Safety: Wear the appropriate PPE when working with acids.

Purpose: ANALYSIS OF FILTER PRESS CAKE

Procedure:

Introduction

This procedure was developed for two control purposes, to provide a measure of the conversion of Magnesite (MgO) to $MgSO_4$ in the digesters, and a measure of removal of $MgSO_4$ from filter cake, both of which affect the economics of the process..

Procedure

Samples (chunks) of Filter cake are obtained from each end and the center of a filter press as a press is being stripped at the end of a press run. The samples are taken to the laboratory and analyzed for:

- Total Solids
- Free moisture
- $MgSO_4$
- Unreacted MgO and/or CaO
- Unreacted Hard burned MgO
- Mineral Matter (Silica. etc.).

Results are recorded in the laboratory computer file

Equipment

Laboratory Scale – B440 Sartorius
Porcelain Crucible – 100 mL
Drying Oven - 100ºC
Muffle Furnace - 500ºC
Laboratory Dessicator
Oven Forceps
Magnetic Stirring Plate with 1” stirrer bar
Glass Beaker – 100 mL
Filter Paper – Baxter 90 mm S/P Grade 361 Qualitative
Erlenmeyer Flask – 250 mL Kimax Wide Mouth
Vacuum Pump – Boekel Hy-Vac 30 liter, Direct Drive. Capacity 26” Hg
EDTA titration assembly, including reagents – See WI 6.10
Supply of DI water
Supply of 1.0 N H_2SO_4 Use Thomas Scientific C748190 1.0 N out of Thomas bottle. Supply of 1.0 N HCl Dilute Thomas Scientific C395L92 (36.5-38%) 1/10 with DI water
Eye protection and rubber gloves.

Method

The three samples are analyzed separately, thus allowing a realistic average to be developed.

1. From each of the three press samples as received, cut a lab sample perpendicular to the cake surface weighing about 30 grams and proceed to analyze each individually as follows
2. Weigh a 100 mL crucible. **Record the weight. - A**
3. Place the lab sample in the crucible, weigh, and **record the weight. – B**

B minus A = the weight of the sample – C

4 Place the crucible and sample in the 100°C oven overnight.
5. Next day, using the forceps, remove the crucible from the oven and place in the dessicator.
6. When cool, weigh the crucible containing the sample, and **record the weight. – D**

D minus A ÷ C = % Total Solids in the Filter Press Cake
B minus D ÷ C = % Free Moisture in the Filter Press Cake

7. Place the dry sample in a 100 mL beaker.
8. Add exactly 100 g. of DI water to the beaker.
9. Break up the cake sample somewhat with the spatula.
10. Add the stirrer bar to the beaker and place the beaker on the magnetic stirrer plate.
11. Start the stirrer and run until the solids are well broken up and dispersed.
12. Weigh a 90 mm filter paper, and **record the weight -E**
13. Place the filter paper in the vacuum filter assembly, and filter the contents of the beaker
14. Place the Erlenmeyer flask on the scale and tare to 0.
15. Add exactly 10 grams of the filtrate at 13 to the flask on the scale.
16. Add about 100 mL of DI water to the flask and titrate with EDTA to a blue end point,
17. Using the formula:

$$\frac{\text{mL of EDTA solution x 1.2036}}{\text{weight B at item 6}} = \text{gms. of } MgSO_4 \text{ in filtrate at item 13 - F}$$

F ÷ A = % $MgSO_4$ in the Filter Press Cake

18. Using the vacuum, wash the filtered solids still on the filter paper thoroughly with DI water.
19. Place the paper and solids in the 100°C oven for 30 minutes or to constant weight.
20. Place the dry filter paper, with solids, on the weigh scale amd **record the weight – G**

(G – E) ÷ A = % Unreacted Solids in Filter Press Cake.

21. Transfer the unreacted solids from the filter paper to a 100 mL beaker.
22. Add 50 mL of 1.0 N H_2SO_4
23. Dispeerse the solids thoroughly and let stand overnight at room temperature.
24. Next day decant and discard as much acid solution as possible.

25. Add 75 or so mL of DI water to the beaker and stir thoroughly.
26. Place a 90 mm filter paper on the scale and **record the weight – H**
27. Place the filter paper in the vacuum filter assembly and filter the contents of the beaker at 26.
28 Rinse the solids on the paper very thoroughly with DI water.
29. Remove the paper and solids from the filter funnel, taking care to glean stray solids.
30. Place the paper and solids in the 100°C oven for 30 minutes, or to constant weight.
31. Place the paper and solids on the scale and **record the weight - I**

(G minus E) minus (I minius H) ÷ A = % MgO and/or CaO in Filter Cake

32. Transfer the unreacred solids from the filter paper to a 100 mL beaker.
36. Add 50 mL of 1.0 N HCl.
34. Disperse the solids and let stand overnight at room temperature.
35. Next day decant and discard as much supernatant acid solution as possible.
36. Add 75 mL or so of DI water to the beaker and stir thoroughly.
37. Place a 90 mm filter paper on the scale and **record the weight - J**
38. Place the filter paper in the vacuum filter assembly and filter the contents of the beaker at 37.
39. Rinse the solids on the paper very thoroughly with DI water
40. Remove the paper and solids from the filter funnel, taking care to glean stray solids.
41. Place paper and solids in the 100°C oven for 30 minutes or to constant weight.
42. Place the dry paper and solids on the scale and **record the weight - K**

(I minus H) minus (K minus J) ÷ A = % Unreacted Hard Burned MgO in the Press Cake

(K minus J) ÷ A = % inert mineral matter (Silica, etc.) in the Press Cake

Preparation of Reagent Acids

Standard 1.0 N Sulfuric Acid

No preparations are necessary for this. Order 1.0 N H2SO4 from Thomas Scientific.

Standard 1.0 N Hydrochloric Acid

HCI is purchased from Thomas as 36.5-38% solution.
Dilute 100 ml of solution form the Thomas container to 1000 ml. in a liter flask.

Preparation of Standard Solutions

EDTA

Weigh 0.10 mol (37.22g) EDTA Disodium Dihydrate salt on the balance and dissolve in about 700 mL of de-ionized water in a 1000-mL beaker. Agitate with mechanical stirring to hasten dissolution. When EDTA has completely dissolved transfer to a 1000-mL

volumetric flask with stopper. Fill to mark with de-ionized water. This is the EDTA standard solution. Store in flask until needed.

Eriochrome Black TS Indicator

Weigh 0.5 grams Eriochrome Black TS and dissolve in about 50 mL of de-ionized water in a 100-mL volumetric flask with stopper. Swirl to mix. When dissolved, fill to mark with de-ionized water. Transfer to drop-flask as needed.

TRAINING DOCUMENTATION

	EMPLOYEE	TITLE	SIGNATURE	DATE
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				