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Application of EAFDS/Lime Integrated System for the Removal of Cu and Mn from Industrial Effluent

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Abstract

Metal pollution is one of the significant concerns affecting the environment and the wellbeing of living things. Copper and manganese are the most common metal pollutants with detrimental effects on the health of human beings. Several methods have been proposed and applied for the treatment of industrial effluent and removal of hazardous metals. One of the most common treatment methods is chemical precipitation. This study is about application of chemical precipitation of copper and manganese metals from industrial acidic effluent in the presence of large concentration of other metals using a steel industry solid waste called Electric Arc Furnace Dust Slag (EAFDS) in conjunction with lime. The study proved that EAFDS alone can remove the target metals considerably and reducing the cost associated with the procurement of other costly chemicals. The concentration of Cu in the raw effluent was 47.2 mg/l. The slag reduced the concentration to 7.8 mg/l achieving 81.7 % removal. The concentration of Mn in the raw effluent was 120.8 mg/l, which was reduced to 12.0 mg/l with the slag only. The two metals were removed achieving 99.7 %with the addition of small amount of $Ca(OH)_2$.

Keywords: Copper; Manganese; Metal removal; Treatment; EAFDS; Neutralisation

Introduction

Human activities are changing the environment on unprecedented level and speed [1,2]. Overpopulation, pollution, climate change and associated Global warming, Ocean acidification and deforestation are the results of human activities. While the industrial revolution brought a positive change for the human race, there is no question that concurrently; it is also having a negative impact on the environment through various unwanted emissions [3-5]. Industrial processes play a major role in the degradation of the global environment even in industrialised countries, growing demand for goods and services putting increased pressure on the environment and the natural resource base [6].

Release of metals or other harmful elements as waste cause environmental problems such as water, air and soil pollution. The effects of water pollution can be detrimental to the environment and to humans, animals, fish, and birds. Water is not suitable if for purposes such as drinking, agriculture, industry and recreation polluted or contaminated with undesirable substances. It also affects the aesthetic quality and beauty of lakes, rivers and other water bodies [7,8]. Due to the non-biodegradability and long biological half-lives for elimination, the accumulation of hazardous metals in the food chain will have a significant effect on human health [9-11]. Hence it is imperative to treat industrial effluent before releasing it into the receiving bodies.

One of the common contaminants of the water system is copper. Copper (Cu) is one of the essential metals to life; it is the third most abundant trace metal in human body after iron and zinc [12]. Cu in small quantities is important to the human body because it helps build strong bones, healthy skin and hair; it is also a component of haemoglobin synthesis and affects endocrine glands function favourably, takes part in the development of enzyme synthesis as well as tissues. If the amount of Cu is higher than the required limit, it becomes toxic.

Wastewater containing large amount of Cu is highly toxic for most plant species, harmful to a variety of living organisms such as microorganisms, fish and humans [13]. Excess Cu can damage to the antioxidant enzyme function, oxidative modification of DNA and a protein, lipid oxidation, activate the redox-sensitive genes, suppress the zinc consumption in the body and causes anaemia by interfering with iron transport [14]. It also causes kidney disease, liver and brain damage, stomach cramps, diarrhoea, haemolysis, nausea and vomiting and upper respiratory tract problems. Excessive Cu may also cause damages to eyes and liver, imbalance in cellular processes causing the Menkes, Wilson's, Alzheimer's, Parkinson's and prion diseases [15].

World Health Organization (WHO), determined the concentration of Cu in drinking water should not exceed 0.05 mg/l [16].

Manganese (Mn) is the 12th most abundant element on the earth. Mn exists widely in nature. As a transition metal, Mn exists in more than five valence states, however mainly as Mn^{2+} or Mn^{3+}. In the environment, it is found mainly in its oxidized chemical forms, as MnO_2 or Mn_3O_4. Mn is essential to human health and is vital for sustaining appropriate functions and regulation of numerous biological processes. It acts as a co-factor in the active centres of various enzymes, and is required for normal development, maintenance of nerve and immune cell functions, and regulation of blood sugar and vitamins, among other functions [17].

Dietary intake of Mn is essential to maintain several important physiological and biological processes, including reproduction, development and formation of healthy cartilage and bone, energy metabolism, urea cycle and antioxidative capacity. Mn also plays a key role in wound healing. Mn is found in nutritional supplements and multivitamin preparations [18].

Even though Mn is an important trace element for the proper functioning of the human body at low levels, at high levels it is toxic. Mn poisoning symptoms include hallucinations, forgetfulness and nerve damage. If its concentration is above certain level in a human body it can cause damage to the brain, liver, kidneys and the nervous system. Mn can also cause serious physiological disorders such as Parkinson's disease, lung embolism and bronchitis. Chronic and acute exposure to Mn may also result in various symptoms of neurotoxicity including cognitive, psychiatric symptoms, extrapyramidal signs, manganism, dystonia, and motor system dysfunction [19].

There is also concern regarding exposure to large amounts of Mn since it may lead to reproductive problems, immunological dysfunction, mutagenicity and carcinogenicity. As a consequence, many environmental agencies throughout the world have regulated limits for Mn concentrationin water. For instance, the USEPA has set the level of Mn in drinking water at 0.05 mg/l. Mn also influences the immune system of marine invertebrates.

Most industrial effluents, especially generated by metal processing industries contain considerable concentrations of Cu and Mn. Due to their effect these contaminants need to be removed or lowered to a concentration set be regulatory bodies through wastewater treatment methods before released to water bodies. Industrial wastewater treatment refers to the mechanisms and processes used to purify wastewater that is produced as a byproduct during production activities. Various wastewater treatment methods are available with varying characteristics of operations. Economic disadvantages of most treatment methods are widely related to expensive equipment, complexities of operations and skilled labour requirement [20]. A simple and cost effective wastewater treatment method will therefore facilitate efficient wastewater treatment and protect the environment from pollution [21]. The purpose of treatment is to eliminate any current or potential threat to human/animal health and the environment [22].

The main treatment methods, which have been employed to reduce or remove hazardous metals including Cu and Mn from effluents, include lime precipitation, ion exchange, adsorption onto activated carbon, membrane processing and electrolytic methods. Most of these methods often involve high capital and operational costs and may be associated with the generation of secondary waste [23,24]. Alkali precipitation is the method widely used for metal removal from acidic effluent by raising the pH to the desired level to form an insoluble precipitate as hydroxides [25]. After adjusting the pH to the required level, the dissolved metal ions are converted to insoluble solid phase *via* a chemical reaction with a precipitant agent such as lime. Typically, the metal precipitated from the solution is in the form of hydroxide [26].

Lime precipitation is one of the most effective methods to treat effluents with a metal concentration of higher than 1000 mg/L. Other advantages of using lime precipitation include simplicity of the process, cheaper equipment, and convenient and safe operations, making it a popular method for metal removal from contaminated wastewater [27].

Different alkaline materials such as $Ca(OH)_2$, $CaCO_3$, NaOH and so on are used to raise the pH of acidic effluent. Research is being carried out continuously to find cost effective, efficient and easy to use replacement for these expensive chemicals and methods. In this study steel industry dust slag, Electric Arc Furnace Dust Slag (EAFDS), was selected as a source of alkali. Steel slag, a type of ferrous slag, is non-metallic byproduct, consisting calcium silicates and ferrites combined with fused oxides of iron, aluminium, manganese, calcium and magnesium that are produced simultaneously with steel [28]. Steel slag contains a significant amount of free calcium and magnesium oxides [29]. Hence, steel slag belongs to medium alkaline materials, with its leachate pH values ranging between 8 to10 [30]. The major components of steel slag are (SiO_2 29.3%, MnO 2.7%, Cr_2O_3 8.8%, Al_2O_3 2.9%, CaO 49.6 %, MgO 4.9%, Fe_2O_3 31.3%, FeO 2.9% and TiO_2 0.7%) [31,32].

Extremely fine dust is formed in an electric arc furnace through vaporization of metal, which is collected in the bag house. In a typical electric arc furnace operation, approximately 2% of the charge is converted to dust forming EAFDS [33]. EAFDS generated during steel production is regarded and listed as a hazardous waste by the regulations of most countries because of the presence of significant amounts of leachable compounds of hazardous metals such as Zn, Ni, Cr and Pb in it [34,35]. However, because of its alkaline nature it can be utilized for neutralization and metal removal from acidic wastewater.

Recycling of waste material in addition to saving natural resources it also saves energy, reduces amount of waste as well as air and water pollutants and reduces greenhouse gases [5]. Recycling wastewater as well as solid wastes has received serious attention from industries in the context of protection of the environment, developing concepts of green chemistry: use of renewable resources and improved water management including recycling of waste materials generated during manufacturing [36].

Aims and Objectives of this Research

The aim of this research was to develop a cost effective and efficient method for the treatment of industrial effluent and removal of Mn and Cu using another industrial solid waste. The material chosen as a replacement for commercial alkalis is Electric Arc Furnace Dust Slag (EAFDS), which is a byproduct generated during the process of steel manufacturing. The aims also include:

- *To determine if dust slag, the byproduct generated by EAF during steel production, can be applied to neutralise acid effluent generated by a steel industry*
- *To determine the optimum condition to remove Cu and Mn from industrial acidic effluent*

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Materials and Methods

Feedstock

All the Chemicals used in this experiment were of analytical grade and used without any further purification. The HCl was obtained from SMM chemicals supplier. The NaOH was bought from Ace Chemical suppliers and the $Ca(OH)_2$ was procured from Sigma.

Effluent and EAFDS were collected from stainless steel industry that uses scrap metal as raw material to produce steel.

Equipment

Knick pH meter 766 Calimatic (Germany) was used to measure pH throughout the process. Rod stirrers used for the agitation were IKARW 20 digital stirrer (Germany). ICP-OES (Spectroacros, Acros-FHS, Germany) was used for the determination of metal content.

Procedure

The study was carried out using 1L plastic beakers and road stirrers. Based on the amount of alkali content in the slag and the acidity of the effluent, the amount of the alkali required for effective neutralization was calculated using alkalinity to acidity ratio of 3:2. All the analyses were carried out in triplicate.

One liter of effluent was measured into a beaker and the calculated amount of slag was added into the container. The contents were stirred continuously at 380 rpm using rod stirrers. Samples were collected at intervals of 10 and 30 minutes as well as every hour for the next 61 hours and every 2 hours until it is 96 hours when the process was continued with the addition of pure lime to reach to the target pH of 12.5. A total of 55 samples from each were collected and analysed. The content was made up to its original 1L volume after each sampling. After 96 hours, the process was continued by adding pure lime ($Ca(OH)_2$) and two more samples were collected at pHs 9.5 and 12.5. The amount of lime required to raise the pH further to 12.5 was only 6.5 g/L.

The collected samples were filtered immediately through Whatman No. 1 filter paper. pH and acidity were analysed using the method given bellow. Metal content was determined using ICP-OES (Spectroacros, Acros-FHS, Germany).

Analytical

The slag was dried in an oven for 2 hrs at 105°C, crushed to powder after cooling and sieved through 300 µm pore size sieve. The amount of free lime and total alkali in the slag were determined as follows.

Total alkali in the slag was determined as $CaCO_3$ by using the following method. 2.5 g of the dried crushed and sieved slag was weighed into a beaker, 100 ml of 1 N HCl was added and boiled for 2 minutes. The solution was cooled to room temperature, made up to the mark of 100 ml with DI water and filtered through Whatman No. 1 filter paper. 40 ml of the filtrate was titrated with 1 N NaOH to pH 7.

The total alkali content as $CaCO_3$ was calculated from the mass and volumes using the formula given in equation

$$\% CaCo_3 = \frac{50 \times C_1 \times V_1 - C_2 \times V_2}{1000} \times \frac{M \times V_3}{V_4} \times 100 \qquad (1)$$

Where C_1 is the concentration of HCl, V_1 the volume of HCl, C_2 the concentration of NaOH, V_2 the volume of NaOH, V_3 the volume of the solution titrated, v_4 the total volume of the sample solution and M the mass of the slag weighed.

Total alkali content, which is the alkali that is capable of reacting with the acid in the effluent; in the slag determined using the method above was 92.4 % as $CaCO_3$.

pH of the raw effluent as well as all the samples taken during the process were also determined by using a pH probe directly. pH of the raw effluent measured using pH meter was 0.64. After adding EAFDS, the solution was stirred at 380 rpm. The pH increased rapidly to 3.0 in 10 minutes, which can be attributed to the presence of water-soluble free lime in the slag. However, the pH increase slowed down once all the free lime was consumed and rose to 3.1 after four hrs 3.5 after 6 hrs, 7.5 after 10 hrs, 7.7 after 12 hrs of agitating and reached to 8.1 after 24 hrs, 8.2 after 48 hrs of agitation and to 8.7 after 96 hrs.

Results and Discussion

Copper

Major sources of wastewater containing Cu are corrosion of water piping, mining and refining of Cu, fertilizer industries, petroleum refining, electroplating, paints and dyes industries, mining and metallurgy, explosives industries, pesticides industries, metal finishing, plastics, etching, iron and steel industries, electronic materials rinse and plating and printing circuits industries [37]. Cu is usually found at high concentrations in most wastewater, because it is one of the most commonly used metals in many industrial applications [38]. There are three important oxidation states of Cu namely 0, $^{+}1$ and $^{+}2$. However, Cu commonly exists as a divalent cation and is generally more mobile in acidic conditions, while at pH above seven; it tends to form minerals like Cu carbonates and hydroxyl-carbonates [39].

It is recognized as one of the most widespread metal contaminants of the environment. Water contaminated with Cu must be treated before being discharged into the environment because of its toxic properties, even at low concentrations [38]. In order to enhance more environmental friendly and cost effective wastewater treatment methods, there has been increased research interest into developing new methods and approaches [40]. Cu concentrations should be less than 2.0 mg/l in mining and electroplating wastewater before discharging into the receiving bodies, as suggested by the WHO, while the USEPA has set a limit of 1.3 mg/l [41].

In wastewater Cu displays high mobility and can enter into the environment easily by forming complex compounds. Therefore, Cu removal from wastewater has a significant importance for curbing its toxicity towards humans, animals and the environment. Due to its deleterious effects on living beings, Cu should be removed or reduced from wastewater to match the standards. Several technologies involving chemical and physical processes such as chemical precipitation, evaporative recovery, solvent extraction oxidation/reduction, coagulation-flocculation, flotation, filtration, adsorption, biosorption, cementation, ion exchange, membrane technologies, reverse osmosis, biological treatment, bioelectrochemical systems and electrochemical treatment technologies have been commonly used for treating wastewater contaminated with Cu prior to discharge into natural water bodies [41]. Chemical precipitation treatment may provide a desired quality effluent with the better cost effective overall process [15]. Hydroxide precipitation is one of the most widespread technologies due to its simplicity, low cost and easy operation.

Since slag has alkaline property similar to hydroxides, one of the aims of this study was to find out if it could be used as a replacement source of alkalinity to remove metals from industrial acidic effluent. Since Cu forms insoluble precipitate in higher pH, EAFDS was investigated to see if it could raise the pH of the industrial acidic effluent high enough to remove Cu from the effluent as a precipitate.

The Cu concentration in the raw effluent was found to be 47.2 mg/l. After steering the mixture of the acidic effluent and EAFDS for 10 minutes the pH was raised to three resulting in rapid decrease in the concentration of Cu reaching to 20.3 mg/l achieving 57 % removal in just 10 minutes. After 24 hours, the pH reached 8.1 and the concentration of Cu in the solution decreased further to 10.0 mg/l achieving 78.8 % removal. The pH increased further to 8.7 before adding $Ca(OH)_2$ into the reaction mixture. The concentration of Cu in the solution dropped further to 7.8 mg/l achieving 81.7 % removal. Addition of $Ca(OH)_2$ increased the pH to 9.5 resulting in more removal of Cu from the solution. Cu Concentration in the solution dropped to 0.2 mg/l achieving 99.6 % removal. pH increased to the target pH 12.5 resulting in a removal of Cu decreasing the concentration to 0.1 mg/l resulting in the final removal of 99.8 % Cu from industrial acid effluent. The effect of pH on the concentration of Cu is given in figure 1.

Manganese

Mn exposure is commonly associated with anthropogenic activities, its geological abundance, and its widespread industrial applicability. Mn ore is considered a crucial industrial element and utilized both metallurgical and non-metallurgical industries. Mn is the fourth major global commercial metal as it has essential application in steel and iron industries. Global yearly utilization of Mn is higher than 1.5 Mega ton and may potentially rise in the future. The manufacturing of Mn compounds, its vast industrial applications, and mining activities have made Mn a significant environmental pollutant. Mn compounds and wastes are released into the water and become toxic metal pollutant in the aquatic environment [42].

Mn exists in the aquatic environment in its dissolved and solid form in oxidation states of $^+2$, $^+3$ or $^+4$ governed by pH and redox conditions mainly as oxides, carbonates and silicates [43].

Different studies are being carried out to develop effective method to remove Mn from wastewater. Some researchers investigated Mn distribution and valence conversions in water. Others studied removal methods, such as aeration and filtration, chemical and/or biological oxidation using bacteria. Several treatment technologies have been used to remove Mn from water, including chemical oxidation, adsorption, membrane filtration and biological treatment. Mn in wastewater can be oxidized by chlorine, converting it to MnO_2. Coagulation process can remove Mn by charge neutralization. Other methods such as adsorption, sedimentation and filtration are also used [44].

Mn can be removed from wastewater by physical, chemical, and biological processes or by a combination of these methods. Typically, Mn (II) ions are chemically removed from effluents by oxidizing them to MnO_2 or precipitation as a carbonate Mn from water. Mn can be removed by oxidation, since Mn (IV) compounds are insoluble [43]. Since Mn (II) oxidises to Mn (IV) in alkaline medium it is imperative to find a material that can raise the pH of the industrial acid effluent to the required pH.

EAFDS was selected because of the high content of total alkali it contains. The concentration of Mn in the raw effluent was 120.8 mg/l. The decrease in the concentration of Mn was very slow until the pH reached 7.9 after 13 hours. The concentration of Mn decreased to 55 mg/l achieving 54.5 % removal. After passing pH 8.0 the concentration dived considerably and reached 12 mg/l at pH 8.7 before the addition of $Ca(OH)_2$ achieving 90.1 % removal. After addition of pure lime, more Mn was removed from the solution. At pH 9.5, the concentration of Mn in the solution was 0.4 mg/l achieving 99.7 % removal. However, the concentration did not show change with further increase in the pH to 12.5 remaining at 0.4 mg/l [45,46]. The effect of pH on the concentration of Mn is given in figure 2.

Conclusion

The treatment method investigated in this study was chemical precipitation technique, which is one of the most common treatment methods to neutralise and remove hazardous metals from wastewater. Since the chemicals used for neutralisation and metal removal incur cost, the aim was to find an alternative replacement which possesses similar property to the commercial costly chemicals. The material selected for the study was Electric Arc Furnace Dust Slag (EAFDS). This material is a byproduct generated by a steel manufacturing industry and usually considered a waste and disposed of into dump sites resulting in the pollution to the environment, disposal levy for the company and loss of potentially useful chemicals embedded in it.

The investigation of the composition of EAFDS indicated that it contains ample amount of alkali which can be exploited as a source for water treatment. Since it is mainly composed of metal oxides the leachate is alkaline. This property was used for the treatment of industrial acid effluent from a steel industry containing various hazardous metals in considerably high concentration.

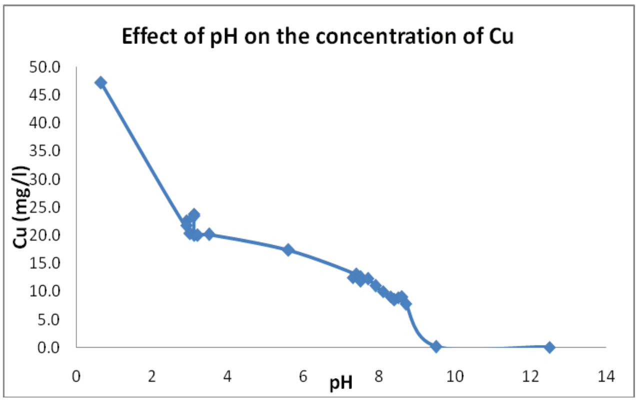

Figure 1: Effect of pH on the concentration of Cu.

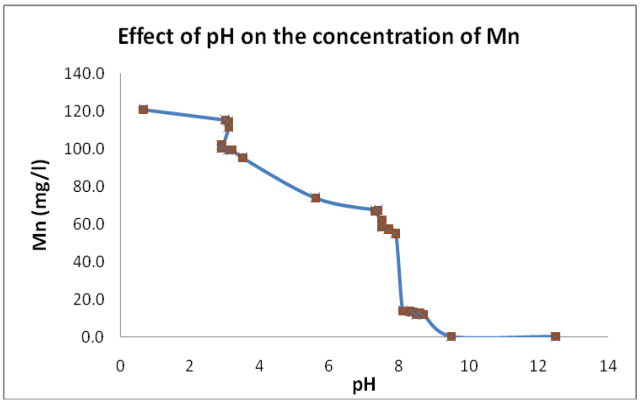

Figure 2: Effect of pH on the concentration of Mn.

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The study demonstrated that EAF dust slag, a byproduct generated during the process of steel manufacturing, can be used for neutralisation of acid effluent from steel industries. The slag alone was capable of neutralizing the highly acidic effluent whose pH was 0.64 effectively raising the pH to greater than 8.1 in 24 hrs and 8.7 after 96 hrs. Hence, EAFDS can be used to neutralise acidic effluent from industries. The process was simple and has no cost to procure alkalis for the process.

The concentration of the target metals Cu and Mn was reduced considerably using the slag only, resulting in the removal of more than 84 % for Cu and 90 % for Mn. To achieve a better removal level $Ca(OH)_2$ was added to raise the pH to the target value of 12.5. Since the pH of the solution was already raised in to alkaline range by EAFDS the amount of lime required was very small saving cost associated with the lime. The addition of lime resulted in further removal of the metals; significantly, resulting in the removal of Cu (99.7 %) and Mn (99.7 %).

Considering EAFDS, a material generated as a waste and has a limited application, particularly in the fields of engineering and construction, using it as a source of alkali has significant advantages. Unlike most of the chemicals used for acid water treatment there is no large cost associated with it. It also does not require a complex process or machinery, working only through thorough mixing. Consequently, this process saves costs particularly for steel industries since it is readily available at their disposal as a by-product of the manufacturing process.

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Compliance with Ethical Standards

No living things or processes that affect the wellbeing of living things or the environment were carried out.

Conflict of Interest

The authors declare there is no conflict of interest whatsoever.

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