## BINDURA UNIVERSITY OF SCIENCE EDUCATION FACULTY OF CHEMISTRY CHEMISTRY DEPARTMENT



# PURIFICATION OF ANIONIC SURFACTANT CONTAMINATED WATER USING Mg/Fe LAYERED DOUBLE HYDROXIDE.

#### DERECK KUDAKWASHE JAMARI

#### B1541921

### SUPERVISOR: Dr. C. MACHINGAUTA

DISSERTATION SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE BACHELOR OF SCIENCE HONORS DEGREE IN CHEMICAL TECHNOLOGY

#### **JUNE 2019**

Approval Form

The undersigned certify that they have supervised, read and recommend to the Bindura University of Science Education for acceptance of a research project entitled:

## PURIFICATION OF ANIONIC SURFACTACNT CONTAMINATED WATER USING Mg/Fe LAYERED DOUBLE HYDROXIDE.

Submitted by JAMARI DERECK KUDAKWASHE

In partial fulfilment of the requirements for the BACHELOR OF SCIENCE HONOURS

DEGREE IN CHEMICAL TECHNOLOGY

...../...../...../

(Signature of Student) Date

(Signature of Supervisor) Date

(Signature of the Chairperson) Date

#### ABSTRACT

The properties of LDHs have proved useful in the elimination of poisonous anions in water bodies and environment. Credit id given to their anion exchange properties and reformation effect. This thesis explores the synthesis, ability of anion sorption by reformation effect and characterization of the materials. The hydrotalcite-like material was successfully synthesized from magnesium and iron salts by co-precipitation. The resulting products were characterized by FTIR to confirm the functional groups present. The synthesized LDH was thermally decomposed at 300°C, 450°C and 500°C. The obtained mixed metal oxide was immersed in a solution containing DS anions. Preliminary analysis of the elimination of DS by the Mg/Fe LDH were carried out and characterized using FTIR and UV- vis. From UV analysis, the changes in concentration of DS with time was used to calculate the efficiency of the mixed metal oxide obtained at respective temperatures. At 300°C efficiency was 48.6%, 59.1% and 90.2% at 500°C and 450°C respectively. The LDH material proved to be a suitable adsorbent.

#### ACKNOWLEDGEMENTS

First and foremost, my sincere appreciation goes to my academic supervisor Dr. C. Machingauta for his time, encouragement and unwavering support up to the time of this research completion, I could not have done much without him. I also extend my heartfelt gratitude to the Bindura University Lab technician, Mr Chayamiti for his support throughout this research. A special thank you goes to my parents for all their financial and moral support and their undying love that gave me the confidence to continue with and complete my work, through the most difficult of times. Above all, I give thanks to the Lord Almighty for the gift of life, health, unconditional love and strength that took me through my education.

#### DEDICATION

This work is dedicated to my family and friends, above all, my Lord Jesus Christ

Table of Contents 1.1 BACKGROUND	
1.2 STATEMENT OF THE PROBLEM	
1.3 JUSTIFICATION	
1.4 AIM OF THE RESEARCH	
1.5 SPECIFIC OBJECTIVES	7
1.6 SCOPE OF STUDY	
CHAPTER 2: LETIRATURE REVIEW	
2.1 SURFACTANTS	
2.2 EFFECTS OF SURFACTANTS	9
2.3 LAYERED DOUBLE HYDROXIDES	
2.4 RELATED WORK DONE	
3.1 REAGENTS AND APPARATUS	
3.2 PREPARATION OF Mg/Fe LDH	
3.3 REFORMATION OF THE LDH	
3.4 FTIR ANALYSIS	
3.7 UV-VIS ANALYSIS	
CHAPTER 4: RESULTS	
4.1 FTIR ANALYSIS	
4.2 UV ANALYSIS	20
4.3 EFFICIENCY CALCULATIONS	21
4.4 DISCUSSION	
4.4.1 FTIR ANALYSIS	
4.4.2 UV- VIS ANALYSIS	
CHAPTER 5: CONCLUSION	

APPENDIX	
Bibliography	

## LIST OF FIGURES

Figure 1: <i>the structure of a surfactant</i>	
Figure 2: <i>structures of micelle (a) and reverse micelle (b)</i>	11
Figure 3: <i>the structure of dodecyl sulphate</i>	
Figure 4: <i>set up for LDHs synthesis</i>	
Figure 5: characterization of LDH	
Figure 6: characterization of LDH-DS	
Figure 7: (a) sodium dodecyl scan curve, (b),(c),(d) and (e) are changes in SDS c	oncentration
with time.	

## 1.1 BACKGROUND

Climatic and seasonal changes together with the growing population of our globe have increased the demand of water. To trounce this problem and meet the demand, recycling of waste water is being used. However, some contaminants like surfactants are being recycled as well as they are not affected by chlorination. They are poisonous to aquatic life as they mucus protective layers that protect fish and other aquatic animals from bacteria and parasites (C. L. Yuan and M. X. Fan, 2014). Increases in surfactant concentrations in water bodies destroy fish grills and eventually kill them. Fish eggs are also destroyed (PY Wang and J Wang, 2007). Surfactants deplete oxygen levels in water bodies. This results in suffocation of aquatic animals and plants like algae (C. L. Yuan and M. X. Fan, 2014). They are also reported to lower the surface tension of water. Surfactants are subjects to bio-accumulation. When they are absorbed by aquatic animals like fish their concentrations intensifies (C. L. Yuan and M. X. Fan, 2014). Water treatment departments are focused on the on removal of solid waste and germs from waste water for drinking only. Other than this purpose the contaminated water is not being treated. The discovery of LDHs have paved a way to multiple researches on their ability of ion exchange (Frederick .L, 2012), intercalation capabilities (LUMBIDZANI M, 2009), controlled release of nutrients, ref and fire retardancy (Frederick .L, 2012). In this work, the reformation capability of LDHs will be used to eliminate anionic surfactants from waste water and water bodies.

Memory effect is also referred to as calcination or rehydration process (Rocha J, 1999). It is when there is intercalation of anions to the calcined LDH in the aqueous environment to regenerate the desired LDH (Machingauta C, 2013). When an LDH material is subjected to temperatures between 400-450 °C, its lattice structure collapses into amorphous mixed oxide  $M^{II} M^{III}_2 O_4$  and a divalent metal oxide (Bostrom T E, 2005). When the mixed oxide is exposed to hydroxyl anions and the desired anions, the LDH lattice is reformed by the sorption of the anions. Carbonate anions had proved to suit the role so well as they can readily decompose releasing carbon dioxide and water vapor which are both stable and non-toxic. Decomposition temperatures are used, there will be partial decomposition of the LDH into structures that cannot undergo reformation (Ogawa M, 2000). This will result in the reduced anion sorption efficiency as the adsorbent surface will be no longer usable (Kura A U, Hussein M Z, 2014). Memory effect of Mg/Fe LDH will be used in this research to remove the anionic surfactants contaminants in water.

## 1.2 STATEMENT OF THE PROBLEM

Surfactants play a crucial role in detergent industry. However, their disposal to water bodies has fuelled up environmental pollution. They are causing water bodies to suffer from foam; they are also poisonous to aquatic life as they destroy protective mucus layers and grills. They are being recycled in drinking water resulting in surfactant dysfunction, lung cancer.

## **1.3 JUSTIFICATION**

Removal of surfactants in laundry and industrial waste water will reduce environmental contamination and will rid the problems posed by surfactants in water bodies. The risk of surfactants being recycled in drinking water will be eliminated.

## 1.4 AIM OF THE RESEARCH

To use the synthesise a mixed metal oxide and use it to remove anionic surfactants from contaminated water bodies using the memory effect of LDHs.

## 1.5 SPECIFIC OBJECTIVES

To synthesise Mg/Fe LDH by co-precipitation method.

To characterise the synthesised LDH

To convert the synthesised LDH to mixed metal oxides by thermal decomposition.

To remove the anionic surfactant SDS in water using the mixed metal oxide

To check whether the SDS anion have been adsorbed by the mixed oxide to form LDH material.

To determine the efficiency of removal of SDS by reformation of LDH.

## 1.6 SCOPE OF STUDY

The study will cover from the synthesis of Mg/Fe LDH using co-precipitation method. The

synthesised carbonate LDH will then be calcined on a temperature range of 300-500°C to allow decarbonation and dehydration. The mixed metal oxides obtained from thermal decomposition will then be allowed to regenerate in the presence of SDS anions and water. FTIR was used for characterisation and MB spectrophotometric method was used to determine the efficiency of SDS elimination.

## **CHAPTER 2: LETIRATURE REVIEW**

#### 2.1 SURFACTANTS

Surfactants are surface active organic compounds that form microstructures when combined with water or other polar solvents. These microstructures include micelles and bilayers (Texter 1999). They pose a non-polar hydrophobic tail of alkychain and a polar hydrophilic head. When immersed in water, the water loving head interacts with water by dipole-dipole interactions (Tadros ,2005).



#### Figure 1: the structure of a surfactant

The alkyl chain tail has a high affinity for non- polar solvents, so to avoid interactions with polar solvents, the surfactant molecule form micelles. (Texter, 1999). In polar solvents micelles are formed whilst in non-polar solvents a reverse micelle is formed.



Figure 2: structures of micelle (a) and reverse micelle (b)

When immersed in solvents, surfactants reduce surface and interfacial tensions as they adsorb on the surfaces and interfaces. There are four classes of surfactants which are anionic, cationic, nonionic and amphoteric. This thesis will focus on anionic surfactants.

Anionic surfactants have a negative charge on the hydrophilic head which enables them to bind to a positively charged particle / compound. They include sulfonic acid salts, alkyl benzene sulfonates, carboxylic acid salts and soaps among others. Anionic surfactants are generally used as detergents, soaps, foaming agents, antistatic agents and emulsifiers. This thesis will use Sodium Dodecyl Sulfate.



Figure 3: the structure of dodecyl sulphate

#### **2.2 EFFECTS OF SURFACTANTS**

Disposal of surfactants in water bodies is causing serious harm on the environment. High concentrations of surfactants in water bodies depletes its oxygen levels. This affect the growth of algae and other microorganisms thus reducing the primary productivity. This disrupts the food chain of aquatic life. Acute poisoning of surfactants results in membrane permeability increase. Hence the material exosmotic and cell structure disintegrates. Foaming of surfactants block sunlight from reaching aquatic plants thus reducing primary productivity of water bodies (Zhao, 2001). Surfactants have a poisoning effect to aquatic animals. They penetrate into gills, blood, pancreas, liver, kidneys and produce a toxic effect. Fish easily absorb surfactants by body and gills. High concentration of surfactants kills fish and its eggs. When fish is exposed to surfactants, its serum transaminase and alkaline acid phosphate activity increases. If the contaminated fish is ingested by humans, it has an inhibitory effect to various enzymes in the human body (Zhao, 2001).

Surfactants pollutes water bodies and lead to various problems. If the concentration reaches 1 mg/L, foams are formed. Combination of foam form an insulation layer that weaken the interaction between the water body and the atmosphere. This results in depletion of dissolved oxygen levels. Surfactants also reduces the water surface tension. They kill the microorganisms in the environment and inhibits the decomposition of other toxic substances (Wang P.Y, 2007).

Long term use of surfactants detergents causes' skin irritation. Sodium dodecyl benzene sulphate penetrates into the body through the skin and cause narrowing of the respiratory system. (M.A Lewis, 1990).Surfactants interrupts the enzyme activity of the body. Thus disrupting the body's normal physiological function. They are also difficulty to degrade when they accumulate into the body (T.M Schimitt, 2001).

#### 2.3 LAYERED DOUBLE HYDROXIDES

They are also referred to as anionic clays or hydrotalcite like materials. They are synthesized by the combination of divalent and trivalent actions (Tong , D.S, 2010). Divalent actions include Magnesium and zinc whilst trivalent examples are aluminum and iron. They are denoted by the general formula:

 $[M^{2+}_{1-x}\;M^{3+}_x\;(OH)]^{X+}\;[\,A^{n-}\,]x/n.\,mH2O$ 

#### Equation 1: general LDHs formula

where  $M^{2+}$  and  $M^{3+}$  denotes divalent and trivalent actions respectively  $A^{n-}$  represents an exchangeable anion and mH20 the number of water molecules (Rives .V, 2001)LDHs compounds have a net positive charge which is often balanced by anions through intercalation or by attraction to negatively charged surfaces .The anion exchange capability of LDHs have drawn much attention to science and technology .Recent researches are focused on the removal of anionic contaminants from water and controlled release of nutrients and chemicals .The anion exchange and Reformation effect of LDHs enables them to remove anions from water .These properties makes them attractive option for water purification .However , LDHs are not selective , anions with strong affinity will adsorb first on their surfaces . Xiang et al. (2009) reported that LDHs can be used to make carbon nanotubes. LDHs had proved to be a better way of removing anion contaminants in water due to their low cost technology which makes them a suitable option for developing countries. They are also easily removed from water by filtration as they form precipitates.

There are several methods that have been discovered for synthesis of LDHs. These methods include Sol gel method (Frederick .L, 2012), urea method (C. L. Yuan and M. X. Fan, 2014), co- precipitation (Frederick .L, 2012) and microwave irradiation (Othman M.R, 2009). This work focuses on elimination and regeneration because it is not complicated, cheap and do not involve volatile solvents. The divalent and trivalent action is mixed in the desired stoichiometric ratio. The mixture is added to caustic solution with interlayer anions of desire. A precipitate is formed and is thermally activated. Carbonate intercalation to LDHs deprives anion exchange ability owing to the strong affinity they exhibit for the carbonate anion (Miyata .S, 1983). Thermal Activation eliminates the carbonate anion and water along it, leading in the synthesis of mixed metal oxide (Erickson K.L, 2004). By subjecting the mixed oxide compound to a source of both water and anions, the LDH lattice is reformed by the sorption of anions and water. This is termed reformation / memory effect (Rives .V, 2001).



#### Equation 2 : Memory effect pathway (Bera, P., 2000)

Memory effect is the ability of the thermally decomposed LDH to regenerate to LDH when exposed to anion source in aqueous environment. When the LDH is exposed to temperatures between 25 -200°C water molecules are eliminated. When exposed to temperature between 200-400°C, there is decarboxylation and hydroxylation is completed. At temperatures between 400-500°C there is calcination of the LDH into mixed oxides. Mg is chosen because of its basic properties, whereas iron was selected due to its high Fischer –Tosh activity, redox properties and the ability to form specific sites in the LDH structure required for catalytic application.



Magnetic stirring plate

#### Figure 4: set up for LDHs synthesis

The anion exchange and reformation property of LDHs enables one to remove anionic contaminants in water and other solvent (Miyata .S, 1983). In reformation, a thermally activated LDH is used to remove the interlayer water and anions to form a mixed metal oxide (Erickson K.L, 2004). Introduction of the mixed oxide to water containing source of anions results in the reformation of the LDH compound. This process can be referred to as memory effect (frost R.L and palmer J. S, 2009).

Sorption by reformation effect is achieved by a thermally activated LDH. Thermal activation can be done by subjecting the LDH to temperatures between 300-500°C. Thermal activation removes the interlayer anions and water to form mixed metal oxides (Erickson K.L, 2004). When the TALDH is exposed to a source of anions and water, there is reformation of the LDH structure. However, there is a risk of partial decomposition of the LDH structure to form oxides that will not undergo reformation if excessive high temperatures are used. Hence, temperature is a crucial factor to be considered.

There are several techniques that are being used to characterize LDHs and also analyses their effectiveness. These techniques include Infrared Spectroscopy (IR), Raman Spectroscopy, AAS, X-Ray Diffraction (XRD) and thermal analysis techniques like Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA).

X-RD is used to identify crystalline materials. LDHs have similar structure, the diffraction patterns portrayed by the XRD is used to identify crystalline impurities in the LDH layer.

By examination of the changes of the  $d_{(003)}$  spacing, additional information is attained. The  $d_{(003)}$  spacing relates to the distance between the cation layers. So, when there is change of the interlayer anions a change in the  $d_{(003)}$  spacing will be noted. The change will be a mark of successful change of anions into the LDH structure. XRD uses the Bragg's law:

#### $n\lambda = 2d\sin\theta$

#### Equation 3: the Bragg's law

where n is an integer,  $\lambda$  is the wavelength of x-rays, d is the interlayer spacing generating the diffraction and  $\theta$  is the diffraction angle.

In TGA, a change in mass of the LDH sample is observed when the sample is subjected to heating slowly over time. The changes in the mass of the sample with time are plotted on a graph to get the TG curve (Willard H.H, 1998). The TG curve is differentiated so that the changes in mass become clearer. However, the TG technique does not identify some reactions that do not result in the changes in mass.

FTIR is used to check for the presence or absence of intercalants in the LDH layer. It is also used to confirm if the LDH structure obtained matches with the expected one. It analyses the molecules by analysis of vibrational changes from the excited molecules. SEM is used to study the shape and aggregation of the LDHs crystallites formed. The difference in the shape and aggregation is used to denote the intermolecular interactions. AAS is used for quantitative determination of the ratio of metal elements in the synthesized LDH. The ratio can be determined by the determination of the concentration of the analyte in the samples. A calibration curve is obtained by running a series of calibration standards through the instrument and plotting the absorption versus the concentration of the standards.

(Costa, F. R., and Abdel-Goad, M.,, 2005), reported that LDHs can be used as flame retardants. LDHs are being used to make Nano composites with improved strength and heat resistance (Fischer, H., 2003). They have also found use in the polymer technology, where they are used as PVC stabilizers and hydrogen scavengers. LDHs are now being used in medicinal applications as smart delivery system (Trikeriotis, M. and Ghanotakis, D. F, 2007). They are used to encapsulate drugs to improve stability, storage and mask unpleasant taste. LDHs are also being used as carriers of nutrients, pesticides and deliver them to their specific target (Wang, B., Zhang, H., Evans, D. G. and Duan, X., 2005). They are also used in environmental remediation, where they are used for sorption of toxic organic compounds and as adsorbents for removal of water contaminants (You, Y., Zhao, H., 2002). LDHs are also used in separation by utilizing their membrane - like nature (Newman, S. P. and Jones, W., 1998). They are also used as thickeners in paint industry. (Venugopal, B. R., Shivakumara, G., 2006)

#### 2.4 RELATED WORK DONE

Water pollution is a global sensitive problem. Several works are being carried out in effort of water purification. (Kulyukhin S.A, 2007), reported the removal of iodide from water using Mg/Al LDHs. The authors proposed that  $I^-$  and  $IO_3^-$  were the major species in the contaminated water. The research reported that LDHs were unable to remove  $IO_3^-$  and  $I^-$  using anion exchange. Credit was given to the low affinity of the LDHs used to anions and steric hindrance, as well as the charge density of the LDH used. The authors proposed that thermally decomposed LDHs will be the solution to these problems and they were successful when they used them.

Another research was also reported by (Wang, B., Zhang, H., Evans, D. G. and Duan, X., 2005) on the removal of chloride from aqueous solutions by anion exchange abilities of Zn/Al LDHs. The LDHs prepared by the authors were characterized by FTIR, XRD and ICP-AES. They investigated on the effect of changing the composition of the LDHs layers. They obtained the best results by using Zn/Al LDH of a ratio of 2:1 which had sorption of 62.2% of the chloride. They also investigated on the effect of pH and proposed that high pH is unfavourable due to competition posed by the hydroxide ions.

(Das.J , 2002) also reported a successful research on the removal of phosphate using iron containing LDH. The authors proposed that excess phosphate can cause eutrophication. The research investigated on the removal of phosphate using anion exchange by iron LDHs. They used ICP-AES for the determination of phosphate removal. Triatafyllidis et al also reported a successful research on the removal of phosphate using thermally activated Mg/Fe LDH.

## **CHAPTER 3: METHODOLOGY**

This chapter comprises of the descriptions of the raw materials and experimental procedures used in the research.

#### **3.1 REAGENTS AND APPARATUS**

98% Sodium hydroxide pellets Acechem South Africa 98% Sodium carbonate Minema South Africa 98. % Ferric Nitrate Nona hydrate Glassworld South Africa 99% Magnesium nitrate Hex hydrate Glassworld South Africa Struan scientific magnetic stirrer NIOLET iS5 Fisher Thermos scientific Fourier Infrared Spectroscopy Genesys 10S UV –Vis Spectrophotometer

### 3.2 PREPARATION OF Mg/Fe LDH

43.056 g of magnesium nitrate and 23.352 g of ferric nitrate were mixed together in 100 ml deionized water in a 250 ml beaker. Thus, the ratio of Mg: Fe is 3:1 respectively. The beaker was set on a magnetic stirrer. 14.953 g of sodium carbonate and 8.134 g of sodium hydroxide were mixed together in 100 ml of deionized water, caustic solution. The caustic solution was added drop wisely into the 250 ml beaker containing magnesium nitrate and ferric nitrate under vigorous stirring. A brown precipitation was immediately formed. The precipitate was allowed to age at 60 °C in a water bath for 24 hours (Costa, F. R., and Abdel-Goad, M., 2005).

Once the precipitate had aged, it was filtered by vacuum filtration. The filtrate weighed and thermally treated in a furnace at a temperature range of 300 - 500 °C for 4 hours to rid it of the carbonate anion and water and form a mixed oxide. The formed cake was then ground into fine powder which was then characterized using FTIR which shows the functional groups in the compound.

#### **3.3 REFORMATION OF THE LDH**

30.537 g of SDS was used to prepare 15M of SDS solution. The solution was added to 50ml of acetic acid, to ionize SDS, in a beaker containing 200 ml of deionized water. 20.684g of the calcinated Mg/Fe LDH was added to the mixture slowly while stirring. An emulsion suspension was immediately formed. The suspension was labelled X and the pH was adjusted to 10.40 by addition of sodium hydroxide at 25 °C. The suspension was allowed to react at ambient temperature for 48 hours whilst adjusting pH to 10 each morning. Once the reaction time had elapsed, the product was retrieved by centrifugation at 3000 rpm for 20 mins. It was thoroughly washed four times with distilled water then rinsed with acetone to wash away organic materials. After centrifugation, the product was dried at room temperature and pressure.

#### 3.4 FTIR ANALYSIS.

Samples were ground into fine powder using a pestle and mortar. A small amount of the finely ground powder was placed on the FTIR disk for FTIR analysis. A spectrum was obtained over a range of 400- 4000 cm<sup>-1</sup> and it represents the average of 16 scans using  $4 \text{ cm}^{-1}$  resolution.

#### **3.7 UV-VIS ANALYSIS**

Serial dilutions were done to prepare different concentrations of SDS solution. 2 ml of each solution were loaded into cuvettes and scanned using Genesys 10S UV –Vis Spectrophotometer. The scan curve in fig 5 was obtained. The maximum point of the curve ,298 nm was used to prepare a calibration graph, which was used in quantitative analysis. 2 ml of each sample containing the regenerated LDH were loaded into cuvettes and the concentration of SDS in the sample was measured.



Figure 5:UV-Vis SDS scan

#### **CHAPTER 4: RESULTS**



Figure 6: FTIR spectra for (a)...... (b)...... (c) ...... (d) ......



Figure 7: characterization of LDH-DS



Figure 6: changes in SDS concentration with time.

#### **4.3 EFFICIENCY CALCULATIONS**

Table ......Showing the effect of calcination/decomposition temperature on exchange efficiency.

Calcination	%	
-------------	---	--

Temp	
300°C	48.6
450°C	90.2
500°V	59.1

#### **4.4 DISCUSSION**

#### **4.4.1 FTIR ANALYSIS**

The synthesized LDH was characterized by FTIR to confirm if the carbonate anions have been intercalated to the LDH lattice. The presences of the carbonate anions are confirmed at peaks around 1300 – 1400 cm<sup>-1</sup> (Labajos F.M and Rives V, 1992). In this work, it was confirmed by peak around 1400 cm<sup>-1</sup> on diagram in fig 6. A peak at 3369.85 cm<sup>-1</sup> was also observed. This peak represents the hydroxyl anions in the lattice. They are often found on a wavelength range of 3300-3400 cm<sup>-1</sup> (Costa, F. R., and Abdel-Goad, M.,, 2005). These peaks are the key peaks that are used to observe the changes of their respective anions concentration in the LDH. When the calcined LDH was characterized, changes in these peaks were noted. The spectra obtained when the LDH was thermally decomposed at 300°C have one peak which is the carbonate peak. The hydroxyl peak had completely disappeared. This shows that dehydroxylation had been completed. The spectra obtained when the LDH was calcined at 450°C portrays a very small peak which represent the carbonate anions. This showed that the decarboxylation process was nearly complete. The spectra did not show the OH peak; thus hydroxylation had been completed also. The spectra obtained when the LDH was calcined at 500°C have neither the carbonate nor the hydroxyl group. Thus both dehydroxylation and decarboxylation had totally completed.

Fig7 shows Spectra's of the LDH-DS of the calcined LDH at 300°, 450° and 500°C. The spectra of the calcined LDH at 300°C shows peaks at 2850 cm<sup>-1</sup> and 2960 cm<sup>-1</sup> which are a result of the C-H stretching. These peaks confirm the alky chains of the SDS anion presence as an interlayer in the LDH-DS lattice. The sulphate and sulphonated groups often portray peaks at wavelengths at 1200- 1180 cm<sup>-1</sup>. Their presence was confirmed by a shoulder peak in the spectra. The spectra also show the OH group, by exposing the calcined LDH in an aqueous medium, the OH anions were intercalated into the LDH lattice as well. The spectras of LDH-DS at both 450 and 500°C showed the presence of the sulphate and sulphonated groups and the OH group. All the spectras showed that DS and OH anions had been successful intercalated to regenerate the LDH lattice. However, these Spectra's does not give the concentrations of the DS anions

extracted from the solution. Thus UV analysis was then carried out.

#### 4.4.2 UV- VIS ANALYSIS

Fig 8 shows DS scan by UV- vis spectrophotometer. A peak at 298 nm was obtained. Thus this wavelength was used to make a calibration curve for quantitative analysis. When the samples of the LDH-DS were analyzed, the changes in the concentration with time in each sample were noted. The obtained concentration changes data in appendix from A1 to A4 was presented on graph in fig 8. From the graph, the greatest concentration change was obtained from the calcined LDH at 450°C, followed by at 500°C then lastly at 300°C. The concentration changes obtained were used to calculate the efficiency of each calcined LDH. The calcined LDH at 450°C had 90.2%, at 300°C had 48.6% and at 500°C had 59.1%. Thus the calcined LDH at 450°C had the optimum efficiency and thus is the suitable temperature for LDH calcination. The efficiency of the calcined LDH at 300°C was the least. This is because of the competition portrayed by the carbonate anions that were still present in the LDH lattice. Carbonate anions have a high affinity to the active sites than the DS anions, thus least amount of DS were intercalated. The efficiency of the calcined LDH at 500°C is greater than that at 300° but less than at 450°. At 500°, some of the LDH material had been over calcinated to very stable metal oxides and thus the regeneration ability is reduced.

#### **CHAPTER 5: CONCLUSION**

Mg/Fe LDH successfully synthesized using co-precipitation method. The synthesized LDH was characterized by FTIR. The spectra showed the presence of CO3 and water intercalation in the LDH lattice. FTIR analysis of the calcined LDH showed the success of removal of the carbonate anion from the LDH interlayers tend to have high affinity to for the carbonate anion, if calcination is not done. It would have been difficult for DS to be adsorbed to the LDH lattice. Decarboxylation and dehydroxylation occurred at temperatures between 350°-400°C.

The thermally decomposed LDH was the investigated as a potential adsorbent for the elimination of anionic surfactants in water .DS was used to represent the anionic surfactant.

The thermally activated LDH was allowed to reform its lattice in the presence of water and DS anions. The new LDH was characterized by FTIR and a new peak of DS was observed. The synthesized Mg/Fe LDH showed a great anion sorption ability. Thus a solution to the anionic surfactant contamination of the environment.

#### RECOMMENDATIONS

The LDH synthesized proved to be an answer to anionic surfactant removal. However, there are several other surfactants other than anionic ones which have not been looked on. Thus there is still room for investigation in other types of surfactants. FTIR analysis is not enough to fully characterize the synthesized LDH. Use of AAS is recommended, so as to check the ratio of the divalent to trivalent metal. Optimum efficiency can be obtained by varying the metal ratios.

#### APPENDIX

#### A1

Spectrum:	SDS .dsp
Description:	Sodium dodecyl sulphate
Operator:	BUSEUV-Vis2-PC\BUSE UV-Vis 2
Created:	5/14/2019 12:05:14 AM
Spectrophotom	eter: GENESYS 10S UV-Vis
Serial number:	2L9S191203
Firmware:	4.006
Baseline:	5/14/2019 12:05:14 AM

Type equation here.



A2: SDS concentrations in calcined LDH at 300°C

Time	Concentration mg/l
12 hrs	13.16 ± 0.05
24 hrs	10.67 ± 0.01
36 hrs	8.20 ± 0.02
48 hrs	8.01 ±0.5

#### A3: SDS concentrations in calcined LDH at 450°C

Time	Concentration mg/L
12 hrs	9.19±0.02
24 hrs	3.37±0.01
36 hrs	2.37±0.05
48 hrs	1.52±0.5

A4: SDS concentrations	in calcined LDH at 500°C
------------------------	--------------------------

Time	Concentration mg/L
12 hrs	12.08±0.01
24 hrs	8.33±0.5
36 hrs	6.65±0.2
48 hrs	6.37±0.5

#### Bibliography

- Bostrom T E. (2005). A study of structural memory effects in synthetic hydrotalcites using environmental SEM. Materials journal. 226–229.
- C. L. Yuan and M. X. Fan. (2014). Journal of Chemical and Pharmaceutical Research,. *Study on characteristics and harm of surfactants*, 2235.
- Costa, F. R., and Abdel-Goad, M.,. (2005). Synthesis and characterization. *Nanocomposites* based on polyethylene and Mg-Al layered double hydroxide, 46: 4447-4453.
- Das.J. (2002). Colloid and intersurface science. Potochemical chAracterization and adsorption

behaviour of Zn/AL, 213-220.

- Erickson K.L. (2004). *a study of structural memory effect in synthetic hydrotalcite using envirometal SEM*, 26-29.
- Fischer, H. (2003). Polymer nanocomposites:. *fundamental research to specific applications.*, 763-772.
- Frederick .L. (2012). synthesis and characterisation of layered double hydroxides, 22.
- frost R.L and palmer J. S. (2009). *Thermal decomposition of hydrotalcites with variable cationic ratios*, 126-129.
- Kulyukhin S.A . (2007). Sorption of radioidide from aqueous solutions on layered magnesium aluminium hydroxide, 449-504.
- Kura A U, Hussein M Z. (2014). Layered double hydroxide nanocomposite for drug delivery systems; bio distribution, toxicity, and drug activity enhancement. *Chemistry Central Journal*.
- Labajos F.M and Rives V. (1992). Effects of hydrothermal and thermal treatments on the physicochemical properties of hydrotalcite -like materials. 1546-1552.
- LUMBIDZANI M. (2009). A critical assessment of the methods for intercalating anionic, 20-25.
- Lv,L. (2002). Removal of chloride ion from aqueuos solution by Zn/AL LDH, 1444-1449.
- M.A Lewis. (1990). ecotoxicol environment and safety. toxicology.
- Machingauta C. (2013). Synthesis, Characterization and Application of Two-Dimensional Layered Metal Hydroxides for Environmental Remediation Purposes. 130.
- Miyata .S. (1983). Clay and clay mineral. *anion properties of hydrotalcite like compounds*, 305-311.
- Newman, S. P. and Jones, W. (1998). *Synthesis, characterization and layered double hydroxides containing organic guests.*, 105-115.
- Ogawa M. (2000). Hydrothermal synthesis of layered double hydroxide-deoxycholate intercalation compounds. *Chemistry of Materials*, 3253–3255.
- Othman M.R. (2009). applied organometallic chemistry. *sythetic hydrotalcites from different routes and their applications as catalyst and gas adsorbents*, 335-346.
- PY Wang and J Wang. (2007). Surface active agents. *theory, synthesis, determination and application of surface active agents*, 5.

- Rives .V. (2001). Material Chemistry and phsycis. *characterisation of layered double hydroxides and decomposition of their products*, 19-25.
- Rocha J. (1999). Reconstruction of layered double hydroxides from calcined precursors. *Journal* of Materials Chemistry, 2499–2503.
- T.M Schimitt. (2001). analysis of surfactants (2nd ed.). newyork: dekker press.
- Tong , D.S. (2010). Applied clay science. *structure and catalytic properties of Sn -containing layered double hydroxides in the presence of dodecyl sulphate*, 569-574.
- Trikeriotis, M. and Ghanotakis, D. F. (2007). *Intercalation of hydrophilic and hydrophobic antibiotics in layered double hydroxides.*, 176-184.
- Venugopal, B. R., Shivakumara, G. (2006). Colloid Interface Sci.,. *Effect of various factors influencing the delamination behavior of surfactant intercalated layered double hydroxides.*, 234-239.
- Wang P.Y. (2007). surface active agents. beijing: petrochemical press.
- Wang, B., Zhang, H., Evans, D. G. and Duan, X. (2005). *Surface modification of layered double hydroxides and incorporation of hydrophobic organic compounds.*, 190-196.
- Willard H.H. (1998). Istremental methods of analysis (7th ed.). california: wadsworth inc.
- You, Y., Zhao, H. (2002). Colloids Surf. *Surfactant-enhanced adsorption of organic compounds by layered double hydroxides.*, 161-172.
- Zhao. (2001). Surfactant application technology,. Beijing: Chemical Industry Press.