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Research Article

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Recovery of Calcium Carbonate from Wastewater Treatment Sludge Using a Flotation Technique

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Abstract

The use of flotation technique for the recovery of calcium carbonate ($CaCO_3$) from wastewater treatment sludge was investigated in this study. The parameters that were investigated included dosage of floating agents (sodium oleate and sunlight dish liquid) and the percentage solids of the slurry. The experiments were performed by floating sieved and un-sieved materials and $CaCO_3$ was determined for both conditions as well as from tailings.

Initial $CaCO_3$ analysis for the bulk material indicated that sieved and un-sieved materials had 63.4% and 32.9% $CaCO_3$ content by weight respectively. The modification of pH was effected by dosing 1g $NaCO_3$ to both 1000 g of sieved and un-sieved materials which was sufficient to raise the pH of the slurry to 9.5. A lower average recovery of 2.33% was observed on un-sieved material after using sodium oleate as a collector when compared to sunlight liquid of 31.6%. Therefore, it was concluded that for un-sieved material sunlight dishwashing liquid was a better collector compared to the latter. The results of this study proved that there is great potential of recovering commercial grade limestone from wastewater sludge.

Keywords: Collector; Conditioner; Froth; Sodium oleate; Sunlight dish liquid; Sieved

Introduction

Complete wastewater treatment does not only involve the treatment and reclamation of the liquid, but also encompasses the processing and disposal of the solids removed or generated during treatment [1]. The flotation process is widely used in industrial wastewater treatment plants, where it is used to remove fats, oil, grease and suspended solids from wastewater. These units are called Dissolved Air Flotation (DAF) units. In particular, dissolved air flotation units are used in removing oil from the wastewater effluents from oil refineries, petrochemical and chemical plants, natural gas processing plants and similar industrial facilities [2,3].

The separation process of froth flotation is a primary method of creating a high-valued concentrates which could contain usable materials [4,5]. Conventional flotation equipment from both Denver and Wemco equipment companies is available for lab and pilot plant studies. Prior to pilot plant testing, flotation parameters such as reagents, conditioning time, pH, and stages are defined at lab scale. In conjunction with the flotation studies, mineralogy, surface chemistry, and liberation determination are used to define feed parameters [6-8].

Lime treatment of wastewater produces relatively large amount of sludge by weight and for that reason, the choice of solids handling methods significantly affects capital and operation costs for the plant as well as the impact of ultimate sludge disposal on the environment [9]. A few water treatment systems have been equipped with sludge treatment facilities for recovering reusable and marketable products [10]. The procedures used at the sludge treatment facilities involve carbonation of the sludge to effect a phase separation between calcium and magnesium values [7]. The magnesium hydroxide component of the sludge is solubilised from the residual insoluble and is disposed off as a by-product waste material while reusable in the water treatment process and also available for sale on the open market. The disposal of the carbonated liquor, however, creates ecological challenges such as sludge dumps and their management.

The treatment of sludge to recover calcium carbonate has been limited to the treatment of sludges obtained from the softening of raw waters that are basically free of turbidity factors. Clay and other turbidity factors present in the raw water are separated as components of the sludge and are carried through the sludge treatment step with insoluble calcium carbonate.

Investigation has shown that froth flotation can be economically and effectively used to produce a relatively high grade calcium carbonate with good recovery from the wastewater treatment sludges when using magnesium carbonate with lime for flocculation [10]. Flotation had its beginning in mineral processing and as such it has been used for a long time in solid to solid separation using stable froths to selectively separate different minerals from each other [10]. It is believed that the cross exchange of flotation experience in the mineral flotation and water effluent treatment should lead to new and improved procedures for industrial waste treatment [11].

The flotation process has enabled the production of high brightness calcium carbonate by removing the silicate impurities from the calcium carbonate ore which would otherwise be responsible for colour imperfections in the finished product [8]. The most important criteria in evaluating the performance of calcium carbonate collector during floatation is the dosage level required and the yield of calcium

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carbonate produced. In froth flotation process, calcium carbonate from the clay or any other water contaminants is conditioned with soda ash and sodium silicate to disperse the clay with the aid of pH adjustment as well. Thereafter, the slurry is conditioned with fatty acid soap which selectively coats the calcium carbonate particle with insoluble soap making it hydrophobic and collectable [8].

Flotation where possible, is carried out in an alkaline medium, as most collectors such as xanthates, are stable under these conditions [12]. The alkalinity condition minimises corrosion of cells, pipe work and other metal handling facilities. Sodium carbonate is usually added to control the alkalinity and to a lesser extend sodium hydroxide or ammonium hydroxide is used for the same purpose. Sulphuric or sulphurous acids are used where lower pH is required [13]. In most cases lime is being used to regulate pulp alkalinity as it is cheaper than most alkalis. Lime can also act as a strong depressant for pyrite and arsenopyrite when using xanthate collectors [14]. Both hydroxyl and calcium ions participate in the depressive effect on pyrite by the formation of mixed films of Fe(OH), FeO(OH), $CaSO_4$ and $CaCO_3$ on the surface [15].

The aim of this study was to recover calcium carbonate which was used after treatment of acid mine water from the sludge. Flotation was selected as a feasible technique to recover the $CaCO_3$ from the waste sludge.

Experimental

Sodium oleate and sunlight dish liquid were used as collectors for calcium carbonate in this study. The effects of collector dosage, modifiers and pH on calcium carbonate recovery were investigated.

Operational procedure for the Denver machine used in floatation studies

The following steps are the general operating principles of the Denver machine used in this study:

- The agitator mixes the slurry and the air is dispersed by an impeller stabilizer;
- The impeller is connected to a vertical hollow shaft that rotates the impeller and feeds low pressure air under the impeller plate;
- As the air flows between the rotating plate and stabilizer, it is broken into finer bubbles by the shearing action and is dispersed throughout the flotation cell;
- As the pulp contacts the impeller, it is intensely agitated and aerated;
- The flow pattern directs the bubbles to the surface and;
- The concentrates collected in the froth column at the surface is discharged at the froth overflow lips.

Figure 1 shows the floatation machine used in this study.

Experimental procedure (Flotation process)

The experiments were carried out in 5 L flotation cell at 10% solids. A mass of 500 g of dry sample was added to 4.5 L of water. It was then milled for 20 min and poured into a 5 L flotation cell. For the initial test, 1 g of Na_2CO_3 and 1 g Na_2SiO_3 were added to the slurry and allowed to condition for 3 min, then 0.5 g of fatty acid soap ($Na_2O_2H_{33}C_{18}$) was added and allowed to condition for 3 min.

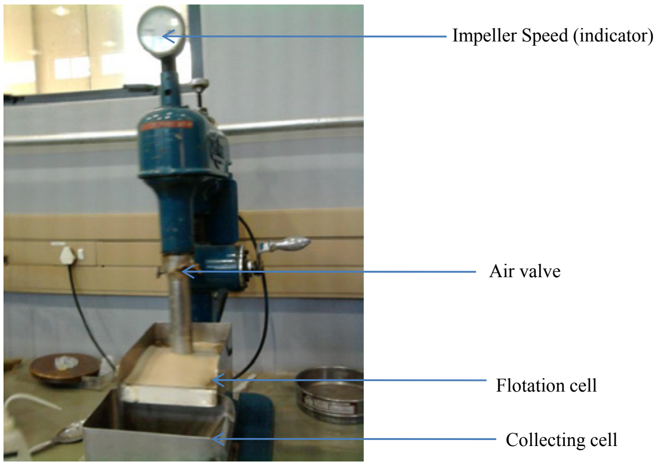

Figure 1: Denver machine used for flotation.

	Effect of sodium oleate	Effect of sunlight		
Test	1.1	1.2	1.3	1.4
Volume (L)	4500	4500	4500	4500
Feed CaCO3	500	500	500	500
Description	Unsieved	Unseived	Unseived	Unseived
Feed CaCO3 content (%)	32.9	32.9	32.9	32.9
Flotation:				
Na2CO3 (g)	1	1	1	1
Silicate (%)		30	30	30
Silicate (mL/L)	0.2	0.2	0.2	0.2
Silicate (mL)	1	1	1	1
Oleate (%)		100	100	100
Oleate (g/L)	0.4	0.1	0.2	0.4
Oleate (g)	2	0.5	1	2
pH Feed (before adding	8.51	8.6	8.6	8.6
Na2CO3)	9.45	9.54	9.54	9.54
pH Feed (after adding	650	650	650	650
Na2CO3)				
Impeller speed (rpm)				

Table 1: Conditions for the experiments on un-sieved material.

The air was then introduced to generate bubbles allowing the formation of froth. The concentrates were collected at the following times intervals, 1, 4, 7 and 20 min. The rotational velocity was set to 650 rpm. This was done on the first three tests with different dosages of $Na_2O_2H_{33}C_{18}$ (i.e. 0.5 g, 1 g and 2 g).

The preceding procedure was used for tests 4, 5 and 6 with sunlight dishwashing soap as the collector agent. The concentrates and tailing of each test were dried, weighed and then taken for analysis to determine the $CaCO_3$ concentration in them.

Experimental procedure (analysis for $CaCO_3$)

Approximately 1.065 g of sample was weighed and added to 50 mL of 1 N HCl. It was then heated to 80C for 15 min. The sample was then allowed to cool and titrated with 1 N NaOH to pH 7.

Results and Discussion

The results and discussion for un-sieved material are as follows

The experimental conditions used for un-sieved sludge are shown in (Table 1). Experiments with sodium oleate with different settings did not produce any significant recoveries from set one shown in (Table 1) and hence, these results were not reported. It was noted that the pH

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was almost constant for the four experiments 1.1 to 1.4 for sunlight. For each experiment, the initial pH was around 8.6 and after addition of soda ash (Na_2CO_3), the pH raised to 9.45. From literature survey, most materials responded well on the pH around 7.5 and 11.5 and calcium carbonate responded well at pH of 9.5, which was then set as target for pH modification in this study [8]. Detailed results on the recoveries are shown on (Table 2 and 3), where each frother concentrate (denoted by FC1, FC2, FC3 and FC4) and tailings concentrate (T) were analysed for $CaCO_3$ content.

Effect of collector dosage on un-sieved material

The total recoveries for the concentrates as collected at different times are shown below in (Table 2 and 3). Tables 2 and 3 show the results of un-sieved material, where the tests were conducted at the same conditions but using different collectors. It was demonstrated that the recovery increased with flotation time, however, the tests conducted with sodium oleate as fatty acid collector showed a very small increase of recovery with time.

Sodium oleate (soap) appeared to have been a weak collector when compared to sunlight dishwashing liquid (detergent). This was also shown by results obtained experiments 1.1 and 1.4 of the un-sieved sludge material, where equal dosages of fatty acids (sunlight and sodium oleate) were added, but with sodium oleate only 2.33% $CaCO_3$ recovery was obtained whereas by sunlight the recovery increased to 31.55%. Detergents have a tendency of emulsifying scum's while soaps lose their froth as they precipitate calcium as calcium salts. The failure by sodium oleate to breakdown calcium carbonated to smaller pieces easily floated in the froth might have lead to the poor recoveries observed in this study.

Figure 2 shows the difference in recovery when using sunlight and sodium oleate at the same dosages. When using dosage of 2 g sodium oleate as collector, it was found that while collecting the froth for 1 minute, approximately 1% recovery of calcium carbonate was obtained and it did not show much different when the froth was collected for 4 and 7 min respectively. When using 2 g sunlight liquid as collector, it was observed that the concentrate which was collected in the first 1 min had a recovery of approximately 3%. The increase in the concentrate collected between 4 and 7 min was quite significant. Even after 7 min, the froth was still there and the concentrate collected for the next 20 min and the recovery increased further to reach12%.

	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		404	32.9	
FC1	1	3.3	18.97	0.99
FC2	4	3.4	21.01	1.13
FC3	7	3.7	20.57	1.20
FC4	20	0	0.00	0.00
FrotherConcentrate		**10.4**	**20.21**	**2.33**
Tails		393.6	15.520	96.67
Head (calc)		404	15.64	99.01

Table 2: Experiment 1.3 of un-sieved material (2 g sodium oleate).

	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		500	32.9	
FC1	1	9.4	27.51	2.95
FC2	4	21.1	26.06	6.28
FC3	7	40.03	23.90	10.99
FC4	20	46.5	22.98	12.20
FrotherConcentrate		**117.3**	**24.21**	**31.55**
Tails		382.7	15.470	67.58
Head (calc)		500	17.52	100.00

Table 3: Experiment 1.4 of un-sieved material (2 g sunlight).

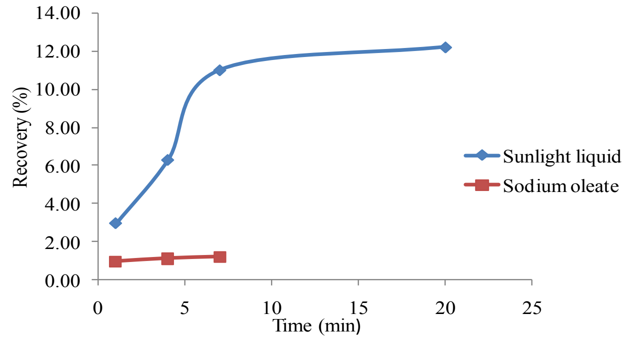

Figure 2: Graph of time versus recovery for experiments of same dosages of two different collectors used.

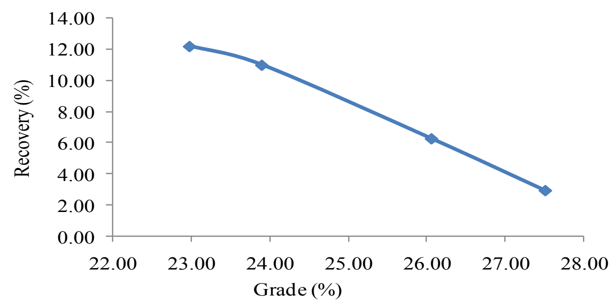

Figure 3: The graph of grade versus recovery for 2 g dosage of sunlight.

	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		500	32.9	
FC1	1	1.6	28.86	0.51
FC2	4	1.7	25.84	0.49
FC3	7	3.5	21.16	0.82
FC4	20	54.1	20.00	12.02
FrotherConcentrate		**60.9**	**20.46**	**13.84**
Tails		439	17.670	86.16
Head (calc)		499.9	18.01	100.00

Table 4: Experiment 1.1 of un-sieved (0.5 g sunlight).

Figure 3 shows the relationship between recovery of $CaCO_3$ and grade in the sludge. At a recovery of about 12% the grade was 23%, but at a recovery of about 2% the grade (quality of calcium carbonate produced) was higher, that is about 27.5%. These results demonstrated that with a higher recovery, a low grade $CaCO_3$ was obtained. This can be explained by the fact that when floating is performed for a long-time, some of the non- $CaCO_3$ materials will also start floating and resulting in a high recovery with a lot of impurities. Some studies have come to a conclusion that typical grade-recovery curve show a relationship of grade being inversely proportional to recovery [12]. The data of sodium oleate as collector could not be shown as the recoveries were too low when compared to the use of sunlight as collector (Figure 3).

The comparison of the effectiveness of sunlight dishwashing liquid as a collector over sodium oleate as a collector is illustrated in (Figure

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4). From the graph, it is noted that with 0.5 g sodium oleate, there was no $CaCO_3$ recovered whilst sunlight managed to produce about 15% recovery.

This can be explained by the fact that impurities in the raw water vary from place to place as well as with seasons and results in the modification of the water treatment processes and the sludge produced thereafter. Hence, variations in the sludge composition would require adjustment in the flotation reagents composition as well.

The experimental conditions for the recovery of $CaCO_3$ use a dosage of 0.5 g of sunlight are shown in (Table 4). From the grade column, the initial concentrate which was collected after 1 min showed a very high grade than the other concentrates. There was grade reduction as the time and the recoveries increase.

The effects of increasing the collector concentration from 0.5 g to 1.0 g are shown in (Table 5).

From (Table 5), it is noted that the concentrates that were collected for the first three time intervals were almost equal although they were not collected for equal in mass (8.4, 8.5, 10.3). This indicates that the pattern of froth floating was not constant and $CaCO_3$ was almost coming out of the cell constantly.

The effect of collector concentration on the recovery of $CaCO_3$ from sludge is illustrated in (Figure 5). It was observed that the recoveries were almost invariant for both dosages in the first 4 minutes. This could be explained by assuming that the conditioning times were too short, therefore, giving inadequate interactions time between the collector and substance of interest $CaCO_3$. After 4 min a rapid increase in the recoveries for both doses was observed.

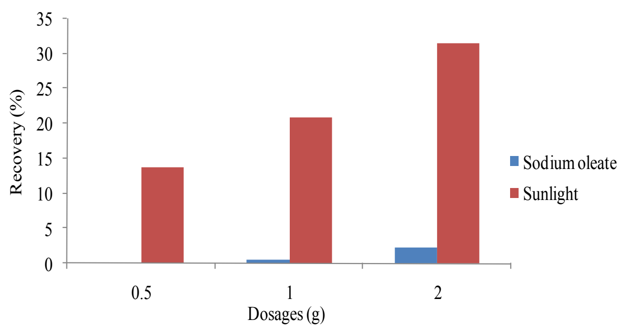

Figure 4: Bar graph of dosage versus recovery to indicate the effectiveness of sunlight over sodium oleate.

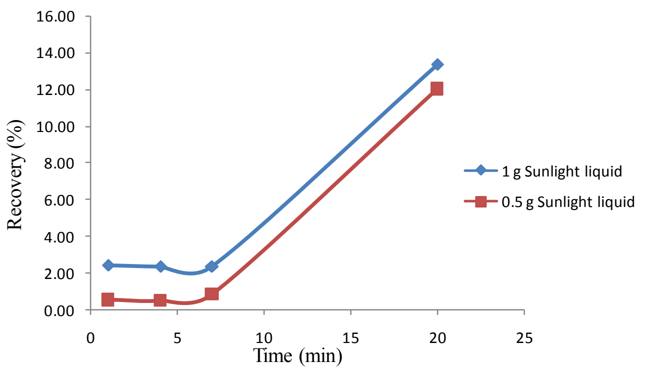

Figure 5: Graph of time versus recovery for 0.5 g and 1.0 g sunlight liquid.

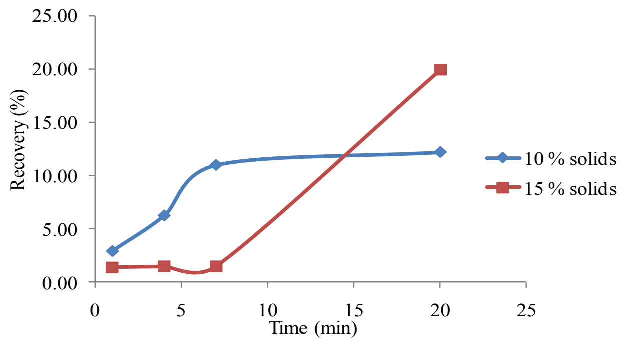

Figure 6: Graph of time versus recovery for slurry at 10 % and 15 % solids.

Products	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		500	32.9	
FC1	1	8.4	26.29	2.40
FC2	4	8.5	25.38	2.35
FC3	7	10.3	21.16	2.37
FC4	20	60.9	20.16	13.35
FrotherConcentrate		**88.1**	**21.37**	**20.91**
Tails		410.8	17.800	79.53
Head (calc)		498.9	18.43	100.00

Table 5: Experiment 1.2 of un-sieved (1 g sunlight).

Products	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		650	32.9	
FC1	1	3.9	23.56	1.45
FC2	4	6.4	15.01	1.52
FC3	7	7.2	13.51	1.54
FC4	20	110	11.44	19.92
FrotherConcentrate		**127.5**	**12.11**	**17.15**
Tails		522.5	10.020	82.85
Head (calc)		650	10.43	100.00

Table 6: Floatation experiment with 15% solids using sunlight.

Effect of percentage solids on the extraction of $CaCO_3$ from un-sieved material

Table 6 contains the results obtained when the slurry made from un-sieved material contained about 15% solids. From (Table 1), the overall recovery was 31.55%, while with slurry at 15 % solids a 17.15% recovery was realised. The slurry with 10% solids produced a higher grade $CaCO_3$ of 24.21% while at 15% solids it gave a lower grade of 12.11% . It was also noted that the recovery and conditioning time showed a direct proportionality relationship.The average $CaCO_3$ grade for these experiment was lower than all the other experiment conducted with different collectors.

The variation of recoveries with time when slurries of 10% and 15% solids were floated by sunlight is shown in (Figure 6). From the graph it is noted that with 10% slurry, recoveries increased more rapidly than 15% slurry until after 14 min. However, the overall recovery for slurry with 10 % solids is more than that of the slurry with 15% solids. The lag of recovery in 15% slurry could be attributed to the need for longer period to condition more concentrated slurry.

The experimental conditions that were used on sieved sludge material are shown in (Table 7). The pH was kept almost constant for these experiments. For each experiment, the initial pH was around 8.4 and after addition of soda ash (Na_2CO_3), the pH raised to 9.45. Detailed results on the recoveries are shown on (Table 8 and 9), where each frother concentrate (denoted by FC1, FC2, FC3 and FC4) and tailings concentrate (T) were analysed for $CaCO_3$ content.

Effect of collector dosage on sieved material

Table 9 indicates the results obtained when using a dosage of 2 g sodium oleate on the sieved material. It was noted that on the last two time intervals, the recovery was constant at 1.81%. The overall recovery obtained was 5.91%, which was lower than what was obtained when un-sieved material was used under similar conditions.

The effects time on recovery of $CaCO_3$ when using a dosage of 4 g sodium oleate on the sieved sludge material in shown in (Table 9). It was noted that the dosage only made a difference of 1.74% to the test performed with a dosage of 2 g. Generally sodium oleate exhibited some poor collection characteristics for $CaCO_3$.

Effect of percentage solids in sieved sludge material on recovery

Figure 7 shows the variation in recoveries with time for the 10% and 15% slurries. The curve lying below indicates that increasing percent solids from 10% to 15% did not make any improvement on recovery and instead the recovery was lowering for every time the concentrates were being collected.

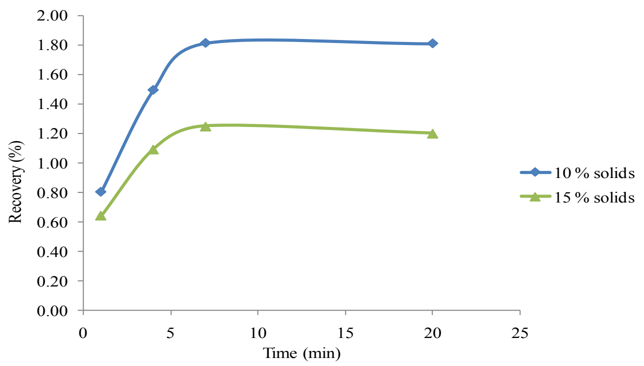

Figure 7: Graph of time versus recovery for slurry at 10 % and 15 % solids (sieved).

Effect of sodium oleate			
Test	1.1	1.2	1.3
Volume	4302	310	4500
Feed $CaCo_3$ (g)	478	500	500
Description	Sieved	Sieved	Sieved
Feed $CaCO_3$ content (%)	63.4	63.4	63.4
Flotation Na_2CO_3 (g) Silicate (%) Silicate (mL/L) Silicate (mL) Oleate (%) Oleate (g/L) Oleate (g)	 1 0.2 1 0.4 2	 1 0.2 1 0.4 2	 1 0.2 1 0.8 4
pH Feed(before adding Na_2CO_3)	8.4	8.5	8.3
pH Feed(after adding Na_2CO_3)	9.45	9.5	9.43
Impeller speed (rpm)	650	650	650

Table 7: Conditions for experiments on sieved material.

	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		444	63.42	
FC1	1	3.5	59.49	0.80
FC2	4	6.2	62.60	1.49
FC3	7	8	58.89	1.81
FC4	20	7.6	61.88	1.81
Frother Concentrate		**25.3**	**60.78**	5.91
Tails		418.7	58.440	94.09
Head (calc)		444	58.57	100.00

Table 8: Conditions used to float sieved sludge material with 2 g sodium oleate.

Products	Time (min)	Mass (g)	$CaCO_3$ Content (%)	$CaCO_3$ Recovery (%)
Feed		450	63.42	
FC1	1	3	55.49	0.64
FC2	4	5.6	50.70	1.09
FC3	7	7.2	45.10	1.25
FC4	20	7.8	40.00	1.20
Frother Concentrate		**23.6**	**46.06**	4.18
Tails		426.4	58.440	95.82
Head (calc)		450	57.79	100.00

Table 9: Effects of time on collection of $CaCO_3$ from sieved using sodium oleate.

Conclusions

From the experimental results obtained on flotation of sieved and un-sieved material it was concluded that:

- Recovering $CaCO_3$ from wastewater treatment sludge is definitely possible with flotation.
- Sunlight dish wash liquid showed a great potential as a collector of $CaCO_3$ from sludge than sodium oleate.
- There is a minimum conditioning time necessary to effect significant recovery of $CaCO_3$.
- pH regulation was essential to improve collection capability of collectors.
- The collector had shown a great influence amongst all the other reagents used, on the tests conducted and sunlight dishwashing liquid was successful on improving the recovery for un-sieved material throughout the experiments.

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