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Removal of Hazardous Pollutants from Tannery Wastewater by Naval Filter Medium (Pumice) Through Adsorption and Filtration Method

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Abstract: *Tannery effluent is one of the most hazardous pollutants containing heavy metals, nutrients, toxic chemicals and others. Therefore identifying potentially efficient low-cost and locally available filter media as an adsorbent for the treatment of tannery wastewater is critical.In present study the efficiency of pumice(volcanic rock) investigated as a filter media for tannery wastewater treatment with selected physicochemical parameters. The volcanic rocks were collected from volcanic cones of refit valley area of Oromia region, Ethiopia and there chemical characteristics were determined using XRF analysis. Batch mode experimental study design has been carried out. The filter media pumice was crushed and graded and effective size was determined by using standard sieve. The composite sample wastewater was collected from Dire tannery, Addis Ababa Ethiopia. The physicochemical analysis of wastewater samples has been done before and after 12, 24, and 72 hours treatment using standard methods. Mean and standard deviations were calculated for each parameter. R statistical software was run for data analysis. Based on this investigation, characterization of the untreated tannery wastewater revealed that the mean concentration of $BOD_{5,}$ COD, TSS, orthophosphate, ammonium, nitrite, nitrate, sulfide, sulfate and chromium were 1081±159.55, 12913±6874.7, 2426±515.2, 168±74, 314±59.9, 1.7±0.29, 124±12.8, 417±130.7, 1307±224 and 35.7±8.6 mg/l respectively. Nitrate removal efficiency of pumice was 95% at RT= 72 hours. However phosphate removal was 63% only. The efficiency of pumice to remove sulfate was from 83-84% in the first 24 and 48 hours retention time pumice achieved 76% chromium reduction respectively. Considering all the selected tannery wastewater parameters for this study, the average treatment efficiency of pumice was 58.8% at RT=24 hours, 61.5% and 67.5% at RT= 48 hours respectively and equivalent result (68.3% efficiency) was obtained after 72 hours.*

Key Words:*Tannery Wastewater, Pumice,,Filter Media, Removal efficiency, Wastewater parameters*

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I. Introduction

Tannery effluent is among one of the most hazardous pollutant of industry. Major problems caused by tannery wastewater containing heavy metals, nutrients, toxic chemicals, chloride, lime with high dissolved and suspended salts, and other pollutants.

Tanning is one of the oldest industries in the world. With the growth of population, the increasing requirement of leather and its products led to the establishment of large commercial tanneries. Two methods are adopted for tanning of raw hide/skin, vegetable tanning, and chrome tanning.Tanneries are typically characterized as pollution intensive industrial complexes which generate widely varying, high-strength wastewaters. Nearly 30 m^3 of wastewater is generated during processing of one tone of raw skin/hide[1].

Tannery operation consists of converting of the raw hide or skin into leather which consume huge amount of water in several stages, generating an enormous amount of liquid effluents which are hazardous to the environment to which they are discharged, consequently, make it as a potentially pollution intensive industry. Tannery effluents again compromise the physical, chemical and biological properties of aquatic environment. Apart from the most toxic heavy metals like Chromium (Cr) chemical impurities of tannery effluents mostly includes the following dissolved substances such as inorganic salt cations (Fe, Zn, Cu, Ca, Na, etc.); anions such as SO_4^2, NO_3^- , PO_4^{3-} and parameters such as, BOD, COD, TSS, TDS etc.[2]Therefore treating tannery wastewater using natural adsorbents is very important to protect the surrounding environment.

In Ethiopia Currently, there are more than 30 tannery industries in operation. Among them the majority found in Oromia region especially Mojo town and around six established in the capital city Addis Ababa. These tanneries have 153,650 sheep and goat skin soaking capacity and 9,725 cowhides soaking capacity per day. Together they also employ 4577 persons.[3]

Every tanning process step, with exception of the crust finishing operations, produces a huge amount of wastewater.[4] The raw tannery effluent causes serious damage to soil, water bodies and the environment at large.

The total wastewater discharge estimation from tanneries is about 400 million m^3/year. About 90% of world leather production uses chrome-tanning processes rather than vegetable tanning.[5] In Chrome tanning process tanneries utilize chromium in the form of basic chromium-sulphate for hide stabilization against microbial degradation and provision of flexibility of the leather. In chrome tanning process about 60% - 80% of chromium reacts with the hides and about 20%- 40% of the chromium amount remaining in the solid and liquid wastes.

Tanneries generate wastewater in the range of 30 – 35 liter per kilogram of skin or hide processed with variable pH and high concentrations of suspended solids, BOD and COD. Major problems are due to wastewater containing heavy metals, toxic chemicals, chloride, lime with high dissolved and suspended salts and other pollutants.[6] Hexavalent chromium from tannery wastewater is one of the major concerns of environmental pollution. This is due to discharge of tannery wastewater in large quantities without or with partial treatment .[7]

Developing countries face numerous challenges related to preserving the environment from industrial wastewater pollution. Like many other developing countries, Ethiopia also grieve from environmental pollution problems of wastewater particularly industrial wastewater. This issue seems to be a subject which has not yet received adequate attention during the development of industries. Certainly very little and/or no investment has been made in the past to wastewater treatment facilities compared to drinking water supply. Therefore, there is a need to develop an efficient and low-cost wastewater treatment technologies for the removal of heavy metals and other pollutants. Among these technologies, adsorption is a user-friendly technique for this purpose.

Adsorption has been identified as one of the most promising mechanism for removal of dissolved heavy metal fractions and nutrients from wastewater. Although commercial adsorbents are available for use in adsorption, they are very expensive, resulting in various new low-cost adsorbents being studied by researchers. Babel and Kurniawan[8] reviewed the technical feasibility of various low-cost adsorbents for heavy metals removal from wastewater and concluded that the use of low-cost adsorbents may contribute to the sustainability of the surrounding environment and offer promising benefits for commercial purpose in the future.

Therefore identifying potentially efficient low-cost and locally available filter media as an adsorbent is critical for proper practice of environmental management by tanning industries. On the other hand ordinary sand for filter media is costly because of construction expansion in the country, not available readily and not efficient in removal of pollutants by adsorption hence there is a need to substitute pumice instead of sand.

1.1 Adsorbents and Adsorption in Wastewater Treatment Technology: Adsorption is recognized as one of the most effective purification and separation technique used in industry especially in water and wastewater treatment. Although the commercially available adsorbents are efficient in removal of heavy metals, they are costly and some cannot be regenerated and recycled. A number of approaches have been recently studied for the development of cheaper and more effective adsorbents for metal removal. Many non-conventional low cost adsorbents, including natural materials, bio-sorbents, and waste materials have been studied and proposed by several researchers.[9]

Adsorption is a user-friendly technique especially for the removal of heavy metals. This process seems to be most versatile and effective method for removal of heavy metal.[10] The adsorption process is being widely used by various researchers for the removal of heavy metals[11] from waste streams and activated carbon has been frequently used as an adsorbent. Despite its extensive use in the water and wastewater treatment industries, activated carbon remains an expensive material. In recent years, the need for safe and economical methods for the elimination of heavy metals from contaminated waters has necessitated research interest towards the production of low cost alternatives to commercially available activated carbon.

Efficient methods of chromium removal from wastewater are important to attain environmental quality standards. Adsorption has been identified as one of the most promising method for removal of dissolved heavy metal from wastewater.[12] It has an advantage over other conventional methods due to its sludge free clean operation. Although commercial adsorbents are available for use in adsorption, they are very expensive, resulting in various new low cost adsorbents being studied by researchers.

1.2 Pumice as a low-cost Adsorbent:Pumice is a light, porous volcanic rock that forms during explosive eruptions. It resembles a sponge because it consists of a network of gas bubbles frozen amidst fragile volcanic glass and minerals (fig. 1). All types of magma (basalt, andesite, dacite, and rhyolite) will form pumice, however it is most commonly formed from rhyolite. During an explosive eruption, volcanic gases dissolved in the liquid portion of magma also expand rapidly to create a foam or froth; in the case of pumice, the liquid part of the froth quickly solidifies to glass around the glass bubbles. Pumice is considered a glass because it has no crystal structure. Like many of the materials considered in this report it is an aluminosilicate.

Figure 1: Typical Pumice Stone: Photo by Mekonnen Birhanie March/2016, Ethiopia.

Studies have shown using substrate rich in iron (Fe), aluminum (Al) or calcium (Ca) concentrations enhances phosphate removal in experimental subsurface wetlands beyond that which can be achieved by using native soils.[13] It was believed that sedimentation of particulate phosphorus and sorption of soluble phosphorus (onto the pumice) were responsible, and that the high concentrations of iron (18.2 %), aluminum (13.7%), calcium (12.7%) and magnesium (7.3%) in the pumice were the source of this high sorption ability. The low specific gravity and high porosity of pumice make it important for a number of applications in water and wastewater treatment processes. Pumice was used as a filter medium and as a support material for microbial growth in water and wastewater treatment.[14] Taking into account the growth of industrialization in Ethiopia and the expected demand for industrial wastewater management, low-cost, appropriate and eco-friendly approaches will play a critical role in the development of future wastewater treatment technology in the country. In this practical approach, this work deal with the principles of adsorption and filtration for the removal of contaminants from tannery wastewater by replacing volcanic ash (Pumice) as a filter media instead of conventional sand.

II. Materials And Methods

2.1 Study Area and Period: This study has been conducted in Addis Ababa University by transporting sample wastewater from Dire tanning industry from May to August 2016.

2.2 Study Design: Batch mode comparative experimental study design has been carried out to determine the efficiency of pumice filter media on the treatment of industrial wastewater, the case of tannery wastewater filtration.

2.3 Experimental materials, Design and setup Establishments:The volcanic rocks were collected from volcanic cones of refit valley area of Oromia region, Ethiopia around Naziret, (Pumice: 8˚14' N 39˚60' E; Scoria: 8˚30' N 39˚19' E) approximately 100 km East of Addis Ababa. The rocks are local volcanic rocks with various chemical and mineralogical structure and transported to Addis Ababa University. The chemical characteristics of filter media were determined by XRF analysis (Table 1).

Table 1: Physical and Chemical Characteristics of Pumice

Chemical Composition	**Percent Weight of Pumice**
SiO_2	64.92
Al_2O_3	10.82
Fe_2O_3	4.62
CaO	5.74
K_2O	4.26
Na_2O	4.92
MgO	0.92
MnO	0.20
P_2O_5	0.14
TiO_2	0.15
H_2O	0.85
LOI*	3.67
pH	7.53
**Physical properties, Particle size = 0.075–0.425mm.	
Porosity (%)	73
Particle density (gcm^{-3})	2.33
Specific surface area (BET) ($m^2\ g^{-1}$)	3.5
Cation exchange capacity (CEC), mequiv. 100 g^{-1}	0.84

**LOI= Loss on ignition: ** E.Alemayehu et al., 2011*

These filter medium was crushed and graded. Effective size was determined by using standard sieve. Based on the analysis the effective size (ES) (d_{10}) of each media was 1.5–4.5 and the uniformity coefficient (UC) (d_{60}/d_{10}) is 3.5 – 4. After grading the filter materials were washed by tap water and dray by sunlight for one week.

Filtration tank was made of metal sheet, with the following dimension, 60 cm height and 28 cm diameter and also was fitted with a half-inch an outlet tap (faucet) 5cm above from bottom of each tank. The filtration tanks was installed at College of natural and computational science, Addis Ababa University. After installation the filter media was filled in the filtration tank 10cm depth with 10 – 25 mm grain size drainage layer at the bottom, 30cm depth filter layer with a grain size of 1.5 – 4.5 mm at the middle and distribution layer (flat coarse gravel) was added 5cm depth at the top of the filter media to protect erosion of filter's top layer, then it is ready for sample tannery wastewater filtration (fig. 2).

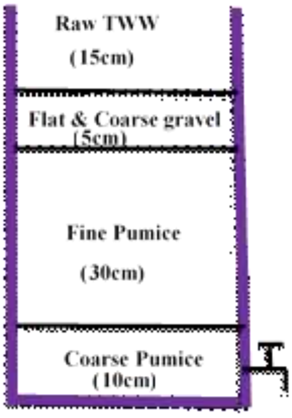

Figure 2: Schematic layout of the Tanker and Components of Pumice filter.

2.4 Wastewater Sample Collection and Filtration: The composite sample wastewater was collected from Dire tannery and transported to Addis Ababa University by using 40 liter plastic 'Jerican'. The onsite measurement of the physicochemical parameters were undertaken. The raw wastewater was added to the two filtration tank at the time and a sample also transported to Addis Ababa EPA water and wastewater analysis laboratory and Water Works Design and Supervision Enterprise(WWDSE) for the raw wastewater characterization.

2.5 Filtrated Sample Collection and Laboratory Analysis: The physicochemical analysis of wastewater samples has been done before and after the treatment with the filter media, using standard methods. Optimum operating treatment time was determined for maximum removal of these impurities by running the experiment for 12, 24 and 72 hours, respectively. Filtrated Sample was taken by 2 liter plastic bottle after each fixed retention time that is after 12, 24, and 72 hours and transported to Addis Ababa EPA Water and Wastewater Analysis Laboratory and Water Works Design and Supervision Enterprise(WWDSE)after taking each sample.
The analytical parameters were pH, DO, BOD_5, COD, TSS, Ammonium N, Nitrite N, Nitrate N, Phosphate, Sulfide, sulfate and chromium. Onsite measurement of the wastewater like temperature, pH and DO were carried out at the site in the tannery environmental quality control laboratory using portable pH meter (Wagtech International N374, M128/03IM, USA) and DO meter (Hach P/N HQ30d, Loveland. CO, USA) for Dissolved oxygen and temperature.
COD, ammonium nitrogen, nitrite nitrogen, nitrate nitrogen, phosphate, Sulfide and Sulfate were measured by using spectrophotometer (Hach model DR/3900 portable spectrophotometer, Germany) according to Hach instructions. BOD_5 and total Cr were analyzed using BOD sensor and inductive stirring system AQUA LYTIC model type ET618-4 and Flame Atomic Absorption Spectrophotometer (AAS), (model AAS NOUA-400, Germany) respectively. Total suspended solids (TSS) were determined according to the Standard Methods for the Examination of Water and Wastewater gravimetric method (APHA, 2005).
The removal efficiency of the filter media for the selected parameters were calculated as:

$$\% \text{ removal} = Ci - Cf/Ci \text{ X } 100$$

Where C_i is the parameter concentration in the untreated wastewater and C_f is the parameter concentration in the treated wastewater.

2.5 Statistical Data Analysis: Mean and standard deviations were calculated to estimate the concentration of each parameter of the samples. The hypothesis has been tested by student t-test using R statistical software: R version 3.2.2 (2015-08-14), Platform: x86_64-w64-mingw32/x64 (64-bit) to determine whether an observed difference between the means of the groups is statistically significant or not, based on the treatment efficiency of the filter materials.

2.6 Data Quality Management: To assure quality of the data by minimizing the errors the following measures had been undertaken: Apparatuses were calibrated; expiry date of reagents had been checked before starting the real analysis and standard control also prepared. Each test had been triplicated.

III. Result And Discussion

3.1: Physicochemical Characteristics of Dire Tannery Wastewater: The raw wastewater was taken from Dire tannery around Asko area, Addis Ababa Ethiopia and transported to Addis Ababa EPA laboratory and WaterWorks Design and Supervision Enterprise(WWDSE)for physicochemical analysis. Based on this investigation the mean concentration of selected physicochemical parameters were presented (Table 2).

Table 2: Characteristics of Dire Tannery Wastewater June/2016.

S.No	Parameter	Concentration (mg/l) Except pH and T^o	Range
1	pH	9.1±3.1	6.5-12.5
2	T^o (^{o}C)	20.6±2.34	19-22
3	BOD_5	1081±159.55	924-1243
4	COD	12913±6874.7	8046-21025
5	TSS	2426±515.2	1849-2840
6	NH_4-N	314±59.9	259-378
7	NO_2-N	1.7±0.29	1.4-1.99
8	NO_3-N	124±12.8	110-135
9	PO_4-P	168±74	112-252
10	Sulfide	417±130.7	334-568
11	Sulfate	1307±224	1118-1555
12	Total chromium	35.7±8.6	28-45

This study revealed that the mean concentration of BOD_5, COD and TSS were 1081±159.55, 12913±6874.7 and 2426±515.2 mg/l respectively (Table 2). This result is basically similar to different studies in Ethiopia with slight difference for different parameters for example a study done at Mojo tannery indicated that the mean concentration of COD was laid between 7950 to 15240 mg/l with the mean of 11123±563.9 mg/l.[15] Another study also undertaken with same tannery wastewater showed that the mean concentration of BOD_5 was1054±448 mg/l.[16] But the concentration of total suspended solid was found from 1849 to 2840 (Table 2) this is a bit greater than some studies for instance a study done in India indicated that 1244 mg/l.[17]

Nutrients like orthophosphate, ammonium, nitrite and nitrate concentration of Dire tannery were characterized in this study, the result revealed that 168±74, 314±59.9, 1.7±0.29, 124±12.8 mg/l respectively. This result is comparable to a study done by Sivakumar *et al.*,[18] which indicates the concentration of nitrate in untreated tannery effluent was 116mg/l. the result of ammonium is in the range of the results done at Bahir Dar tannery wastewater characterization (96-420 mg/l).[19] According to Arasappan and Kalyanaraman,[20] the nitrite concentration of untreated tannery wastewater was 1.3 mg/l almost parallel to this study finding which accounts 1.7±0.29 mg/l (Table 2). Whereas the concentration of orthophosphate in this study was 168±74 mg/l, this result shows that the concentration of phosphate in Dire tannery wastewater is higher than other study results done previously to characterize another tannery wastewaters. The variation may be due to the utilization of phosphorus containing chemicals for different purposes and tanning activities in Dire tannery.

The total suspended solid in Dire tannery found to be 2426 mg/l this result is more or less similar with results of tannery wastewater analyzed by Saritha and Meikandaan.[21] The concentration level of both sulfide and sulfate were 417±130.7 and 1307±224 mg/l respectively. In this case the amount of sulfide found in this study wastewater was more or less equivalent to study done by Islam *et al.* [22] that is 380±50 mg/l. Arasappan and Kalyanaraman,[20] also characterize the tannery wastewater based on their result the concentration of sulfate was 1517mg/l which is almost parallel to this investigation. In terms of chromium concentration, Dire tannery comprised 35.7±8.6 mg/l is similar to other different results presented from various tannery wastewaters in Ethiopia for example a study done by Seyoum Leta *et al.* [15] indicates 32.2±5.7 mg/l. On the other hand two more study results found to be in the chromium concentration ranges of this investigation result 28-45 mg/l (Table 2).[16][23]

Even though Wastewater of each tannery process consists of varying pH and temperature values, this study results (9.1±3.1 and 20.6±2.34^{o}C) respectively were analogous to different studies. Likewise a large variation exists in values of physicochemical parameters in general like BOD_5, COD, TSS, phosphate, sulfide, sulfate, etc. in every tannery wastewater characteristics; this may be because of different tanning process, methods, technology and raw material utilization by various Tanning industries.

3.2: Tannery Wastewater Treatment by Filtration Technique Using Pumice as a Filter Media: Laboratory scale batch experimental studies was undertaken on the adsorbent nature of pumice to investigate the optimum pH, contact time, both adsorbet and adsorbent dose etc. for water and wastewater treatment. But there is no and/or little studies done on pumice adsorbent in real situation with real wastewater. This study investigates the potential of pumice by considering selected parameters of real tannery wastewater with three different retention time to filter out the untreated wastewater (Table 3).

Table 3: Tannery Wastewater Treatment Efficiency of Pumice as a Filter Media

Parameters	**Mean Influent Concentration**	**Efficiency**					
		RT= 24 hours		RT= 48 hours		RT= 72 hours	
		Mean Effluent Concentration	% Removal	Mean Effluent Concentration	% Removal	Mean Effluent Concentration	% Removal
BOD_5	1081±159.55	475±48.8	56	461±46.5	57	415±26	62
COD	12913±6874.7	9380±7798	27	9034±7787	30	7102±5740	45
TSS	2426±515.2	841±126.5	65	776±125	68	727±109.4	70
NH_4-N	314±59.9	185±41.2	41	226±8.7	28	242±27.3	23
NO_2-N	1.7±0.29	0.207±0.002	88	0.078±0.003	95	0.049±0.0045	97
NO_3-N	124±12.8	74±6.4	40	54±5.7	56	5.9±0.82	95
PO_4-P	168±74	98.6±42.5	41	82±42.8	51	57.7±18.4	66
Sulfide	417±130.7	124±17.72	70	120±15.5	71	117±51	72
Sulfate	1307±224	207±45.2	84	218±31.7	83	226±25	83
T. Chromium	35.7±8.6	8.4±1.83	76	8.4±1.77	76	10.6±0.55	70

BOD_5, andCOD removal efficiency of pumice is not satisfactory because the biological activities in the filtration tank is insignificant except on the top of the filter media there may be some biological activities on it that is why the maximum reduction of BOD_5 and COD (62% and 45%) were shown after 72 hours retention time. More over the removal efficiency of these parameters were increased with increasing the retention time that can be also one evidence for little microbial activities in the system especially at the top part of the filter media. On the other hand 70% of the TSS was removed in this system, this is directly related to the straining mechanism of filtration. The indicated amount of solids may be strained in the pumice aggregates and the efficiency may also increase, if the particle size of the filter media reduced but this may also has a draw backs of head loos.

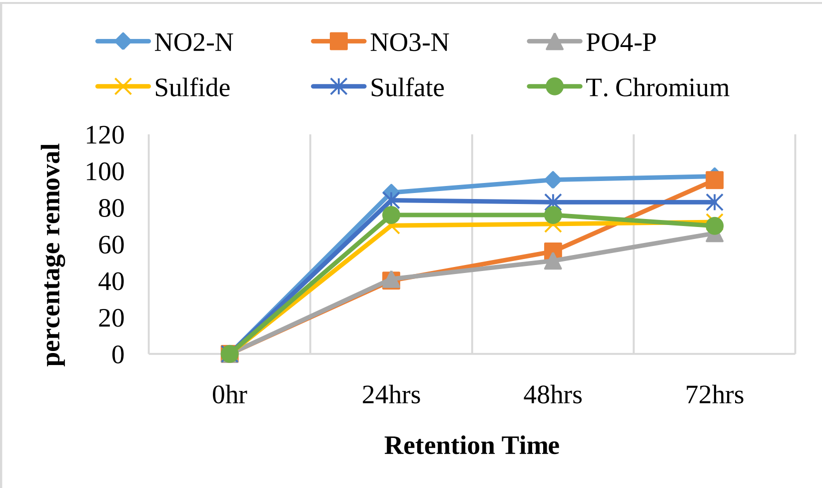

Figure 3: Nutrient, Sulfur and Chromium Removal Efficiency of Pumice Based on Retention Time

Promising reduction of nutrients like nitrate and phosphate, sulfur (sulfide and sulfate) and chromium from tannery wastewater by filtration using pumice media was achieved (Table 2 and Fig. 3). Nitrite and nitrate removal efficiency were 97% and 95% respectively t RT=72hours. This result is parallel to the result of a study done by Wasse Bekele *et al.*,[24] in that study the percentage of nitrate ion removal from aqueous solution using Ethiopian acid activated bentonite clay adsorbent media was determined to be 80% at an optimum condition. Other parallel study also showed that from four filter materials, calcite, zeolite, sand and iron filings, were investigated using laboratory batch tests to evaluate their efficiency in the removal of nitrate and phosphate from the simulated storm water at different initial concentrations under the same 24 hours exposure time. The range of removal for nitrate was from 39% to 65% for calcite, from 42% to 77% for zeolite, from 40% to 70% for sand, and from 74% to 100% for iron filings.[25]The minor difference between this study and other study may be due to the nature of filter media and difference in contact time and method too.

Whereas in the removal of phosphate, this investigation was achieved 66% from tannery wastewater this result is more or less similar to study done in Jimma University Ethiopia that shows only 49.3% reduction from 4mg/l aqueous solution after 24 hours contact time with similar filter media,[26] this difference may be due to the concentration and retention time differences between the two studies. In another study that was tested the four filter materials. The removal of phosphate ranged from 35 % to 41 % for calcite, 59%to 100% for zeolite, 49% to 100% for sand, and 73% to 100% for iron filings.[25] From the indicated four filter materials the result of this paper can be compared in the range of the result obtained from iron filings. That means pumice is similar adsorption characteristics with iron filings. The removal of nitrate and phosphate may be mainly attributed to adsorption, ion exchange and precipitation.

Other wastewater parameters treated with pumice filter media in this investigation were Sulfide and sulfate. Even though the removal of sulfate ion is the main challenge in industrial wastewater treatment, this finding revealed that the maximum reduction efficiency of pumice for these parameters were (72% at RT= 72 hours and 84% at RT= 24 hours) respectively. This removal may be achieved based on the good sulfate adsorption nature of the pumice and its chemical composition. On the other hand metal ions from the filter material may react with dissolved sulfide ions to form metal sulfides as colloidal suspension which were coagulated and precipitated and finally filtered out, this mechanism may be contributed mainly for sulfide removal in the filtration tank. In a study cared out to investigate the effect of chemical modification method on sulfate removal efficiency of adsorbents, the removal of sulfate ion using Fe-modified carbon residue was notably higher compare with unmodified carbon residue and commercially available activated carbon.[27]

Batch experimental laboratory scale studies were undertaken in different world on the potential of pumice to reduce the concentration of heavy metals including chromium from aqueous solution in the laboratory. But in this study the chromium removal efficiency of pumice from real tannery wastewater was done, the finding indicates that 76%, 76% and 70% removal potential at RT= 24hours, 48hours and 72hours respectively. Based on this study result the chromium removal efficiency of pumice was decreased with increasing retention time this may be because of adsorbent saturation. According to Esayas Alemayehu *et al*.,[28] the maximum adsorption result of chromium by pumice was 80% at low pH. The difference in adsorption yield between the two studies may be due to the different samples to be tested that is the real wastewater filtration without any adjustment used in this study and the aqueous solution with known chromium concentration and optimized environmental factors like adjusted pH, temperature, adsorbent dose etc. for the better efficiency utilized by those authors and the other main difference is the method of the investigation.

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