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GRAVIMETRIC DETERMINATION OF SULPHATE IN SOME SELECTED INDUSTRIAL EFFLUENTS

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ABSTRACT

Gravimetric Analysis as one of the Analytical method for the Quantitative Determination of sulphate ions from aqueous media being the most simplest, rapid and low-cost method. This study evaluates some selected parameters and the results were compared with some regulatory standard; Federal Ministry of Environment (FME) in Nigeria and the World Health Organization. The Concentration of the selected industrial effluent was determined using the aforementioned method. The values of Density, TSS and TS were in the range of 0.950 g/ml – 0.976 g/ml, 67 ppm – 2051 ppm and 728 mg/L- 2700 mg/L respectively. While the percentage of sulphate obtained from each sample were 14.65%, 7.35%, 6.19, 17.93% and 29.01% for NASCO, DANA, KRPC, NILEST and KCV respectively. A new technological treatment process is recommended for these industries to provide good ways on waste management that are economically viable.

Keywords: Sulphate Ion, Industrial Effluent and Gravimetric method.

INTRODUCTION

Sulphate ions is a polyatomic anion with the empirical formula SO_4^{2-} which are used in many industrial processes, both as raw materials (mining and metallurgy industrial activities) and as auxiliary materials (in electroplating industry, chemical industry, building materials industry, etc.) (Ercikdi *et al*., 2009). Therefore, in many of these industrial processes, the concentration of sulphate ions is an important parameter that must be determined easily and with high precision (Caceres *et al*., 2015). On the other hand, wastewaters resulting from such industrial activities also contain sulphate ions in different concentrations, and their determination is of real interest, technological and environmental point of view.

Sulphate, however, is present as part of the sulfur cycle in the atmosphere (ECL, 2009). Both atmospheric and terrestrial processes are present in the sulphur cycle. The cycle starts with the erosion of sulfate (evaporates) and sulfide containing rocks and minerals within terrestrial processes. This is a process that releases the stored sulphur into the environment. The sulpur then comes into contact with the air and is converted into sulphate (SO_4^{2-}). The sulphate is taken up and converted into organosulpur compounds by plants and microorganism. The organic sulphur that carries the sulphur into the food chain is consumed by plants and animals.

As plants and organisms die, some of the sulphur is released back into the environment as sulphate. The breakdown of vegetation in swamps and tidal flats releases hydrogen sulphide (H_2S) gas into the environment. Hydrogen sulphide converts to sulphate in aqueous environments. The other major natural contributor to the sulphur budget in the environment is volcanoes. The fumarolic activity of volcanoes introduces SO_x (SO_2, SO_3) and hydrogen sulphide gases to the atmosphere. These gases eventually convert to sulphate ions in water and precipitate as alkali sulphate salts.

Various forms of sulphur-containing chemicals are commonly used in the production of fertilisers, fungicides, algae control and insecticides, as well as in the production of glass, paper and wood pulp industries, soap and detergent, in medicine, in hide-skin processing and water treatment (WHO, 2004). One-third of all sulphur that reaches the atmosphere, including 90% of sulphur dioxide, stems from human activities. These include industries such as mining and mineral processing, agriculture, and paper and pulp. Sulphur enters the atmosphere primarily through the burning of fossil fuels, incineration of refuse, and the processing of minerals and metals. The burning of coal and petroleum by industry and power plants creates large amounts of toxic gases such as sulphur dioxide (SO_2) and sulphur trioxide (SO_3) which react with atmospheric water and oxygen to produce sulphuric acid (H_2SO_4) which lowers the pH of soils and raises sulphate levels of surface waters.

The excessive levels of these pollutants in the environment are causing a lot of damages to human and animal health, plants and trees including tropical rainforest locations (Tropical Rainforest Animals, 2008). The indiscriminate handling and release of industrial effluents or wastewater into surrounding terrestrial or aquatic habitat has been implicated as one of the major sources of environmental pollution. According to the United States Environmental Protection Agency (USEPA, 2006), effluent is defined as wastewater treated or untreated that flows out of a treatment plant, sewer or industrial outfall. Industrial wastewater varies both inflow and in pollution strength and can therefore be categorized as extremely complex mixtures containing inorganic and organic compounds (Nielsen and Rank, 1994). In Nigeria, over 80% of the industries release solid wastes, liquid effluents and gaseous emissions directly into the environment without any treatment (Federal Ministry of Water Resources [FEPA], 1991).

Industrial effluents carry various types of contaminants such as; metals, organic and inorganic matter, Polycyclic Aromatic Hydrocarbons (PAHs), microorganisms, etc. into the environment especially the aquatic systems (Ho *et al*., 2012). The complexity of industrial effluents as a result of its various contaminants makes it quite impossible to carry out a hazard assessment based on chemical analysis (El-Shahaby *et al*., 2003). Due to the versatile use of sulphate in almost all the industries as an industrial reagent, it is believed that the effluent of such industries must contain some reasonable amount of sulphate. It is important to view the possibility to minimize it is an effect on the environment through recycling and recovery so that it can be used in the same industry or another industry and can also be used for other purposes.

SAMPLE COLLECTION

The Effluent Discharged by the industries through gutters was obtained from five selected industries namely, (NASCO Company Jos Plateau State, DANA Steel Company Katsina State, KRPC Kaduna State, NILEST Zaria Kaduna State and KCV Katsina State). The samples were collected in a clean plastic bottle which had been carefully rinsed with distilled water and kept for the experiment.

PROCEDURE

Gravimetric Determination of Sulphate

The prepared sample was stirred thoroughly to ensure homogenous mixture, 20 ml from this solution was measured with measuring cylinder and transferred into the beaker (250 ml) and 150 ml of distilled water was poured, two drops of Hydrocloric acid was added. The mixture was heated to boiling point in a hot solution. 30 ml of Barium Cloride solution (5%) was added slowly through the centre of the solution using a pipette, the solution was stirred gently with glass rod while adding the Bariumchloride. 5 ml of Silvernitrate was added to the precipitation medium. The hot solution was placed in a water bath at low temperature for an hour to allow for complete precipitation; the solution was gently decanted through filter paper of known weight and kept overnight for oven drying.

Equation of the Reaction:

$$SO_4^{2-} + BaCl_2 \longrightarrow BaSO_4 + 2Cl^-$$

Gravimetric Factor (GF)

$$GF = \frac{\text{Molecular weight of Sought Substance}}{\text{Molecular weight of a Compound}}$$

Molecular weight of Sulphate = 32+ (16×4) = 96

Molecular weight of $BaSO_4$ = 137 + 32 + (16×4) = 233

GF= 96/233 = 0.4125

DETERMINATION OF DENSITY OF A SOLUTION

50 ml (empty) dry and clean beaker was weighted and recorded, the sample solution was measured from the same beaker (of known weight) weight of beaker plus sample was measured and recorded. The weight of sample is determine by subtracting the weight of empty beaker from the weight of a beaker with solution. The density was found by dividing mass of the solution by the sample volume.

Density ρ = Mass of sample in Gram/ Sample volume in millitre

$$\rho = \frac{m(g)}{V(ml)}$$

TOTAL SUSPENDED SOLID (TSS)

Total Suspended Solid is a measurement of the organic or inorganic material larger than a specific size repeated in mg/l. 20 ml of each sample was filtered through a filter paper that is initially weighted. The filter was dried in the oven at temperature of 105^{0}C overnight. The filter was removed and allowed to cool to room temperature in desiccators and weighted to constant weight. The increase in the mass of the dry filter paper was later recorded and used for calculating TSS.

TSS Is calculated using the formula

$$\text{Total Suspended Solids (mgL}^{-1}\text{)} = \frac{(A\text{-}B) \times 1000}{\text{Volume of Sample (ml)}}$$

TOTAL SOLID

20 ml of the sample was collected into an evaporating dish which was weighted already. The sample was allowed to evaporate to dryness over a hot plate. The residue was then allowed to cool at room temperature and weight until a constant

weight is obtained. The weight of the empty dish was subtracted from the weight of the dish plus the residue to obtain the weight of the solid sample.

TS is calculated with the formula.

$$\text{Total Solids } (mgL^{-1}) = \frac{(A\text{-}B) \times 1000}{\text{The Volume of Sample (ml)}}$$

Where A = weight of dried residue + Evaporating dish (mg), B= weight of the evaporating dish (mg)

RECOVERY TEST

0.5 g of anhydrous sodium sulphate was weight and put in 250 ml beaker, 20 ml of distilled water measured and poured and 20 ml of effluent was also measured and pour inside the same beaker. About 100 ml of distilled water was then added to the mixture, it was then stirred thoroughly. The solution was then heated to boiling and a gravimetric method was to recover the sulphate using Bariumchloride.

Equation of the reaction

$$Na_2SO_4 \xrightarrow{\text{Dissociation}} 2Na^+ + SO_4^{2-}$$

$$BaCl_2 + SO_4^{2-} \longrightarrow BaSO_4 + Cl^-$$

RESULT

DENSITY

Table 1. Density of the Solution of each Effluent

Density of the solution obtained g/ml	Effluent
0.961 g/ml	NASCO
0.974 g/ml	DANA
0.970 g/ml	KRPC
0.976 g/ml	NILEST
0.950 g/ml	KCV

Density is simply the amount of substance in kilogram per unit volume in liter knowing the density of the solution could help in determine the percentage of sulphate in each sample. The mean value obtained of the waste water for the five industries were 0.961 g/ml, 0.974 g/ml, 0.970 g/ml, 0.976 g/ml, and 0.950 g/ml for NASCO, DANA, KRPC, NILEST and KCV respectively, where NILEST recorded the highest value of 0.976 g/ml while KCV has the lowest value of density 0.950 g/ml (Table 1).

TOTAL SUSPENDED SOLID

Table 2. TSS of the Solution of each Effluent

Total Suspended Solid (mg/L)	Effluent
2051 ppm	NASCO
760 ppm	DANA
67 ppm	KRPC
775 ppm	NILEST
164 ppm	KCV
WHO Discharge Standard	**60 mg/L**
FME Discharge standard	**0.75 mg/L**

TSS is a measure of particulate matter suspended in water; it is used to describe the extent of pollution in wastewater. In addition ,TSS serve as a good indicator for the turbidity of the water (21) The TSS values for the effluent obtained from the five industries were, 2051 ppm, 760 ppm, 67 ppm, 775 ppm, and 164 ppm respectively (Table 2). This indicates that the industries were efficient in reducing the level of TSS but this elimination was not sufficient as the TSS in the effluent from all the selected industries except KRPC which did not comply with WHO (60 mg/L) and FME (0.73 mg/L) respectively. The high level in TSS could be attributed as the nature of activities taking place in the industry.

TOTAL SOLID

Table 3. TS of the Solution of each Effluent

Total Solid (mg/L)	Effluent
2700 mg/L	NASCO
728 mg/L	DANA
942 mg/L	KRPC
2422 mg/L	NILEST
860 mg/L	KCV
FME Discharge standard	**1500 mg/L**

Solids in effluent could either be organic or inorganic in nature, such solids may be present in dissolved or suspended form (44) The TS values recorded from the five industries were 2700 mg/L, 728 mg/L, 942 mg/L, 2422 mg/L and 860 mg/L respectively(Table 3). However three industries; DANA, KCV, and KRPC recorded the lowest value of 728 mg/L, 860 mg/L and 942 mg/L respectively were below the permissible requirement of 1500 mg/L for TS required for ground water to be used for domestic purpose while NASCO and NILEST were ineffective with the required standard by FME.

RECOVERY TEST AND PERCENTAGE OF SULPHATE IN EACH EFFLUENT

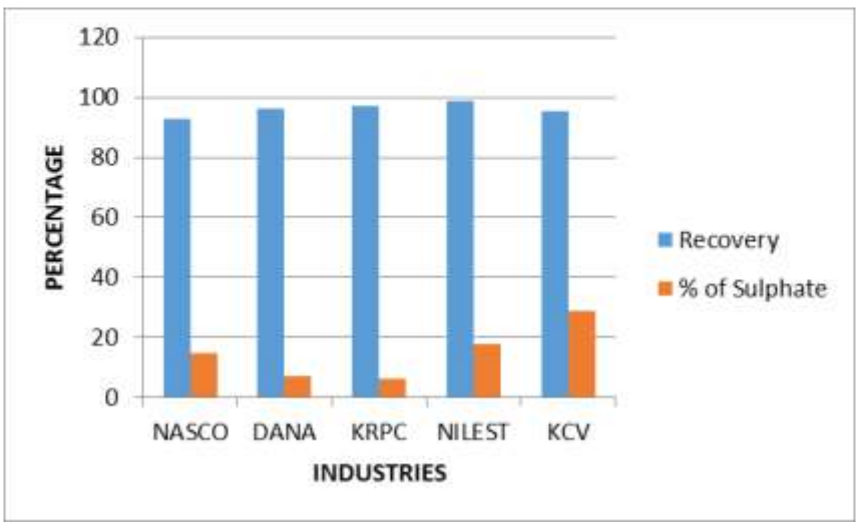

Figure 1. Recovery test and percentage of sulphate in each effluent

Resource Recovery is the systematic division of waste which was intended for disposal for a specific next use, it is the processing of recyclables to extract or to recover materials and resources. These activities are performed at a resource facility. Resource Recovery is not only environmentally important but it is also cost-effective. The recovery test values for the five industries were 92.96 %, 96.14 %, 97.15 %, 98.79 % and 95.53 % respectively (figure 1) with NILEST recorded the highest value. However on economic perfection the result pointed that in every 20 ml of KCV wastewater there is 29.01 % of sulphate is present, this is as a result of the nature of activities in the industry which implies the industry is in much use of sulphate containing compounds in their course of production.

CONCLUSION:

Environmental Pollution is a major problem in many developed and developing countries around the world where nations are striving to arrive at an effective regulatory regime to control the discharge of industrial effluents into their ecosystem. The obtained experimental results have shown that Gravimetric method is one of the simplest, rapid and low-cost method for sulphate determination in industrial effluent, the sulphate was found in appreciable concentration which indicates that there is need for these industries to create an effluent treatment plant to enable recovery and reuse of sulphate into different purposes thereby averting its harmful effect on the environment.

However, the result revealed the high level of effluent introduced into the environment; the result also proved non-compliance to various regulatory standards as some of the physicochemical parameters investigated exceeded the levels recommended for discharge. Hence a new technological treatment process is therefore recommended for these industries to provide good ways on waste management that are economically viable and environmentally friendly.

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