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## Adsorption of Heavy Metals from Industrial Waste Using Fly ash Geopolymer Pebbles

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## ABSTRACT

The improper disposal of waste water by tannery and coolant oil industries in nearby water sources which contains heavy metals, made us to choose the adsorption process using geo polymer for the heavy metal removal. In the 1st phase, stock solution for heavy metals like Cu2+, Ni2+ and Pb2+ were prepared. On the other side geo polymer pebbles were also prepared according to the proportions along with. Using geopolymer pebbles, the solution will be retained for certain hours and it will titrated to find out their initial and final ion concentration. Similarly in 2nd phase, Industrial waste water will be retained with the geopolymer pebbles and then they are titrated to find out their initial and final ion concentration. The samples were tested by titration and by Atomic Adsorption Spectrophotometry methods. Those results were also been compared.

## **Keywords**

Adsorption, heavy metal, geo polymer.

## **1. INTRODUCTION**

The rapid growth of an industrial society leads to a significant increase in the demand for water. However, industrial wastewater has a tremendously negative impact on the environment. Researchers around the world have often proposed many kind of solutions in managing problem of industrial effluents. However in most cases suggested solutions are either too expensive to be applied in industrial scale. For example, disposal of improperly treated wastewater can cause soil contamination, or especially wastewater containing heavy metals results in severe environmental damage. Electroplating, battery, printed circuit board (PCB), and metal surface coating treatment are the main sources of heavy metal wastewater. Heavy metals can be removed from wastewater with a large number of approaches, including chemical coagulation, ion exchange, solvent extraction, and adsorption. In recent years, adsorption has become the central research focus due to its protocol simplicity, effectiveness, and low cost. Geopolymer, an inorganic polymer, has threedimensional polymeric structure and pores formed by the condensation of aluminosilicate mineral powder in addition to an alkali solution at a temperature below 100°C has also possess good adsorption character. The geopolymer synthesis process is simpler than that of zeolite .Therefore, wastewater treatment using a geopolymer pebbles as an adsorbent should be more feasible. Fly ash possess adsorption character and can easily be shaped into

stable pellets along with geo polymer. After ageing for a short period of time, these pellets can be used as adsorbents for the removal of heavy metals from industrial effluents. Experiments in aqueous solutions have shown excellent adsorption behavior concerning the removal of heavy metals like nickel, copper, lead, chromium (III). The advantage of this technology is after saturation the pellets can be easily removed

from the liquid phase. In an attempt to contribute to the reduction of environmental pollution and production cost decrease, researchers have turned their attention to more effective exploitation of industrial byproducts by adsorption process.

## 2. MATERIALS USED

## 2.1 Class C fly ash

Class C fly ash generally contains more than 20% lime (CaO). Unlike Class F, self- cementing Class C fly ash does not require an activator. Alkali and sulfate (SO4) contents are generally higher in Class C fly ashes. The fly ash to water were in the ratio 3.3 to 3.5 for the best results in the adsorption of heavy metals along with geopolymer and the pH of the standard solution were 6-8.

## 2.2 Manufactured sand

With the ban on sand mining implemented by different states, the use of manufactured sand has been enhanced. The usage of manufactured sand has meet the specification of IS 383: 1970 clause 2. The sand particles which are passing 4.75mm sieve has been used for the preparation of geo polymers.

## 2.3 Sodium Hydroxide

This white colored pellets are available in different concentration. The usage of 10 molarity NaOH is preferred for better results. It is highly soluble in water, with a lower solubility in ethanol and methanol, but is insoluble in ether and other non-polar solvents. Similar to the hydration of sulfuric acid, dissolution of solid sodium hydroxide in water is a highly exothermic reaction in which a large amount of heat is liberated posing a threat to safety through the possibility of splashing. The resulting solution is usually colorless and odorless with slippery feeling upon contact with bare hands.

## 2.4 Sodium Silicate

These materials are available in aqueous solution and in solid form. The pure compositions are colorless or white, but commercial samples are often greenish or blue owing to the presence of iron containing impurities. It is readily soluble in water producing an alkaline solution. It is one of a number of related compounds which include sodium orthosilicate, Na4SiO4, sodium pyrosilicate, Na6Si2O7. Sodium silicate is stable in neutral and alkaline solutions. In acidic solutions, the silicate ion reacts with hydrogen ions to form silicic acid, which when heated and roasted forms silica gel, a hard, glassy substance.

## **2.5 Buffer Solution**

70 grams of Ammonium chloride was dissolved 568 ml of concentrated ammonia solution and diluted to the mark with redistilled water and transferred to polyethylene bottle for storage until further use.

## **2.6 EDTA Solution**

3.723 gram disodium ethylene diammine tetra acetate also called as disodium salt and dissolved in distilled water and make up to 1000ml to get EDTA solution 0.01M.

## **3. METHODS**

#### 3.1 Phase 1

It consist of two steps

- 3.1.1 Preparation of geo polymer pebbles.
- 3.1.2 Preparation of stock solutions of various heavy metals.(  $Cu^{2+},\,Ni^{2+}$  and  $Pb^{2+}$  )
- 3.1.1 Preparation of geo polymer pebbles
  - Fly ash of class C which is more pozzolonic in nature has been mixed with the M-sand which meets the specification of IS 383: 1970 in the ratio 1: 1.5.
  - The entire weight of fly ash should not exceed 40% of the weight of NaOH & Na<sub>2</sub>SiO<sub>3</sub>.
  - The ratio of fly ash to solutions were 1: 0.35.
  - The ratio of NaOH & Na<sub>2</sub>SiO<sub>3.</sub> Were in the ratio 1: 2.5.
  - These components are mixed thoroughly to form a cementetious substance called geopolymer.
  - Larger the surface area larger is the adsorption. So the pebbles are to be casted around 2.5-3 cm in diameter.
  - The pebbles are to be ambient cured for 24 hours and steam cured for 24 hours to make it strong enough for the adsorption process.



Figure 1. Geopolymer pebbles after steam curing



Figure 2. Adsorption apparatus

• The stock solution which ae prepared are to be half filled in the apparatus along with 25 geopolymer pebbles to make the heavy metals to get adsorbed.

## 3.2 Phase II

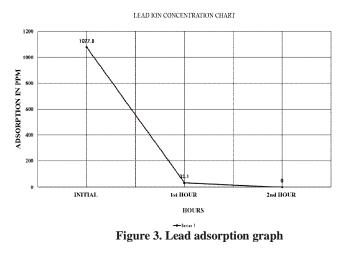
- The waste waters were been collected from industries like tannery, coolant oil and printing industries.
- These waste waters are to be flocculated by adding activated carbon weighing 10-12 grams due the presence of suspended particles and oil content and then they are allowed to get settled. These wastes are to be filtered properly and then they are to be titrated for determination of initial ion concentration against EDTA.
- Then the waste water are to be filled in the cylinder for the height of <sup>3</sup>/<sub>4</sub> of its total height and then 25 pebbles were added and allowed to get retained for several hours. The samples were been collected in regular interval and then they are allowed for testing

## 4. RESULT & DISCUSSION

#### 4.1 Determination of lead (titration)

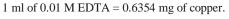
10 ml of Pb solution was taken in a conical flask and diluted to 50ml with distilled water. Then two ml of ammonia buffer pH 10 and muroxide indicator were added. The solution was then titrated with 0.01M EDTA solution until the color changes from pink to violet. The quantity of Pb was calculated from the volume of EDTA used.

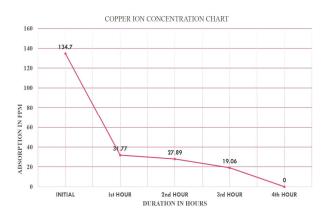
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## 4.2. Determination of copper (titration)

10 ml of Cu solution was taken in a conical flask and diluted to 50ml with distilled water. Then two ml of ammonia buffer pH 10 and muroxide indicator were added. The solution was then titrated with 0.01M EDTA solution until the color changes from yellow green to violet. The quantity of Cu was calculated from the volume of EDTA used.





**Figure 4: Copper Adsorption Graph** 

#### 4.3 Determination of nickel (titration)

10 ml of Ni solution was taken in a conical flask and diluted to 50ml with distilled water. Then two ml of ammonia buffer pH 10 and muroxide indicator were added. The solution was the



Figure 5. nickel adsorption graph

n titrated with 0.01M EDTA solution until the color changes from yellow green to steel blue. The quantity of Ni

was calculated from the volume of EDTA used.

1 ml of 0.01 M EDTA = 0.05871 mg of nickel.

The above mentioned heavy metal concentration were also determined using Atomic Adsorption Spectrophotometer (AAS) and the results were also comparable to the titration values.



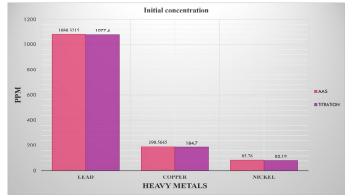


Figure 6. Initial ion concentration comparison between AAS & Titration

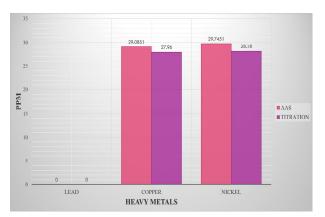


Figure7: Ist hour ion concentration compparision between AAS & Titration

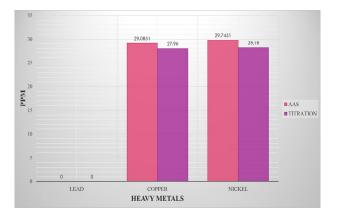


Figure 8: 2<sup>nd</sup> hour ion concentration comparison between AAS & Titration

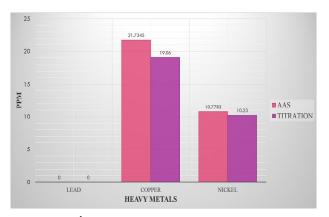


Figure 9: 3<sup>rd</sup> ion concentration comparison AAS & Titration

# 4.4 Determination of chromium ion concentration by AAS

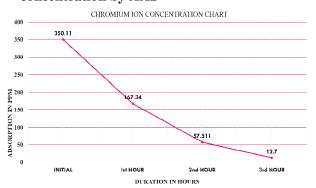


Figure 10: Chromium adsorption by AAS

# 4.5 Determination of heavy metal in Tannery industrial waste by AAS

For the "wet blue" process leathers are to be treated with chromium. After retaining the waste water in geopolymer pebbles some samples were tested in AAS for the presence of heavy metal and its concentration. The Adsorption of Chromium in Tannery waste water by Atomic Adsorption spectrophotometer at the Initial as well as the fourth hour is found out to be as follows.

Initial Concentration	= 28.0	ppm
2nd hour concentration	= 19.4	3 ppm
4th hour concentration	= 11.45	5 ppm

This result confirms that the adsorption of heavy metal chromium from tannery waste.

# 4.6 Determination of heavy metal in Coolant oil waste by AAS

A coolant is a fluid which flows through or around a device to prevent it from overheating, transferring the heat produced by the device to other devices that use or dissipate it. An ideal coolant has high thermal capacity, low viscosity, is low- cost, non-toxic, and chemically inert, neither causing nor promoting corrosion of the cooling system. Some applications also require the coolant to be an electrical insulator.

The Adsorption of Nickel in Coolant oil waste water by Atomic Adsorption spectrophotometer at the Initial as well as the fourth hour is found out to be as follows.

Initial Concentration= 64.1 ppm2nd hour concentration= 31.78 ppm4th hour concentration= 17.54 ppm

This result confirms that the adsorption of heavy metal nickel from tannery waste.

## 5. CONCLUSION

Geopolymer mortar pebbles using manufacturer sand can be used as an adsorbent. Geopolymer mortar pebbles effectively removes the heavy metals like Lead, Chromium, Nickel and Copper. The optimum holding time for various metal for the maximum removals are

Heavy metals		optimum hours	
1.	Lead	3nd hour	
2.	Copper	5th hour	
3.	Nickel	5th hour	
4.	Tannery waste	4th hour	
5.	Coolant oil	4th hour	

Geopolymer mortar pebbles can be used in removing heavy metals from tannery waste, coolant oil. The titration method for the evaluation of heavy metals and atomic adsorption spectrophotometry showed agreeing values. Fly ash can also be effectively used in the adsorption process in addition with geopolymer pebbles.

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