

# Comparing The Efficacy of Different Alkali Metals on Acid Mine Drainage Neutralization

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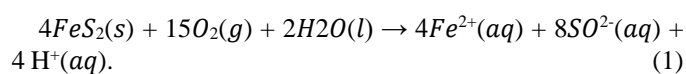
**Abstract**—The presence of sulphate in Acid Mine Drainage (AMD) has negative effects on the environment. Sulphate can cause acidification of soil and water bodies, and increase the mobility of metals, such as iron and manganese, in the environment, potentially leading to contamination of water, soil and death of biodiversity. The sulphate in AMD can be recovered for beneficial uses through precipitation. The efficiencies of sodium hydroxide, calcium hydroxide and synthetic MgO in AMD neutralization were investigated in this study with the purpose of understanding which is more efficient in sulphate recovery. Characterization of solid samples was done using X-Ray Fluorescence (XRF) and Fourier-Transform Infrared Spectroscopy (FTIR) Liquid samples: raw and treated AMD was analysed using Inductively Coupled Plasma Mass-Spectroscopy (ICPMS). The AMD used in the study had an acidic pH of 2.34,  $SO_4^{2-}$  was found to be 7141 mg/L and Fe was 2604 mg/L. The results indicated that amongst the studied materials, the neutralization efficiency obeyed the following sequence:  $NaOH \geq Ca(OH)_2 \geq MgO$ . Sulphate recovery was observed to be in the following order:  $Ca(OH)_2$  (12.76%)  $\geq NaOH$  (10.282%)  $\geq MgO$  (6.281%). In conclusion, neutralizing agents have strengths and weaknesses and the type used in AMD treatment has an effect on the type of products formed, with  $Ca(OH)_2$  being most reliant in sulphate recovery according to this study.

**Keywords**—Alkali metals, AMD, compare, neutralization and sulphates.

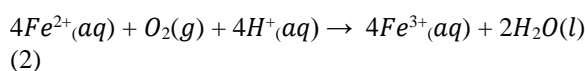
## I. INTRODUCTION

Acid Mine Drainage (AMD) originates from the oxidation of tailings, waste rock, or remnants from underground or tunnel excavations that contain minerals abundant in sulfides, primarily pyrite ( $FeS_2$ ), when they come into contact with both oxygen and water [1]. Water pollution can happen in the voids left behind by deep or underground mining, when water flows through coal mining waste on the surface, or when water is used for mining operation [2]. Acid mine drainage is the wastewater contaminated with heavy metals released from mining activities that are detrimental to the environment.

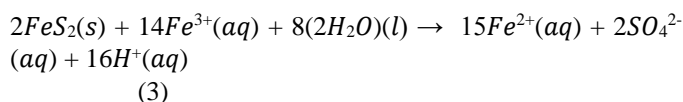
The formation of AMD can be represented by the following reactions:



The oxidation of sulphide to sulphate solubilises the ferrous iron ( $Fe(II)$ ) as shown in (1), which is subsequently oxidised to ferric iron ( $Fe^{3+}$ ) as shown in (2)



The ferric cations produced can also oxidise additional pyrite and itself being reduced into ferrous ions.



The net effect of these reactions is to produce  $H^+$  and maintain the solubility of the ferric iron [3].

The formation of acid mine drainage is a complex process, but the presence of sulphate is an important factor [4]. When pyrite (iron sulphide) and other sulphide minerals are exposed to air and water, they undergo a process called oxidation, which produces sulphuric acid. This acid then reacts with other minerals in the rock, forming more acidic compounds and releasing heavy metals into the water. The sulphate released during this process can also contribute to the production of hydrogen sulphide gas, which can be toxic to plants, animals and humans [5].

The presence of sulphate in acid mine drainage contributes to the negative effects of AMD pollution in several ways. The high concentration of sulphate in AMD can cause the formation of acid rain, which can damage plants, soil, and infrastructure [6]. In addition, sulphate can interact with other elements in the environment, such as calcium, to form gypsum. This process can cause the loss of base cations, such as calcium, from the soil, which can lead to a decrease in the pH of the soil and a decrease in the fertility of the soil [7].

AMD is characterized by an acidic pH that can be below 2 and high levels of electrical conductivity and metal ions such as  $Fe^{2+}$ ,  $Fe^{3+}$ ,  $Mn^{2+}$  and  $Al^{3+}$  among others [8]. Acid mine effluents are able to find their way into the environment and water bodies affecting and altering their chemical profiles and natural state. Discharge of AMD into water resources could have deleterious effects on the aquatic environment and drinking water supplies.

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Soils with AMD contamination lacks essential elements crucial for healthy plant growth. Consequently, the long-term environmental consequences of AMD include challenges in re-establishing vegetation and facilitating land rehabilitation [9]. High concentrations of dissolved metals in water from AMD can be easily absorbed by aquatic organisms and accumulate in them inducing toxic effects that impair growth, metabolism, reproduction, or even survival of living organisms, impacting the entire trophic chain [10].

The composition and pH of AMD, which can change depending on the geology of the mine sites and the mining techniques used, determine the extent of environmental pollution that it causes [11]. The remediation of AMD is thus crucial for protecting and maintaining the environment from the harm of the pollutant. Based on the requirements for chemical addition, infrastructure, maintenance, and monitoring, AMD treatment can be categorized as active or passive systems [12]. In order to lessen the harmful effects on the receiving environment, active treatment procedures have been used, such as the ongoing addition of chemicals and substrates to precipitate metals, membrane processes and ion exchange.

Active treatment of AMD involves the introduction of alkaline reagents to raise the pH and induce the precipitation of dissolved metals in the form of hydroxides, which can then be separated through either gravity settling or dissolved air flotation. The ideal pH for this precipitation process, as suggested by [10] typically falls within a range of 6 to 9 and is contingent on the specific dissolved metals targeted for removal. The selection of AMD treatment approach, along with the choice of chemicals and pH levels, significantly impacts the quality of the discharged water.

García-Valero et al., [9] reported various techniques that have been experimented with or applied to address AMD at its source, including the utilization of limestone and alkaline waste materials from industrial processes like fly ash, cement kiln dust, green liquor dregs, and bauxite residues. Active methods for treating acid mine drainage offer notable advantages, particularly in their capacity to efficiently address large volumes of AMD.

In addition, active methods allow for greater control over the treatment process, so it is possible to achieve a high level of treatment efficiency. Active treatment methods can be designed to remove specific pollutants, so they can be tailored to the specific characteristics of a particular AMD site. Another advantage of active methods is they often have a faster treatment time than passive methods, making them suitable for sites where there is a need for rapid treatment [13] - [14].

Sodium hydroxide (NaOH), magnesium oxide (MgO), and calcium hydroxide (Ca(OH)<sub>2</sub>) are all commonly used as neutralizing agents in acid mine drainage treatment. Each of these agents has different properties and advantages, and the most suitable agent for a particular situation depends on a number of factors, including the chemistry of the AMD, the pH target, and the desired end use of the water and sludge [15]. Sodium hydroxide, magnesium oxide, and calcium hydroxide are commonly used as neutralizing agents in AMD treatment for a variety of reasons. Firstly, these reagents are readily

available and relatively inexpensive. They are also effective at neutralizing the acidity of AMD and can achieve the desired pH target. Lastly, they are capable of removing a range of metals and other contaminants from the water [16].

## II. MATERIALS AND METHODS

### A. Sampling

The AMD sample was collected from Khwezela Colliery, a two-part open-cast thermal coal mine located at eMalahleni in the Mpumalanga Province, South Africa. The precise geographical coordinates are -25.926243 latitude and 29.155635 longitude.

### B. Modification Of Magnesite

Magnesite was calcined or heated at high temperatures to produce magnesium oxide (Synthetic MgO). Calcination of magnesite can improve its reactivity and purity. 50 g of magnesite was put in a furnace for 15 minutes at a temperature of 1050 °C. This was done in accordance with literature as reported by [17]. Post calcination, the magnesite was left to cool in the furnace to a temperature that was conducive for handling where after it was allowed to cool further at room temperature (25 °C) conditions.

### C. Aqueous Sample Characterization

Filtered samples of the raw and treated AMD were characterized using ICP-MS (iCAP 7000 Series, ANATECH, South Africa) analysis for metal composition quantification. pH, TDS and EC of the AMD were determined using a HACH HQ40d multimeter probe.

### D. Solid Sample Characterization

All solid samples relevant to the objectives of the experiments were collected and analysed using XRF (S1 Titan handheld XRF, Bruker, Germany) and FTIR (Bruker instrument Alpha platinum Art).

### E. Batch Neutralization Experiments

For quality assurance of results, experiments were conducted in triplicate to ensure consistency and reliability of results. The neutralization experiments involved the use of the following neutralizing agents: NaOH, Ca(OH)<sub>2</sub> and synthesised MgO. The dosages used to neutralize the AMD ranged from 2.5 g to 15 g with an increment of 2.5 g per beaker. 500 mL of AMD was poured into glass beakers, the relevant masses of neutralizing agents were poured into the beakers then subjected to agitation using a flocculator at 200 rpm for 72 minutes. Neutralized samples were allowed to settle for 30 minutes before being filtered using 100 mm filter papers. The samples were measured for pH, EC and TDS.

### F. Liquid Sample Preparation

After the neutralization of AMD, the samples were prepared for ICP-MS analysis. Samples were diluted by a factor of 100, by adding 1 mL of the sample to 99 mL of diluent. This was achieved by filtering the samples using a 0.45 µm pore size nitrate cellulose filter membrane. 1 mL of each sample was

filtered into a 100 mL glass bottle followed by pipetting 5 mL nitric acid and allowed to settle for 1 minute. 96 mL of deionized water was measured into a volumetric flask and added into the nitric acid-sample mixture. The samples were refrigerated at 4 °C until analysis.

### G. Preparation Of Solid Samples

Post-neutralization, samples were allowed to sit for 30 B. minutes to allow precipitates to settle at the bottom of the beakers forming sludge and the excess water removed. The remaining sludge was collected in evaporation dishes to be dried in an oven for 24 hours at a temperature of 100 °C. The dried sludge was then manually milled and sieved to acquire particle sizes of <150 µm using a 150 µm manual sieve. The final products were collected and stored in closed centrifugal tubes until analysis.

### H. Sulphate Recovery

Sulphate recovery was evaluated by determining the amount of sulphate removed from the AMD using (4) and percentage composition of neutralization residue for potential useable sulphate minerals that can be isolated and extracted.

$$\% \text{ sulphate} = [(C_0 - C) / C_0] \times 100 \quad (4)$$

Where  $C_0$  = initial concentration of sulphate and  $C$  = is the final concentration of sulphate after treatment.

## III. RESULTS AND DISCUSSIONS

### A. Characterization Results

#### AMD Characterization

Analysis of the physicochemical properties of the AMD which included pH, EC, and TDS was conducted. It was found that the AMD had a pH of 2.34, an EC of 990 µS/cm, a TDS concentration of 693 mg/L and a sulphate concentration of 7141 mg/L. The sulphate concentration is more than 28 times the value that is stipulated by SANS241 standards. The low pH can mobilize heavy metals, such as iron, lead, and manganese, which can then be transported into neighbouring environments. High concentration of heavy metals were recorded as follows: Zn - 20.7 mg/L, Cu - 4.4 mg/L, Mn - 69.4 mg/L, Al - 277.7 mg/L and Iron - 2604.5 mg/L which are all above the standards set by SANS241. The ICP-MS results for the AMD that was reacted with the neutralizing agents indicated an increase in sulphate concentration which was contrary to the expected results. The sulphate concentration in the neutralized AMD increased from 7141 mg/L to 51778 mg/L for synthetic MgO and 3774594 mg/L for NaOH which is a drastic jump from the initial sample. The increase in sulphate concentration might have been as a result of re-oxidation of reduced sulphur compounds. AMD may contain reduced sulphur compounds, such as thiosulphates or elemental sulphur. During the neutralization process, these reduced sulphur compounds may have undergone re-oxidation reactions, leading to the formation of sulphate ions. This can contribute to the sulphate concentration increase. The other reason might be due to equilibrium reactions. The neutralization of AMD typically

involves the addition of alkaline materials to raise the pH and reduce the acidity. As the pH increases, chemical equilibria involving sulphate ions, hydroxide ions ( $\text{OH}^-$ ), and other chemical species may shift, resulting in the release of sulphate ions. For example, hydroxide ions can react with sulphate ions to form bisulphate ions ( $\text{HSO}_4^-$ ), and subsequently, sulphate ions.

### C. XRF Analysis

TABLE I shows the elemental composition (wt%) of Raw magnesite, Synthetic MgO, AMD reacted: Synthetic MgO, NaOH and  $\text{Ca}(\text{OH})_2$

TABLE I  
ELEMENTAL COMPOSITION (WT%) OF RAW MAGNESITE,  
SYNTHETIC MGO, AMD REACTED: SYNTHETIC MGO, NAOH AND  
 $\text{CA}(\text{OH})_2$

Element Composition (%)	Raw Magnesite	Synthetic MgO	AMD reacted MgO	AMD reacted NaOH	AMD reacted $\text{Ca}(\text{OH})_2$
MgO	26.078	47.853	19.383	3.761	1.323
$\text{Al}_2\text{O}_3$	1.200	1.2	2.933	3.615	2.799
$\text{SiO}_2$	11.954	12.739	7.62	6.582	6.582
S	0.00	0.005	6.281	10.282	12.763
CaO	1.091	1.506	1.003	2.673	15.372
$\text{Fe}_2\text{O}_3$	1.712	1.884	20.06	41.426	16.005

As shown in TABLE I, the major constituents of the AMD reacted sludge are Si, Al, Fe, Ca, Mg, and S. These are some of the main components in acid mine drainage that are of concern. The NaOH reagent was more effective in the precipitation of iron.  $\text{Ca}(\text{OH})_2$  was effective in the precipitation of sulphate in the form S. In some cases, sulphate in AMD is reduced to produce elemental sulphur. There are several chemical processes that can be used to recover elemental sulphur from AMD. One common process is called flotation [18]. In flotation, the AMD is mixed with a chemical reagent, such as sodium ethyl xanthate, which attaches to the elemental sulphur particles. These particles then rise to the surface of the water, where they can be collected. The collected particles can then be dried and sold as elemental sulphur. Other processes that can be used to recover elemental sulphur from AMD include ion exchange and membrane filtration. Elemental sulphur has commercial value, primarily in the production of sulfuric acid

### D. FTIR Analysis

Fig. 1 is an FTIR graph of raw magnesite, synthetic MgO and sludge produced from reacting NaOH,  $\text{Ca}(\text{OH})_2$  and synthetic MgO with AMD.

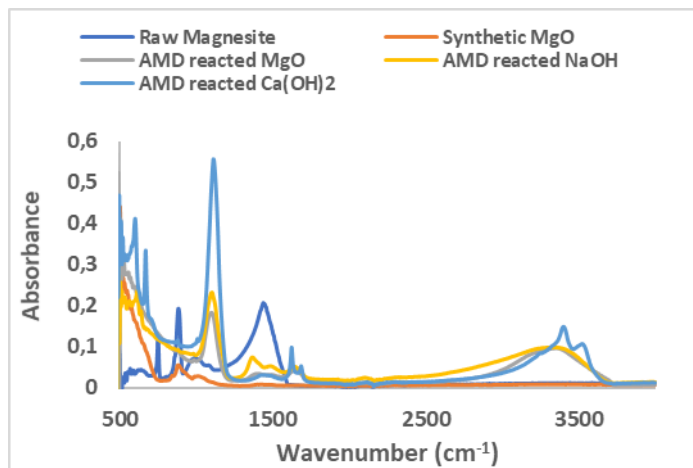


Fig. 1. FTIR analysis of Raw magnesite, synthetic MgO and sludge produced from reacting NaOH, Ca(OH)<sub>2</sub> and synthetic MgO with AMD.

As depicted in Fig. 1, distinct peaks associated with carbonate (CO<sub>3</sub>) minerals were detected at approximately 748.69, 885.37, and 1456.58 cm<sup>-1</sup> in the raw magnesite [18]. Subsequently, these peaks were observed to vanish after the calcination process on the synthetic MgO, indicating a swift release of CO<sub>2</sub> from the magnesite structures. The presence of a peak at 3672 cm<sup>-1</sup> signifies the existence of hydroxyl groups in the synthesized material, which can be attributed to the formation of MgO concurrent with the release of CO<sub>2</sub>. The changes in peak presence and position attest to the formation of MgO rendering the calcination process successful. The peak at 1103.73 cm<sup>-1</sup> can be attributed to the stretching of hydroxyl group present in the AMD reacted synthetic MgO which is an indication of a reaction between the AMD and synthetic material taking place. The vibration and bending of O-H in water molecule appeared at 3388.80 and 1652.86 cm<sup>-1</sup>. OH stretching at region 3628 – 3260 cm<sup>-1</sup> shows the presence of hydroxyl groups on the AMD reacted synthetic MgO [19]. Peaks were observed at 614.04, 1103.65, 1379.06 and 3347.68 cm<sup>-1</sup> for the AMD reacted NaOH. The peak at 614.04 cm<sup>-1</sup> can be attributed to the presence of inorganic compounds such as Fe<sup>3+</sup> and Al<sup>3+</sup>. The peak at 3347 cm<sup>-1</sup> is in the O-H stretching region, which is commonly associated with hydroxyl groups. This peak could be indicative of the presence of hydroxyl groups in compounds formed during the neutralization process [19]. The peak at 601.80 cm<sup>-1</sup> is typically associated with metal-oxygen (M-O) stretching vibrations. The peak might be indicative of the formation of metal hydroxides. It could be a signature of the Ca-O stretching vibration in calcium hydroxide (Ca(OH)<sub>2</sub>) or other metal hydroxides that might have formed as a result of the neutralization process.

#### E. Neutralization Experiment

##### Evaluation Of AMD Neutralization With Synthetic MgO

The effect of synthetic MgO dosage on the pH, EC & TDS of AMD is shown in Fig. 2 and Fig. 3 respectively.

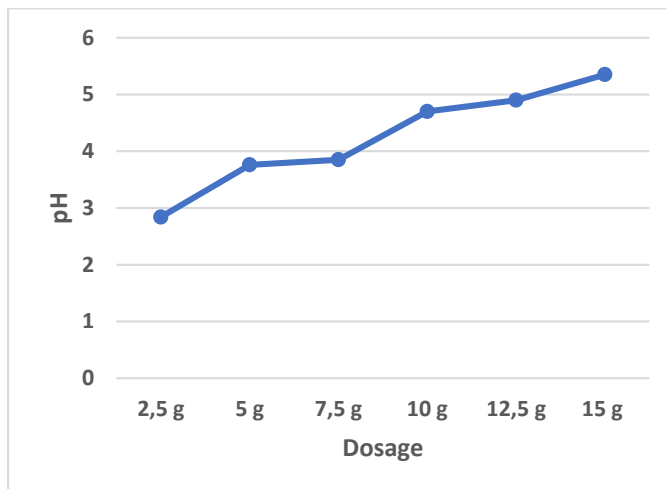


Fig. 2. The effect of synthetic MgO dosage on the pH of AMD sample.

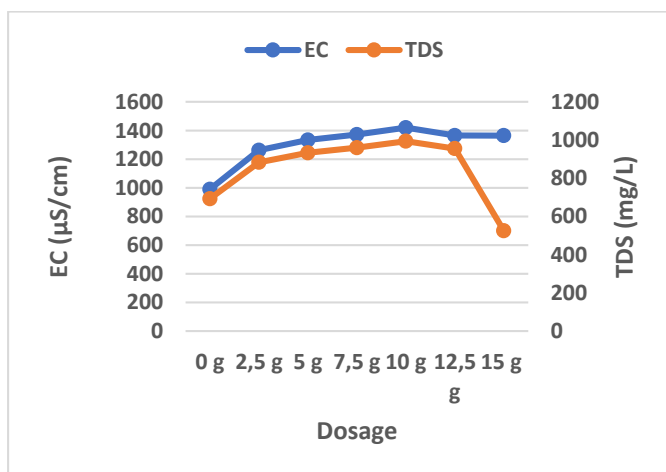


Fig.3. The effect of synthetic MgO dosage on the EC and TDS of AMD sample.

Synthetic MgO elevated the AMD pH from 2.34 to a maximum of 5.35 with a dosage of 15 g. Based on Fig.2, a dosage that is above 15 grams is required to effectively elevate the pH to a range between 6 and 8 to be able to meet SANS241 standards. Magnesium oxide can increase the total dissolved solids and electrical conductivity when used to neutralize acid mine drainage because it dissolves in water to form magnesium ions (Mg<sup>2+</sup>) [20]. These ions increase the TDS and EC because they are positively charged and can conduct electricity. In addition, MgO can react with other compounds in the water, such as carbon dioxide, to form additional minerals that also increase the TDS and EC.

#### F. Evaluation Of Neutralization Of AMD With NaOH

The effect of NaOH dosage on the pH, EC & TDS of AMD is shown in Fig. 4 and Fig. 5 respectively.

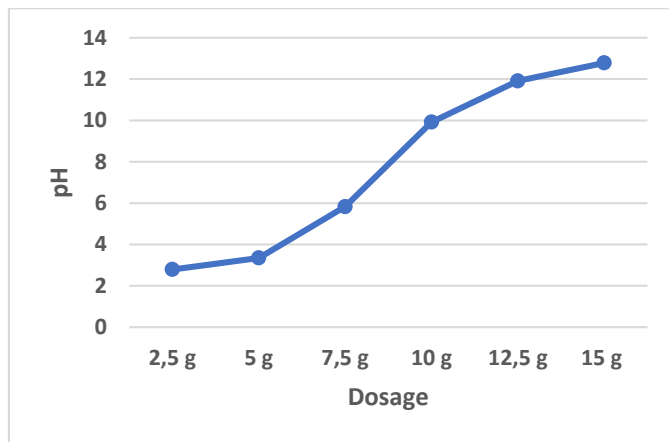


Fig. 4. The effect of NaOH dosage on the pH of AMD sample.

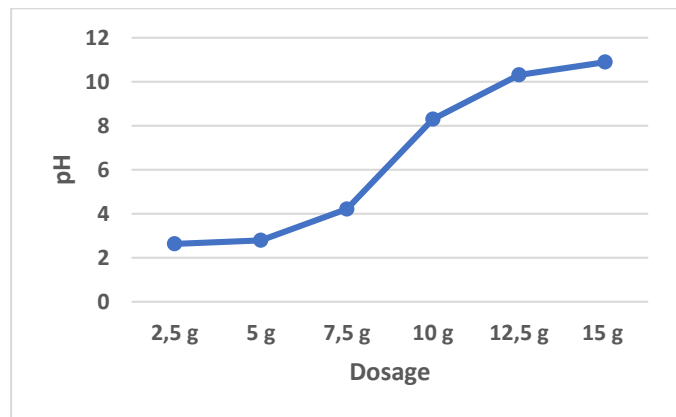


Fig. 6. The effects of  $\text{Ca}(\text{OH})_2$  dosage on the pH of AMD sample.

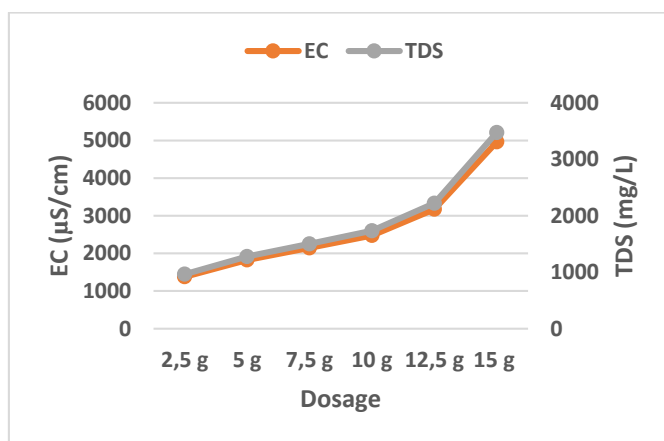


Fig. 5. The effect of NaOH dosage on the EC and TDS of AMD sample.

NaOH was able to effectively neutralize the AMD by elevating the pH to 12.78 with a dosage of 15 g. The pH of 6 to 8 is observed between the dosages of 7.5 g and 9 g. This pH is in accordance with SANS241 standards and is used as a determining factor for the extent of neutralization. The NaOH works by reacting with the hydrogen ions in the water when added, forming water molecules and sodium ions. This reaction increases the pH of the water, effectively neutralizing the acidity. TDS and EC are observed to increase in Fig.5 when NaOH was used to treat AMD. This occurs because the reaction forms sodium ions, which increase the dissolved solids in the water. In addition, the ions can carry an electric charge, which increases the conductivity [21]. Although NaOH is effective in neutralizing the acidity of AMD, it does have the side effect of increasing TDS and EC in treated water.

#### G. Evaluation Of Neutralization Of AMD With $\text{Ca}(\text{OH})_2$

The effect of  $\text{Ca}(\text{OH})_2$  dosage on the pH, EC & TDS of AMD is shown in Fig. 6 and Fig. 7 respectively.

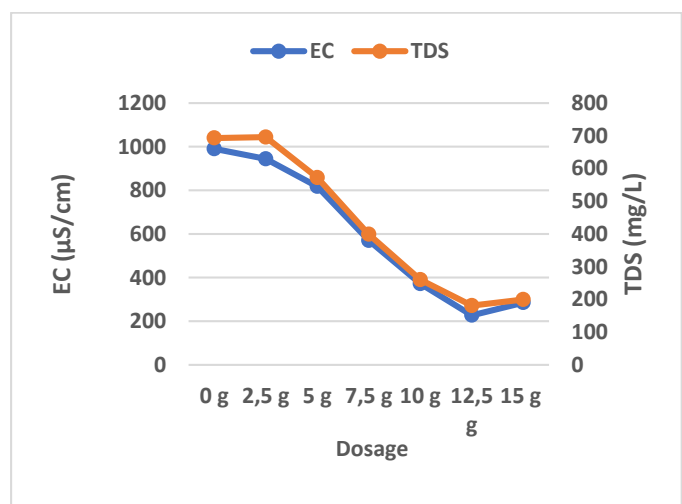


Fig. 7. The effects of  $\text{Ca}(\text{OH})_2$  dosage on the EC and TDS of AMD sample.

$\text{Ca}(\text{OH})_2$  was able to effectively neutralize the AMD sample by elevating the pH from 2.34 to a maximum of 10.89 with a dosage of 15 g. Based on the graph a dosage of 8 g to 10 g is adequate to elevate the pH to a desired value of 6 to 8 in accordance to SANS241. The hydroxide ions ( $\text{OH}^-$ ) from the  $\text{Ca}(\text{OH})_2$  react with the hydrogen ions ( $\text{H}^+$ ) in the AMD, reducing the acidity of the water. The reaction produces calcium ions ( $\text{Ca}^{2+}$ ) and water ( $\text{H}_2\text{O}$ ). In addition, some of the calcium ions combine with carbonate ions ( $\text{CO}_3^{2-}$ ) to form calcium carbonate ( $\text{CaCO}_3$ ) which can further neutralize the acidity [22].

TDS and EC are observed to decrease in Fig. 7 when  $\text{Ca}(\text{OH})_2$  is used to neutralize AMD which maybe as a result of calcium ions ( $\text{Ca}^{2+}$ ) not carrying a charge that contributes to EC, unlike the negatively charged hydroxide ions ( $\text{OH}^-$ ) that result from NaOH neutralization. In addition, calcium carbonate ( $\text{CaCO}_3$ ), which is formed as a by-product of  $\text{Ca}(\text{OH})_2$  neutralization, can precipitate out of solution, and settle to the bottom [23]. This also reduces the quantity of dissolved solids in the water.

From these comparative results, it can be concluded that all the tested products managed to increase the pH of AMD and attenuate the levels of inorganic contaminants.  $\text{Ca}(\text{OH})_2$  and  $\text{NaOH}$  were highly effective in neutralizing the AMD whilst the synthetic  $\text{MgO}$  indicated to be not as effective by failing to raise the pH within permissible limits according to SANS241.

Sulphate recovery from AMD is an important part of AMD treatment process as it is a key factor in the acidity of the AMD. Sulphate recovery not only is part of AMD remediation, it has a potential economic viability if the process yields substantial results. Sulphate recovery results indicated sulphate that could be recovered as S in the following order:  $\text{Ca}(\text{OH})_2$  (12.76%)  $\geq$   $\text{NaOH}$  (10.28%)  $\geq$   $\text{MgO}$  (6.28%). An integrated approach such as incorporating the use of alkaline reagents and electrocoagulation can be advantageous in removing the remaining dissolved sulphates.

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