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Research Article

Determining heavy metal concentrations and physicochemical properties in wastewater

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Abstract

Wastewaters are frequently harmful to both the environment and human health since they are both directly and indirectly released into surface waters. The aim of this study was to determine physicochemical properties and to assess the levels of heavy metals in wastewater. Wastewater samples were collected from Koka and Mojo from the Oromia region, Ethiopia. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) was used to determine the concentration of these heavy metals. The conductivity of wastewater obtained from the Mojo and Koka sites ranged from 1141.33 to 1498.32µS/cm and 1066.33 to 1243.72µS/cm, respectively. The maximum and minimum BOD effluent from Mojo and Koka sites were 1044.78mg/L and 794.73mg/L, and 883.00mg/L and 772.67mg/L, respectively. The COD value was found to range between 1466.08mg/L and 1615.38mg/L in the Mojo area and 1352.65mg/L to 1530.83mg/L in the Koka area, respectively. High BOD levels are a sign of contamination and could indicate a lack of oxygen for living things. In every one of the sample sites, it exceeds the recommended level. High COD levels suggest hazardous conditions and the presence of organic compounds that are resistant to biological processes. The maximum TDS of the effluents, which is more than the recommended limit, were found to be 2417.08mg/L and 2317.06mg/L in Mojo and Koka areas, respectively. Overall concentrations of heavy metals (As, Pb, B, Zn, Cd Hg, Cu, Ni, Co, Fe, Mn and Cr) in mg/L were found to be in the permissible range except for mercury (Hg). The wastewater had heavy metal Hg that was higher than the limits advised by the WHO and US EPA. The studies' findings imply that the effluents are harmful by nature and need considerable treatment before being released into the ecosystem on land.

Introduction

Environmental pollution is one of the major concerns in the world due to urbanization, industrialization, high population density, improved living conditions, and economic development [1]. A large amount of water pollutants mostly toxic heavy metals are directly or indirectly discharged to the water body through natural or anthropogenic activity, which are very harmful to the human body and other life forms even in low concentrations as there is no effective removal mechanism [1,2]. Natural sources of heavy metals in the water body include natural weathering of rocks, soil erosion, and volcanic eruptions, and anthropogenic activities are metallurgical processes, industry, agriculture, printing, photographic materials, combustion of fossil fuel, forest fires, mining activity, automobile emissions and sewage [2,3].

Wastewater irrigation has been used widely for many years in the world particularly in urban agriculture for the cultivation of vegetables or agricultural crops to facilitate food security [4]. Wastewater irrigation has the advantages of contributing essential plant nutrients and organic matter to the soil, as well as providing significant water resources. Yet, excessive heavy metal buildup in agricultural soil caused by wastewater irrigation may not only contaminate the land but also compromise the quality and safety of food [5].

Heavy metal accumulation in plants varies among plant species [6]. Because people and animals consume agricultural goods, they are exposed to the heavy metals that are directly absorbed by agricultural crops during the plant's life cycle. The ability of different plants to absorb heavy metals is measured by either plant absorption or soil-to-plant transfer factors of the heavy metals. Heavy metals, due to their toxic and mutagenic effects even at very low concentrations, are among the major contaminants of food supply items and are considered a major problem to our environment [7]. They are not biodegradable, and have high potential for accumulation in the different body organs, leading to unwanted side effects. The implication associated with heavy metal contamination is of great concern, particularly in the agricultural production system [8].

Therefore, it is crucial to comprehend the heavy metals status in vegetables obtained from wastewater irrigation areas in order to ensure the safety of the food.

Materials and methods

Study area

The study area was Modjo (Lome woreda) and around Koka Town (Ejersa Joro Kebele). Modjo is located 73 km East of Addis Ababa, Oromia Regional state, Ethiopia. It has a latitude and longitude of 8°39′N 39°5′E with an elevation between 1788 and 1825 meters above sea level. The factory is a medium-sized leather factory. It is situated near the Mojo River and channels directly to the River course. Koka (Ejersa Joro Kebele) is located 85 km Southeast of Addis Ababa with a grid reference of 8°27.154´ latitude and 39°03.894´ longitude. This area is characterized by a semi-arid climate having an altitude of 1630 msl, an average annual rainfall of 800mm, and minimum and maximum temperatures of 17.5°C and 26°C, respectively Figure 1.

Sample collection

The industrial wastewater (effluent) samples were collected from Mojo and Koka (Koka Ejersa) areas. Water samples (500 mL) were collected in the plastic bottle using a vinyl glove from different areas of each selected site during January 2022. A total of four composite samples were collected; measurement points for the sampling were designated as P1 to P4. Wastewater samples were collected at the point designated as P1 or outlet of the industry, 5 meters away from the River point designated as P2, 10 meters away from the point designated as P3, and at 20 meters away from the point designated as P4. The empty plastic bottles were first acid washed with 5% HNO_3 to avoid contamination and then rinsed with double-deionized water. About 40 samples of wastewater were collected from different selected sectors in duplicates, all of which are used for the irrigation of vegetables. Each sampling sites span 200 meters from the upper to the lower stream of the river. Some important parameters like pH, Electrical Conductivity, and Temperature were measured on the spot. The samples were directly transported to the laboratory and about 1 mL of concentrated HNO_3 was added to the samples to avoid any kind of microbial growth.

Analysis of physicochemical properties

Electrical Conductivity (EC): High electrical conductivity (EC) water has an impact on irrigation, permeability, and soil structure. The electrical conductivity of wastewater

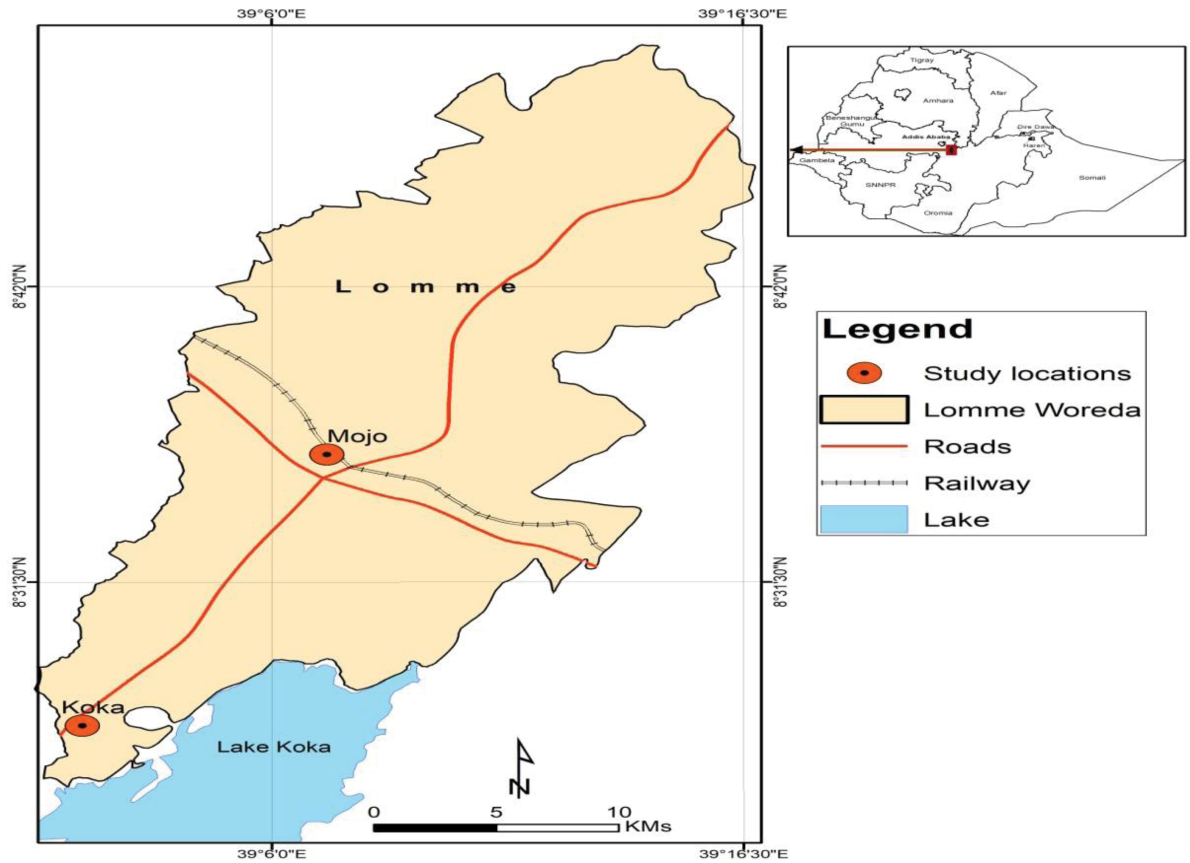

Figure 1: Map of sampling areas.

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was measured using Mettler Toledo Seven Excellence Multiparameter in micro siemens per centimeter (μscm^{-1}) [9].

Chemical Oxygen Demand (COD): The introduction of an excess of potassium dichromate ($K_2Cr_2O_7$) is a common method of determining COD. Titration with ferrous ammonium sulfate is used to measure the excess amount of dichromate once the reaction is finished [10]. The mass of oxygen used per liter of solution is represented by the COD unit of measurement, milligrams per liter (mg/L).

$$COD\ (mg\ /\ L) = \frac{(A-B)\,x\,C\,x\,8000}{Volume\ of\ the\ sample\ (mL)} \quad (1)$$

Where A is the volume of titrant used for the sample (mL); B is the volume of titrant used for the blank sample (mL); and C is the normalcy of the ferrous ammonium sulfate.

Dissolved Oxygen (DO): The DO in the samples of collected wastewater was assessed using Winkler's technique. In the Winkler method, the DO in the water sample is "trapped" by a sequence of chemical reactions that produce an acid product when iodine is present. After that, the iodide solution was titrated using the proper neutralizing agent. The amount of DO in the water sample is equal to the color shift representing the endpoint [11].

Biochemical Oxygen Demand (BOD): Biochemical oxygen demand (BOD) reflects the amount of oxygen used by bacteria and other microorganisms while they break down organic material at a specific temperature and under aerobic (oxygen-containing) conditions. The level of BOD in wastewater was determined according to [11].

Determination of total hardness: The hardness of water is conveniently expressed in terms of dissolved salts of calcium and magnesium.

Calcium and magnesium are the main dissolved minerals found in substantial quantities in hard water. The total hardness of water was determined by titrating it with an EDTA (ethylene diamine tetra acetic acid), a complexing agent standard solution [12].

Determination of Total Dissolved Solids (TDS): The healthy and bad components in water are collectively known as total dissolved solids (TDS). They can be water-soluble minerals, salts, metals, cations, anions, or other organic or inorganic compounds and it was quantified by gravimetric method.

$$\text{Total Dissolved solids}(mg\ /\ L) = \frac{(A-B)\,x1000}{Volume\ of\ the\ sample\ (mL)} \quad (2)$$

Where A is the combined weight of the dried residue and the evaporating dish (mg), and B is the evaporating dish's weight (mg).

Determination of Total Solids (TS): The gravimetric method is used to measure the TS content in water. A pre-weighed Petri dish was filled with a sample of water, and it was heated in an oven set to 180 C. After cooling in the desiccator, the residue was weighed to a constant weight.

Determination of Suspended solid (TSS): Suspended solids are defined as water solids that a filter can capture. The wastewater sample was filtered through a pre-weighed filter to calculate TSS. The residue that was left on the filter was dried in an oven at 103°C - 105°C according to the gravimetric method.

Metal analysis

Sample digestion: Water samples (50 mL) from each sampling bottle were well-mixed thoroughly by shaking. A Multiwave 3000 microwave system (Anton Paar, Graz, Austria) programmable for time and power between 600 and 1400 W and equipped with 16 high-pressure polytetrafluoroethylene vessels (MF 100) was used for sample digestion. A 2 mL filtered aliquot of water sample accurately weighted was pipetted into a microwave digestion Vessel. Nine mL of 10 M HNO_3 and 3 mL of 10 M HCl were added. The vessels were capped and placed in the microwave digestion system and digestion undertake at 1800c for 45 minutes until a clear solution was observed. After digestion, the samples were filtered with Whatman No.42 filter paper and the clear solution was diluted to a 50 mL volumetric flask with 2% HNO_3 blank digestion was also carried out in the same way. The blank solution contained all reagents except wastewater. All samples were digested in triplicates. The digested samples were analyzed for toxic heavy metals by using (ICP-OES) (Model: ARCOS FHS12, USA).

Instrument's operating conditions: The instrumental (ICP-OES) conditions for the analysis of multi-elements and the working range concentration of each element with correlation coefficients are presented in Tables 1,2, respectively. The correlation coefficient (R^2) values calculated from the calibration curves of each analyte element were at least 0.998 (Table 2). These values ensured the linearity of the calibration curves.

Method validation: In the present study due to the absence of certified reference materials for wastewater samples in our laboratory, the validity of the digestion procedure, precision, and accuracy of ICP-OES were assured by spiking samples with the standard of known concentration. The spiked and non-spiked water samples were digested following the same procedure employed in the digestion of the respective samples

Table 1: Instrumental operating conditions for the analysis of metal in the sample of wastewater.

Parameters	Value
Plasma power	1400W
Pump speed	30rpm
Coolant flow	13 L/min
Auxiliary flow	0.8 L/min
Nebulizer flow	0.73 L/min
Optical temperature	14.0-16.00C
Nebulizer pressure	2.0-4.0 Bar
Main Argon Pressure	6.0-8.0Bar
Replicates	3

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Table 2: Concentrations of the working standard solutions and Coefficient of determination of the calibration curve for analysis of wastewater samples.

Element	Concentration (mg/L)	Coefficient of determination (R^2)
Cr	0.08,0.28,0.56,0.84,1.12 and 1.4	0.999
Cd	0.08,0.28,0.56,0.84,1.12 and 1.4	0.999
Zn	0.16, 0.56, 1.12,1.16,2.24 and 2.8	0.999
Fe	0.24, 0.8, 1.6,2.4,3.2 and 4	0.999
Pb	0.08,0.28,0.56,0.84,1.12 and 1.4	0.999
Cu	0.16, 0.56, 1.12,1.16,2.24 and 2.8	0.999
As	0.19,0.64,1.28,1.92,2.56 and 3.2	0.998
Mn	0.24, 0.8, 1.6, 2.4, 3.2 and 4	0.999

and analyzed in similar conditions. Then the percentage recoveries of the analytes were calculated [13].

$$\text{Recovery} = \left(\frac{\text{CM in the spik samples} - \text{CM in the nonspik sample}}{\text{Amount added}}\right) \times 100\% \quad (3)$$

Where, CM = concentration of metal of interest

The Pollution Load Index (PLI)

The pollution load in the study area was calculated using the pollution load index (PLI) for a number of contaminants. PLI, or pollution load index, was calculated for the entire sampling site as the nth root of the product of the n CF.

$$\text{PLI} = (CF_1 \times CF_2 \times CF_3 \times \cdots \times CF_n)^{1/n} \quad (4)$$

Where; Cn represents the concentration of metal n in the sample.

This empirical index offers a straight forward, comparative way to gauge the degree of heavy metal pollution [14].

Integrated pollution load index (IPI)

According to the methodology utilized by many researchers [15-17], the mean of all pollution load index values for all of the metals taken into consideration was established. IPI is classified as a low contamination (IPI ≤ 1.0), a moderate contamination (1.0 < IPI ≤ 2.0), a high contamination (2.0 < IPI ≤ 5), and an extremely high level of contamination (IPI > 5).

Pollution evaluation index (PEI)

In this study, the water quality is categorized using the pollution evaluation index (PEI) [18], which is derived from Equation (2) and is a useful tool for determining the extent of HMs pollution.

$$\text{PEI} = \sum_{i=1}^{N} \frac{Hc}{Hmac} \quad (5)$$

Where Hc and Hmac are the measured value and maximum permissible concentration of each HM, expressed in µg/L, respectively. According to the PEI results, there are three levels of pollution: low (HEI 40), middle (40 HEI 80), and high (HEI > 80) [19].

Results and discussion

Physicochemical properties of wastewater

Due to their major impact on the requirements for water quality, the physicochemical parameters are regarded as essential characteristics. Table 3 shows the physicochemical properties such as temperature, EC, pH, COD, BOD TSS, TS TDS, TH, and TA of the wastewater. The conductivity of wastewater obtained from Mojo and Koka sites ranged from 1141.33 ± 7.84 to 1498.32 ± 5.20 µS/cm and 1066.33 ± 10.75 to 1243.72 ± 81.61 µS/cm, respectively. Greater conductivity was correlated with greater wastewater turbidity, and vice versa. Water's conductivity, a metric measuring the fluid's capacity to carry electricity, is directly proportional to the concentration of ions present. The quality of water is immediately impacted when its conductivity changes noticeably. The wastewater sample taken from the Mojo site had greater values for EC, COD, BOD, TSS, TS, TDS, and TH than the one taken from the Koka site. The maximum and minimum BOD effluent from Mojo and Koka sites were 1044.78 ± 18.06 mg/L and 794.73 ± 23.30 mg/L, and 883.00 ± 11.17 mg/L and 772.67 ± 20.41 mg/L, respectively. The BOD of the studied wastewater samples was greater than the permissible limit of 30 mg/L [20]. High BOD levels are a sign of contamination and could indicate a lack of oxygen for living things. The COD value in the sample effluent was found to range between 1466.08 ± 8.54 mg/L and 1615.38 ± 8.38 mg/L in the Mojo area and 1352.65 ± 29.68 mg/L to1530.83 ± 27.54 mg/L in Koka area, respectively. In every one of the sample sites, it exceeds the recommended level. High COD levels suggest hazardous conditions and the presence of organic compounds that are resistant to biological processes [21]. The maximum TDS of the effluents, which is more than the recommended limit, were found to be 2417.08 ± 55.64 mg/L and 2317.06 ± 74.59 mg/L in Mojo and Koka areas, respectively [22]. The temperature values of wastewater in both sites were found from 26.47°C to 30.07 °C which were within the 20 °C to 32 °C suggested by the WHO [23]. Wastewater samples' pH ranged from 7.15 to 8.85 which was greater than the limit set by WHO 6.5 to 8.536 range [24].

Positively correlated variables from Table 4 that have $p < 0.05$ have a tendency to rise together. One variable often tends to decline while the other rises for couples with negative correlation coefficients and $p < 0.05$. There is no significant difference between the variables for couples with $p > 0.05$. Coefficients that are statistically significant ($p < 0.05$) are bolded.

Metal concentrations in wastewater

Table 5 provides information on the levels of heavy metals (As, Pb, B, Zn, Cd, Cu, Ni, Co, Fe, Mn, and Cr) in wastewater samples. The Food and Agriculture Organization, World Health Organization, and U.S. Environmental Protection Agency (FAO, 1985; WHO, 2006; US EPA, 2007 and Environment and Health in China, 2009 [25-28] had established acceptable limits, and the readings that were found fell below those ranges. The average Hg concentration in the wastewater samples was greater than

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Table 3: Wastewater physicochemical properties (concentrations are in mg/L, except pH, temperature, and EC).

Sample site	pH	Variables measured mean ± SD								
		Temp (°C)	EC µS/cm	BOD	COD	TSS	TS	TDS	TH	TA
KW1	8.40 ± 0.17	27.87 ± 0.40	1200.56 ± 50.46	883.00 ± 11.17	1396.40 ± 26.33	697.59 ± 31.10	2705.31 ± 166.05	2187.41 ± 83.59	342.57 ± 22.41	1547.83 ± 71.34
KW2	8.85 ± 0.09	26.73 ± 0.85	1243.72 ± 81.61	834.79 ± 21.86	1530.83 ± 27.54	672.86 ± 15.93	3145.36 ± 48.15	2266.60 ± 56.18	407.41 ± 15.97	1470.20 ± 81.73
KW3	7.69 ± 0.15	30.07 ± 0.46	1129.21 ± 5.26	772.67 ± 20.41	1352.65 ± 29.68	713.99 ± 39.69	2999.73 ± 119.64	2317.06 ± 74.59	334.59 ± 18.76	1408.73 ± 44.55
KW4	8.09 ± 0.06	28.20 ± 0.44	1066.33 ± 10.75	796.58 ± 13.51	1421.66 ± 12.99	602.65 ± 34.33	3045.55 ± 90.20	2297.14 ± 96.17	395.78 ± 11.42	1453.77 ± 64.30
MW1	8.39 ± 0.19	29.03 ± 0.40	1141.33 ± 7.84	794.73 ± 23.30	1466.08 ± 8.54	714.51 ± 67.67	2871.43 ± 74.18	2216.79 ± 50.46	436.29 ± 31.35	1609.97 ± 67.63
MW2	7.15 ± 0.07	26.50 ± 0.70	1309.86 ± 32.67	957.06 ± 51.27	1517.15 ± 15.09	824.28 ± 35.24	3020.44 ± 46.32	2275.53 ± 85.24	368.81 ± 35.89	1348.53 ± 54.62
MW3	7.78 ± 0.17	26.47 ± 0.67	1410.69 ± 55.74	1017.19 ± 26.17	1537.85 ± 12.96	792.42 ± 44.31	3046.22 ± 126.50	2363.27 ± 68.48	421.11 ± 37.24	1414.73 ± 24.74
MW4	8.14 ± 0.08	28.03 ± 0.78	1498.32 ± 5.20	1044.78 ± 18.06	1615.38 ± 8.38	728.57 ± 45.03	3171.69 ± 70.99	2417.08 ± 55.64	473.37 ± 54.90	1474.67 ± 36.84

Table 4: Pearson correlation coefficients between physicochemical properties in wastewater collected from Mojo and Koka Rivers.

	Temp	*pH*	*EC*	*BOD*	*COD*	*TSS*	*TS*	*TDS*	*TH*	*TA*
Temp	1									
pH	0.071	1								
EC	-0.537	-0.181	1							
BOD	-0.611	-0.312	0.954	1						
COD	-0.624	0.042	0.853	0.780	1					
TSS	-0.404	-0.651	0.624	0.637	0.395	1				
TS	-0.271	-0.080	0.452	0.320	0.635	0.029	1			
TDS	-0.088	-0.311	0.660	0.598	0.564	0.216	0.783	1		
TH	-0.205	0.294	0.555	0.468	0.809	0.022	0.523	0.511	1	
TA	0.361	0.738	-0.277	-0.322	-0.136	-0.430	-0.529	-0.504	0.278	1

Table 5: The mean concentration of heavy metals in (mg/L) in wastewater from different sampling sites.

Sampling site	As	Pb	B	Zn	Cd	Hg	Cu	Ni	Co	Fe	Mn	Cr
KW1	0.001	0.001	0.001	0.280	0.001	0.035	0.004	0.003	0.002	0.531	0.025	0.001
KW2	0.001	0.001	0.011	0.248	0.001	0.039	0.028	0.003	0.002	0.604	0.022	0.002
KW3	0.007	0.006	0.001	0.224	0.007	0.045	0.080	0.006	0.004	0.577	0.023	0.009
KW4	0.001	0.001	0.001	0.262	0.002	0.031	0.001	0.001	0.001	0.129	0.045	0.012
MW1	0.001	0.001	0.001	0.254	0.001	0.030	0.030	0.001	0.001	0.343	0.024	0.045
MW2	0.001	0.001	0.001	0.235	0.001	0.030	0.043	0.002	0.001	0.392	0.026	0.001
MW3	0.001	0.001	0.034	0.357	0.002	0.039	0.026	0.002	0.002	0.267	0.013	0.038
MW4	0.001	0.001	0.014	0.268	0.001	0.031	0.040	0.004	0.002	1.819	0.074	0.004
[25]	0.100	5.000	2.000	2.000	0.010	-	0.200	0.200	0.050	5.000	0.200	0.100
[26]	0.01	0.010	-	-	0.003	0.001	2.000	0.020	-	-	-	0.050
[27]	0.05	0.006	-	5.000	0.010	0.00003	1.300	0.200	-	0.3	-	0.050
[28]	-	1.000	-	-	0.030	0.005	-	1.000	-	-	-	0.500
[29]	0.100	5.000	0.7-3.00	2.000	0.010	-	0.200	0.200	0.050	5.000	0.200	0.100

KW: wastewater from Koka River, MW: wastewater from Mojo River

0.001 mg/L, exceeding the WHO [26], 0.00003 mg/L US EPA [27], and 0.005 mg/L [28] guidelines.

Pearson correlation coefficients from Table 6 that have $p < 0.05$ indicates a positive correlation between metals which means when one variable rises or falls, the other does the same. Variables with correlation coefficient magnitudes between 0.5 and 0.9 were strongly correlated. Variables with correlation coefficient magnitudes between 0.1 and 0.5 were low correlations. On the other hand, two variables that move in opposition to one another, so that when one variable rises, the other lowers, have a negative correlation and $p < 0.05$. There is no significant difference between the variables for couples with $p > 0.05$. Coefficients that were statistically significant ($p < 0.05$) are bolded. The Pearson correlation coefficients were conducted using Excel.

Conclusion

This study demonstrates that the wastewater's physicochemical parameters (temperature, EC, pH, COD, BOD, TSS, TS, TDS, and TH) were determined and the results were found above the acceptable guidelines at all sampling locations. These numbers are the most terrifying and pose

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Table 6: Pearson correlation coefficients between heavy metal concentrations in wastewater collected from Mojo and Koka. Rivers.

	As	Pb	B	Zn	Cd	Hg	Cu	Ni	Co	Fe	Mn	Cr
As	1											
Pb	1.000	1										
B	-0.241	-0.241	1									
Zn	-0.415	-0.415	0.848	1								
Cd	0.976	0.976	-0.135	-0.279	1							
Hg	0.731	0.731	0.264	0.060	0.749	1						
Cu	0.792	0.792	-0.052	-0.432	0.711	0.508	1					
Ni	0.787	0.787	-0.044	-0.318	0.703	0.697	0.733	1				
Co	0.866	0.866	0.086	-0.131	0.836	0.886	0.684	0.928	1			
Fe	-0.004	-0.004	0.136	-0.110	-0.106	-0.109	0.278	0.507	0.240	1		
Mn	-0.177	-0.177	-0.096	-0.172	-0.200	-0.475	-0.060	0.128	-0.130	0.777	1	
Cr	-0.115	-0.115	0.376	0.488	-0.032	-0.065	-0.064	-0.459	-0.214	-0.366	-0.344	1

a serious risk to the ecosystem. Water samples from the Mojo and Koka localities were examined for the presence of heavy metals (As, Pb, B, Zn, Cd, Hg, Cu, Ni, Co, Fe, Mn, and Cr). The levels of those metals in wastewater samples were within the permissible ranges established by FAO and WHO. On the other hand, mercury (Hg), whose permitted limit in wastewater is established at 0.00003 mg/L by the US EPA, was a public health concern in the research area. Therefore, before releasing factory wastewater into the environment, effluent management and adequate treatment are absolutely necessary. The studies' findings imply that the effluents are harmful by nature and need considerable treatment, especially in physical and chemical treatment stations before being released into the ecosystem on land.

Data availability statement

The data used to support the findings of this study are included in the article. The used materials are available in the Ethiopian Institute of Agricultural Research Laboratory.

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