

# Manufacture of Fly Ash Based Coagulants for Use in Water Treatment

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#### Abstract:

Fly ash was explored as a raw material for the production of three fly ash-based coagulants being raw, calcined and calcined- sodium hydroxide complex coagulants. Calcined fly ash and calcined fly ash - sodium hydroxide complex coagulants were prepared by calcining raw fly ash at a temperature of 800°C and all three were acid washed with hydrochloric acid before being used as coagulants. A fly ash sample from a local coal fired power plant was characterization by means of an XRD and FTIR. The characterization results revealed the presence of various compounds which included quartz, hematite, magnetite, anhydrite, and ettringite with a large characteristic peak showing quartz at approximately  $26^{\circ} \approx 2\theta$ . The behaviour of the prepared coagulants in treating waste water was investigated at different turbidity ranges of low (19 NTU), medium (49 NTU) and high (80 NTU) in jar test experiments. Coagulant doses were also varied at these different turbidity levels to determine the dosage that gives the lowest turbidity for all the three coagulants. From this study, we observed that an increase in coagulant dose from 0.05 to 0.25g/l led to a decrease in residual turbidity. Calcined fly ash was more effective at low and medium turbidity at a dosage of 0.20 g/l whereas Calcined fly ash - sodium hydroxide complex coagulant was found to be more effective with the highest % reduction of 99.8% at a higher turbidity for the same dose.

Keywords: Bentonite Clay, Coagulant, Fly Ash, Waste Water.

## I. INTRODUCTION

Water is often contaminated with hazardous materials such as organic and inorganic compounds. This problem arises from the increasing production of non-biodegradable hazardous contaminants which originate from sources like human waste, industrial effluents, paper, and vegetable matter [1]. The treatment of such water to an environmentally acceptable standard is very important from an environmental and economic point of view [2]. Fly ash—is a by-product from a thermal power plant produced from the thermal combustion of coal. Botswana's coal based power plants produces around 700 000 tons of fly ash per annum [3] and the ash produced requires more ash ponds or dykes for its disposal. The light weight particles of fly ash can contribute to air pollution when exposed to wind in the ash ponds. Due to the presence of heavy metals in fly ash, contact with water sources can damage aquatic life and the basicity can affect the physio-chemical characteristics of the soil [4]. Due to the presence of aluminium and iron oxide in fly ash, it can be utilized as a coagulant for use in waste water treatment [5]. This conversion of fly ash to a



coagulant will not only be a solution to the problems mentioned but will help reduce the utilization of chemical coagulants used in Botswana thereby saving costs. Raw and pre-treated fly ash coagulants were prepared for use by acid washing with hydrochloric acid and their performance was assessed by testing their effect on turbidity reduction and the pH using a jar test apparatus.

## **II. EXPERIMENTAL METHODS**

#### A. Materials

Fly ash samples were collected from a local coal fired power station and these samples were used in the manufacture of fly ash based coagulants. Fly ash contains alkali and alkaline earth materials therefore hydrochloric acid was used to wash off the alkaline material contained in the fly ash. Bentonite clay was used to simulate the waste water for which the effectiveness of the manufactured coagulants would be tested.

#### B. Characterisation of fly ash

An X-Ray Diffractometer (XRD) was used to identify the mineral phases of fly ash and the crystalline structure (whether it's amorphous or crystalline) while a Fourier Transform Infrared Spectroscopy (FTIR) was used to identify the functional groups present in the fly ash.

#### C. Preparation of a fly ash based coagulant

Three fly ash based coagulants were prepared using three different samples. The first sample was raw fly ash, the second sample (calcined fly ash) was prepared by heating the fly ash in an oven at a temperature of 800 °C for 2 hours and the third sample (calcined fly ash – sodium hydroxide complex) was prepared by mixing the fly ash with sodium hydroxide at a ratio of 100:6 and calcining the mixture at 800 °C for 2 hours. 100 g of each of the three samples was reacted with 300ml of hydrochloric acid (4 M) at 80 °C and was magnetically stirred at a speed of 180rpm for 45 minutes to remove the alkaline materials contained in fly ash. The samples were then filtered and the solid residue was dried in an oven for 2 hours at a temperature of  $115 \,^{\circ}$ C.

#### D. Preparation of simulated waste water

10 g of bentonite clay powder was magnetically stirred in 1000 ml of water for 15 minutes and was left for 24 hours before use to precipitate out the larger bentonite particles.

# *E. Test for the effectiveness of the fly ash based coagulants in waste water treatment*

A jar test experiment was conducted to test the effectiveness of the fly ash based coagulants produced. 1000 ml of simulated wastewater was stirred with the coagulant prepared in a beaker. Different dosages of the prepared coagulant were used in 6 beakers each containing (0.05, 0.10, 0.15, 0.20 and 0.25 g/l) at turbidity of 19, 49 and 80 NTU for each of the coagulants. Agitation was done after the pre-determined dosage of fly ash was added to the wastewater sample in order to mix the effluent and coagulant. This agitation was done to produce a uniform suspension before the settling characteristics of the flocs/solids. The jar test experiment was carried out by rapid mixing at 250 rpm for 2 minutes for thorough mixing and slow agitation at 100 rpm for 15 minutes for formation of flocs. The mixture was then allowed to settle for 30 minutes and pH and turbidity of the samples were measured using a pH and a turbidity meter respectively. The same procedure was carried out for all the three different coagulants. Fig. 1 shows the jar test experiment.



Fig. 1: Jar test experiment

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## **III. RESULTS AND DISCUSSION**

The aim of the experiment was to treat wastewater by accelerating the particle settling rate and reduce contaminants in wastewater. By using coagulants and flocculants, particle sizes are increased due to their agglomeration resulting in faster settling rates [6]. Parameters used for evaluating the effectiveness of suspended solids removal in wastewater treatment include settling time, coagulant dose, turbidity and pH.

#### A. Manufactured fly ash based coagulants

Fig. 2 shows the samples of the three manufactured fly ash based coagulants.



Fig.2: Fly Ash based coagulants (a) raw fly ash (b) calcined fly ash (c) calcined fly ash-sodium hydroxide complex

# B. X- Ray Diffractometer of the Fly Ash based Coagulants

The diffractogram in *Fig. 3* shows that the three fly ash sample consists mainly of Quartz (SiO<sub>2</sub>), Hematite (Fe<sub>2</sub>O<sub>3</sub>), Magnetite (Fe<sub>3</sub>O<sub>4</sub>), Anhydrite  $(CaSO_4)$ and Ettringite  $(Ca_6Al_2(SO_4)_3(OH)_{12})$ 26H<sub>2</sub>O) with a large characteristic peak of quartz (SiO<sub>2</sub>) at 26°  $\approx$  20. The large characteristic peak suggests the presence of a large amount of amorphous material and according to [7], the bulk phase composition would represent a more amorphous phase in fly ash. Calcined fly ash coagulants show diminished peaks or decrease in intensity and this may be attributed to the breakdown of some mineral components and the removal of amorphous silicon by sodium hydroxide through leaching.

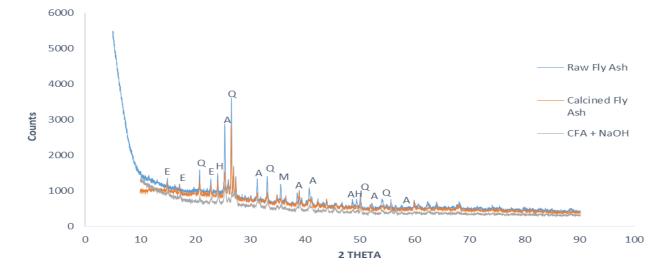


Fig. 3: X-Ray Diffractometer for Fly ash samples



# C. Spectral Analysis of the Fly Ash based Coagulants

Fig. 4 shows the spectral bands that were identified from the FTIR spectroscopy and they were used to identify the functional groups present in the three fly ash based coagulants. CFA and CFA – Sodium Hydroxide complex present peaks at 3739.5 and 3735.7 cm<sup>-1</sup> respectively that are associated with bonded water and indicating that the exposure of hydrolysed silica to compressive stress increased water dissolution into the silica structure as hydroxyl [8][9]. A peak at 3390.4 cm<sup>-1</sup> shows the presence of hydrogen bonded OH stretching vibrations for alcohol and phenolic groups whereas asymmetrical stretching vibrations of C – C aromatic rings are present at 1409.8 cm<sup>-1</sup> for RFA and 1498.5 cm<sup>-1</sup> for CFA – Sodium Hydroxide complex. Carbonyl groups (Esters, Ethers, Phenols and Carboxylic acids) have IR absorptions associated with C – O stretching vibrations and they are shown by strong peaks at 1083.8 and 1054.9 cm<sup>-1</sup> in RFA and CFA respectively. Strong C – H stretch vibrations representing Alkenes are seen at the peaks 960.4 and 673.1 cm<sup>-1</sup> for CFA – Sodium Hydroxide complex while a medium vibration at 673.1 cm<sup>-1</sup> in CFA – Sodium Hydroxide complex is attributed to C – Cl stretch. Inorganic compounds produce characteristic infrared spectrums when they form covalent bonds within ions. The inorganic ion SO<sub>4</sub><sup>2-</sup> is represented by 1083.8 cm<sup>-1</sup> while PO<sub>4</sub><sup>3-</sup> is shown by 1083.8, 1054.9 and 960.4 cm<sup>-1</sup>.

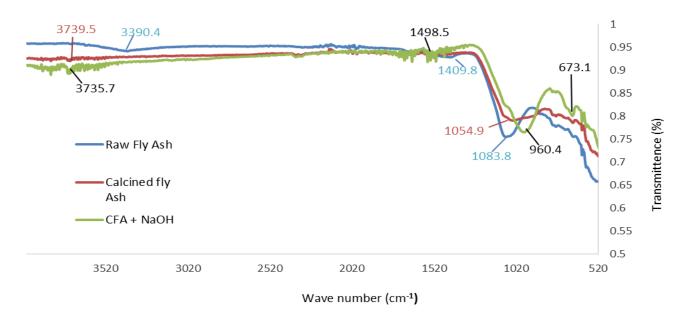


Fig. 4: Spectral Analysis of the Fly Ash Based Coagulants

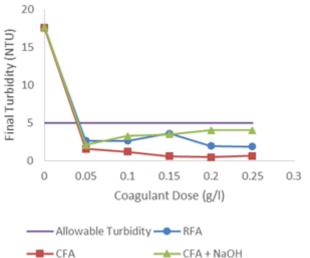
# D. Effects of the three Coagulants in Water Treatment at Low turbidity (19 NTU)

The effect of a coagulant dosage on turbidity removal was investigated for the three fly ash based coagulants and the results are shown in *Fig. 5*. A general trend shows that an increase in dosage resulted in a decrease in the residual turbidity. Coagulant chemicals with charges opposite to those

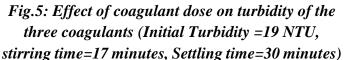
of the suspended solids are added to the water to neutralize the negative charges on non-settleable solids (such as clay and colour-producing organic substances). Increasing the dosage of the coagulant would imply that more charges are employed to neutralize the negative charges in waste water which will make the particles to agglomerate and settle, this will in turn reduce the turbidity of the coagulated



mixture as the suspended solids would have settled [10]. The dosage of raw fly ash that gave the lowest turbidity was found to be 0.25 g/l with a residual turbidity of 1.85 NTU. As the dosage of calcined fly ash increased from 0.05 g/l to 0.20 g/l, the residual turbidity decreased from 1.58 to 0.47 NTU from an initial turbidity of 19 NTU. The dosage that gave the lowest turbidity with calcined fly ash was found to be 0.20 g/l at a turbidity value of 0.47 NTU. Calcined Fly Ash-Sodium Hydroxide complex displayed a different behaviour in residual turbidity with an increase in dosage. The lowest turbidity value was obtained at 0.05 g/l and with further increase in dosage the turbidity increased. According to [11], a decrease in residual turbidity with an increase in coagulant dose might be due to the high concentration of residual  $Fe^{3+}$  ions in the coagulant. The high concentration of residual Fe<sup>3+</sup> in calcined fly ash Sodium Hydroxide complex could have been re-liberated when the fly ash was calcined. Calcined fly ash coagulant showed a greater coagulative capacity than others and a dosage of 0.20 g/l resulted in turbidity less than 0.5 NTU. Nevertheless all these coagulants were able to meet the standards set by Botswana Bureau of Standards (BOBS) of 5 NTU as shown in Fig. 5.



# ---- CFA ---- CFA + NaOH



# E. Effects of the three Coagulants in Water Treatment at Medium turbidity (49 NTU)

Fig. 6 shows the performance of the three fly ash based coagulants at a turbidity of 49 NTU. In raw fly ash coagulated mixture, a decrease in residual turbidity from 1.97 to 0.89 NTU from an initial turbidity of 49 NTU was observed for coagulant doses between 0.05 and 0.25g/l-whereas calcined fly ash coagulant had a decrease in turbidity from 1.81 to 1.31 NTU was seen at dosages between 0.05 to 0.20 g/l. At a dosage of 0.25 g/l, the turbidity increased to 0.53 NTU. Calcined fly ash sodium hydroxide complex coagulant had a great decrease in turbidity from 3.28 to 1.9 NTU at dosages of 0.05 and 0.10 g/l, this changed as the turbidity increased to 2.2 NTU at a dosage of 0.15g/l. Beyond this dosage there was a slight decrease in turbidity from 1.26 to 1.25 NTU at a dose of 0.20g/l to 0.25g/l. Calcined Fly ash coagulant was more effective in medium turbid water This can be attributed to the reason that there is enough suspended solids to adsorb the  $Fe^{3+}$  so there is little colloidal Fe left in solution due to the excess Fe<sup>3+</sup> in the coagulant. Despite calcined fly ash coagulant being the most effective at this turbidity, all the three coagulants were able to meet the threshold of coagulated mixture set by Botswana Bureau of Standards (BOBS).

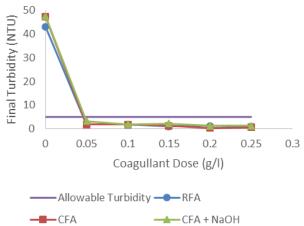
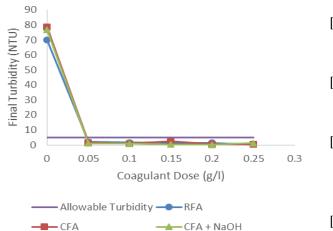


Fig. 6: Effect of coagulant dose on turbidity of the three coagulants (Initial Turbidity =49 NTU, stirring time=17 minutes, Settling time=30 minutes)



# F. Effects of the three Coagulants in Water Treatment at Medium turbidity (80 NTU)

The effect of coagulant dosage on turbidity removal was investigated at a high turbidity of 80 NTU. Results obtained are shown in Fig. 7 and it shows that raw fly ash as a coagulant had a slight decrease in turbidity from 0.98 to 0.34 NTU for doses between 0.05 and 0.15 g/l at an initial turbidity of 80 NTU. A sudden slight increase in turbidity was observed at 0.20 g/l but beyond this dose, turdidity decreased to 0.32 NTU. Calcined fly ash coagulant showed a similar behavior to the raw fly ash coagulant in such a way that the turbidity reduction from 1.7 to 0.45 NTU was seen at a dose of 0.05 to 0.15 g/l, an increase in turbidity to 0.47 was observed at a dose of 0.20g/l at the same initial turbidity and beyond this turbidity a decrease of turbidity to 0.2 NTU was observed. Calcined fly ash sodium hydroxide complex coagulant had a decrease in turbidity from 1.92 to 0.54 NTU at doses of 0.05 to 0.20g/l. It was only at a dosage of 0.2 g/l where there was an increase in turbidity to 1.17 NTU. In [1] comparing the three fly ash based coagulants, calcined fly ash sodium hydroxide complex coagulant was found to be more effective with the [2] lowest residual turbidity of 0.54 NTU at a dose of 0.20g/l.



# Fig. 7: Effect of coagulant dose on turbidity of the three coagulants (Initial Turbidity is 80 NTU, stirring time=17 minutes, Settling time=30 minutes)

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#### CONCLUSION

The three manufactured fly ash-based coagulants were utilized in treating simulated waste water prepared from bentonite clay. The study was based on reducing the turbidity levels to the required BOBS standards of less than 5 NTU and the coagulants proved effective as they all met the required BOBS standards. Calcined fly ash was found to be more effective at low and medium turbidity at a dosage of 0.20 g/l as it resulted in turbidity less than 0.5 NTU whereas calcined fly ash-sodium hydroxide complex coagulant was found to be more effective at removing high turbidity resulting in a percentage turbidity reduction of 99.8% at a dose of 0.20 g/l. Low turbid waters are usually hard to coagulate due to low concentrations of stable particles therefore the increased charges in calcined fly ash-sodium hydroxide complex could not coagulate low concentration of stable particles.

#### REFERENCES

- A. E. A. Nayl et al., "Adsorption studies on the removal of COD and BOD from treated sewage using activated carbon prepared from date palm waste,"Environ. Sci. Pollut. Res,. November, 2017..
- A. Baghvand, A. D. Zand, N. Mehrdadi, and A. Karbassi, "Optimizing coagulation process for low to high turbidity waters using aluminum and iron salts,"
  Am. J. Environ. Sci., vol. 6, no. 5, pp. 442–448, 2010.
- [3] O. Chelenyane, "Electricity Generation And Distribution stats brief, Quarter 1 2017," Statistics Botswana ., pp. 1–16, June 2017.
- [4] M. Ahmaruzzaman, "A review on the utilization of fly ash," Prog. Energy Combust. Sci., vol. 36, no. 3, pp. 327–363, 2010.
- [5] L. Li, "Production of a new wastewater treatment coagulant from fly ash with concomitant sulfur dioxide removal from flue gas," Diss. Abstr. Int., 2008.
- [6] B. J. Pillai, D. Ph, and N. Company, "Flocculants and Coagulants: The Keys to Water and Waste Management in Aggregate," no. December, 1997.
- [7] R. T. Chancey, P. Stutzman, M. C. G. Juenger, and D. W. Fowler, "Comprehensive phase characterization



of crystalline and amorphous phases of a Class F fly ash," Cem. Concr. Res., vol. 40, no. 1, pp. 146–156, 2010.

- [8] T. S. Malarvizhi and T. Santhi, "Lignite fired fly ash modified by chemical treatment for adsorption of zinc from aqueous solution," Res. Chem. Intermed., vol. 39, no. 6, pp. 2473–2494, 2013.
- [9] B. Bayat, "Comparative study of adsorption properties of Turkish fly ashes: I. The case of nickel(II), copper(II) and zinc(II)," J. Hazard. Mater., vol. 95, no. 3, pp. 251–273, 2002.
- [10] T. L. Engelhardt, "Coagulation, Flocculation and Clarification of Drinking Water," pp. 1–57, 2010.
- [11] M. Fan, R. C. Brown, T. D. Wheelock, A. T. Cooper, M. Nomura, and Y. Zhuang, "Production of a complex coagulant from fly ash," Chem. Eng. J., vol. 106, no. 3, pp. 269–277, 2005.