric flask using filter paper. The blank was prepared in a similar way by digesting 1000cm^{3}of deionized water in place of the sample (Akan*et al.,* 2007b).

The digested water sample was aspirated into the atomic absorption spectrophotometer (model: Varian AA240FS) and absorbance measured at the individual metal wavelength using the appropriate hollow cathode lamps of Pb, Cr, Ni and Cd. Calibration curves for the different metals were generated from metal standard solutions, and from the result, the concentrations of metals in the samples were calculated.

The levels of arsenic in the water samples were determined as described by Basset*et al*. (1983).

Statistical Analysis

All analyses were performed in triplicates and the results expressed as mean±SD. The excel data analysis was used. The differences in EC, TDS, PO_4^{3-}, SO_4^{2-}, Cl^-. NO_3^-, DO, BOD, As, Cd, Cr, Ni and Pb concentrations among the different samplingpoints were tested by analysis of variance method, (ANOVA), Tukey test and t-Test was used to determine pair wise differences betweenpoints. A value of $p<0.05$ was considered statistically significant.

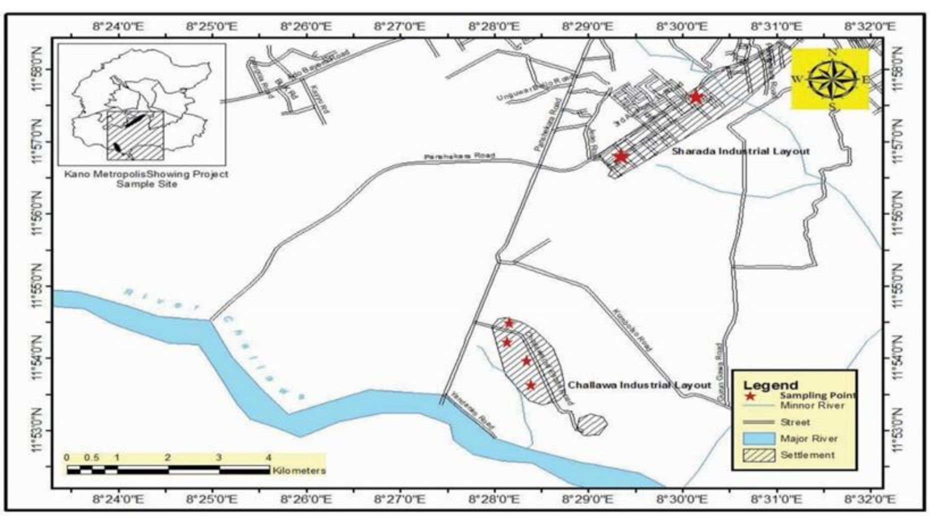

Figure 1: Map of Sharada and Challawa Industrial areas showing the sampling points

RESULTS AND DISCUSSIONS

Temperature of the tannery effluents was found to be within the WHO and USEPAlimit (<40^{0}C) for all the tannery effluents discharged from the companies into the Challawa river body. The values ranged from 36.00±0.00 – 37.67±0.58^{0}C for treated and 36.33±0.58 – 39.00±0.00^{0}C for untreated (Table 1). This may be from boiled liquor used for leather processing and the values are in agreement with the work of Akan *et al.* (2007a).

The pH of the tannery effluents discharged into the Chalawa river body from the tanneries under study was low for untreated effluents (2.63±0.15 – 5.60±0.20) and within permissible limit(6.0 – 9.0), for treated effluents i.e. 8.53±0.06 – 9.33±0.64, (Table 1), which indicates that treatment using biological method was efficient (Hayelon and Adhena, 2014; Mohamed *et al.,* 2016). The low pH value in untreated effluents may be due to use of acids in the tannery processing. Arasappan and Kalyanaraman, (2015) reported pH of 7.08 and 7.07 for treated tannery effluent. The discharge of such effluent with acidic pH into ponds, rivers and streams for irrigation may be dangerous to aquatic biota such as zooplankton and fishes (Hemamalini and Sneha, 2014).

Untreated effluent showed higher levels of conductivity than treated (Figure 2).Both treated and untreated effluent have conductivity values above WHO limit of 1200µS/cm except FTa and UQT,an indication that there were discharge of high levels ofcations and anions in the effluents, FTa and UQT may have efficient effluent treatment. There was no significant difference in conductivity values among sites GBTa, GBTb and MDTb. Similarly, conductivity levels at sites LQT wassignificantly higher than those obtained from the rest of the sampling sites and this may be attributed to poor treatment facility, while the value obtained at sites BBT, MDTa and FTBa are not significantly different from one another. The high conductivity of the effluents can alter the chelating properties of water bodies and createan in-balance of free metals availability for flora and fauna (Akan *et al.,* 2007a). This is in agreement with the study carried out by (Arasappan and Kalyanaraman, 2015; Noorjahan, 2014; Mohammed *et al.,* 2017).

The total dissolved solids (TDS) values in the effluentsare shownin Figure 2. The TDS values for treated effluent were lower than untreated effluent. The value of untreated effluent was higher than the WHO/USEPA limit of 600mg/L. Sites GBTa, GBTb, MDTa, MDTb and BBT are not significantly different from one another but are slightly lower than the levels obtained at site LQT. This may be attributed to efficient effluent treatment at sites GBTa, GBTb, MDTa, MDTb and BBT.Total dissolved solids are mainly due to carbonates, bicarbonates, chlorides, sulphates, phosphates, nitrates, nitrogen, calcium, sodium, potassium, iron suspended solids from skin and dissolved processing salts(Kannan*et al.,* 2009). This agreed with (Rajeswari 2015; Mohammed *et al.,* 2017).The t-test showed strong positive correlation between conductivity and TDS (r= 0.9995), the higher the conductivity the higher the TDS, at 5.0% probability and 8.0% degree of freedom.

Table 1: Mean Temp. andpH,of the Tannery Effluents fromSharada and Challawa Industrial Areas.

Sampling Sites	Temp. ^{0}C	pH
GBTa	36.7±0.58	8.53±0.12
GBTb	36.7±0.58	3.07±0.06
Fta	36.0±0.34	8.87±0.23
FTb	36.3±0.23	3.23±0.08
MDTa	37.7±0.66	9.33±0.60
MDTb	39.7±0.85	2.63±0.15
UQT	38.0±0.66	5.60±0.20
BBT	39.0±0.76	4.80±0.10
LQT	38.0±0.59	4.77±0.21
USEPA2004/ WHO, 2008	< 40.0	6.50-9.50

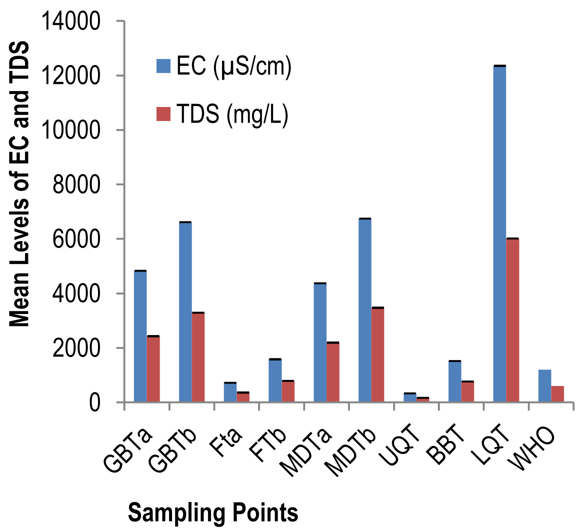

Figure 2: Mean Levels of EC and TDS in the Tannery effluents from Sharada and Challawa Industrial Areas

The Sulphate levels of the tannery effluents were higher in untreated than the treated (Figure 3). For untreated the value was 448.45±2.55mg/L and for treated 246.78±2.28mg/L. The values were below the WHO maximum permissible limit (500mg/L) except BBT and MDTb for discharge of tannery effluent into rivers. The sulphate levels at sites GBTa, GBTb, FTb, BBT, and LQT showed no significant difference among the sites at $p<0.05$ while sites FTa, UQT, GBTa and MDTa were also not significantly different from one another, however, site UQT has the lowest sulphate concentration. The concentration of sulphate at site MDTb was significantly higher than those obtained from other sites except sites BBT, LQT and FTa.The high Sulphate level may be attributed to the excessive use of sulphuric acid and tanning powder containing high levels of Sulphate (Bosnic*et al.,* 2000). This agreed with the studies conducted by (Akan *et al.,* 2007a)

The phosphate values of the tannery effluents were below the WHO permissible limit of 5mg/L for discharge of effluent into rivers except GBTa, GBTb and LQT (Figure 3). The phosphate levels in all the sampling sites showed no significant difference at $p<0.05$. The observed phosphate levels were in agreement with (Akan *et al.,*2007a).Chloride level in the tannery effluent was higher in untreated effluent than the treated effluent. The value for untreated was higher than WHO maximum permissible limit (600mg/L) for discharge of effluent into rivers(Figure 3). Chloride levels at sites MDTb and LQT were not significantly different from each other but were significantly greater than those obtained from other sites.The high chloride content may be due to the use of large quantities of common salt (NaCl) in hide and skin preservation and pickling process. Sites FTa has the lowest chloride levels even though it was not significantly different from those obtained at sites UQT and BBT. The chloride values obtained agreed with the work of (Kawser*et al.,* 2011; Rajeswari, 2015).

The nitrate content of the untreated tannery effluent was higher than the treated effluent (Figure 3). The nitrate levels in all the sites were not significantly different from one another at $p<0.05$ The high nitrate content in tannery effluent may be due to the presence of ammonia from deliming materials and nitrogen contained in protein materials from liming and hair removal process. ANOVA and Tukey test showed there was a significant difference between sulphate, phosphate, chloride and nitrate, ($p<0.05$) between the sampling sites.

The effluent mean dissolved oxygen concentrations were 13.51mg/L for treated tannery effluent and 8.95mg/L for untreated (Table 2). The dissolved oxygen levels at sites GBTa was significantly greater than the values recorded in other sampling sites though it was below the USEPA 2004; WHO 2008 limit of 50mg/L. Dissolved oxygen levels at sites GBT, FTa, MDTa, BBT and LQT were not significantly different from one another. The values observed is in agreement with (Akan et al., 2007a; Mohammed *et al.,* 2017). Low dissolved oxygen in effluent when discharged into rivers has adverse effects on aquatic biota as less oxygen will be available for aquatic life.

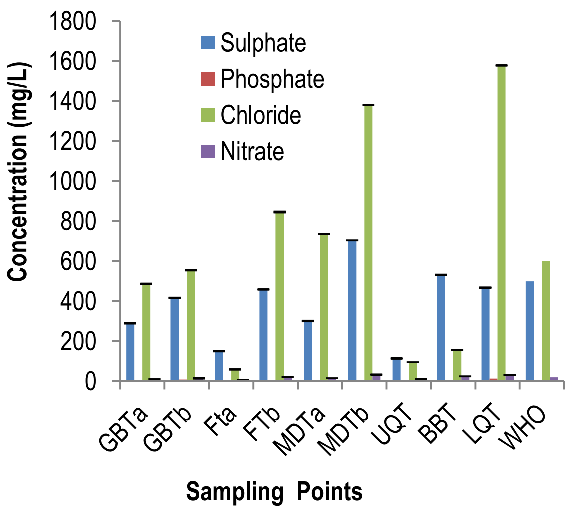

Figure 3: Mean Sulphate, Phosphate, Chloride and Nitrate of Tannery effluents from Sharada and Challawa Industrial Areas

Mean BOD values for treated tannery effluent was 6.80 mg/L while that of untreated was 2.47mg/L (Table 2). The BOD levels at sites GBTa was significantly different from those observed in other sites at $p<0.05$. The unhairing and tanning of hides and skin involved a lot of organic matters which gives rise to high BOD,and as a result, strips oxygen from polluted water and deprives aquatic life of oxygen for survival (Varsha*et al.*, 2017). High level of BOD (600-1622mg/L) was observed by (Noorjahan, 2014). The t-test analysis indicated that there was positive correlation between DO and BOD (r= 0.767).

Arsenic concentrations in tannery effluents (Figure 4) showed that only GBTa, FTa, and MDTa have their arsenic levels within the USEPA 2004;WHO, 2008 threshold limit of 0.1mg/L which ranges from 0.0659-0.1110mg/L with treatment efficiency of 40.63%, 0.0545-0.1000mg/L with 45.50% and 0.0955-0.1409mg/L with 32.22% respectively, while other tanneries sampled were above the limit (NESREA, 2007). Arsenic levels in all the sites studied showed no significant difference. High arsenic concentration in tannery effluent may be attributed to the use of arsenic added to the float to reduce bacterial development in tanning process. Bhatnagar*et al.* (2013); Manjushree et al. (2013); Emmanuel and Abdul-qadir (2017) obtained similar arsenic levels in tannery effluent. Arsenic has toxic effect associated with lung, kidney, bladder, skin disorders and increased risk of cancer (ATSDR, 2003; Scragg, 2006).

Table 2: Mean DO and BOD of the Tannery Effluents from Sharada and Challawa Industrial Areas

Sampling Sites	DO (mg/L)	BOD (mg/L)
GBTa	$21.3±0.29_{e}$	$12.3±0.21_{g}$
GBTb	$12.1±0.26_{cd}$	$4.07±0.17_{f}$
Fta	$9.03±0.17_{c}$	$0.77±0.09_{f}$
FTb	$2.60±0.16_{a}$	$4.40±0.12_{f}$
MDTa	$10.2±0.20_{c}$	$1.30±0.08_{f}$
MDTb	$14.1±0.22_{d}$	$3.67±0.12_{f}$
UQT	$5.10±0.13_{ab}$	$3.37±0.17_{f}$
BBT	$8.43±0.16_{bc}$	$2.27±0.12_{f}$
LQT	$11.3±0.21_{c}$	$3.03±0.14_{f}$
USEPA2004/ WHO, 2008	50	100

Least Significant Difference (LSD) = 3.934
Mean with different letter(s) are statistically significant, $p<0.05$

Cadmium concentrations in tannery effluents samples were as show in (Figure 4). From the figure it can be seen that all the effluents had their cadmium concentrations below the WHO maximum permissible limit of 1.0mg/L with the range of 0.0205-0.0470mg/L and treatment efficiency of 56.38%. Cadmium levels do not also differed significantly in all the sampling sites.Manjushree *et al.,* (2013 Simul-Bhuyan and Shafmentaliqul-Islam (2017); Mohammed *et al.,* (2017) obtained similar Cd levels in tannery effluents and treatment efficiency of 33.25%. 0.07mg/L and 0.081mg/L were reported by (Sahu*et al.,* 2007; Amanial, 2015).

Chromium concentrations in the tannery effluents are shown in Figure 4, it can be observed that chromium level in all the sites were higher than the WHO maximum permissible limit of 1.0mg/L except GBTa (0.625±0.01)mg/L and FTa (0.925±0.01)mg/Lwith treatment efficiency of 56.14% and 30.18% respectively. The drop in the treatment efficiency may be due to improper maintenance of the treatment plant or used of substandard chemicals. The levels of chromium insiteMDTb was significantly greater than those recorded in other sites, while the levels in sites GBTa, GBTb, MDTa, BBT and LQT were not significantly different from one another at $p<0.05$. Range of 1.26-2.17mg/L and 1.55mg/L were also reported by Sahu*et al.,* (2007); Monika *et al.,* (2011). Higher range of 3.33-5.79mg/L was also reported by Bernard and Ogunleye (2015).Values of 5.56mg/L and 7.21mg/L were reported by Bhatnagar*et al.* (2013) and Deepali (2010) respectively. The higher values of chromium may be attributed to large amount of chromium salt(chromium sulphate) used in tannery tanning operations (Modal *et al.,* 2005; USEPA, 2010;). At high concentration and long term exposure, chromium can cause cancer (IARC, 2012).

Nickel concentration in the tannery effluentsispresented in Figure 4. From the results it showed that nickel concentrations in all the tannery effluents were below the WHO permissible limit of 1.0 mg/L with range 0.0029-0.0144mg/L and treatment efficiency of 79.86%. Range of 0.004-0.0095mg/L was reported by Monika *et al.* (2011). Other values reported in literatures were 0.05mg/L, 0.68mg/L and 0.85mg/L (Sahu*et al.,* 2007;Bhatnagar*et al.,* 2013; Amanial, 2015) respectively. There was no significant difference in the levels of nickel in all the sites.The presence of nickel in tannery effluent may be attributed to chemicals used in the tanning and post tanning processing of leather (UNIDO, 2005). At high concentration, nickel may cause damage to DNA and cell structures (Monika *et al.,* 2011).

Lead concentrations in all the tannery effluents were above WHO (2008) limit of 0.1mg/L (Figure 4) with the range of 0.6250-0.8501mg/L and treatment efficiency of 26.48%. Lead levels in FTa was significantly lower than those observed in the other sites while those recorded at sites GBTa, FTb, MDTa and UQT were not significantly different from one another. A range of 0.67-3.10mg/L was reported from the same study area (Bernard and Ogunleye, 2015). Lead affects central nervous system, particularly in children and also damages liver, kidney and the immune system and at higher concentration lead may result in metallic poisoning which can possibly cause cancer in human (Bakare-odunola, 2005). Results indicated significant difference ($p< 0.05$) among the metals (As, Cd, Cr, Ni and Pb) in effluents from the different tannery companies in Sharada and Challawa industrial area,

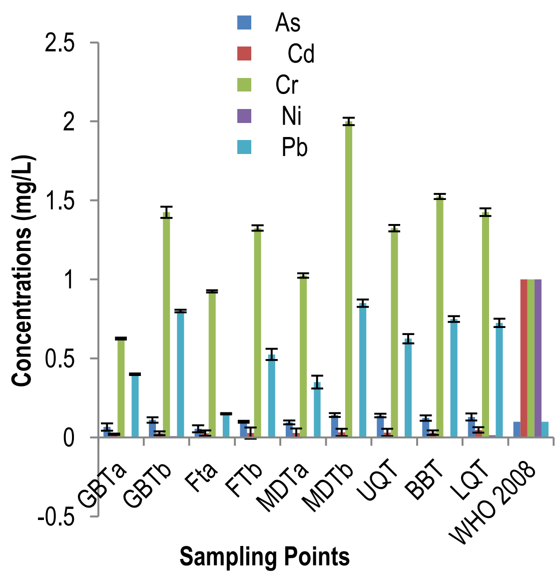

Figure 4: Concentrations of As, Cd, Cr, Ni and Pb in the Tannery effluents from Sharada and Challawa Industrial Areas.

CONCLUSION

The results of physicochemical parameters and heavy metals content in tannery effluent samples were found to be high in untreated than treated. These results indicated that treatment methods employed by some of the tannery companies in treating their effluents do not remove the parameters and heavy metals to WHO, USEPA, NESREA and FEPA permissible level before discharging the effluent into the river, and these could have serious environmental andhealth effects.

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